UNIVERSITI TEKNOLOGI MARA

FORMULATION OF MINIMUM SIGNATURE SOLID ROCKET PROPELLANT BASED ON AMMONIUM PERCHLORATE AND SORBITOL WITH MAGNESIUM AS A METAL ADDITIVE

MUHAMMAD ZAKWAN BIN AZIZI

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CONFIRMATION BY PANEL OF EXAMINERS

I certify that a Panel of Examiners has met on 15 October 2025 to conduct the final examination of Muhammad Zakwan Bin Azizi on his Doctor of Philosophy thesis entitled "Formulation of Minimum Signature Solid Rocket Propellant Based on Ammonium Perchlorate and Sorbitol with Magnesium as a Metal Additive" in accordance with Universiti Teknologi MARA Act 1976 (Akta 173). The Panel of Examiners recommends that the student be awarded the relevant degree. The Panel of Examiners was as follows:

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ABSTRACT

Minimum-signature smoke solid propellants were developed using ammonium perchlorate (AP) and sorbitol, integrated with metal additives such as magnesium (Mg). Different fuel compositions were also blended with metal catalysts, notably ferric oxide (Fe_2O_3). Minimum smoke solid propellants are already widely available in the solid rocket propulsion field. Minimum-smoke solid propellants are widely available but remain complex and costly to produce, with much related research kept confidential in developed countries. An optimized AP/sorbitol-based formulation containing 77 wt% AP and 5 wt% Mg exhibited the highest burning rate (0.126 cm/s) at ambient conditions. The heat of combustion increased with oxidizer content up to zero oxygen balance, and the addition of metal additives enhanced the heat of combustion 4142.57 kJ/mol and specific impulse 183.56 s. Mechanical properties were evaluated using tensile and Shore A hardness tests. The results showed that incorporating 5 wt% magnesium and 1 wt% ferric oxide significantly improved mechanical durability. In particular, the combined addition of Mg and ferric oxide in formulation SP8 produced the highest tensile stress at 1.116 N/mm² along with increased hardness, indicating enhanced structural integrity. AP/sorbitol based solid propellant formulations were synthesized and characterized using FTIR, XRD, and CHNO-S analyses. The incorporation of magnesium significantly enhanced thermal stability, increasing the decomposition and ignition temperature to 243.59 °C. In contrast, 0.05 wt% ferric oxide acted primarily as a catalyst and did not directly contribute to energy release. Kinetic analysis using the Kissinger–Akahira–Sunose methods showed activation energies ranging from 75 to 161 kJ/mol, with formulation SP6 exhibiting the highest values, attributed to its energy-dense combustion pathway. The static firing tests demonstrate that the incorporation of magnesium significantly enhances propellant performance, producing a peak thrust of 4.40 N and a specific impulse of 29.60 s. Formulations employing a weaker oxidizer generated a lower thrust of 2.61 N compared to 3.00 N for formulations with a stronger oxidizer, despite using the same fuel composition. Among all samples, SP4 and SP5 exhibited the highest light transmittance and lowest particle mass, qualifying under Advisory Group for Aerospace Research and Development (AGARD). In contrast, SP6 to SP8, which included metal additives, showed increased smoke opacity and particle mass, falling under AGARD Class BA or BB. The findings in this research of AP/sorbitol-based propellants, enhanced with optimized metal additives, as promising low-signature alternatives for regional space and defence applications.

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LIST OF SYMBOLS

Symbols

%wt	Weight Percent
CHNS-O	Carbon, Hydrogen, Nitrogen, Sulphur, and Oxygen Elemental Analysis
DSC	Differential Scanning Calorimetry
Ea	Activation Energy
EDS	Energy Dispersive X-ray Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
FWO	Flynn–Wall–Ozawa Method
g	Gram
Isp	Specific Impulse
KAS	Kissinger–Akahira–Sunose Method
mg	Milligram
OB	Oxygen Balance
SEM	Scanning Electron Microscopy
SP	Solid Propellant Sample Code (e.g., SP4, SP5)
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction
ΔH	Heat of Combustion

LIST OF ABBREVIATIONS

Abbreviations

ADN	Ammonium Dinitramide
AGARD	Advisory Group for Aerospace Research and Development
AN	Ammonium Nitrate
AP	Ammonium Perchlorate
BEM	Ballistic Evaluation Motor
CL-20	Hexanitrohexaazaisowurtzitane
GAP	Glycidyl Azide Polymer
HMX	Cyclotetramethylene-Tetranitramine
HTPB	Hydroxyl-Terminated Polybutadiene
KNO_3	Potassium Nitrate
PEG	Polyethylene Glycol
RDX	1,3,5-Trinitro-1,3,5-Triazine
SP	Solid Propellant

LIST OF NOMENCLATURES

Nomenclatures

CO	Carbon Monoxide
CO_2	Carbon Dioxide
Fe_2O_3	Ferric Oxide
H_2O	Water
HCl	Hydrogen Chloride
K	Kelvin (Temperature Unit)
Mg	Magnesium
min	Minute
NO_x	Nitrogen Oxides
s	Second
T	Temperature
β	Heating Rate in Thermal Analysis

CHAPTER 1
INTRODUCTION

1.1 Research Background

The first rocket was invented in China back in the 13th century [1]. They were used as short-range missiles in the military, as well as a firecracker. Gun powder was used as a solid fuel for solid rocket propellant at an early stage of its development, but as time passes, the invention improves as modern civilization modernizes at a rapid pace. Since then, rocket technology has evolved to fulfil the demand in a variety of fields including not only defence but also in space exploration, meteorology, and education [2].

Rocket motors are classified into solid, hybrid and liquid [3]. One type of rocket fuel engine used in rocket systems is solid rocket propellant. It converts chemical energy into kinetic energy produce at exit nozzle under isentropic flow. It can be divided into heterogeneous and homogeneous mixture solid propellant. The oxidizer, fuel, plastic binder, and additive materials make up heterogeneous solid propellant, also known as composite propellant [4]. Oxidizer and fuel are combined and formed into a solid rigid mixture. To achieve high thrust force, the solid is burned in a chemical reaction with an additive to improve combustion performance [5]. Solid rocket propellant is easy to handle, safe due to chemical stability, easy to store, low shock resistant, and solid rocket motor does not require complex equipment such as a liquid pump [6]. Because of these advantages, solid propellant was used as engine fuel in missiles or short-range rockets.

Many studies have been conducted to investigate the solid propellant burning rate, solid propellant formulation, solid propellant performance, and physical properties of solid propellant. A mixture of fuel, oxidizing agent, and additive demonstrated outstanding ballistic performance [7]. In a solid propellant system, an oxidizing agent provides enough oxygen to the fuel to allow combustion to occur in outer space no oxygen, while an additive is added to improve the physical and chemical properties of the propellant. Moreover, the burning products of solid propellant generate smoke, which must be controlled in order to achieve the desired objective and specific qualification. Smoke produce from solid propellant combustion is not only harmful to the environment, but it also exposes the position of the armed forces to enemies [8].

The smoke produced by the combustion of solid propellant is hazardous to humans and environment. Besides that, its smoke also exposes the military position to adversaries. Solid propellant plumes that are chlorinated, aluminized, and highly acidic are hazardous to human health, the environment, and the ozone layer [9]. The exhaust gas produced by solid combustion is corrosive and hazardous to the environment. This issue has prompted researchers to create green solid propellants with the goal of reducing and eliminating toxic gases.

The combustion concept requires fuel, heat, and oxygen. Fuel reacts rapidly with oxygen, producing heat as a byproduct. Smoke produced during the incomplete combustion of substances indicates a lack of oxygen to oxidize the substance. As a result, complete combustion with sufficient oxygen need to oxidize substances produces little smoke and only yield water vapor and carbon dioxide [10]. Hence, oxygen balance is an important analytical tool for determining the type and completeness of combustion.

Oxygen balance is an important thermochemistry parameter, required to study smoke production of combustion propellant material [11]. By knowing amount of oxygen balance for oxidizing agent, researcher can predict the constituents of solid propellant burning. The nature of products will depend upon the amount of oxygen available during reaction [12]. This supply of oxygen will depend in turn upon the quantity of oxidizing atoms which present in propellant molecule. This oxygen balance is defined as the amount of oxygen, expressed in weight percent, liberated as result of the complete conversion of the explosive material to carbon dioxide and water. The oxygen balance provides information on the types of gases liberated [13]. A large negative oxygen balance indicates insufficient oxygen for the complete combustion to occur. This is because oxygen plays a critical role in the combustion process, acting as one of the three fundamental components alongside fuel and heat required to sustain the reaction. Consequently, toxic gases such as carbon monoxide will be liberated. For ammonium perchlorate (AP), it has high oxygen balance with positive sign [14]. It indicates that it has high proportion of oxygen more than required for complete oxidation of its fuel elements. Moreover, AP mixtures with fuel produce high performance of solid propellant combustion compared to another oxidizing agent. Hence it necessary in application in solid rocket motor field.

Solid propellants with a minimum of smoke were developed and produced decades ago. Many research papers [15] on minimum signature propellants have recently been published [16], [17]. It has become a hot topic in research [18] to reduce

the smoke produced by solid propellant combustion [19], [20], [21]. Efforts to develop modern solid propellants continue around the world in search of the best solution. Future green oxidizer development must meet new criteria such as environmental friendliness, low hazard, low cost, high performance, compatibility, and stability [22], [23], [24]. Currently, missile rocket defence technology necessitates minimum signature, environmentally friendly solid propellant without sacrificing performance.

Many efforts have been carried out to produce solid minimum signature propellant to be applied in defence by using more high energetic material and greener materials [25]. However, in Malaysia there are several constraints including raw materials to produce green oxidizing agents [26]. Examples of high energetic materials are HMX(cyclotetramethylene-tetranitramine), RDX (1,3,5-trinitro-1,3,5-triazine), FOX-12 (N-guanylurea-dinitramide), FOX-7(1,1-diamino-2,2-dinitroethylene) and CL-20(Hexanitrohexaazaisowurtzitane) [27], [28], [29]. Among the constraints are availability, production cost, expertise, safety and many more[30]. In addition, the process to produce the material is very complex. Its process is also time-consuming and dangerous. The most suitable materials to be use and have oxygen balance close to AP, are Ammonium dinitramide (ADN) and AN (Ammonium Nitrate). Oxygen balance for ADN is +25.8% and Ammonium nitrate is +20% positive oxygen balance means perfect combustion as mentioned above[31]. Unfortunately, these two materials also have limitations to be used in this research.

As for ADN, it is not stable when it is melt [32]. The same goes for its performance as propulsion solid. Furthermore ADN is strong hygroscopic, difficult storage and incompatibility with curing agent [14]. This is also the same goes with AN. Many studies state that AN and ADN can produce product that does not emit smoke at the expense of combustion quality. The combustion rate for AN based as an oxidizing agent is weak. On the contrary, for AN based, the biggest problem faced by propulsion researchers is a solid phase transformation [33]. It will leave excess product in solid form. This will cause a problem with the solid rocket motor.

AP is a common oxidizing, agent it's been used in rocket field since many decades ago [34], [35]. Consequently, obtaining AP is not challenging. Its ease of production, compatibility with a wide range of additives, and positive oxygen balance have maintained AP as the primary oxidizer for solid rocket propellants [36] . AP also offers several performance and practical advantages high specific impulse, reliable performance, and straightforward manufacture which have secured its continued use

across aerospace launch systems and military applications. Beyond propulsion, AP is employed in airbag inflators, ejection-seat charges, and certain demolition and construction blasting applications [37], [38].

Most of the fuel used to mix with AP is HTPB (hydroxyl-terminated polybutadiene), plastic polymer, and metal fuel. A well-known composition for solid rocket propellant is AP/HTPB with aluminium metal as an additive metal [39]. This mixture performs well in terms of combustion. HTPB is also poisonous and damaging to human health. Hence another option is to utilize sugar alcohol like sorbitol to mix with AP [40]. Despite its excellent performance, it emits visible smoke. An attempt to reduce chlorine smoke from AP is to use alkali metal to attract chlorine gas by reacting with it [41]. Example of alkali metal that have been used as scavengers are lithium and magnesium which is the most chosen scavengers [42]. In addition, AP work in synergy with catalyst and others metal additive [43]. Additionally, AP serve as a filler that gives the propellant physical strength and is crucial in adjusting the propellant burning rate using multimodal particle size distribution [44]. Since the primary topics of recent AP research have been catalyst and thermal AP decomposition [45]. Hence it is important to get better understanding to produce better performance of solid propellant combustion. However, AP has a significant flaw that makes it particularly risky at superfine sizes [43]. In addition, several other factors must be taken into account before a solid propellant may be produced [46]. The component and the stage of the production process are a couple of examples of parameters need to be considered. Because of this versatility, AP is the preferred oxidizing agent to be employed in this study.

Sugar alcohols like sorbitol, dextrose, xylitol, mannitol, erythritol, and maltitol are commonly used in the food industry and as sweeteners. It was commonly used to replace table sugar as a sweetener for diabetics. Sorbitol is a type of sugar-based alcohol that is commonly used to substitute table sugar in diabetics. In this rocketry field, it is typically combined with potassium nitrate and used in the invention of solid propellants [47]. Because of its high caramelize point compare to sucrose it is an excellent candidate for solid propellant formulation [48]. Given its applicability to substitute table sugar (sucrose), it is widely available in stores and malls [49]. Furthermore, its price is very low, allowing individuals to purchase sorbitol. However, the potassium nitrate/sorbitol mixture generates a lot of smoke. Smoke species such as carbonate, carbon, and carbon monoxide are becoming a problem. Because potassium nitrate is a weak oxidizing agent, this smoke species is the result of incomplete combustion due to lack of oxygen

supplies to the fuel. As a result, the use of sugar alcohol such as sorbitol use as a solid propellant for rocket combustion fuel typically results in a lower combustion rate [50]. On the other hand, it will reduce the performance of the rocket's solid propellant. Catalysts such as iron oxide, copper oxide, and aluminium oxide are used to increase the rate of combustion of solid rocket propellant. Furthermore, additives such as energetic polymeric binder are added not only to improve the burning performance but also to improve the physical properties of the propellant.

Beside that due to its availability, easy to get, not toxic or harmful to human and has high caramelize point, it becomes perfect choice to replace sucrose as fuel. The majority of research on sorbitol as a solid propellant has focused on its performance, optimum formulation, casting method, physical properties, burning rate, and production method [51], [52], [53]. Aside from that, the ignition method has emerged as a hot topic for research in sorbitol-based propellants. Moreover work on sorbitol propellant meet new level [54]. Effort to combine sorbitol with nitrous oxide in hybrid system is a sign of research in this field is moving forward sustainably [55]. In fact, it attracted more interest from researchers to be involved in this study. Many studies on solid rocket propellant employ potassium nitrate as an oxidizing agent [56], [57], [58]. This ingredient is called as KNSB (potassium nitrate sorbitol) propellant. KNSB solid propellant has a significant downside in that it produces a lot of smoke when burned due to the fact that it weak oxidizing agent [58].

Researchers in this field overlook to investigate the combustion product of KNSB propellant [59]. There has been little effort made to reduce smoke from the combustion of KNSB propellant. Recently researchers have added metal powder, such as magnesium, lithium or sodium to ensure that the smoke is scavenged by metal reaction [60]. To comply with the authorities' rules and regulations, research in minimum signature (minimum smoke) of solid propellant must be done and investigated to produce better quality solid rocket propellant for use in defence and aerospace. The idea is to combine a strong oxidizer, such as AP, with sorbitol. AP is a good candidate because it has a positive oxygen balance of +34%, indicating high oxygen content [61]. The study provides new insights into oxygen balance alignment, smoke opacity behaviour, thermal decomposition interactions, and the suitability of sorbitol as a green fuel for AP-based formulations

No study has combined sorbitol (green fuel) with ammonium perchlorate (strong oxidizer) to evaluate the feasibility of a low-signature, reduced-smoke composite

propellant. Current sorbitol-based propellants produce significant smoke due to weak oxidizers like KNO_3, while high-performance oxidizers such as ADN and AN face issue related to instability, hygroscopicity, and poor combustion. AP remains the most viable oxidizer, but AP-based propellants face challenges in smoke generation and chlorine-based exhaust species The idea is to use AP as an oxidizer, which can provide high oxygen, and it is anticipated that this oxidizer will produce a synergetic propellant with the fuel and that with catalyst to produce clean combustion with minimum soot trace. This research aims to develop and characterize a minimum signature solid propellant by combining sorbitol with ammonium perchlorate and metal additives. The methodology includes material synthesis, thermal analysis, combustion experiments, smoke-opacity measurement, and static testing evaluation. The novelty of this study lies in introducing sorbitol typically used with weak oxidizers incorporated into an AP-based system to achieve cleaner combustion, reduced smoke, and improved environmental compatibility while maintaining acceptable combustion performance for defence and aerospace application

1.2 Motivation for This Work

The responsibility to develop and find alternative methods to produce solid rocket propellants is part of this researcher's obligation for the future of the country in aerospace and defence field. As a progressive and forward-looking nation, Malaysia has strategically prioritized research and development to address emerging global and regional challenges. In line with this vision, the government launched the National Space Policy 2030, which outlines four strategic pillars aimed at strengthening the national space ecosystem: (1) advancing space technology, (2) enhancing space infrastructure, (3) mastering the space sector, and (4) expanding space applications. This policy framework is designed to cultivate local expertise, promote scientific advancement, and stimulate economic growth through innovation in space-related domains. To further reinforce this national agenda, complementary policies have been implemented across various ministries. These include the Malaysian Aerospace Blueprint 2030 (MAB2030) under the Prime Minister’s Department, the National Science, Technology, and Innovation Policy (MOSTI), and the National Defence Policy (MINDEF). Notably, this research aligns directly with the second core strategy of the National Space Policy 2030 and supports Strategy 4 of MAB2030, particularly Initiative

4.3, which focuses on capability development in avionics, mission systems, sensor integration, electronic warfare (EW), ground systems, and missile/rocket propulsion technologies.

As a patriotic Malaysian, this project aligns with national policies aimed at advancing Malaysia's technological capabilities in rocket propulsion and solid propellant development. It directly contributes to Malaysia's national agenda of strengthening original capabilities in space and defence technologies, while also supporting the broader objective of positioning the country as a leading space technology hub in Southeast Asia.

1.3 Problem Statement

The development of efficient, low-signature solid propellants is crucial for modern aerospace and defence applications. Although traditional solid propellants are widely used, they often produce high infrared and visible signatures, which reduces their suitability for stealth operations. The incorporation of alternative fuel binders like sorbitol and metal additives has the potential to enhance combustion characteristics while reducing detectable emissions. Nonetheless, there is a lack of comprehensive research on the formulation and synthesis of AP/sorbitol-based propellants with Mg metal additives to achieve minimum signature effects. The influence of metallic additives on the combustion behaviour, thermal stability, and exhaust emissions of AP-based solid propellants remains insufficiently understood. The optimum ratio of fuel, oxidizer, and metallic additive required to achieve clean combustion is needed to discover. Furthermore, a comparative analysis of propellants with and without metallic additives will be carried out. Addressing this knowledge gap is essential for improving propellant performance while minimizing environmental and operational drawbacks.

The performance and stability of solid propellants are significantly influenced by their thermal and physical properties, as well as their kinetic decomposition behaviour. AP and sorbitol-based propellants are commonly used due to their energy density and processing advantages. However, the addition of metal additives, such as magnesium, introduces complex thermal reactions that can affect combustion efficiency, burn rates, and decomposition kinetics. Regarding their potential benefits, there is little study on the thermal stability, mechanical integrity, and kinetic decomposition features of AP/sorbitol-based propellants with Mg metal additive.

Understanding how these formulations behave under different thermal conditions is essential for improving their combustion efficiency, ignition characteristics, and safety performance. Besides that, kinetic decomposition parameter for AP/sorbitol/Mg formulations are very limited, creating a significant knowledge gap. To address this, the present study will perform using TGA measurements at multiple heating rates. The experiments will allow strong determination of thermal stability, ignition sensitivity, AP/sorbitol/Mg propellants.

The performance of solid propellants in propulsion systems is important, since characteristics like as thrust performance, combustion stability, and exhaust signature determine their suitability for aerospace and defence applications. Traditional AP-based propellants frequently emit significant infrared and visible emissions, making them easily detected. The use of green biodegradable fuel like sorbitol and Mg as chloride scavenger in AP-based formulations has demonstrated potential for enhancing combustion performance while lowering signature emissions. Nevertheless, there is a scarcity of detailed testing of minimum signature solid propellants. While theoretical research and thermal analyses offer insights on decomposition behaviour, real-world performance testing, such as static firing tests and ballistic evaluation motor tests, is required to evaluate their efficiency, thrust generation, and emission characteristics. Without such empirical evidence, the true usefulness of these formulations in practical applications is questionable. Static test validation of lab-scale formulations and systematic smoke opacity measurements for each propellant variant are currently lacking. This study therefore conducts controlled static-fire tests with time-resolved optical and particulate instrumentation to quantify signature and combustion performance.

1.4 Research Objectives

The purpose of this research is to synthesis, evaluate, formulate, characterize, and test a minimum smoke from combustion AP/sugar alcohol-based solid propellant that is reliable and safe for laboratory testing for educational purposes. The following objectives must be met to meet the criteria for this study.

a) To formulate an ammonium perchlorate sorbitol propellant composition with metal additives for minimum signature solid rocket propellant characteristics.

b) To characterize thermal and physical properties of solid propellant of ammonium perchlorate mix with sorbitol and magnesium metal.

c) To test the performance of minimum signature solid propellant using static firing test and smoke opacity meter.

1.5 Research Question

i) How can the amount of ammonium perchlorate will affect the providing of oxygen to sorbitol to undergoes complete combustion and how magnesium metal play a role in scavenge the chloride gas?

ii) What is the energy, physical and chemical analysis result of solid propellant from the formulation, with and without metal additive?

iii) What is the amount of smoke concentration, burning rate and specific impulse of solid propellant.

1.6 Significance of Study

The significances of this study are to provide a novel minimum signature solid propellant that is low in cost, has a simple manufacturing method, optimum new formulation of fuel/oxidizing agent/metal additive and has readily available raw materials. As a result, mass manufacture of solid propellant is feasible. Beside that it delivers a clean combustion product at the exhaust outlet of a rocket/missile for improved performance in the military and aerospace industries in Malaysia for better future. On the other hand, the outcome of this study will be able to assist researchers around propulsion field in better understanding prediction for combustion products in propellant.

Furthermore, the study's thermal and kinetic decomposition analysis offers critical information on thermal stability, ignition behaviour, decomposition mechanisms and mechanical properties which are essential for improving propellant safety and performance. The use of static firing tests and ballistic evaluation motor tests ensures

that the research findings are experimentally validated, bridging the gap between theoretical predictions and real-world applications.

The findings from this study are expected to enhance existing models of solid propellant combustion, providing valuable data for researchers working in propellant chemistry, aerospace engineering, and defence technology. Additionally, the development of a minimum signature propellant formulation can contribute to future innovations in green high energetic materials. By contributing to both fundamental and applied research, this study lays the groundwork for future advancements in solid propellant technology, supporting the development of safer, more efficient, and environmentally friendly propulsion systems.

Eventually this work going to be provided minimum signature composite solid propellant that complies with insensitive munitions standards, with a focus toward safety and accident prevention to bring Malaysia align in research of defence technology with other developed countries.

1.7 Limitation

This research is subject to several limitations that define the scope and constraints of the study. Firstly, the formulation and synthesis of the solid propellant are conducted on a laboratory scale, meaning that findings may not fully represent large-scale production or real-world manufacturing conditions. The study is also limited to specific metal additives, particularly magnesium, and does not explore other potential additives that might influence combustion energy and signature reduction. Additionally, the research focuses solely AP and sorbitol as the primary oxidizer-fuel combination, without considering other potential oxidizers or hybrid formulations that could provide further insights into minimum signature characteristics. In addition to not considering all the toxic species likely to be produced by the ignition of a predominantly organic matrix, the model does have several limitations. First, it is an equilibrium-based system and does not consider those synthesis pathways which may be governed predominantly by kinetic processes.

Secondly, it assumes Ideal gas behaviour on the part of all the gases produced, this assumption is not likely to be accurate over the entire range of conditions existing inside the rocket motor, However, from a practical standpoint, this may not be a severe limitation. For example, the magnitude of non-Ideal gas effects depends primarily on

the density and the temperature in the system. For the system in question, the largest densities occur in the chamber.

In terms of performance testing, the study relies on static firing tests and ballistic evaluation motor tests conducted in controlled laboratory conditions. While these tests provide insights into burn rate, thrust performance, and emission characteristics, they do not account for external environmental factors such as humidity, extreme temperatures, or variations in atmospheric pressure, which may influence real-world combustion performance.

The study is limited in its application to specific defence and aerospace propulsion systems. While the findings contribute to the advancement of minimum signature solid propellant technology, the research does not extend to actual rocket motor integration, large-scale propulsion system testing, or military field applications. The insights gained are primarily intended for fundamental research and development, providing a foundation for further studies in energetic materials, combustion science, and emission control in propulsion systems.

Moreover, one of the notable limitations encountered in this research is restricted access to comprehensive scientific literature and technical data, particularly those related to advanced solid propellant formulations, combustion, and performance testing. This limitation arises due to the sensitive nature of the subject matter, which is closely associated with defence applications and missile technology. As a result, many high-level studies and experimental findings are either classified, governed by export control regulations. Consequently, the scope of this research had to rely primarily on publicly available data, open-source publications, and declassified materials, which may limit the depth of comparative analysis with internationally developed defence-grade systems

1.8 Assumption

In this research, several assumptions are made to ensure the validity and feasibility of the study. First, it is assumed that the AP/sorbitol/Mg metal additive formulation will be homogeneous and stable, allowing for consistent combustion behaviour without unexpected variations. Additionally, the incorporation of metal additives, such as magnesium, is expected to enhance combustion performance while minimizing the visible and infrared signature. It is also assumed that the synthesis

process will not introduce unintended impurities that could significantly alter the thermal decomposition or combustion characteristics of the propellant.

Furthermore, in the characterization phase, it is assumed that the thermal decomposition behaviour of the formulated propellant will follow predictable kinetic models, facilitating accurate analysis. The thermal stability and mechanical reliability of the propellant are expected to remain within acceptable limits under normal storage and operational conditions. Additionally, the inclusion of metal additives is assumed to modify the decomposition kinetics in a controlled manner, without compromising the overall safety and stability of the propellant.

For the performance testing phase, it is assumed that the static firing test will provide reliable thrust and burn rate data, accurately reflecting real propulsion conditions. Similarly, the ballistics evaluation motor test is expected to yield meaningful insights into pressure generation, burn efficiency, and exhaust signature characteristics. It is also assumed that the minimum signature effect will be measurable and significant when compared to conventional AP-based propellants. Lastly, it is assumed that environmental factors, such as humidity and temperature fluctuations, will have minimal impact on combustion performance under controlled testing conditions. These assumptions serve as the foundation for the research and help guide the methodology in achieving reliable and meaningful results.

1.9 Ethical Committee

This study conforms to the highest ethical standards, ensuring integrity, transparency, and responsibility throughout. The Ethical Committee approved the research to ensure that it followed established ethical norms. The environmental effect of the experimental techniques was carefully considered, notably in terms of handling and disposal of hazardous chemicals such as AP and other solid propellant components. Furthermore, all data obtained during the study were kept strictly secret and utilised only for the purposes specified in the research goals. The committee's ethical monitoring guarantees that the study meets all legal and moral duties, encouraging appropriate research procedures.

1.10 Thesis Scope

In this work, sorbitol is used as the fuel, and AP serves as a powerful oxidizing agent to create solid propellants with a low signature. AP is supplied by STRIDE (Science & Technology Research Institute for defence), while Sorbitol is supplied by HEMREL UITM. In addition, formulation thermodynamics properties of mixture will be calculated to support the fact that AP/Sorbitol is compatible in produce low signature. The formulation process is limited to laboratory-scale production, ensuring strict adherence to safety protocols and handling procedures for energetic materials.

The thermal and physical characterization of the propellant includes the analysis of thermal stability, decomposition behaviour, and structural integrity using techniques such as differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and kinetic decomposition studies. Additionally, the mechanical properties of the propellant, such as tensile strength and hardness, will be evaluated to determine its suitability for propulsion applications. The study focuses on metal additives, sorbitol, and their effects on AP-based solid propellant, without extending to alternative oxidizers or hybrid fuel systems.

To understand the kinetic decomposition behaviour, this study evaluates the reaction mechanisms, activation energy, and combustion stability of the formulated propellant. The impact of mixture between sorbitol and metal additives AP-based solid propellant on burn rate, ignition characteristics, and decomposition pathways will be analysed, with a particular emphasis on minimizing exhaust emissions and improving combustion efficiency. Computational simulations of kinetic behaviour are beyond the scope of this research.

The performance evaluation of the propellant will be conducted through static firing tests and ballistic evaluation motor tests, which will assess burn rate, pressure-time characteristics, thrust performance**,** and smoke emission reduction. These tests will be conducted under controlled laboratory conditions, and real-world aerospace applications, such as large-scale rocket motor testing, are not included in the scope of this study. Additionally, environmental, and long-term aging effects on the propellant are beyond the study’s focus.

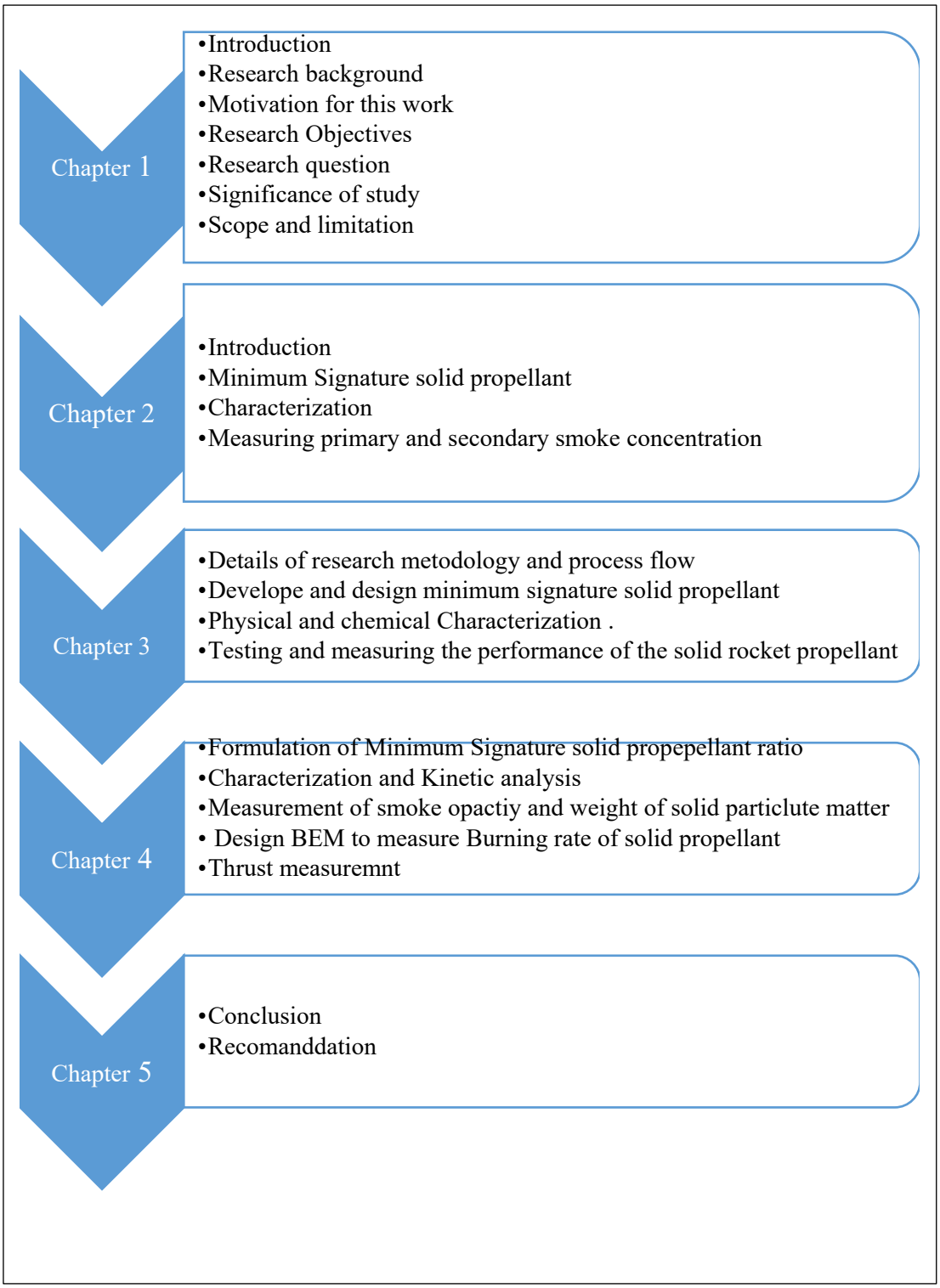
1.11 Thesis Outline

The thesis for this research is on the study of solid propellant combustion with minimum signature and its performance, with the goal of providing a better knowledge of combustion mechanics and solutions for reducing combustion signature in solid rocket propellant combustion. The importance of solid propellants in pyrotechnics and propulsion systems is discussed in the Introduction, with particular attention to the difficulties in attaining steady combustion and a low environmental effect. It highlights the significance of the study in improving combustion energy and lowering signatures while outlining the research goals.

The Literature Review goes into existing research on solid propellant compositions, combustion mechanism, noting gaps such as inadequate data on light transmittance and difficulty in reducing combustion byproducts. It also emphasises the significance of optimising propellant mixtures to improve performance.

The Methodology section details the experimental setup, which includes propellant formulation preparation and combustion analysis using the Digital Light Transmittance equipment, burning, and testing.

The Results & Discussion section contains extensive results about combustion properties. The effects of formulation factors on combustion behaviour are carefully investigated. Conclusions summarize key finding on minimum signature solid propellant formulation.


Chapter 1
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CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

This chapter present a review of the previous works on solid propellant which is categorized into 3 topics: the overview of minimum signature solid propellant, the performance and characterization of minimum signature solid propellant and quantification of the primary and secondary smoke of solid propellant. The chapter start with the overview of the minimum signature solid propellant in section 2.2. Accordingly, a review on performance and characterization of solid rocket propellant is written in section 2.3. In section 2.4 present the quantification of the primary and secondary smoke from combustion of solid propellant. the chapter culminate with the identification of gaps in the literature in section 2.5.

2.2 Overview of Minimum Signature Solid Propellant

A rocket is a device with an accelerating force known as thrust, which is achieved by ejecting a hot gas at high speed, high temperature and high pressure. A rocket or missile can be divided into two types: solid propellant and liquid propellant. Solid propellants are preferred because of their reliability, simplicity, easy-to-use, availability, lower production cost, chemically stable, high thrust and ability be stored for future use [62]. A solid rocket propellant system is applied in various types of fields and disciplines. Solid rocket propellant is used in tactical missiles, cloud seeding, space shuttle rocket boosters, air bag generators and hail suppression.

Solid propellant can be categorized into two classes: homogenous and heterogeneous propellant as depicted in Figure 2.1. Homogenous (double-base) propellant, first discovered by Alfred Nobel in 1890, is made up of nitrocellulose and nitro-glycerine [63]. Nitrocellulose is a sensitive material, thus limiting its applications. Nitrocellulose, due to its sensitivity and inherent instability, imposes limitations on its applications. To address this issue, nitro-glycerine was introduced to serve as a plasticizer for nitrocellulose, transforming it into a more manageable slurry that can be easily extruded into the desired shape, a formulation known as percussor for minimum

signature powder used in solid propellant field. Double-base propellants can further undergo modification, leading to the development of composite-modified double-base (CMDB) propellants through the addition of additive metals, thereby enhancing their performance characteristics [64].

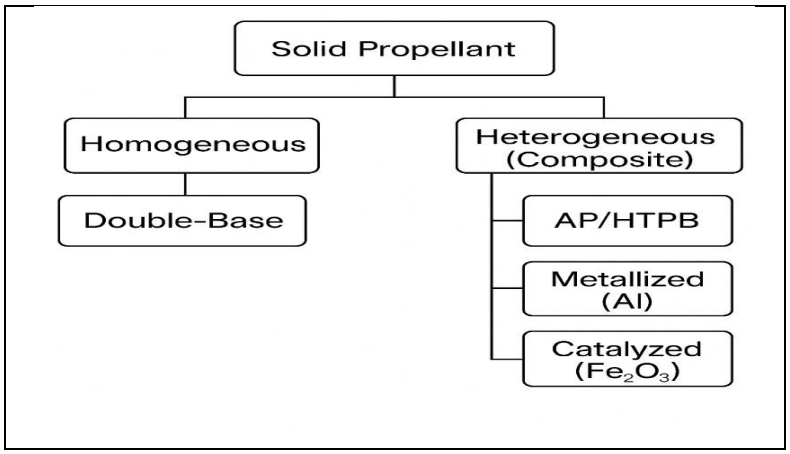


Figure 2.1 Classification Of Solid Propellant For Rocket Propulsion

A composite propellant is characterized as a heterogeneous propellant consisting of an oxidizer, powdered fuel and additive metal. In solid propellants, the oxidizer plays a crucial role, typically constituting a weight percentage within the range of 60-80%, while the solid fuel component generally falls below 25% by weight [4]. Solid rocket propellant does not need an external source of oxygen for combustion since there is oxidizer to supply oxygen in comparison with liquid propellant. Furthermore, most composite propellants produce a lot of smoke during combustion. Grain fabrication method of solid propellant can be divided into two methods: case bonding and free standing [65]. For case bonding method, solid propellant grain is directly put into the combustion chamber, whereas for the free-standing method, propellant grain is cast separately from the Combustion chamber.

Additives alkali earth metal, polymeric binder, iron oxide, and plasticizers were added to the formulation to enhance their performance, such as burning rate and improve their mechanical properties [24]. A common composite propellant that has been used for 50 years is, known as AP/HTPB. AP act as an oxidizer and hydroxyl terminated poly butadiene (HTPB) acts as a binder fuel. Binders not only act as a matrix for the solid propellant mixture but also improve propellant mechanical properties and influence the combustion rate of propellant [24]. The binder must have a high C/H ratio in its backbone so that exhaust gas release contains a lower average molecular weight

and no heavy gas is released [66]. Solid propellant performance is increased by metal additives like aluminium powder. The well-known formulation for this conventional composite propellant is 69-70% (AP), 16-20% (Al Powder) and 11-14% (HTPB). This is a remarkable finding in solid composite rocket propellant system with a specific impulse of 277 s and a pressure 68atm [67].

The major drawback of using composite propellant is the production of smoke. Smoke produced by a combustion of solid propellant has high impact to humans and the environment. Chlorinated, aluminized, and highly acidic plumes produced by solid propellant are harmful to human health, the environment, and the ozone layer [9]. When hydrogen chloride from the solid propellant burning it will react with atmospheric moisture, it will produce a highly toxic acidic gas. Exhaust gas produced from solid combustion is corrosive and harmful to the environment. This problem has prompted researchers to develop green solid propellant, aiming to reduce and eliminate toxic gases [9].

Minimum smoke solid propellants have been developed and have been produced many decades ago. Many research papers on the minimum signature propellant have been published recently [68]. It has become a hot topic in research to reduce the smoke produced by the combustion of solid propellant [69]. Efforts to develop modern solid propellants are continuing around the world to find the best solution. The development of futuristic green oxidizers must meet new criteria, which include being environmental friendly, low hazard, low cost, high performance, compatible and stable from external stimuli [70]. Powell et al. [71] analysed reduced vulnerability minimum smoke propellants, emphasizing the role of Insensitive Munitions (IM) compliant formulations to mitigate detonation risks upon impact or exposure to high temperatures. Their research underscores the growing need for safer propellant formulations that not only reduce emissions but also enhance storage stability and operational safety.

Currently, missile rocket defence technology requires minimum signature, environmentally friendly solid propellant without sacrificing performance and handling hazard which also knowns as green propellant. The combination of green propellant and preventing giving away the position from which missiles (minimum signature) are launched becomes an objective in this work. This work summarizes recent working progress on the production of environmentally friendly and minimum signature modern solid propellant.

2.2.1 Development of Minimum Signature Solid Propellant

The futures design of rocket and weapon systems requires the use of energetic materials with high energy output that are environmentally friendly which reduce vulnerability during storage or transportation. Extensive programs have evolved worldwide for the development and introduction of insensitive Munitions (IM) Ordinances that fulfil performance expectations in response to unplanned hazardous stimuli [72]. The main function of the oxidizer in propellants is to provide the required oxygen for the perfect combustion of fuel. Therefore, the oxidizer must release as much oxygen as possible for combustion. Besides, the stability and melting point above 200˚C of the oxidizer are also important for practical use in solid rocket propellant, during manufacture and during its wear (shelf life). It is always better to have an oxidizer with a high melting point, as propellant curing takes place at high temperatures. The density of the oxidizer must be high enough at 1.95 g/cm^3 to accommodate the high solid load. In addition, it must have a low heat of formation and a high oxidation potential [20].

The present oxidizer that has been used conventionally is ammonium perchlorate. Ammonium perchlorate became the best oxidizer in solid rocket propellants because of specific features like high burning rate, high thermal stability, high energy density, good compatibility, low sensitivity to shock, and long shelf life [8].The drawback of using AP propellant is that it produces chlorinated exhaust gas products, which have a huge impact on the environment and produce a signature for missiles to be tracked. To address these issues, researchers have explored various approaches, including alternative oxidizers, energetic additives, and advanced bonding agents, to develop propellants that minimize their exhaust signature while maintaining high combustion efficiency.

Early efforts to formulate minimum signature propellants focused on replacing AP with alternative oxidizers. Helmy et al. [73] studied the potential of nitramines such as HMX and RDX to reduce smoke emissions while sustaining high-energy output. Building on this work, Bivin et al. [74] developed Class 1.3 minimum smoke propellants using hydroxy-terminated nitramine polyester (ORP-2) and treated ammonium nitrate as oxidizers. Their formulations demonstrated improved mechanical properties and met stringent military standards for survivability and low-detection propulsion systems. Parallel advancements have explored gelled propellants as an alternative to traditional solid propellants. Hodge et al.[75] investigated gelled

propellants for tactical missile applications, which provided the density impulse of solid propellants while allowing controlled combustion similar to liquid propellants. Their work emphasized the safety and mission flexibility benefits of gelled fuels, though their handling and storage complexities remain a challenge.

Additionally, Menke et al. [76] focused on ammonium dinitramide (ADN)-based propellants, highlighting their high thermodynamic efficiency and low-emission characteristics compared to AP-based formulations. The push for environmentally friendly oxidizers has also driven the adoption of ammonium dinitramide ADN as a viable alternative to AP. Nagamachi et al. [77] emphasized the benefits of ADN, particularly its chlorine-free composition, which eliminates hydrochloric acid emissions and enhances specific impulse performance. Advances in crystal reshaping and microencapsulation techniques have further improved ADN's stability and processability, making it a promising candidate for future propellant formulations. Another work by Menke et al.[78] introduced a new class of AP/CL20/GAP mixed with ADN formulations, which provided burn rates up to 45 mm/s at 10 MPa while maintaining a significantly reduced exhaust signature. These propellants offer a balance between high combustion energy and low visibility exhaust, making them suitable for tactical missile applications. Use of ADN as green oxidizer was further investigate by Cican et al. [79] highlighted the potential of ammonium dinitramide (ADN) as an alternative oxidizer to AP and to calculate the amount HCl produce from combustion of ADN/GAP solid propellant in comparison with the classic formulation of AP/HTPB. The result demonstrating low-smoke and high-energy characteristics with minimum smoke and no HCl were produced from this formulation without scarifying solid propellant performances which has a high specific impulse.

Another effort was done by Williams et al. [80] introduced a lead-based additive for minimum smoke compositions, which effectively reduced smoke emissions while enabling sustained combustion at lower pressures. Similarly, Landsem et al.[81] developed neutral polymeric bonding agents (NPBA) for smokeless composite propellants, which enhanced mechanical properties without negatively impacting combustion efficiency. These findings demonstrate the importance of chemical modifications and stabilizers in achieving optimized performance for minimum signature solid propellants. Similarly, Abd-Elghany et al. [82] explored 2,2,2-trinitroethyl-formate (TNEF), which showed superior specific impulse at 250.1s compared to AP based solid propellant at 156.9 s while eliminating toxic chlorine

emissions. Youssef et al. [83] further optimized low-signature base bleed propellant formulations, which effectively minimize secondary smoke emissions in artillery applications, highlighting a shift toward cleaner and more efficient propellant formulations. The research used of sorbitol as fuel in minimum signature solid propellant was done by Oyedeko et al. [84] investigated the impact of sorbitol-based propellants, demonstrating how variations in oxidizer-to-fuel ratios affect thrust performance and exhaust emissions. Although sorbitol has been explored as a clean, low-toxicity fuel, existing research primarily pairs it with weak oxidizers like KNO_3, resulting in heavy smoke and low combustion efficiency. Very limited work investigates sorbitol in high-oxygen systems, and no study has evaluated sorbitol combined with ammonium perchlorate for minimum-signature applications. This absence of data indicates a clear gap in understanding how a strong oxidizer green fuel system behaves in terms of thermal decomposition and combustion cleanliness.

Overall, the reviewed literature shows substantial progress in developing minimum-signature solid propellants through alternative oxidizers, energetic additives, polymeric bonding agents, and advanced scavenging strategies. While materials such as ADN, CL-20, GAP, nitramines, and gelled propellants have demonstrated reduced smoke and improved environmental performance, they remain limited by issues of stability, cost, manufacturability, or storage. Ammonium perchlorate continues to be the most practical oxidizer due to its high performance and compatibility, despite generating HCl during combustion. Sorbitol, although recognised as a clean and low-toxicity fuel, has only been studied with weak oxidizers such as potassium nitrate, which leads to significant smoke formation and poor combustion efficiency. Critically, no prior work has examined sorbitol in combination with a strong oxidizer such as AP for minimum-signature applications, leaving a clear gap in understanding of its combustion behaviour and smoke-suppression potential. This gap establishes the need to investigate AP–sorbitol formulations with suitable additives to determine whether a cleaner-burning, minimum-signature solid propellant can be achieved without compromising performance.

2.2.2 Formulation of Minimum Signature Solid Propellant

The first minimum signature solid propellant was produced by nitration of cellulose in the 19th century. Known as a homogenous solid propellant, it is relatively

unstable and dangerous to produce. Alfred Nobel first produced a remarkable finding by mixing 40% nitrocellulose with 60% nitro-glycerine plasticizer to make it more stable which has been used until now as a double base propellant [85]. Additive materials like ammonium perchlorate, Al, and stabilizer were added to enhance the propellant's performance such as burning rate and energy density [86][87]. It is used in guns, canon, and in solid rocket propellant. Since it does not produce smoke, it is suitable to be applied in defence especially in production of bullets and missiles [87], [88].

However, double base propellants have major problems regarding stability, especially when handling nitro-glycerine. Liquid inclusion of nitro-glycerine causes major problems in propellant performance and stability [89]. In double base propellants, sodium nitrate ester 1,4-dinitro-to-2,3-dinitro-2,3bis(nitratomethylene) butane (SMX) is replaced by the nitro-glycerine above 40% wt, and their interaction with nitrocellulose was investigated [89]. The aging of double base propellant has also become a major problem. Nitrocellulose is decomposing, and chemical stability is degrading with time. Nitrogen dioxide formed during the aging process is reacting with stabilizer to prevent this compound from building up in rocket chamber[90]. Aromatic amine with diphenylamine and ethyl-centralite is a typical additive stabilizer in USA [90]. To overcome these problems, the amount of stabilizer should be decreased during manufacturing, and nitrocellulose molar mass should be decreased [89]. Thus, a proper manufacturing service lifetime can last up to 20 years.

After WW2 ended, double based propellant was replaced by composite solid propellant because of its great performance and quality [91]. But they have huge problems regarding smoke visibility. Much research on how to reduce smoke from the combustion of solid propellant has been done in the past. Generally, the composition of composite solid rocket propellant is ammonium perchlorate, hydroxyl-terminated polybutadiene, and aluminium as solid fuel. The addition of aluminium produces a lot of smoke and has become a major problem for this system. Productions of aluminium oxide produce dense smoke, making the trail visible and exposing the position of the armed forces. By replacing aluminium with the 0.5% zirconium silicate and 2% strontium carbonate, firing with minimum signature stability of propellant is achieved [92].

Besides aluminium oxide, production of HCl gas from the burning of AP propellant is also becoming a huge problem [17]. This is due to the fact that secondary

smoke formation from exhaust plume is a major disadvantages in the defence sector as well as for the environment [17], [23], [93]. Researchers use lithium to scavenge the chlorine gas from the reaction. The formulation used was Al/Li 26.6 %, coarse AP 49.2 %, fine AP 12.3 %, and HTPB 11.7 % for 20 gram solid propellant can reduce 95% HCl formation with an improvement of 7% specific impulse compared to conventional AP/HTPB/Al propellant [23], [94]. Researchers try to produce synergetic effect by combining AP with AN (Ammonium Nitrate). Many literatures already mentioned that ammonium nitrate is minimum signature oxidizer that is good for the environment, but it has significant drawbacks such as low combustion rate and phase transformation at low temperature[95]. An attempt was made to combine the properties of 30% AP and 55-80% AN in single ingredient[95], [96], [97] The result produced is not impressive enough with a burning rate 8 mm/s although it is the minimum signature propellant.

Another formulation is using nitramine as an energetic material. 60% nitramine, 20% copper oxide as a catalyst, and 20% black carbon produce burning rate 10.4 mm/s and specific impulse 232s [98]. Solid loaded carbon gel is an attractive way to produce minimum smoke but also has an impulse almost identical to the formulation using Al metal [75]. The addition of nitramine based high energetic material RDX and HMX to composite double-based propellant formulations increase energy density, burning rate and reduce exhaust smoke [99]. CL-20 (china lake compound) is new oxidizer that has high energy content, chlorine free, and minimum signature exhaust [100]. It has 14 % energy per mass and is 20% higher than HMX. This propellant is non-toxic, does not pollute environment, is lead free and contain no solid particulate free[101]. It also produces the minimum signature gas and has only a faint shock diamond pattern that is visible. Without dense smoke trails and bright flames this propellant become a close candidate to replace the current solid propellant[102]. CL-20 was mixed 90% with HTPB binder, which cures using standard isocyanates[101].

HMX was used to have dual effect on performance and improve strength. The result clearly shows that adding HMX in formulations enhances mechanical properties and improves the impact sensitivity of the ingredient [103]. By adding HMX, the performance is comparable with AP/HTPB solid propellant. Another work reported that using polyurethane as a binder produces great results [42]. This work studied a method to detect the density of smoke from solid propellant. High energy materials used are HMX, AP, AN, and NANO3 with polyurethane as a binder and Mg/Al as metal fuel. The result of this study concludes that the formulation of 60wt% AP, 17wt% Mg, 9wt%

HMX and 14wt% binder is good in the production of minimum signature exhaust gas. Recently, reports of using synergy effects on both sugar fuels have also been conducted by researchers. The synergistic effect of both sugar fuels, sucrose, and sorbitol, on rocket performance has also been studied. The remarkable result was achieved by using a conventional oxidizer, potassium nitrate. The composition of 65 wt% KNO_3, 30% wt sucrose, 3% wt sorbitol and 1 % of iron oxide as catalyst provides excellent solid rocket performance while producing less smoke [58].

Table 2.1 shows the solid propellant formulation ratio and their effect on the production of smoke according to AGARD. From the table, it can be summarized that oxidizing agent compatibility with fuel and additives is the deciding factor in achieving great performance and a clean combustion product. The additives in that table are not restricted to metal only, but polymers and plastics have also been used to achieve desired physical and combustion performance. Information from Table 2.1 showed that weak oxidizers have low performance with high smoke concentration, for example potassium nitrate/sucrose solid propellants have smoke class BA. Additives like metal oxide, polymeric material, plasticizer, and surfactant affected the propellant performance and smoke quality.

According to the NATO standard classification of minimum signature propellant, composite propellant falls into quality class CC and AC for primary and secondary smoke[104]. The improvement from level A to C is directly proportional to the amount aluminium percentage in the formulation. The classification of A level smoke is obtained when there is <1% aluminium in the formulation [105]. Results from Table 2.1 clearly show a significant change in primary smoke concentration as the number of aluminium increases. The smoke class of AP/HTPB solid propellant increased from A class to C class as the amount of aluminium increased from 1% to 18% [105][106]. The primary smoke concentration has increased since the production of aluminium oxide solid particle as a combustion product. From the Table 2.1, the secondary smoke is independent of the amount aluminum content. It stays constant because HCL gas is dependent on the amount of ammonium perchlorate. It is difficult to reduce secondary smoke classification for condensable gas, especially HCL gas from ammonium perchlorate [105].

Production of HCL gas from the combustion of ammonium perchlorate based can be reduced by synergically combining with other energetic materials due to AP having many excellent properties despite the production of HCL gas. From Table 2.1,

it indicates the smoke class of AP based propellant improves slightly to the AB class when mixed with a strong oxidizing agent CL-20 (Hexanitrohexaazaisowurtzitane or HNIW) and the energetic binder GAP (glycidyl azide polymer) [71]. Gettwert et al.[106] study the performance of GAP and ADN(ammonium dinitramide) with low smoke signature conclude that GAP based propellant can replace AP/HTPB propellant with low sensitivity. From the same work, the addition of aluminum metal causes the production of primary smoke class C, although strong oxidizers and binder were used. Research done by Lip et al. [107] mixed AP with FOX(1, 1-diamino-2, 2-dinitroethylene) as oxidizer and used GAP binder as a fuel produce smoke class AA with no secondary smoke. It proves that AP is compatible with mixing with many other energetics material and binder. Fox-7 is an insensitive, highly energetic material used in the production of minimum signature propellant. Another study was mixed with Fox-7 and AN. In this literature, GAP was formulated with an insensitive energetic filler, Fox-7/ AN as an oxidizer produce smoke class AA [39]. GAP is an energetic organic binder polymer used as a fuel and as energetic material in many solid propellant systems. Many researchers investigated GAP to produce a smokeless rocket combustion system[25], [39], [71], [108] Referring to Table 2.1, GAP is used in many solid propellant systems to produce smokeless class AA with different types of oxidizers. Few works use GAP as a binder which mixed with strong oxidizers proves to be effective in producing high quality of minimum signature solid propellant. Another polymeric energetic binder used to produce the minimum signature solid propellant is Polyethylene Glycol (PEG). Works done by Yim et al.[109] used PEG mixed with RDX/HNIW as oxidizing agents to produce smoke class AA. In this work, zirconium metal and potassium sulfate were used as additives that show no effect on smoke quality.

All works on production green minimum signature solid propellant can be summarized by the fact that types of oxidizing agents, fuel, and most importantly, additives play major factors in the concentrations of smoke. Metal additives like aluminum make a major contribution to primary smoke production while AP contributes to the production HCL gas, which can be reduced by a synergistic mix with an energetic binder and oxidizer.

Table 2.1
Solid Propellants Formulation Ratio And Their Effect On Smoke Production According To AGARD And Performance.

Propellants Formulation wt%			**Results and**	**Ref**
Fuels	**Oxidizers**	**Additives**	**remarks**	**years**
HTPB 16%	AP 83%	Aluminum (Al) 1%	Smoke Class: AC	[105]
HTPB 9.5%	AP 68%	Al 18%	Smoke Class: CC	[106]
GAP 47%	AP 33%	CL-20 20%	Smoke Class: AB	[25]
GAP 25%	RDX/HMX 60%	BuNena 15%	Smokeless Class: AA	[108]
GAP (Binder)	RDX/ PSAN	TMETN/ BTTN	Smoke Class: Na	[71]
GAP 15%	FOX 68%	Plasticizer/ Stabilizer 15%/ 2%	Smoke Class: AA	[107]
GAP 15%	FOX/ AP 48%/ 20%	Plasticizer/ Stabilizer 15%/ 2%	Smoke Class: AA	[107]
Sucrose 35%	KNO_3 65%	-	Smoke Class: BA	[9]
Sorbitol 35%	KNO_3 65%	Surfactant	Smoke Class: Na	[110]
Polyester 25%	AN 75%	Nitro- Plasticizer	Smoke Class: AA	[111]
GAP 35%	AN 65%	BuNena Plasticizer	Smoke class: AA	[39]
Resin 28%	AN 46%	Nitrocellulose 50%	Smoke Class: AC	[112]
Poly glycidyl nitrate (PGN)	AN -	CL 20 60%	Smoke Class: AA	[113]
GAP 24%	ADN 60%	Al 16%	Smoke Class: CA	[106]
GAP 25.6%	ADN 50%	FOX12/Plasticizer 20%/4.4%	Smoke Class: AA	[106]
GAP 25.5%	ADN 50%	HMX/Plasticizer 11.5%/4.5	Smoke Class: AA	[106]
Polyethylene Glycol (PEG) 37.1%	RDX/HNIW 27.9%/30%	ZrC/K_2SO_4/Additive 1.0% / 1.1% /2.9%	Smoke Class: AA	[109]

In summary, the evolution of minimum signature solid propellants has shown that smoke production is fundamentally governed by the interplay between oxidizer type, fuel selection, and additive chemistry. Double-base propellants offered smokeless combustion but suffered from long-term instability and safety issues, leading to the dominance of composite AP/HTPB/Al systems after World War II. However, aluminium oxide particulates and HCl emissions from AP have created serious challenges for both environmental impact and concealment on the battlefield. Numerous approaches ranging from metal oxide additives, lithium scavengers, nitramine oxidizers, CL-20, GAP binders, and AN/FOX-7 synergetic systems have demonstrated varying degrees of smoke reduction, with several achieving AGARD and NATO smoke classes AA, AB, or AC depending on composition. The collective findings across these studies clearly indicate that aluminium content strongly influences primary smoke, while AP governs secondary smoke through HCl production, which is difficult to suppress without replacing or modifying the oxidizer system. Overall, the literature establishes that achieving a true minimum-signature propellant requires careful optimization of oxidizer/fuel compatibility and strategic use of additives to reduce both particulate and gaseous emissions. This understanding provides the foundation for exploring new formulations particularly those combining strong oxidizers with cleaner-burning fuels to achieve low-smoke combustion without compromising performance

2.3 Performance and Characterization of Minimum Signature Solid Propellants

Solid particulate, toxic compounds and vapor are products of solid propellant combustion that emit gas into the environment. Smoke is a suspension of many small solid particles in a gas, while vapor is a set of condensed liquid droplets suspended in a gas, for an example clouds, fog, or mist [114]. The emission of smoke from the nozzle is affected not only by the characteristics of the specific rocket propulsion system or its propellant formulation but also the flight path, flight velocity, altitude, weather condition, humidity, and particular vehicle configuration. Most solid rocket propellant gases contain 5-35% water vapor and solid particle which act as nuclei upon which water vapor can condense thus making plume more visible and easier to be tracked [114]. Gas like carbon dioxide and water vapor are produced from complete combustion

of carbonaceous material. Minimum smoke or soot is seen from the complete combustion. Complete combustion occur when there is enough oxygen present in the system [115], [116]. For the burning of the solid rocket propellant, it falls under deflagration. Deflagration is defined as small number of substances of unconfined condition that suddenly ignite caused by flame, spark, shock, friction, or high temperature. When a propellant burns in a confined, the rate of deflagration will increase with the degree of confinement, and the hot gaseous product emerge away from the regressing surface [115], [116]. Hence, most of the burning in solid rocket propellant system consumes little oxygen, therefore gaseous like carbon monoxide and metal salt particles were produced [117]. Furthermore, the exhaust plume of the rocket can generate visible Infra-red, ultraviolet radiation signatures and gases such as carbon dioxide and water. The signature may be detected by radar or expose the position to the enemy.

The Advisory Group for Aerospace Research and Development (AGARD) [118], an agency of NATO, proposes the terminology for solid propellant combustion exhaust products. This method distinguishes between primary and secondary smoke based on the combustion of solid propellant products, as depicted in Figure 2.2. The AGARD smoke classification system (AA–CC) is determined from two independent measurements: primary smoke and secondary smoke. Primary smoke refers to condensed solid particulates formed during combustion mainly metal oxides such as Al_2O_3, MgO, carbonaceous residues, and other condensed-phase fragments—and is quantified using optical opacity measurements, laser light-scattering techniques, or gravimetric collection of particulate mass. A higher particulate concentration corresponds to a higher primary smoke class (A → low, C → high). Secondary smoke, on the other hand, originates from gaseous chlorine-containing combustion products such as HCl, ClO, and NOx that condense or become visible in the exhaust plume. These gases are measured using techniques such as FTIR gas analysers, laser absorption spectroscopy, titration of HCl absorbed in water, or thermochemical modelling (e.g., CEA, CHEETAH). Propellants producing large amounts of HCl are placed into higher secondary smoke classes (A → low, C → high). The combination of these two independent parameters determines the final classification (e.g., AA, AB, BC, CC), reflecting overall exhaust visibility and signature level

For primary and secondary smoke three categories were proposed: A, B and C. A category is least smoky, while a C category is most smoky. According to AGARD,

primary smoke is the result of condensed solid particle materials, for example metals, carbon, and metal oxide thereby resulting in the formation of a particle cloud in the atmosphere downstream from the nozzle. The main contributor to primary smoke is catalyst, anti-instability, additive, after burning suppressant and metals to increase thermodynamic performance. Gaseous products found in exhaust rockets are mostly metal compound are copper hydroxide, potassium hydroxide and aluminium oxide. Besides that, anti-instability additives do not decompose in the combustion chamber and contribute as exhaust products. Other than propellant products, material motor parts that are exposed to flame may ablate and generate smoke. Their effect is significant, typically in the form of carbon soot, silica, and iron oxide [104]. Finally, the igniter may also play an important role in the generation of primary smoke.

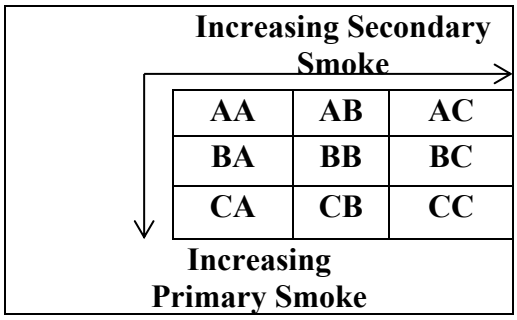


Figure 2.2 Classification of Solid Combustion Exhaust Product

For secondary smoke, AGARD states that it results from the condensation of water and acid vapor. It occurs from the interaction between the exhaust plume and the atmosphere. It is different from primary smoke, which is the result of solid particles, and it is independent of ambient conditions. Secondary smoke forms when the local vapor pressures of the condensable species exceed their saturation vapor pressure [104]. Secondary smoke mainly contains water vapors, chloride salt, and fluoride salt. Secondary smoke can form within a primary smoke to give total sum of individual effect on visibility and obscuration. The formation of secondary smoke is totally dependent on condensable species in motor exhaust, concentration of solid product, relative humidity, and ambient temperature [104].

During a reaction in the combustion chamber, solid propellant breaks apart into its constituent atoms. This is quickly followed by the rearrangement of atoms into more stable compounds. These molecules are usually water, carbon dioxide, carbon monoxide, nitrogen gas, hydrogen gas, carbon, aluminum oxide and sulfur dioxide.

They can be found in all combustion and explosive product materials. The nature of the product will depend on the amount of oxygen content during the reaction take place [11]. Alternatively, the amount of oxygen content will depend on quantity of oxidizing atoms that are present in solid propellant molecule [11]. The quantity of oxygen consumed in combustion reactions will affect the type of gases released to environment. Not enough oxygen in molecules to oxidize reactants will produce toxic gases such as carbon monoxide instead of carbon dioxide and water.

Most energetic materials contains, C, H, N and O atoms [119]. During the reaction, the molecules use oxygen provided by the oxidizing agent. Heat release and heat absorbed during reaction known as heats of formation. When energetic materials do not contain enough oxygen to combust, they will produce carbon monoxide, carbon soot, and hydrogen gas. The heat energy released during the formation of these gases is known as “heat of explosion”. Conversely, reactant burn in excess oxygen to form substance like carbon dioxide, and water, liberated heat added with the heat of explosion would be equal to the heat of combustion [11], [119].

2.3.1 Green Oxidizing Agent Solid Propellants Performance

Rocket propellant is a tool that develops thrust through the rapid expulsion of matter. This high speed fluid is created by the high pressure combustion of liquid or solid propellant, consisting of fuel and oxidizer components in the combustion chamber [120]. Specific impulse is an important parameter for rating the efficiency of rocket propellant, which indicates how many pounds of thrust are obtained by the consumption of one kilogram of propellant in one second. Basically, propellant formulations with different mass ratios and additive play a major role in the performance of solid rocket propellant [121]. Table 2.2 gives the details of the performance characteristics of a few solid propellant formulations using advanced green oxidizers and energetic binders in comparison with solid propellant base line. Addition of energetic binder GAP (Glycidyl Azide Polymer) and CL-20 (Hexanitrohexaazaisowurtzitane) to AP formulation synergistically improve the propellant performance compare. Oyedeko et al. [121] performed the characterization study on the performance of potassium nitrate-sucrose with 2% carbon and 3% magnesium as an additive material. Burning products from this formulation generate a lot of smoke due to the presence of carbon and magnesium. Excessive gases like magnesium oxide, carbon monoxide, chloride gas and magnesium

carbonate could be harmful to the environment. Hence, new green oxidizers with better solid propellant performance need to be studied more thoroughly.

Table 2.2
Performance of Solid Propellant with Advanced Oxidizing Agent

Composition WT%	Specific Impulse(s)	Gap Identified	Ref
14% HTPB 68%AP 18% Al (Base line)	264.5	Nano-Al increased burning rate up to 100%; higher Isp; enhanced ballistic performance.	[122]
58%GAP 22%AP 20%AL	266.0	Novel PGN-based energetic plasticizers improved thermal stability and compatibility; raise Isp	[123]
22%GAP 56%AP 22% (70:30) Al/Mg	260.3	GAP-coated Al improved energy release; faster reaction under ultrafast stimulus; superior underwater blast performance.	[124]
55%GAP 23%ADN 20%Al	276.5	ADN is highly sensitive, hygroscopic, chemically unstable, storage-limited, and costly.	[76]
55% GAP 25%CL-20 20%Al	267.1	High cost, complex foaming, extreme sensitivity, and high energy unsuitable for minimum-signature tactical propellants	[125]
60% GAP 25%HNF 15%Al	277.2	HNF/HNE increased energy performance; improved Isp and stability.	[126]

Russian scientist in the 70s had come out with an oxidizer ADN (ammonium dinitramide) is inorganic strong oxidizer. ADN is a promising candidate for a green oxidizer alternative to AP solid rocket propellants. It's not only environmentally friendly but also overcomes the limitation in performance possessed by AP/HTPB. Few scientists had done characterization studies on ADN based propellants. They found that ADN decomposition happened above the melting point. Gaseous emission of N_2O, NO_2, H_2O, CO_2, CO, N_2 and NO were detected during ADN decomposition [127]. Coating agents in the ADN pellet improve the thermal stability of ADN at temperatures above the decomposition temperature, while at low temperature the addition of coating agents seems has no effect on the thermal stability of ADN [128]. Result of this study concludes that ADN crystal and pellet are sensitive to impact. Moreover the ADN/GAP propellant has significant minimum signature exhaust product of solid propellant burning compared to AP/HTPB [106]. V.Gettwert et al.[106] conducted an experiment on performance of ADN/GAP propellants in comparison AL/AP/HTPB. The results from this investigation show that the specific impulse for the ADN/GAP propellant is 2247 Ns/kg significantly higher compared to the AL/AP/HTPB propellants which is 2070 Ns/kg.

ADN based propellants offer great performance for rocket propellants with high impulse, and no secondary smoke is produced [76]. It works well with polymeric binder such as GAP and HTPB gives excellent results on the combustion performance of ADN propellant [89], [90]. Koji Fujisato et.al [131] investigated the combustion characteristics of ADN based solid propellants with HTPB as a solid propellants binder for this research. They found that the burning rate of HTPB/ADN was faster than that of HTPB/AP. In addition, thermal properties of binder affect their combustion rate characteristics, thus more research needs to be studied on the compatibility of binder with ADN propellant. It is becoming a major workhorse today for rocket propellant applications to replace AP based propellant. ADN is classified as 'class C' minimum hazard material. The products of ADN based combustion are usually ammonia, water, N_2O and NO_2. The problem regarding ADN is that it is very sensitive to moisture, rapidly decomposes, and has chemical incompatibility with some compound in composite propellants [132], [133]. Hence, efforts have been made to improve the chemical stability of ADN. Jessica de Olivera Silva et al. [133] investigate ADN recrystallization and microencapsulation with HTPB by using simple coacervation method of separating macromolecules into two immiscible liquid . ADN was made into spherical pellets to ease processing and casting. Chemical stability between the capsule (HTPB) and ADN happened at 60˚C. Hence, ADN pellet are key product for environmental friendly and low signature solid composite propellant for defence and space applications [133], [134].

Another effort was done by Thao T.Vo et al. [135] using nitration of Fox-7 to produce tetranitroacetimidic acid (TNAA) for replacing ammonium perchlorate in solid rocket propellant systems. TNAA was found to have a slightly higher specific impulse at 209 s and was slightly more stable than ammonium perchlorate based. An attempt has been made to use Fox-7 as a high energy oxidizer for replacing the minimum signature double base propellant in solid rocket motors. In this work, Fox-7 was substituted for nitramine filler to be used in the GAP-RDX formulations [108]. The result exhibited reduced shock sensitivity with increasing content of Fox-7, while losing performance efficiency. FOX-7 was first produced by Laytpoz in 1998. FOX-7 is a great oxidizing agent and has been considered to be used in solid rocket propellant system [136]. This compound's performance is comparable to the high energetic material HMX, RDX and PETN [137]. FOX-7 was mixed with the binder containing AP/AL to study its performance, thermochemical, and ballistic properties [138]. The

result from this work explained that the incorporation of FOX-7 instead of AP cause a reduction in combustion energy, specific impulse, burning rate and temperature [138], [139]. In other work, research to investigate the properties of propellant formulation based HTPB/FOX-7 using DSC and a sensitivity test apparatus has been done. The result showed that impact sensitivity was over 25.0 J and friction sensitivity was less than 68%. If compared with the HTPB/FOX-7 formulation with the RDX formulation the mechanical sensitivity and electrostatic discharges of FOX-7 based are decreased. Recently, a report about the comparison effect of FOX-7 and HMX on the properties of AP/AL/binder concluded that the specific impulse value of 251 s, density of 1.91 g/cm3 and burning temperature of 3600 k can be synthesized using 60% FOX-7 and 19% active binder [140]. In another literature, formulation of FOX-7/AP/GAP propellants with 68% to 70% solid loading including 20% to 42% AP and TMENTN/1, 2, 4-butandiole trinitrate (BTTN) nitrate ester plasticizers was prepared [141]. These formulations showed a high burning rate at 20mm/s, better chemical stability, low thermal sensitivity, and low smoke combustion. Jensen et .al [108] report that FOX-7 is not suitable to substitute for minimum signature GAP/RDX composite rocket propellants that have lower shock sensitivity but have good mechanical properties [142].

Research to find the minimum signature combustion product and environmentally friendly oxidizing agent has moved to a new level. A new oxidizing agent, Hexanitrohexaazaisowurtzitane (CL-20), was synthesized to meet the current requirement for a solid propellant that is less smoky and greener for the environment. Literature studies on comparative performance of CL-20/RDX-based solid propellants were done [143]. Analyses of the combustion performance of CL-20-based solid propellant show an increase, as the composition of CL-20 is increased. CL-20 is a promising oxidizing agent to replace other energetic materials such as AP, HMX or RDX to meet new requirements which are eco-friendly, high rocket performance, and minimum signature propellants for application on futuristic missiles and space mission.

CL-20 is cage-like structure that can release energy at a much higher level than HMX or RDX. Its high heat of formation (+454 KJ/mole) is due to the strain introduced by the cage like structure and presents 6 N-NO_2 groups [144]. It is high density by (>2.04g/cc) energy material due to the close packing of the constituent atom shape like cage structure[125], [144]. It is the cleanest combustion efficient oxidizer for future propellants. Furthermore, it has a good oxygen balance (-11%), good thermal stability

and is practical to apply in solid rocket propellant. It is bonded with other plastic additives such as GAP which 85-95% CL-20 [125]. CL-20 formulation showed a higher burning rate compared to HMX and AP based propellant[145]. A series of nitramines arranged around a cyclo-butane ring from Cis and Trans-isomer. The Cis configuration of CL-20 is commonly used to boost energy in rocket propellant. Figure 2.3 show a summary of green oxidizers with theirs physical and chemical properties.

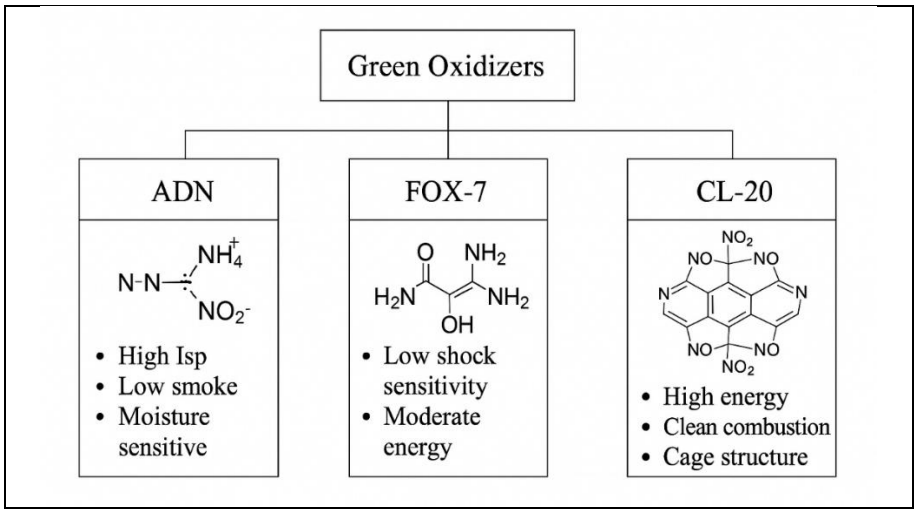


Figure 2.3 Classification Of Green Oxidizers For Solid Rocket Propellant

Olena Kositsyna et al. [146] have calculated specific impulse for 45 different compositions of solid propellant based on green oxidizing agents (ADN, Hydrazinium Nitro-Formate (HNF), CL-20) and polymer binders (HTPB, GAP, poly-3-nitratomethyl-3-methyloxetane(Poly-NIMMO)). The results from the calculation of 45 compositions could be used as preliminary forecasting of energetic characteristic performance as well as their ratios. This work concludes that maximum values of energetic characteristics were obtained for compositions based on an energetic binder at 80% oxidizer content. In addition, the oxidizer-binder ratio is optimal at 80%: 20%, and the most favorable components are HNF, HNIW, GAP, and poly-NIMMO. HNF is an oxidizer that is far superior to ADN. It was produced back in the 80s in the Netherland. It was synthesized by acid-based reaction between hydrazine and trinitromethane in ethylene dichloride. HNF has improves by 7% in specific impulse compared to conventional AP/HTPB propellant [147]. It has a heat of formation (ΔH°_{f}18kcal/mol) higher than ADN, higher melting point, and is less hygroscopic than ADN. Its stability highly depends on the purity of final product during production [126]. This is due to the dependence on hydrazine content. Besides that, HNF is compatible

with the GAP polymeric energetic binder. The HNF/AL/GAP combination is a very promising high-performance propellant, according to the European Space Agency (ESA). HNF has many advantages such as lower signature, no pollution, and high specific impulse compare to Ammonium perchlorate [148], [149], [150]. This is a strong oxidizer; it has high burning rate from a formulation of HNF/GAP/AL result as reported in the literature. The burning rate and ballistic properties of HNF are dependent on particle size. Affected HNF size (mean size 5-10 μm) lowers the pressure exponent of the propellant. Hence burning rate of the propellant can be tuning by adjusting HNF particle size [151]. Aside from being a good high-performance propellant, it has disadvantages such as sensitivity towards impact and friction [152].

Over the last few years, substantial work has been performed on producing novel green energetic oxidizers, binders, and plasticizers to comply with the regulations, together with insensitive munitions. Many attempts have been made to produce a new green solid propellant without compromising the propellant's performance. Regarding the toxic gas produced by the combustion of solid rocket propellant, which is harmful to humans and the environment. It is notable that the smoke produced will be exposed in the soldier position to the enemy. In terms of defence, smoke produced from propellant combustion becomes a signature for enemies tracking of missile trajectory, revealing the launch location of the missile, and causing the loss of control of optically guided missiles. Thus, finding the green, minimum signature, and high-performance solid rocket propellant is a top concern and needs further effort to achieve its objectives.

Table 2.3 shows the part of oxidizer/fuel composition with additive by weight percent with their propellant performance and smoke production class according to Advisory Group for Aerospace Research and Development (AGARD) after solid propellant combustion. It illustrated that oxidizer and fuel play important roles in determining the production of smoke from combustion of propellant. Due to its natural properties as weak Oxidizing agent Potassium Nitrate depict the low performance and produce more smoke compared to other oxidizers. From the Table 2.3 it shows that, most high energetic oxidizers with high oxygen balance produce no or less smoke during combustion of propellant. With the help of an energetic fuel for combustion the reaction produces higher performance with less smoke at all. Besides that, the presence of AP as oxidizing agent proved that combustion product produced chloride gas. The addition of additive materials like plastic binder or additive metal shows significant reduction in chloride gas content. Hence compatibility of oxidizing agent with fuel and

additive is major role in deciding whether the combustion produce smoke or not. On the hand they also provide good solid propellant performance with high specific impulse.

Table 2.3
Composition And Performance Characteristic Of Solid Propellants With Difference Greenoxidizers.

Composition wt.%				Specific Impulse Isp(s)	Smoke Production	Ref.
Oxidizer	Fuel/Binder	Additive	Others			
46% AN	16.2% HTPS *(50%Nc/18%IP DI/28.2%HTPS)*	24% Al	1%Dessavin 1%Glycerin 1%Silicon	253.93 s	0% HCl Class AA	[112]
65% KNO_3	35% Sorbitol	-	-	110 s	0% HCl Class CB	[48]
58% HNF	24% Energetic Binder	18% Al	-	273.1s	-	[153]
41% TKX-50 16% Ap	10% GAP	18% Al	15% NG	278s	3% HCl Class AB	[154]
54% CL-20 3% AP	10% GAP	18% Al	15% NG	275s	4% HCl Class AB	[154]
63% ADN	18.6% Ammonia	18.4% Methanol	-	250s	0% HCl Class AA	[155] [76]

Although ammonium perchlorate remains widely used due to its stability and proven performance, its production of HCl and dense exhaust plume continues to motivate the search for greener alternatives. Modern oxidizers such as ADN, FOX-7, CL-20, and HNF offer improved specific impulse, reduced smoke, and cleaner combustion, particularly when combined with energetic binders like GAP or poly-NIMMO. However, each candidate faces limitations including moisture sensitivity, thermal instability, mechanical sensitivity, or reduced performance when substituted directly into AP-based formulations. Studies consistently show that propellant performance and signature depend strongly on oxidizer–fuel compatibility, oxygen balance, binder interaction, and the role of additives in modulating combustion behaviour. Table 2.2 and Table 2.3 clearly highlight that high-oxygen, high-energy oxidizers tend to produce minimal smoke, whereas weak oxidizers such as KNO_3 yield

poor performance and heavy emissions. Overall, while considerable advancements have been made, the literature reinforces the need for continued exploration of green oxidizer–fuel systems that balance performance, stability, safety, and minimum exhaust signature for next-generation defence and aerospace applications.

2.3.2 Burning Rate Measurement Using Ballistic Evaluation Motor (BEM)

The Ballistic evaluation motor (BEM) is very popular in rocket propellant field, especially for the evaluation of ballistic properties. These give an insight into the burning rate of propellant at various operating temperatures and pressure ranges [156]. Among all kinds of data that can be obtained experimentally by a Ballistic Evaluation Motor (BEM), there is one especial property interpreted as burn velocity versus time, also known as burning rate. This property is related to the chamber pressure and temperature during the steady state burning grain (the rigid mass of propellant with specific form, geometry and burning area). Research concerning burning grains can be obtained by experimental tests, using a Crawford bomb (with this equipment, the combustion pressure is previously fixed) or one Ballistic Evaluation Motor by applying analytical relations between the pressure curve and propellant burning time [120].

Based on industry requirements, a Ballistics Evaluation Motor (BEM) was created to determine performance characteristics and stable regression behavior across a wide pressure range in a single test [48]. More Ballistic evaluation Motors provide a more feasible operating environment, both in terms of gas and temperature. In contemporary propellant laboratories, several methodologies coexist and are utilized to evaluate the propellant's ballistic performance [157]. The performance of a Ballistic Evaluation Motor depends on factors such as burning rate modifiers, plasticizers, curing agents, and nozzle design [158]. A solid rocket motor nozzle is finely formed behind component of the thrust chamber that controls the expansion of exhaust products, converting the energy forms created in the combustion chamber into kinetic energy effectively hence providing thrust to the vehicle. This is accomplished by creating the necessary nozzle geometric profile; the typical rocket motor nozzle is convergent-divergent. This nozzle is subjected to extremely high pressures and a quick, dense gas flow at high temperatures. A nozzle must be made of a material that can sustain structural and thermal load.

Shekhar et al.[156] study start port propellant grain for Ballistic evaluation motor (BEM), used 2.5 kg solid propellant and has dimensions of OD 115 mm, ID 60 mm, length 200 mm with throat diameter around 30 to 50 mm. This study found that at pressure between 8-10 Mpa need to burn solid propellant around 7-9 s. Another study Terzic et al.[159] predict ballistic parameter of different grain solid propellant using ballistic evaluation motor (BEM) with designed nozzle for between 14- 29 mm. From this study the maximum pressure achieve is 21 Mpa with burning time 6s. The longest burning time recorded in this study was 15 s and the highest thrust recorded is 19200 N. Same study done by Ame'rico et al.[49] measure burning rate of potassium nitrate/sucrose solid propellant using developed BEM. In this study the BEM used 5 different nozzle sizes to generate different chamber pressure. In this investigation, the burning rate is plotted against the chamber pressure to validate the result of burning rate from numerical estimation. Further study to investigate the effect of casting technique on solid propellant performance was done by Ramesh et al. [160] therefore to achieve a relatively high burn rate of about 13-14 mm/s at 7000 kPa, the propellant's solid loading was increased from 85.15% to 87.27% using ammonium perchlorate and process aids, without altering burn rate or mechanical attributes. In this study, the altered mixture was tested for several parameters including end-of-mix viscosity, density, mechanical, and ballistic qualities. The cured propellant's density increased from 1.74 g·cm-3 to 1.79 g·cm-3, but mechanical characteristics remained unchanged. The findings of this investigation show that the modified composition's performance index has increased from 416 to 437, which is supported by the results of ballistic evaluation motors weighing 2 kg. Similar study

Raj Alexander et al.[161] developed a ballistic evaluation motor for testing a solid propellant based on ammonium perchlorate with a high burning rate and density loading. In this investigation, the BEM measured a high burn rate of 23 mm/s at 35 bars. Moreover, burn rate measurement at 10 bar, 20 bar, and 35 bar is accomplished by evaluating three alternative nozzle diameters (7 mm, 6 mm, and 5.5 mm). Olde et al.[110] employed a potassium nitrate-based sorbitol solution to monitor burning rate in BEM and analyze the underlying cause of reported BEM ignition failures. This investigation discovered an issue with the ignition of potassium nitrate sorbitol solid propellant with surfactant and fine potassium nitrate. This inspired the development of laser ignition research to evaluate KNSB combustion behavior. Propellant samples of various compositions were ignited using a 1064 nm laser. Samples with fine particle

dispersion and surfactant had a distinct flame structure become the reason for failure ignition. This study suggests that reducing incoming flux to 250 W/cm2 would result in an estimated ignition transient of around 0.5 s for uncoated KNSB propellant, as a compromise between detecting transient behavior and restricting heat conduction throughout the sample [162]. The literature clearly shows that the Ballistic Evaluation Motor (BEM) is a critical experimental tool for determining the burning rate, pressure behaviour, and overall ballistic performance of solid propellants under realistic operating conditions. Various studies demonstrate that BEM performance is strongly influenced by grain geometry, nozzle configuration, chamber pressure, propellant formulation, and ignition characteristics, with reported burning rates and thrust outputs varying widely depending on these parameters. Research also highlights the importance of modifying oxidizer loading, particle size, binder compatibility, and casting techniques to achieve stable regression and improved ballistic efficiency. Furthermore, several investigations reveal challenges associated with ignition sensitivity, pressure stability, and formulation-dependent response, reinforcing the need for controlled and well-designed BEM testing.

2.3.3 Effect of Oxygen Balance on Solid Propellant Performance

The oxygen balance is a method in the high energetic material field to evaluate the properties of materials [10]. It tells the researchers whether the energetic materials can complete combustion or vice versa. It is divided into positive and negative oxygen balances, which serve as predictors of the type of combustion [10]. A positive oxygen balance (OB) indicates that they could be used as energetic oxidizers. Positive OB of oxygenated materials can produce less harmful gas than negative OB. A very high positive oxygen balance means it is a high energy dense oxidizer. All carbon, metal, and hydrogen can be converted into carbon dioxide, water, and metal oxide by an energetic with a zero or positive oxygen balance[163]. Simultaneously, by introducing nitro chemicals into the formulation, the oxygen balance can be modified [164].

By paying attention to the type of oxygen connections in the explosive molecules, it can be described as a linear function of oxygen balance. The oxygen balance cannot be too negative or too positive, and only values near to zero are preferred, the stoichiometric mixture ratio between oxidizing agent and reducing agent is used [13], [165]. By referring Figure 2.4 the relation of Oxygen Balance of many

strong oxidizing agents in relation with Heat of explosion. As the oxygen balance approaches zero the heat of explosion increases regardless positive or negative oxygen balance. These parameters can help the researcher to regulate the amount of oxidizing and reducing agent to get optimized value of performance.

The minimum amount of oxidizer required to achieve complete combustion in a solid propellant is governed by the stoichiometric oxygen balance of the formulation. Studies consistently show that propellants must maintain an overall oxygen balance close to zero $OB \approx 0\%$ to ensure that the oxidizer provides sufficient oxygen to fully oxidize the fuel without generating soot or carbon monoxide [166]. For composite AP-based propellants, this typically requires an oxidizer content of about 68–72 wt% [167], while sugar-based KNO_3 propellants require more than 72 wt% KNO_3 due to their oxygen deficiency [58] . High-oxygen oxidizers such as ADN and AN achieve complete combustion at lower thresholds approximately 55–60 wt% for ADN and 60–65 wt% for AN [76], [107] These findings demonstrate that oxidizer content is the primary determinant of combustion completeness and directly influences smoke formation and exhaust quality

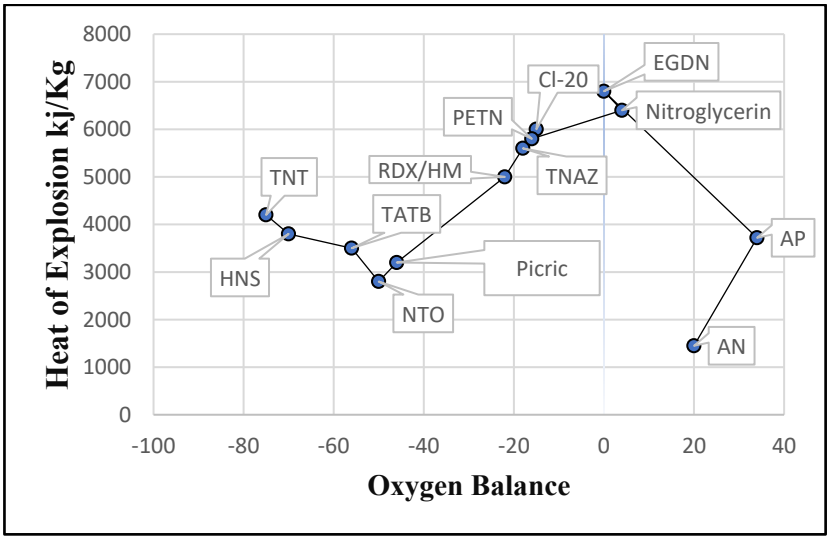


Figure 2.4 Heat Of Explosion For Different Energetic Materials With Respective To Oxygen Balance [13]

In terms of thermodynamics, the use of the thermodynamic equation of state of the reaction products has made it possible to evaluate the chemical properties of propellant. As depicted in Figure 2.4 and Figure 2.5 Heat of explosion reaches a maximum with an oxygen balance approaching to zero because this corresponds to the

stoichiometric oxidation of carbon to carbon dioxide and hydrogen to water [168]. Furthermore, in study of performance explosive properties the result show that -NH2, -C=O, and -N-O groups improve oxygen balance and hence increase heat of formation [169]. Consequently, it may result in the desirable outcome of better stability, higher density, and hence higher detonation velocity and pressure. The study on the flow properties of solid rocket motor plumes shows that Hydrogen/oxygen/chlorine chemistry has a substantial influence on solid rocket motor plumes [170]. The findings indicate that the choice of kinetic model for combustion propellant has a greater impact in oxygenated plumes. The calculation conditions for this study, which the theoretical would be near to the measured calorimetric heat were identified through a comparison of the measured and calculated values of the heat of combustion for a few propellants [171]. H.Muthurajan et al.[168] used computer code LOTUSES to predict the explosive mixtures properties such as heat of formation, velocity detonation. This computer code optimized the value of mixed explosive combination to get high value velocity detonation and compute amount of oxygen balance for the explosive mixture. The study revealed that as oxygen balance approach to zero the detonation velocity is increased [172]. Another work done by Alipoor et al. [173] calculate detonation velocity of mixed explosive for TNT and AN (Ammonium Nitrate) using chemical equilibrium application software. The result conclude from this work is that oxygen balance in condensed explosive is key parameter to determine the optimized detonation properties and [173]. Qiong et al.[174] substituted aminofurzan into N-trinitroethyl with good oxygen balance to study its performance of detonation and heat of formation. The compound contains a good oxygen balance within the range -15.31%-0% and shows good thermal stability at 159-230°C with high positive heat of formation (268-1259.5 kJ/mol). Furthermore nitramine substation helped the compound to has zero oxygen balance with high detonation velocity 9486 ms^{-1} [174]. From the same author, Qiong introduce the trinitromethyl group into 1,3,4-oxadiazole has very high positive oxygen balance (39.12%) with high detonation velocity 9266 ms^{-1} [175]. On the other hand, there is no standard approach for calculating the oxygen balance of metal-containing energetic materials due to the uncertainty of the detonation of product metals. The most common technique, however, implies that the metals be transformed into metal oxides [176].

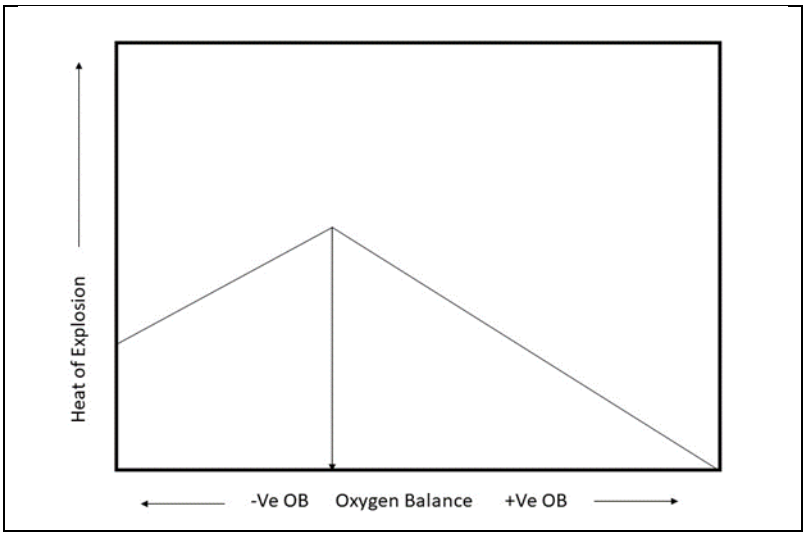


Figure 2.5 Effect of Oxygen Balance on Heat of Explosion

Because the type of product created from propellant combustion is heavily dependent on the oxygen balance of the propellant, knowing the composition of gaseous products formed is critical for determining heat of formation and other chemical attributes. There are numerous guidelines for figuring out the decomposition products, but they only give an understanding of the decomposition products. They are necessary when calculating the heat of combustion, but they provide no information regarding the energy released during decomposition. Hyun et al. [177] compare various methods for calculating the performance of energetic materials. In this work, the products were determined using several rules Kamlet-Jacobs (K-J), Kistiakowsky-Wilson (K-W), and modified-Kistiakowsky (modified K-W).The model K-W rule was found to be the most accurate when compared to others [177]. Another comparison approach for estimating velocity and detonation pressure of high energetic materials was investigated by employing the kamlet method based on K-W principles [178]. The results of the heat of detonation calculation demonstrate that the K-W technique was more accurate. Paraschiv et al. [179] assess three combustion models for determining the heat of explosion. The authors employed the Le Chateliar-Millard method in this paper, which yielded a decent result with a 7% error for determining the heat of combustion. The L-M model produced the best results, which were extremely close to the experimental results. The Kamlet-Jacobs model produces results with a significant level of inaccuracy. The conclusion of this experiment is that the chemical composition of the propellant is critical in predicting performance parameters [179].

Chen et al. [43] optimize oxygen balance by changing the A-site cation in

molecular perovskite by trimming carbon/hydrogen and adding oxygen atom into perovskite molecule. In this work calculation on energetic materials performance is using density functional theory (DFT) and Kamlet-Jacob equation. The study found that oxygen balance of molecular perovskite changes from -22.0% to -3.9% and increases in crystal density from 2.02 to 2.07 gcm^{-3}. This study revealed how to determine the A-site cations in a perovskite structure rationally for altering the properties of molecular perovskite high-energetic materials, providing vital indications for building more sophisticated energetic materials for practical usage.

Table 2.4 shows the various oxidizing agents with their oxygen balance and performance. Depicted from the table, DOTZ (1,4,2,3,5,6-dioxatetrazinane) oxidizer with zero oxygen balance has high specific impulse with high heat of formation. On the other hand, ammonium perchlorate has the lowest performance compared to others despite it having a positive oxygen balance.

Table 2.4
Oxygen Balance And Performance Of Advanced Oxidizing Agents

Oxidizer	Oxygen Balance (%)	Specific Impulse Isp	Limitation and research gap	Heat of Formation KJ/mol	Ref
Ap	+34.04	158.76	Extremely high OB oxidizers with high predicted impulse. Not physically stable and no propellant integration	-290.45	126]
HMX	-21.63	265.72	-	75.02	126]
RDX	-21.61	265.06	-	61.53	126]
CL-20	-10.95	272.81	-	415.80	126]
$[NF_2O]^+$	+47	264	Extremely high OB oxidizers with high predicted impulse. Not physically stable and no propellant integration.	-624	180]
DOTZ	0	297.9	High-performance, zero OB oxidizer with predicted Isp=298 s. No real combustion system; unstable synthesis	265.7	181]
DNDOTZ	+43.9	230.9	Shows link between detonation products and performance. no AP or green fuel systems	410.5	181]
TNT	-174	268.8	High-energy and chlorine-free; good specific impulse. ADN is moisture-sensitive, expensive	-67	182]

Materials with oxygen balance values near zero consistently exhibit higher heat of explosion, greater detonation velocities, and more efficient conversion of fuel elements into fully oxidized gaseous products. Studies show that the required oxidizer fraction for achieving OB ≈ 0% varies widely among systems, with AP composites typically needing 68–72 wt% oxidizer, KNO_3–sugar propellants requiring over 72 wt% KNO_3, and high-oxygen oxidizers such as ADN and AN reaching complete combustion at substantially lower loadings. Numerous computational and experimental approaches including thermochemical models, EOS calculations, and predictive formulations such as the K-W rule confirm that oxygen balance strongly correlates with performance indicators such as heat of formation, detonation velocity, and plume composition. Furthermore, molecular design strategies, such as nitro-substitution and perovskite cation modification, demonstrate the ability to tune oxygen balance and thereby optimize energetic behaviour. Overall, the studies highlight that achieving an appropriate oxygen balance is essential not only for maximizing propulsion performance but also for controlling smoke, toxic by-products, and plume visibility—factors that are central to the development of minimum-signature, environmentally responsible solid propellants.

2.3.4 Thermal Decomposition & Kinetic Analysis

Thermal decomposition and stability studies have been integral to understanding the long-term viability of minimum signature propellants. Tomaszewski et al. [183] investigated the effects of processing solvents on nitrocellulose decomposition, providing insights into thermal stability and degradation mechanisms. Such studies are critical in ensuring that minimum signature formulations maintain their structural integrity and safety over extended storage periods**.**

The thermal breakdown of AP that occurs at the orthorhombic-to-cubic phase transition temperature of 240°C is referred to as low-temperature decomposition. This stage ends with a 30 wt.% loss and the cessation of decomposition. When the temperature climbs over this point, the decomposition process continues until it is completed. It is generally important to understand the mechanisms of ammonium perchlorate (AP) low-temperature breakdown and how the addition of additives may affect decomposition rates. Proton transfer and electron transfer mechanisms are the two main pathways for AP low-temperature breakdown that have been proposed in the

literature [184]. According to the proton transfer mechanism, ammonia and perchloric acid are formed when a proton is transferred from the ammonium cation, causing breakdown. This method is more commonly acknowledged. However, the electron transfer mechanism suggests that the molecule breaks down into NH4 and ClO4 radicals at low temperatures because of an electron transfer.

Differential Scanning Calorimetry (DSC) and Thermogravimetric analysis (TGA) are useful method for evaluating the kinetic parameters and thermal properties of energetic materials. It has been shown by Kissinger [185], Ozawa [186], and Flynn [112] that DSC relies on the linear correlation between peak temperature and heating rate. The extensively used Ozawa method, also referred to as an iso-conversional method, is especially noteworthy for its use in linear heating rate activation energy estimation. Dynamic analysis has been useful in understanding the total thermal degradation kinetics of polymers and composites, even if thermal analysis alone may not fully explain the entire mechanism of thermal deterioration. Important parameters including the frequency factor (A), activation energy (E), and overall reaction order can be reliably determined using this method.

The use of kinetic models has provided deeper insights into the reaction mechanisms of AP-based propellants. Additionally, Wang et al. [187] analysed the thermal decomposition of HTPB/AP and HTPB/HMX mixtures using TG-FTIR and Raman spectroscopy. Their results indicated that HTPB decomposes in a two-stage process, with significant gaseous product formation under an inert atmosphere. The presence of AP influenced the reaction pathways, leading to different gas-phase and condensed-phase interactions. These insights contribute to improving fuel-rich propellant formulations by optimizing oxidizer content and decomposition efficiency. Xue et al. [188] developed a micro-scale combustion model for AP/HTPB composite propellants, analysing the flame structure and transient burning rate behaviour. The study demonstrated that a decrease in environmental pressure from 3 MPa to 2 MPa led to a sharp decline in transient burning rate, with flame extinction occurring below a critical temperature of 700 K. Their findings emphasize the need for pressure-dependent kinetic models in predicting combustion instabilities in solid propellants.

The decomposition of AP-based propellants has been widely studied to understand its multi-stage reaction mechanisms. Zhang et al. [189] investigated the thermal decomposition kinetics of naturally aged AP/HTPB base bleed composite propellants using DSC under different heating rates. The study revealed that the

decomposition occurs in three stages: phase transition of AP at 513 K, low-temperature decomposition below 587 K, and high-temperature dissociative sublimation near 830 K. The activation energy of naturally aged samples was found to be lower than laboratory-aged samples, indicating the effects of long-term aging on degradation kinetics. Another significant study by Gonçalves et al. [190] analysed the thermal decomposition kinetics of aged and non-aged AP/HTPB composite propellants using DSC and the Ozawa method. The activation energy of non-aged samples was 134.5 kJ/mol, while aged samples showed a reduced value of 79.0 kJ/mol, highlighting how aging affects the energetic properties and stability of solid propellants. These findings emphasize the importance of storage conditions and aging mechanisms in propellant performance.

The thermal breakdown process of AP is quite complex [191]. The perfect mechanism of thermal decomposition of AP has not been understood totally yet. According to the proton-transfer mechanism stated by Jacobs et al. [192] AP decomposes into ammonium ion and perchlorate ion show in (2.1) , followed by proton transfer from ammonium ion to perchlorate ion (2.2), leading to ammonia and perchloric acid formation in (2.3) . The remaining ammonia and perchloric acid are sublimated into the gas phase in (2.4). The lower decomposition temperature of AP is as follows:

$$NH_4ClO_4 \rightarrow NH_4^+ + ClO_4^- \quad (2.1)$$

$$NH_4^+ + ClO_4^- \rightarrow NH_3 + HClO_4 \quad (2.2)$$

$$4HClO_4 \rightarrow 2ClO_3 + 2ClO + 2H_2O + 3O_2 \quad (2.3)$$

$$2NH_3 + 2O_2 \rightarrow N_20 + 3H_2O \leftrightarrow NH_4NO_3 + H_20 \quad (2.4)$$

At high temperatures:

$$4HClO_4 \rightarrow Cl_2 + 5O_2 + 2ClO_2 + H_2O \quad (2.5)$$

$$2Cl_2 + 2H_2O \rightarrow 4HCl + O_2 \quad (2.6)$$

$$2NH_3 + 2Cl0_2 \rightarrow N_2O + Cl_2 + 3H_2O \quad (2.7)$$

$$N_2O + O_2 \rightarrow NO + NO_2 \quad (2.8)$$

The main reasons of the completion of AP thermal degradation are the catalyst's role in O_2 production and gas absorption on its surface. The prior studies [193] suggest that the catalytic mechanism of metal oxide for AP can be generally characterized as follows: in the middle of the AP thermal decomposition process, the metal oxide interacts with ammonia, perchloric acid, and the corresponding decomposition products via hydrogen bonding or coordination with Lewis acid sites; this disrupts the AP decomposition reaction balance and causes the reaction equilibrium to shift in a direction that will speed up the AP decomposition. Additionally, a few metal oxides are common semiconductor materials that have hole conductivity, which makes them efficient carriers of electrons [194]. These characteristics speed up the electron transfer involved in a few events that occur during the breakdown of AP. In conclusion, it is possible to alter the AP breakdown route when metal oxides are present.

Ammonium perchlorate is a flexible oxidising agent that interacts with a variety of materials to improve performance [195]. Several previous investigations have shown that AP efficiently work together with metals additive [196], [197], metal oxides [197], [198], [199], organic polymers [190], [200], mineral metals [45], [201], and a variety of other energetic materials [191], [202]. Moreover, it mixes well with other energetic substances such as CL-20 and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX), resulting to a higher burning rate than traditional AP solid propellants [203]. The role of metal oxide catalysts in enhancing the decomposition and combustion characteristics of AP-based propellants has been extensively studied. Padwal & Varma et al.[204] examined the thermal decomposition of HTPB-AP composite solid propellants catalysed with Ferric Oxide (Fe_2O_3). Their results indicated that nano-Fe_2O_3 (4 nm) had a stronger catalytic effect compared to micro-Fe_2O_3, shifting the exothermic peak to a lower temperature and increasing the burning rate. These findings suggest that nanoscale catalysts enhance energy release and combustion efficiency.

Ferric oxide is one of metal oxide shows an impressive catalytic proficiency in both AP/sorbitol and AP/sorbitol/Mg solid propellant. It lowers the kinetic parameters of solid propellant AP/Sorbitol/Magnesium and lowers the decomposition temperature of the solid propellant. Hence AP/Sorbitol/Mg can work synergistically with Ferric oxide to improve the solid propellant performance for better applications in rocket testing. It demonstrated strong catalysis in AP catalytic reduction. It was well accepted that it may interact with ammonia gas from AP decomposition in (2.9). The reaction was followed:

$$Fe^{3+} + NH_3 \rightarrow Fe^{2+} + NH_2 + H^+ \quad (2.9)$$

At high temperatures AP decomposes into gases. Thus, hypothetically Fe_2O_3 reacts with HCl gas, scavenging the HCl from the system hence reduce the HCl concentration in (2.10). Ferric oxide does not only function as a catalyst that lowers the decomposition temperature of ammonium perchlorate (AP) it also has an indirect but important influence on smoke output [205]. Secondary smoke reduction requires reactive metals such as Mg, Li, or Al–Li alloys that can chemically bind HCl to form metal chlorides. Therefore, Fe_2O_3 improves rate of combustion and may help lower particulate-based primary smoke, making it necessary to pair Fe_2O_3 with scavenging metals in minimum-signature formulations

$$Fe^{3+} + 3\,Cl^- \rightarrow FeCl_3 \quad (2.10)$$

Similarly, Almeida et al. [197] investigated the effect of iron oxide burning rate catalysts in nano and micro-scale on AP-HTPB propellants. The study applied the Flynn-Wall, Ozawa, Kissinger, and Starink methods to determine the kinetic parameters, revealing that nano-Fe_2O_3 significantly reduced the activation energy of AP decomposition, thereby improving the burning rate and reducing ignition delay. The incorporation of transition metal oxide influenced the combustion wave structure, demonstrating their role as burning rate enhancers. Additionally, they improve heat of combustion, activation energy, and mechanical qualities. Therefore, Fe_2O_3 improves combustion rate and may help lower particulate-based primary smoke, but it does not reduce HCl-based secondary smoke, making it necessary to pair Fe_2O_3 with scavenging metals in minimum-signature formulations. This provides as proof for researchers, suggesting that AP is an extraordinary material that is compatible with a wide range of compounds.

At higher temperatures decomposition, there is a reaction between AP and Magnesium [206][207] show in (2.11) and (2.12). Through adding magnesium as a metal additive, HCl neutralization improved by generating the reactive base magnesium hydroxide Mg $(OH)_2$ (2.13). After Mg particles are incorporated with AP solid propellants, magnesium chloride is produced as a product of combustion show in (2.14). The reactions between magnesium atoms and HCl are as follows:

$$Mg + NH_4ClO_4 \rightarrow MgO + MgCl_2 + HCl \quad (2.11)$$

$$2Cl_2 + 2H_2O \rightarrow 4HCl + O_2 \quad (2.12)$$

$$MgO + H_2O \rightarrow Mg(OH)_2 \quad (2.13)$$

$$Mg(OH)_2 + HCl \rightarrow MgCl_2 + H_2 \quad (2.14)$$

Magnesium influences both the thermal decomposition and exhaust chemistry of AP-based propellants. Its exothermic oxidation accelerates AP breakdown, while its strong affinity for chlorine enables the formation of magnesium chloride, effectively reducing HCl emissions. Through these combined effects, Mg enhances combustion energy and significantly decreases secondary smoke, supporting its use in minimum-signature propellant formulations. The metal hydroxide formed reacts with hydrogen chloride to give magnesium chloride and water it will neutralize and reduce the HCl concentration. Thus reduce the effect of HCl production in combustion of AP based propellant.

Overall, the literature shows that the thermal decomposition of AP and AP-based propellants is a multi-stage, complex process influenced by decomposition mechanisms, binder interactions, aging effects, and the presence of catalytic additives. DSC, TGA, and kinetic modelling consistently indicate that additives particularly metals and metal oxides such as Fe_2O_3 lower activation energy, shift decomposition to lower temperatures, and enhance burning efficiency. Studies on AP/sorbitol and AP/sorbitol/Mg formulations further demonstrate that metal additives modify thermal behaviour and improve combustion characteristics. These findings confirm that AP is a highly adaptable oxidizer whose decomposition behaviour can be effectively tuned through additive selection, supporting its suitability for developing improved minimum-signature propellants. Lastly, the metal additives help to improve the AP-based solid propellant, making it more appropriate for rocketry applications.

2.3.5 Physical Properties of Solid Propellant

The physical properties of solid propellants are crucial for ensuring structural stability, safety, and reliable performance in rocket propulsion systems. These mechanical characteristics are strongly influenced by the choice of oxidizer, binder, plasticizer, bonding agents, and metal additives. While metallic additives are

traditionally introduced to enhance combustion behaviour, they also play an equally important role in determining the mechanical response of the propellant under operational stressed.

The incorporation of metallic additives significantly impacts the mechanical behaviour of solid propellants. Zhang et al. [208] synthesized multi-functional bonding agents that, when integrated into HTPB/AP/RDX/Al propellant formulations, led to a 35.4% increase in tensile strength and a 62.0% enhancement in elongation at break. The bonding mechanism between amine-modified polybutadiene and the metal particles created stronger interfacial adhesion, which improved mechanical stability and prevented crack initiation. In the meantime, another study is conducted to compare the effects of metal additive 5% by weight in AP-based solid propellant using magnesium (Mg) and Aluminum (Al). The obtained results from the study revealed that propellants containing Mg exhibit a higher tensile strength and lower strains than those containing Al [209]. Further studies on effect of metal additive size on mechanical properties was done by Armstrong et al. [210] examined the mechanical integrity of metal-energized formulations, demonstrating that particle size and distribution affect stress localization and deformation resistance. A finer metal particle size resulted in enhanced fracture toughness, while larger particles contributed to stress concentrations and potential crack initiation. Similarly, Göçmez et al. [211] examined the effect of oxidizer content and fine-to-coarse ratio of AP particles on burning rate and mechanical properties. They found that higher solid loading (up to 82%) increased tensile strength and burning rate while reducing elongation at maximum stress. This highlights a trade-off: higher oxidizer content improves energy output but makes the propellant more brittle.

Further study by Xiao et al. [212] on CL-20/HMX cocrystals and composites demonstrated that metallic inclusions affect the elastic modulus, tensile strength, and failure strain. Molecular dynamics simulations revealed that cohesive energy density (CED) and binding energy (E-bind) between energetic molecules decrease with increasing temperature, indicating a trade-off between thermal stability and mechanical resilience. Additionally, a study on aluminized propellants by Glotov et al. [213] investigated the role of aluminium agglomeration in mechanical degradation. The researchers found that large agglomerates led to localized stress concentrations, causing premature cracking and failure under mechanical loading. However, when aluminium particles were properly coated or nano-structured, they exhibited improved dispersion within the binder matrix, leading to higher impact resistance and tensile stability.

Bonding agents are critical in enhancing adhesion between the oxidizer, binder, and metallic fuel**.** It plays crucial role in enhancing mechanical strength of solid propellant [214]. From the same research using epoxy as binder stating that the higher hydrogen-bond density correlated with increased Young's modulus and tensile strength. This study provides insights into how modifying binder chemistry can optimize mechanical behaviour without compromising energetic performance. Recent studies have explored the effect of sorbitol content on the tensile strength, modulus, and flexibility. Sorbitol is commonly used as a plasticizer to improve the flexibility and processability of starch-based materials. A study by Izamuddin et al. [215] investigated the effect of different sorbitol weight percentages (60 wt%, 65 wt%, 70 wt%, and 75 wt%) on the tensile properties. Their findings revealed a decreasing trend in tensile strength as sorbitol content increased. This indicates that higher sorbitol concentrations weaken intermolecular forces, reducing tensile strength. The plasticization effect of sorbitol disrupts the hydrogen bonding network between amylose and amylopectin molecules, leading to increased molecular mobility. Research by Abd elall et al.[216] demonstrated that methyl aziridinyl phosphine oxide (MAPO) improved Young's modulus and tensile strength. From same study highlighted that the choice of solvent in bonding agent production affects mechanical properties. The use of polar solvents improved strain values, while copper chloride ($CuCl_2$) at 4.5% yielded maximum strain. However, increasing $CuCl_2$ beyond this concentration led to reduced mechanical performance Also, Yadav et al. [217] reviewed various bonding agents, emphasizing that neutral polymeric bonding agents (NPBA) and hydantoin-based additives enhanced strain-stress characteristics by improving interfacial adhesion and reducing dewetting.

Overall, the literature demonstrates that the physical and mechanical properties of solid propellants are strongly governed by the interaction between oxidizers, binders, plasticizers, bonding agents, and metallic additives. Metal fuels such as Al, Mg, and B not only enhance combustion performance but also significantly influence tensile strength, elasticity, fracture behaviour, and failure modes. Studies consistently show that particle size, dispersion quality, and interfacial adhesion play critical roles in determining stress distribution and crack initiation within the propellant matrix. Higher solid loading and finer particle sizes tend to improve strength but often reduce elongation and increase brittleness, highlighting the inherent trade-off between mechanical robustness and energetic performance. Bonding agents and binder chemistry further modify these relationships by strengthening molecular interactions

and improving load transfer between components. Additionally, plasticizers like sorbitol can increase flexibility but may weaken tensile properties at higher concentrations. Together, these findings emphasize that achieving optimal mechanical stability in minimum-signature propellants requires careful balancing of formulation parameters to ensure structural integrity without compromising combustion efficiency.

2.4 Reduction and Quantification of the Primary and Secondary Smoke of Solid Propellant

The combustion of solid propellants produces both primary and secondary smoke, which consists of various gaseous and particulate emissions. These emissions are a major concern in military, aerospace, and space propulsion applications, where reducing the visibility and environmental impact of exhaust plumes is critical. Primary smoke is directly generated from the combustion of oxidizers, fuels, and binders within the propellant formulation, while secondary smoke results from condensation, chemical recombination, and atmospheric interactions after the exhaust gases leave the nozzle [218]. The reduction of these smoke emissions is essential for stealth applications, improved propulsion efficiency, and regulatory compliance in both defence and civilian aerospace sectors.

The primary contributors to smoke formation in composite propellants are metallic fuel additives [219], such as Al, oxidizers like AP, and polymeric binders such as HTPB. The incomplete combustion of metal fuels leads to the formation of aluminium oxide (Al_2O_3) agglomerates, which are responsible for the visible smoke in exhaust plumes. Additionally, secondary smoke is formed due to the reaction of combustion by-products with atmospheric moisture and oxygen, generating submicron-sized particulates and condensed-phase aerosols. To mitigate these effects, researchers have focused on alternative oxidizers, nano-structured metal additives, and advanced combustion stabilizers to optimize combustion energy and reduce smoke emissions.

Quantification of primary and secondary smoke involves spectroscopic analysis, laser-based diagnostics, and particulate sampling techniques to measure optical opacity, particle size distribution, and emission composition. Advanced experimental setups, including high-speed imaging and infrared (IR) absorption spectroscopy, allow for real-time tracking of smoke formation and dissipation [220]. The development of minimum-signature propellants requires an integrated approach, combining material formulation

strategies and exhaust gas characterization to minimize both thermal and optical signatures.

2.4.1 Reduction of Hydrogen Chloride Gas in Combustion Product of Solid Propellant

AP is an inorganic chemical with the formula NH_4ClO_4 that is utilized in solid propulsion systems as a strong oxidant. It is a white crystalline material that readily dissolves in water. The reaction of ammonia and perchloric acid produces ammonium perchlorate (AP). Because of its improved mechanical and ballistic qualities in comparison to other oxidizers, AP is a well-known oxidizer. It has been used in solid rocket composite propellants for many decades ago. Ammonium perchlorate acts as an oxidizer while Hydroxyl-terminated polybutadiene (HTPB) and aluminium acts as a metal fuel [221].

According to many researchers, [114]–[116] the development of AP kinds of propellant faces three major environmental challenges:

- Impacts on the ground, such as groundwater contamination or mishaps brought on by incorrect propellant processing and handling,
- Biological impacts, such as propellant toxicity and corrosiveness,
- Environmental effects, which are typically induced by the interaction of waste products from burning with the surrounding air, particularly the ozone layer.

According to Abd-Elghany et al. [82] the combustion of an AP-based solid propellant system emits about 100 T of HCl gas into the upper atmosphere. It is thought that AP may interfere with thyroid gland function in humans [38]. This is because the combustion of AP-containing propellants releases hydrochloric acid (HCl) as exhaust gas, which pollutes the surrounding air. The heated gases released during rocket launches emit dangerous pollutants. Furthermore, poisonous rocket emissions have a deleterious impact on the upper atmosphere as well as the flora and fauna near the rocket launch site [225], [226]. The build-up of these harmful substances in the atmosphere results in ozone depletion, acid rain, climate change, and adverse impacts on human health. In addition, the HCl gas released by rocket exhaust causes air pollution to rise by 1% with each launch. Dennis et al. [227] report that the exhaust from a rocket launch pad using composite propellant contains 60 T of HCl [227]. The peak HCl

concentrations in the exhaust plumes of eight Titan III rockets ranged from 25 to 0.5 mg/L or parts per million (ppm) for 3 to 300 minutes [227], [228]. Furthermore, the European Space Launcher Ariane-5, which includes 476 T of AP-based solid composite propellants and generates 270 T of concentrated HCl during burning, highlights the risk of HCl.

Toxic HCl emissions have prompted researchers to develop several solutions, one of which is the use of green propellants. Three methods exist for converting conventional AP-based propellants into green propellants: scavenging, neutralising, and non-chlorine propellants [83], [209].

2.4.1.1 Neutralization HCl Gas by Metal Additive

Metal is used to neutralize HCl gas to reduce the concentration of chloride gas. The oxide reacts with chloride to form metal chloride. According to the experimental results, the neutralized reaction could eliminate HCl from the product gases if the aerosol MgO/H2O/HCl is generated. Consequently, when magnesium particles are added to AP-based propellant, the amount of HCl molecules decreases. The phrase "neutralized propellants" refers to this type of propellant. Among the combustion products of the AP propellant is HCl gas, which is transformed into magnesium chloride, a stable and environmentally friendly substance [209]. Although it has been demonstrated that these formulations significantly reduces HCl emission during the combustion process, they are also distinguished by their low cost of production and fabrication. Furthermore, prior research by Terry et al. [23] and Rodic et al. [209] discovered that aluminium (Al), magnesium (Mg), and lithium (Li) metals are the most promising alkali metals for usage as scavenging agents in AP solid propellants.

Several studies have found that including metal additions in composite propellant reduces the percentage of HCl in the combustion output [229]. According to Terry et al. [23] metal alkali halophilic materials are chosen based on their capacity to efficiently remove chlorine ions and create alkali metal chlorides (i.e., salts) during the combustion process. Sodium nitrate (NaNO3) is partially substituted for ammonium perchlorate (NH4ClO4) in scavenging propellants. This approach is used in the combustion chamber to eliminate HCl by producing sodium chloride (NaCl). Sodium nitrate, chlorine-free oxidants, and metal additions can scavenge or neutralize 1-10% of the HCl. Because of its high density, wide availability, and convenience of use, using

NaNO3 as an HCl scavenger is favourable. Thus, alkali metal nitrates such as lithium nitrate (LiNO3) and sodium nitrate (NaNO3) have been used to replace a stoichiometric amount of AP in order to introduce extremely halophilic material in a stable manner [230]. Another study used lithium carbonate and sodium carbonate to eliminate HCl gas in solid heterogeneous rocket combustion. The results of this study conclude that 20% lithium carbonate by mass successfully eliminates HCl gas. while for sodium carbonate, it needs to be 25% to eliminate all HCl [206]. Beside that ferrous oxide is well known catalyst neutralize with chloride gas to form metal chloride [231]. Another research is using Zinc oxide and alumina as sorbent to remove HCl gas at high temperatures. The metal provide place for the highly reaction with acid HCl gas for various absorption applications[41]

Further research found that nanoscale alkali metal hydride mixed with aluminium can be used as a scavenger to remove HCl gas effectively. Only 8% % mass of alkali metal hydride can remove 28% of the HCl gas from the combustion product [232]. Chalghoum et al.[233] study on the effect of complex metal chloride elimination on HCl exhaust gas combustion product reveal that complex metal chloride such as Mg_2FeH_6, $LiMgAlH_4$, and $LiAlH_4$ show a synergetic effect with high performance and a good HCl scavenger effect [233]. Terry et al. [23] reduce HCl gas from combustion AP oxidising agent and **(**Hydroxyl-terminated polybutadiene) HTPB using binary metal Al/Li; 80/20% by mass manages to reduce HCl at 95%. From the same work, it was concluded that Li 15% by mass above will have a greater impact on removing HCl gas**.** In AP composite propellant formulations, the aluminium-lithium binary alloy was assessed both theoretically and empirically as a high-performance halide scavenger, which means it can significantly lower HCl production and raise the ideal specific impulse. Further research to reduce HCl gas using binary metal Al/Li; 80/20 % succeed to reduce 65-70 % HCl [234]. This work uses new method to measure HCl which is laser-absorption-spectroscopy to accurately measure HCl concentrations as illustrated in Table 2.5. In addition binary metal, Mg/Al is also used as HCl gas scavenger greatly reduce the amounts of gas [226].

Figure 2.6 summarises the type of chemical species that capable of scavenging the H Cl produced during the combustion of AP-based propellants. These scavenger function by neutralizing or capturing chlorine-containing species to reduce secondary smoke and corrosive exhaust. Metal scavengers such as Al, Mg, and Li react directly with HCl to form stable chlorides, while alkali nitrates and carbonates promote

oxidation or neutralization pathways. Complex hydrides provide highly reactive sites that bind chlorine at elevated temperatures. Sorbent materials, including ZnO, ZrO_2, and Al_2O_3, further reduce HCl by physically or chemically adsorbing acidic gases. Together, these categories represent the primary strategies employed to minimize chlorine emissions in AP-based propellant systems.

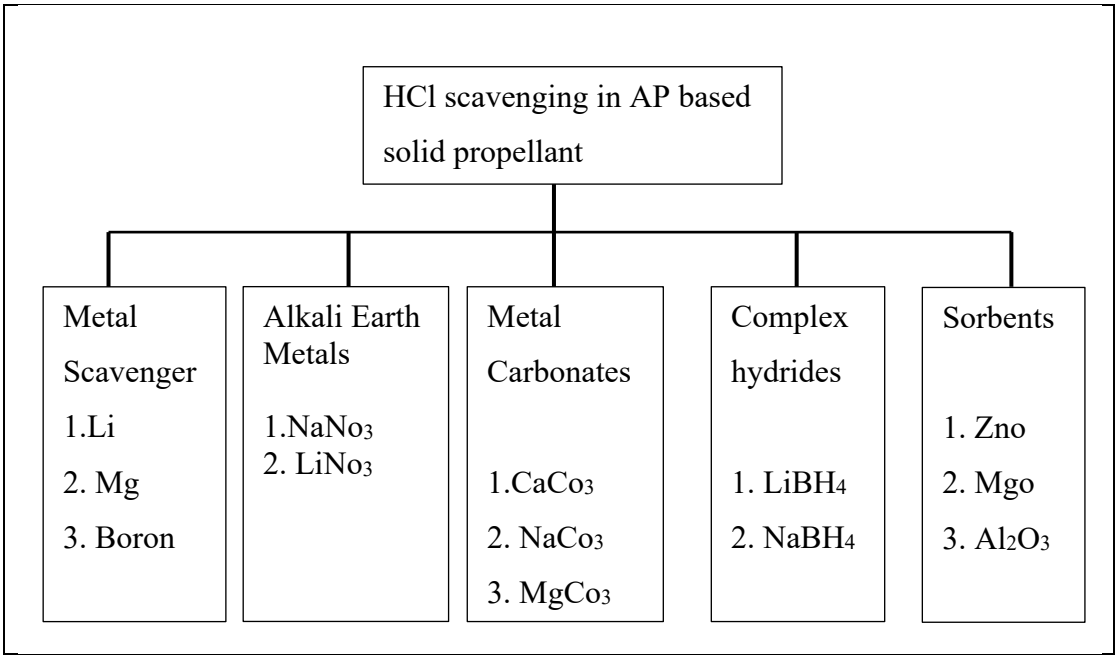


Figure 2.6 Classification Of Hcl Scavenging Species.

2.4.2 Method for Measuring Hydrogen Chloride Gas Concentration

From formulations show in Table 2.5 perfect combustion and complete HCl removal to those revealing interesting variations, this study investigates a range of possibilities. This compilation explains a spectrum of solid propellant formulations, distinguished by varying concentrations of ammonium perchlorate (AP), binders, and metallic additives. The focus is on the effect of these formulations on hydrogen chloride (HCl) concentrations and the methods employed for measurement. Notable formulations include one with AP, magnesium, and iron oxide, demonstrating complete combustion with 100% removal of HCl. The methods employed for HCl measurement, including combustion gas solubility, gas analysers, and theoretical calculations, add a layer of scientific depth to the exploration.

From Table 2.5 it shows magnesium metal (Mg) effectively removes HCl from the exhaust gas of solid propellant combustion. Hansen et al. [235] use magnesium and

ferrous oxide as catalysts to successfully remove 100% of HCl gas. From the research utilization of sodium nitrate within the examined solid propellant formulations is noticed to result in an important reduction of hydrogen chloride (HCl) gas removal effectiveness. This observation implies that the incorporation of sodium nitrate as an additive exerts a mitigating influence on the neutralization and elimination of HCl during the combustion process. Besides that, metallic compounds, particularly those of a complex or binary nature, exhibit a noteworthy effectiveness in the removal of hydrogen chloride (HCl) gas. The synergistic effect of combining two or three metals as compounds demonstrates a remarkable enhancement in the efficiency of HCl gas removal. This observation underscores the potential benefits of leveraging compound metal formulations to optimize the removal of HCl, showcasing a promising avenue for further exploration in the development of advanced solid propellant compositions [23], [233], [234].

To determine the efficiency of metal scavengers on hydrogen chloride (HCl) gas, a rigorous assessment necessitates the implementation of a precise measurement methodology to accurately determine the extent of HCl concentration removal. The selection of an effective method is paramount in achieving reliable results. Various approaches, including combustion gas solubility, gas analysers, and theoretical calculations, have been employed to estimate HCl concentrations in previous research. To address this, experimental validations are crucial to ensure the real-world applicability of theoretical models. By comparing theoretical predictions with empirical observations, researchers can enhance the reliability of their findings, uncover distinctions in the scavenging process, and identify potential limitations in theoretical calculations. This integrated approach, combining theory and experimentation, strengthens the overall understanding of HCl removal mechanisms and ensures more robust scientific conclusion. For example, Vellaisamy et al. [236] employed both the Mohr titration method and chemical equilibrium application to measure hydrogen chloride (HCl) concentrations. The results from their work revealed significant discrepancies between the two methods. This discrepancy underscores the importance of careful consideration when selecting measurement techniques, as different methods may yield varying outcomes. The choice of method can significantly influence the reported HCl concentrations, emphasizing the need for researchers to critically evaluate and validate.

Table 2.5
Effect Of Metals Additive And Method Of Measurement On Hcl Concentrations

Solid Propellant Formulation wt. %	Metal Additive wt.%	Method of measurement HCl concentration and research gap.	HCl mole Fraction% Removed	Ref
AP 38.6 %	Al 21.0% $NaNO_3$ 28.1% Fe_2O_3 0.25%	Combustion gas is soluble in water. Identified gas-phase/condensed-phase reactions. Chemical analysis of AP exhaust reactions.	69.3%	[235]
AP 65 %	Mg 25.25 % Fe_2O_3 0.5 %	Combustion gas soluble in water	100%	[235]
AP 62.5%	Al 15% Mg 11.54%	Combustion gas soluble in water	44.5%	[235]
AP 61.5 % HTPB 11.7%	AL/Li 26.8 % (20/80) %	Thermochemical Calculation (CHEETAH) *Dissolved HCl in distilled water (Validation)	95%	[23]
AP 69% Binder 26%	Mg 5%	Gas Analyzer. HTPB binder; no primary smoke work (Gas mestdx 4030)	45%	[237]
AP 67% RDX 7% Binder 26%	-	Gas Analyzer HTPB binder; no primary smoke work. (Gas mestdx 4030)	84%	[237]
AP 65 % HTPB 35 %	AL/Mg 15 % (5/10) %	Theoretical, NASA chemical equilibrium Application (CEA)	90 %	[226]
AP 65 % HTPB 35 %	AL/Mg 15 %	Gas Bubbling Mohr Titration Mg > Al in scavenging; Fe_2O_3 catalytic	51 %	[226]
AP 60% HTPB 10%	$LiAlH_4$ 30%	Theoretical calculation Chemical equilibrium Application (CEA) Mg, Fe_2O_3 reduce HCl	98 %	[233]
AP 68 % HTPB 14 %	AL/Li 18% (20/80) %	Laser absorption spectroscopy. Accurate HCl measurement	70 %	[234]

Based on the limitations identified in previous studies from Table 2.5, this research adopts a measurement approach that minimizes underestimation error and provides results that closely represent the actual gas-phase HCl produced during combustion. Methods such as Mohr titration and water-solubility techniques, although widely used, quantify only the dissolved fraction of HCl and therefore tend to

underestimate total emissions. Thermochemical calculations (CEA/CHEETAH) provide valuable theoretical predictions but cannot account for real combustion kinetics, heterogeneous reactions, or incomplete scavenging. Gas-phase spectroscopic techniques have consistently been shown to provide the most accurate measurements because they directly detect HCl in the exhaust plume. For these reasons, the method selected in this study prioritizes direct gas-phase analysis, ensuring higher accuracy, reduced dependency on absorption efficiency, and better comparability with modern diagnostic standards. This approach provides a more reliable representation of HCl formation and removal in AP/Sorbitol/Mg combustion, addressing the methodological inconsistencies highlighted in earlier literature.

Ruesh et al. [234] have introduced a novel approach for accurately measuring hydrogen chloride (HCl) concentration through the utilization of laser absorption spectroscopy. Their work demonstrates that, employing aluminium and lithium metal as HCl scavengers, the method effectively measures a substantial portion of HCl, specifically achieving a 70% removal. This innovative method, relying on laser absorption spectroscopy, not only represents a technological advancement in HCl concentration measurement but also showcases the effectiveness of specific metal combinations in scavenging HCl in the studied context. Such findings contribute to the scientific foundation for tailoring propellant formulations with enhanced environmental considerations and improved combustion performance. By employing a method that aligns with the specific characteristics of metal scavengers and their interactions with HCl, researchers can ensure a comprehensive and accurate evaluation of the efficacy of these scavengers in mitigating HCl gas concentrations.

The exploration of green oxidizers, and their role in clean combustion reactions has not only contributed to a deeper understanding but has also paved the way for future innovations in propellant formulations. By comparing the performance of new green oxidizers to conventional counterparts, the review highlights the ongoing pursuit of cleaner and more sustainable alternatives. The Advisory Group for Aerospace Research and Development (AGARD), an agency of NATO, plays a critical role in shaping the terminology for solid propellant combustion exhaust products. Their proposed method distinguishes between primary and secondary smoke, categorizing them into A, B, and C, with A being least smoky and C being most smoky. Primary smoke primarily results from condensed solid particle materials such as metals, carbon, and metal oxides. AGARD identifies catalysts, anti-instability additives, after-burning suppressants, and

metals as main contributors to primary smoke. Gaseous products in rocket exhaust, including copper hydroxide, potassium hydroxide, and aluminium oxide, are predominantly metal compounds.

The emphasis on achieving clean combustion not only aligns with environmental regulations but also addresses the priority of developing insensitive munitions. The dedication to balancing performance with environmental responsibility reflects a forward-looking approach, ensuring that the future of solid rocket propellants is not only technologically advanced but also environmentally conscious. Furthermore, this review lies in the focus on reducing hydrogen chloride (HCl) gas concentration in combustion products. Recognizing the environmental and health implications associated with HCl emissions, the integration of strategies for HCl reduction marks a significant step towards achieving an eco-friendlier propulsion technology. The exploration of alkali metals scavengers and neutralization processes by metal oxides plays a pivotal role in this reduction effort, demonstrating a holistic approach to minimizing the environmental impact of solid rocket propulsion.

In essence, the comprehensive review serves as a valuable resource for researchers, engineers, and policymakers involved in the propulsion field, providing a roadmap for further advancements in minimum signature solid rocket propellants and the realization of cleaner and more sustainable combustion technologies.

2.5 Conclusion Remark

In summary, ammonium perchlorate (AP) has demonstrated remarkable compatibility with a wide range of fuels, binders, and additives, a clear knowledge gap exists regarding its use in combination with sorbitol and magnesium. No published formulation to date has evaluated AP–sorbitol composite propellants incorporating Mg as a chlorine-scavenging metal, leaving the combustion behaviour, thermal stability, and smoke-reduction mechanisms of this system uncharacterized. The interaction between sorbitol's oxygen balance and Mg's combustion chemistry also remains unresolved, particularly because Mg reacts through pathways that differ significantly from hydrocarbon-based fuels. This raises a fundamental uncertainty about whether oxygen availability is sufficient to avoid incomplete combustion and residue formation in AP/Sorbitol/Mg systems.

Furthermore, although oxygen balance is recognized as a major determinant of propellant performance, its role in modifying burn rate in the presence of metallic additives has not been systematically studied, especially for systems combining positive-oxygen-balance oxidizers with carbohydrate-based fuels. The decomposition behaviour of sorbitol, which produces low-molecular-weight intermediates before full combustion, may further complicate reactions with AP and Mg. No existing studies have examined whether these intermediates influence ignition stability, burn-rate consistency, or aging behaviour in AP/Sorbitol/Mg formulations**.**

Another significant gap concerns the thermal degradation pathway. While Mg is known to act as a chlorine scavenger, its catalytic influence on the decomposition kinetics of AP/Sorbitol mixtures is unknown, and no kinetic modelling or DSC/TGA studies have been published to clarify whether Mg accelerates, delays, or destabilizes the decomposition process. Existing kinetic literature is overwhelmingly focused on AP/HTPB or nitrate-based propellants, leaving AP/Sorbitol/Mg combustion mechanisms, flame structure, and transient burning characteristics largely unexplored.

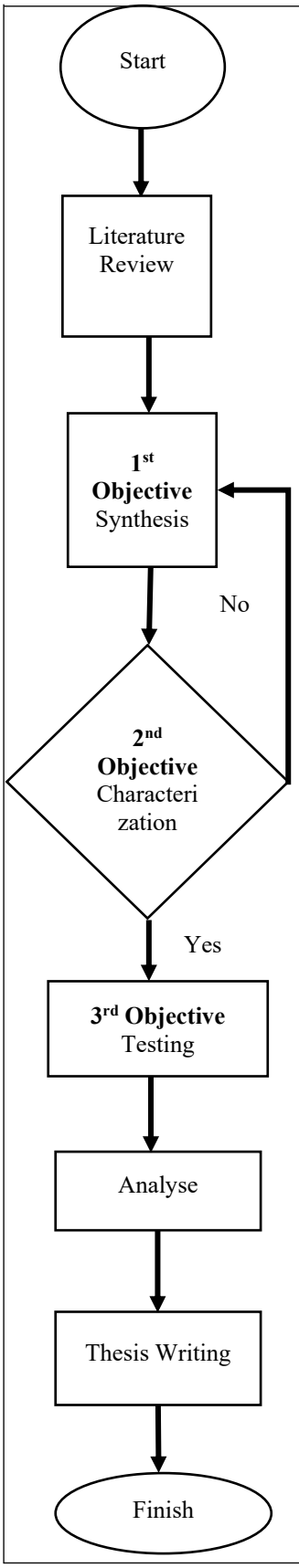
Finally, although smoke reduction is a recurring topic in minimum-signature propellant research, there is no standardized methodology for quantifying primary and secondary smoke, as studies rely on inconsistent techniques (opacity measurements, laser diagnostics, or particulate sampling). This lack of standardization prevents meaningful comparison across formulations and limits the ability to evaluate the environmental impact of new propellant systems.

Taken together, the key knowledge gap identified in the literature is the complete absence of scientific data on AP/Sorbitol/Mg propellants, specifically concerning their combustion behaviour, thermal decomposition mechanisms, oxygen-balance interactions, and smoke-generation characteristics. Addressing this gap forms the core motivation and novelty of the present research.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 Introduction

This chapter outlines the experimental methods used in the development of minimum signature solid propellant from AP-based mix with sorbitol and Mg metal. The method starts with the synthesis of AP/sorbitol/Mg solid propellant. The method described in Section 3.2 was implemented using three approaches to determine the optimal ratio of ammonium perchlorate (AP), sorbitol, and magnesium (Mg) metal. This optimization was conducted with respect to achieving the ideal oxygen balance, elemental stoichiometric coefficient greater than one (fuel lean) and maximizing combustion energy release. The first approach involved a parametric analysis to determine the optimal ratio with respect to energy, specific impulse and combustion product using the Propellant Evaluation Program (PROPEP) software. The second approach utilized thermodynamic mathematical calculations, while the third approach validated the heat of combustion through experimental measurements using a bomb calorimeter. Next, the method in section 3.3 proceed to the characterization of the sample. The characterization process was divided into two categories: chemical and physical characterization. The chemical characterization involved analysing the surface properties, elemental composition, and thermal decomposition behaviour of the propellant. Surface topography and morphology were examined to assess structural features, while elemental analysis was conducted to determine the composition of the solid propellant. Additionally, the physical characterization was performed using tensile testing and Shore hardness measurements to evaluate the mechanical properties of the propellant. Thermal stability analysis was conducted to provide insights into the properties of the solid propellant, offering crucial information for the subsequent stages of propellant testing. Additionally, kinetic analysis contributed valuable data to ensure the safe testing of the solid propellant in the next phase. The subsequent method in section 3.4 is divided into three subsections, focusing on evaluating the burn rate, assessing thrust performance, and quantitatively measuring smoke concentration. The methods utilized in this research are summarized in Figure 3.1.



Input 1st Objective	Method	Outcome
Parametric Analysis	Evaluation of theoretical performance and formulation ratio using PROPEP	Identified Optimum formulation ratio of solid propellants
Thermochemistry Calculation	Used of mathematical concept energy for solid propellant	Energy analysis for solid propellant
Sample solid propellant	Bomb Calorimeter for Heat Energy validation	Validate the ratio for solid propellants

Input 2nd Objective	Method	Outcome
Optimized sample	*FTIR *CHNO *TGA *XRD *DSC *SEM	*Morphology& Topology *Internal energy *Thermal Properties *Composition
Kinetic analysis	Kissinger and Ozawa method at different heating rates	*Activation energy *Equilibrium constants.

Input 3rd Objective	Method	Outcome
Static Test	*Measure the thrust produced from combustion Solid Propellant	* Thrust of each propellant with different formulations
Solid residue, smoke, and gas concentration	*Measure the light intensity pass through smoke. * Glass wool to trap solid particle. *Solid residue observes using SEM	* Smoke opacity and weight of trapped solid particle. *Elemental composition of solid residual

Figure 3.1 Research Frame work for Development of Solid Rocket Propellant

3.2 Synthesis of Minimum Signature Solid Propellant

Solid propellant was prepared in in the laboratory in weigh basis of 100 gram for the combustion to quantify the smoke concentration and thrust with burn rate performance. First, new formulation of minimum signature solid propellant is analysed using PROPEP to determine the performance parameter and burning product constituent at optimum ratio. In the next step, the optimum ratio which give the lowest amount combustion products and heat energy is evaluated by using thermochemical calculation and compared with the result from PROPEP. Bomb calorimeter was used to find the value of heat combustion for validation. The following sub-chapters present the heat energy calculation, chemical equation of the reaction and experiment to measure heat of combustion.

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3.2.1 Propellant Evaluation Program (PROPEP) Software

PROPEP is software to determine chemical equilibrium for the combustion of solid rocket propellant and most possible effective propellant formulation ratio [238].

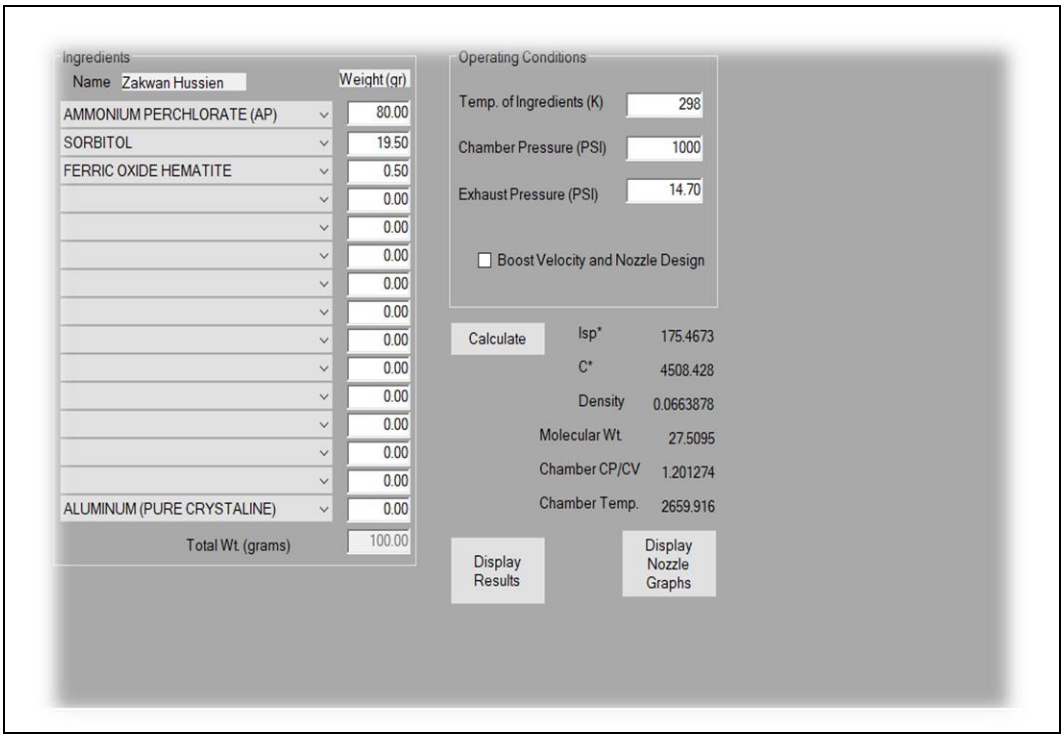


Figure 3.2 PROPEP Software Interface

In PROPEP, the total propellant mass is normalized to 100 units for calculation purposes. This value does not represent an actual physical mass but only expresses the relative mass fractions of each component in the formulation. By using this software rocket solid propellant performance can be predicted shown in Figure 3.2. Furthermore, it helps researcher to evaluate theoretical performance such as specific impulse and characteristic velocity. Default operating conditions values are set up as shown below. Total mass of propellant is 100 gram which is represent the mass percent of constituent.

- Temperature condition: 298K is room temperature.
- Chamber pressure: 1000 psi
- Exhaust pressure: 14 psi (1 atm ideal expansion at sea level)

From the Figure 3.2, shows the PROPEP interface used to evaluate the theoretical combustion performance of the solid propellant under the defined operating conditions of 298 K ingredient temperature, 1000 psi chamber pressure, and 14.7 psi exhaust pressure, PROPEP calculates the equilibrium properties of the propellant. The predicted gas density (0.066 lb/ft³) and molecular weight (27.51 g/mol) show that the exhaust is dominated by light species such as H_2O and CO_2, consistent with a minimum-signature formulation producing minimal particulate matter. The Cp/Cv ratio of 1.20 further confirms a gas composition typical of triatomic combustion products. Overall, Figure 3.2 demonstrates how PROPEP predicts the thermodynamic and performance behaviour of the AP/Sorbitol/Fe_2O_3 mixture, providing a theoretical foundation for understanding its combustion characteristics prior to experimental testing. The chemical ingredient for oxidizer, fuel and catalyst is filled in propellant formulation with total weight up to 100 grams. Result from this software tells the performance of new solid formulation. In addition, the most important is it's shown a chemical species with amount produce inside the product of the propellant combustion which is very important for this study.

3.2.2 Thermochemical Calculation for Solid Propellant Performance

Thermochemical calculation is a crucial tool in solid propellant research, providing valuable insights into combustion efficiency, burn rate behaviour, exhaust composition, and overall propulsion performance. By predicting key combustion parameters such as adiabatic flame temperature, heat release, and specific impulse,

facilitates optimization the oxidizer-to-fuel ratio for maximum efficiency. In AP/Sorbitol/Mg-based propellants, thermochemical calculations assist in evaluating the optimum ratio between AP, sorbitol, and Mg metal which provide the oxygen balance value, elemental stoichiometric coefficient (fuel-rich or fuel-lean), energy release, and amount of combustion product. The determination of oxygen balance (OB), equivalence ratio (ϕ_e), and related thermochemical parameters was performed using manual stoichiometric calculations, supported by Microsoft Excel for numerical processing and tabulation. No external thermochemistry software was employed for these computations. Instead, customised Excel worksheets were developed to systematise the calculations, verify mass balance, and ensure consistency across all formulations analysed

3.2.2.1 Oxygen Balance

The oxygen balance (Ω) provides information on the types of gases that are liberated**.** The set of guidelines was used to help understand the issue in the breakdown of $C_aH_bN_cO_dX_e$ combustion products with relation to the oxygen balance of AP/Sorbitol solid propellant [146], [182]. The oxygen balance indicates the types of gases that are liberated. To optimize the performance of propellant compositions, the atomic composition of the mixture must first be calculated. Thus, by controlling the oxidizing agent content in the propellant's mixture, it will change the value of the oxygen balance. The oxygen balance of the solid propellant of ammonium perchlorate/sorbitol formulation was calculated using (3.1), where **Ω** is oxygen balance is representing amount of oxygen required for complete oxidation, a is amount of carbon, b is amount hydrogen, d is amount of oxygen and e is amount of metal which represent any additive. M is relative atomic mass for mixture [12]. The propellant composition is optimized to achieve an oxygen balance as close to zero, as the heat of combustion reaches its maximum when the oxygen balance is approaching zero [11]. This is because it corresponds to the stoichiometric oxidation of carbon to carbon dioxide and hydrogen to water [13].

$$\Omega = \frac{[d - (2a) - \mathrm{e} - \left(\frac{b}{2}\right)] \times 1600}{\mathrm{M}} \tag{3.1}$$

3.2.2.2 Element Stoichiometric Coefficient

The energetically related properties of propellants, fuels, and explosives are evaluated using a straightforward method that considers the chemical valences of the fuel, and the oxidizer components present in a combustible mixture. It provides a quick way to get the elemental stoichiometric coefficient and makes it easier to balance complex combustion equations stoichiometrically. This method effectively predicts whether a mixture is fuel-lean, fuel-rich, or stoichiometrically balanced. It has been demonstrated that the total oxidising or reducing valences of different stoichiometrically balanced combustible systems has a linear relationship with their calorimetric values. This relationship has been effectively applied to determining the calorific value of fossil fuels. According to Bakhman et.al [239], elemental stoichiometric coefficients $\emptyset_e$ are parameters that indicate the intramolecular interaction between the oxidizer and fuel. For this work, this is a more user-friendly approach for multicomponent systems that can be used to determine the elemental stoichiometric coefficient. The elemental stoichiometric coefficient $\emptyset_e$ is defined in (3.2).

$$\emptyset_e = \frac{\sum n_{\circ} \upsilon_{\circ}}{(-1)\sum n_r \upsilon_r} \tag{3.2}$$

Where υ and n are the valence and number atoms of the element, whereas o and r refer to the oxidizing agent and reducing agent element, respectively. The denominator is multiplied by (-1) to convert the negative sign of the reducing element given by fuel. A mixture is fuel rich if $\emptyset_e < 1$, fuel lean if $\emptyset_e > 1$, and stoichiometrically balanced at $\emptyset_e = 1$, [239], [240], [241].

In order to clarify the decomposition product, a set of rules was used. According to the Springall Roberts rule of decomposition a set of chemical equation was developed

for this solid propellant reaction. The Springall–Robert's rule is an empirical method used to estimate the amount of oxidizer required for complete combustion in solid propellant formulations. It assumes that all carbon must oxidize to CO_2 and all hydrogen to H_2O, allowing a quick determination of whether a mixture is oxygen-rich, oxygen-balanced, or oxygen-deficient. This rule provides a simple preliminary check on oxidizer–fuel ratios before applying more detailed thermochemical calculations First step chemical equation in (3.3) was developed in this study for basic AP-based sorbitol solid propellant without metal additive. This chemical equation is crucial in early stage of this study as such it provide useful information of reaction product and used to calculate many important thermodynamic parameters for this work. It became blueprint for the next method to study the effect of metal additive. By using the (3.3), the calculations in (3.2) can be done. Hence the value of elemental stoichiometric coefficient $\emptyset_e$ can be computed. The detail calculation of stoichiometric formulation shown in appendix 1.

$$26NH_4ClO_4 + 5C_6H_{14}O_6 \rightarrow 30CO_2 + 26HCl + 13N_2 + 74H_2O \quad (3.3)$$

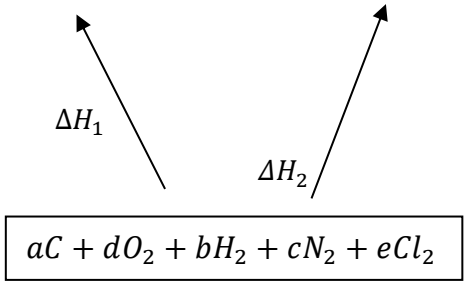
3.2.2.3 Thermochemistry

Thermochemistry is the branch of science that studies heat changes occurring during chemical reactions. The concept revolves around three key parameters: internal energy (E), heat content or enthalpy (H) and work done (W). Nevertheless, the oxygen balance does not provide information about the amount of heat actually released and composition gas product during a reaction [242], [243]. The number of moles of gaseous products produced during combustion, the average molar mass of gas products, and the explosion heat all have a significant impact on explosion performance [177]. In a constant-pressure environment and in accordance with Spring Robert's Rule, the following equation (3.4) expresses the enthalpy of formation for each constituent. This method aligns with the approach used by Ying Li et al. [244] and Dany Frem et al. [245]. Therefore, Heat of explosion can be determined by the difference between the total of energies for the formation of explosive components and the sum of energies for the formation of the explosion product.

The heat of reaction is the net heat absorbed or evolved during a chemical reaction. It can be divided into two types of reactions: endothermic and exothermic

reaction. According to Hess's law in (3.5) is used to calculate the heat of reaction for solid propellant mixture.

$$C_aH_bN_cO_dCl_e - -\Delta H_{Rxn} \rightarrow aCO_2 + \left(\frac{b}{2}\right)H_2O + \frac{e}{2}Cl_2 + \frac{C}{2}N_2 \quad (3.4)$$



$$\Delta H = H_{Products} - H_{reactants} \quad (3.5)$$

ΔH represents a negative for exothermic reactions and a positive for endothermic reactions. Whereas $\Delta H_{Product}$ and $\Delta H_{Reactant}$ represent heat of formation for each product and reactant. All heat of reactions is compared at standard state, which is typically defined at 298K and 1atm pressure. All explosive chemical reactions are exothermic. The heat evolved or absorbed when 1 mole of a compound is created from its element in the standard state is known as the heat of formation of a compound. Heat of formation (ΔH_f) is an important thermochemical parameter for an explosive because it affects the heat of explosion and heat of detonation.

Hess's law is used to calculate enthalpies of product or reactant. It states that "if a chemical reaction is carried out in stages, the algebraic sum of the amounts heat evolved in separate stages is equal to the total amount of heat evolved when the reaction occurs directly". Hess's law states that the heat of a chemical reaction can be obtained by the sum of the heats of reaction of several subsequent partial reactions starting with the same reactants and leading with to same product as depicted in Figure 3.3.

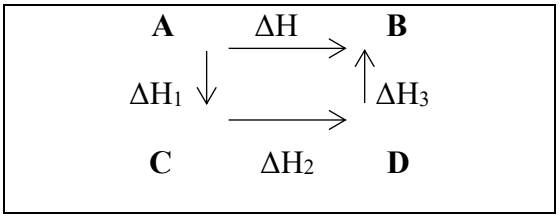


Figure 3.3 Hess's Law Cycle

It is common practice to use constant pressure conditions for rocket propellant burning in the combustion chamber of a rocket motor under conditions of free expansion to the atmosphere [11]. For this reason, the equation (3.6) show the $\Delta H_{reaction}$ is Heat of combustion, $\Delta H_{fproducts}$ is Heat formation of product and $\Delta H_{reactant}$ is Heat of formation component of solid Propellant.

$$\Delta H_{Reaction} = \sum \Delta H^{\theta}_{f(Product)} - \sum \Delta H^{\theta}_{f(Solid\ Propellant)} \tag{3.6}$$

$$\mathrm{Q} = \frac{\Delta \mathrm{H_d} \times 1000}{\mathrm{MW}} \tag{3.7}$$

Eventually, (3.7) is used to calculate final Heat release from the combustion of solid propellant where ΔH_d is Heat of reaction according to Hess's law, MW is molecular weight of solid propellant mixture and Q is Heat of explosion. The calculated Heat of explosion is compared with Heat of explosion from bomb calorimeter in section 3.2.4.

3.2.3 Solid Propellant Production

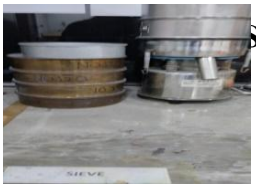
Granule Sorbitol and ammonium perchlorate powders with an average particle size of 90 μm were used as feedstock materials in this work. Sorbitol and ammonium perchlorate were weighed according to the ratio calculated with optimum oxygen balance.

A sample composed of AP/Sorbitol/Mg serves as the primary material in this study, while the remaining four samples incorporate with ferric as metal oxide catalyst and varying AP concentrations. These variations are introduced to facilitate comparative analysis, enabling the evaluation of the interaction effects between the metal additives and different AP content on the overall combustion and mechanical properties of the propellant. A total of five samples were prepared with different weight percentages following the calculation of the oxygen balance content of the mixtures. The sample with optimized oxygen balance was mixed by electric mixer with magnesium and ferric oxide as a power enhancing material. Figure 3.4 show that Sorbitol was first heated at a temperature of 120 °C until it melted. Immediately

ammonium perchlorate was added slowly to avoid accidents because ammonium perchlorate auto ignition temperature is 240°C and it was stirred evenly until it formed slurry-liked paste followed by additive metals. After melting the sorbitol and incorporating the oxidizer and additives, the mixture was allowed to cool slightly until it reached a slurry-like consistency suitable for shaping. The propellant was cast into cylindrical Mold and formed using a cold-press technique. This method involved applying uniform mechanical pressure at room temperature to compact the formulation and remove trapped air. The grains were then left to cool and solidify completely inside the Molds. This approach ensured consistent grain geometry, improved mechanical integrity, and eliminated defects that could arise from uneven cooling or incomplete compaction.

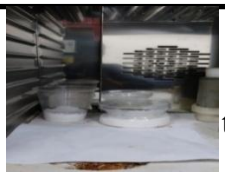
The temperature of the mixture must be carefully monitored at all times to ensure it remains well below the 240 °C auto-ignition temperature of AP. The melted solid propellant was first recrystallized for few minute. The recrystallization step was essential because sorbitol is hygroscopic, and residual moisture can alter propellant viscosity during mixing, promote premature decomposition reactions, and negatively impact ignition and burning rate consistency. For safety consideration all experimental work was conducted using appropriate PPE, including heat-resistant gloves, chemical-resistant nitrile gloves, safety goggles, a full-face shield, and an antistatic laboratory coat. To minimize ignition risk, antistatic grounding straps and electrostatic discharge-safe footwear were used during handling of ammonium perchlorate and magnesium powders. A particulate respirator was worn throughout powder preparation to prevent inhalation of fine oxidizer and metal particle.

In this solid propellant preparation, AP was set as excess reactant while sorbitol as limiting reactant. AP was set slightly in excess to create a fuel-lean mixture, ensuring cleaner combustion and reducing soot formation. The additional oxygen also supports the full oxidation of sorbitol to reduce formation of carbon soot. Thus, the choice of excess AP was intentional to enhance combustion cleanliness and minimise exhaust signature. Selecting sorbitol as the limiting reactant in AP/Sorbitol-based solid propellants ensures complete fuel combustion, optimized oxygen balance, and enhanced thermal efficiency. By maintaining an oxidizer-rich environment, this approach minimizes unburned residues, soot, and secondary smoke formation, leading to cleaner combustion and improved energy output.



Seiving Ammonium Perchlorate

>AP powder is seived, using
2 mm seive to break the chunks

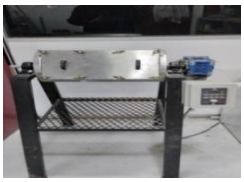


Preparation of Sorbitol

> sorbitol is placed in oven at temperature 50˚C for 1 day to remove moisture. due to hygroscopic

Weighing the Ingredients

>Weighing each of materials according to weight
Percent wt% as calculated based on thermochemical parameters



Mixing the Ingredient

> Run for 1 hour at 30 rpm
> mixing with electric mixer

Propellant Production

>Mixture is melting at temperature 120 ˚C.
> Compulsory to wear PPE glove, mask, google .

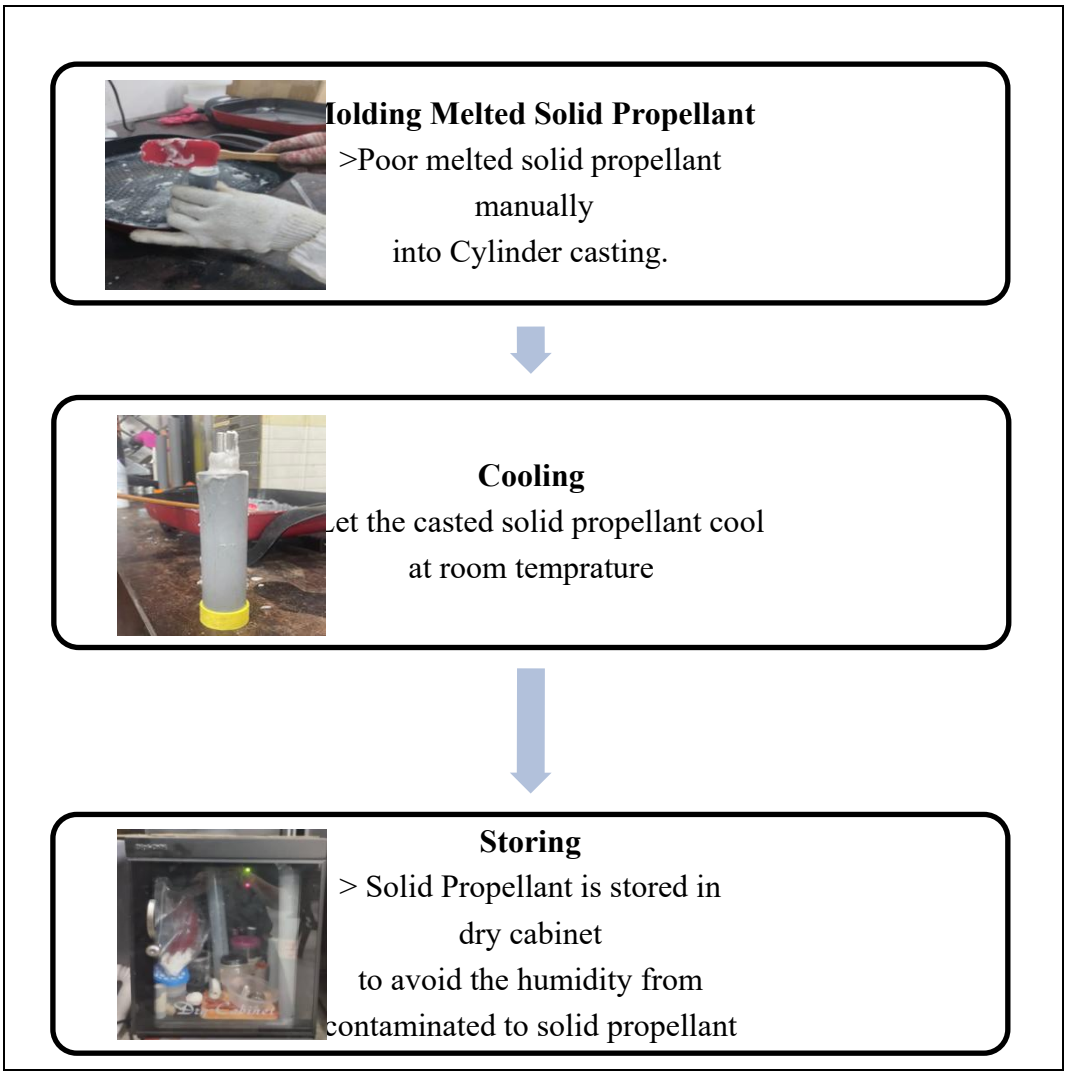


Figure 3.4 Process Flow For Propellant Production

3.2.4 Measuring Heat of Combustion

Energy performance is assessed through the Heat of Explosion (Qv), determined using an adiabatic calorimetric oxygen bomb under pressurized oxygen. Each sample's Qv was measured three times, and the average results were reported. The IKA C 2000 oxygen bomb calorimeter was employed to measure the combustion heat value of the 0.5g sample using the resistance nichrome wire ignition method, and subsequently, the combustion heat ratio was calculated [244]. The experiment took place within an ambient temperature range of 20 to 30°C, and an oxygen pressure of 3.0 MPa was applied under oxygen gas atmosphere [245] [246]. The result then compared with section 3.2.2.3.

Plate 3.1 Oxygen Bomb Calorimeter

3.2.4.1 Open Burning for Burn Rate Determination of Solid Propellant

The burn rate investigation was initially conducted at ambient pressure and room temperature as a preliminary assessment to characterize the combustion behaviours of the solid propellant. This initial evaluation serves as a foundational step before performing burn rate measurements under high pressure and temperature conditions in next section. By establishing baseline data in controlled conditions, this approach helps ensure experimental safety and prevents potential hazards during high-pressure testing.

The cured samples were cut into smaller pieces with diameters of 1.0 × 1.0 × 4.0 cm, and the burning rate was measured at ambient pressure, as depicted in plate 3.2. This method was adopted by [247].

Plate 3.2 Measurement of Burn of Solid Propellant at Ambient Temperature and Pressure

3.3 Characterization of Solid Propellant

The characterization of solid propellants is essential for evaluating their chemical, physical, thermal, and kinetic properties, as these factors directly influence combustion performance, stability, and efficiency. A characterization approach provides critical insights into the morphology, Thermal decomposition behaviour, reaction kinetics, and mechanical strength of the propellant formulation. All experimental analyses in this study were performed in duplicate to ensure repeatability and reliability of the results. For DSC and TGA, each formulation was tested twice, and the thermal curves were compared to confirm consistency. FTIR spectra were obtained from two independently prepared pellets, while SEM observations were carried out on two separate sample surfaces to verify morphological uniformity. Mechanical testing was also conducted on two specimens per formulation. These replicated measurements ensured that the reported findings are representative and not influenced by single-sample variation

The chemical characterization of the propellant was performed using SEM and EDX to examine surface morphology and elemental distribution. These techniques allowed the evaluation of particle dispersion, formulation homogeneity, and the influence of metal additives on the material structure**.** Additionally, thermal analysis is performed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) to study the thermal decomposition profiles of the propellant components. These techniques help identify onset decomposition temperatures, reaction pathways, and the impact of additives on thermal stability.

To further understand the reaction kinetics of the propellant, kinetic analysis is conducted to determine activation energy (Ea), reaction order, and decomposition rate constants. This is achieved through model-based and model-free approaches using Kissinger and Flynn-Wall-Ozawa (FWO). By analysing the kinetic parameters, this study provides a deeper understanding of the propellant's combustion efficiency, thermal degradation mechanisms, and safety margins under various operating conditions.

For physical characterization, mechanical properties such as tensile strength and Shore hardness are evaluated to assess the mechanical properties of the propellant. These properties are critical in ensuring that the propellant can withstand storage, handling, and operational stresses.

By integrating chemical, thermal, kinetic, and mechanical characterization, this methodology ensures a comprehensive evaluation of the propellant's properties. The findings from these analyses will support the optimization of solid propellant formulations for enhanced combustion performance, safety, and structural durability, ultimately contributing to the development of high-performance and minimum-signature propellants.

3.3.1 Mechanical Testing

The mechanical properties of AP/Sorbitol solid propellant, as well as the effect of metal additions on solid propellant strength, are critical in the solid rocket propellant area for better quality and safety feature. Tensile test and shore durometer hardness test were applied in this section. All samples were conditioned prior to analysis to eliminate moisture effects, as sorbitol is highly hygroscopic. After casting, the propellant specimens were placed in a drying cabinet and held under controlled temperature and low-humidity conditions until a constant mass was achieved. This conditioning step ensured that absorbed moisture did not influence thermal behaviour, mechanical properties, or combustion characteristics during testing.

3.3.1.1 Tensile Test

For testing brittle materials, including certain types of solid propellant, test speed must be carefully chosen to ensure the accurate measurement of their tensile

properties without causing any premature failure or unrealistic results. The ASTM D638 standard specifies different test speeds for various materials. For brittle materials, the slower test speed is generally recommended to avoid any rapid stress application, which can possibly lead to inaccurate results. ASTM D638 Type IV was selected because the use of sorbitol as a sugar-based fuel causes the propellant to behave similarly to a candy-like material, where a relatively soft outer layer forms despite a brittle interior. In this study, Type IV geometry provided the most practical and reproducible sample shape for brittle propellant specimens, ensuring uniform stress distribution and reducing the likelihood of grip-induced failure The recommended test speed for brittle propellants is often in the range of 1 to 5 mm/min [248].

Additionally, for the Ammonium Perchlorate/Sorbitol-based solid propellants, relevant ASTM standard for testing mechanical properties, including tensile testing, is ASTM-D638 Type IV and the required standard test specimen is as illustrated in Figure 3.5 [215], The tensile and hardness testing procedures followed a defined specimen geometry consistent with the requirements of ASTM D638 for polymer-based materials. As illustrated in Figure 3.5, the tensile specimen was prepared in the shape of a Type IV dog bone, which is appropriate for small such as AP/sorbitol propellant grains. The specimen dimensions overall length 115 mm, gauge length 33 mm, gauge width 6 mm, and tab width 19 mm match the standard Type IV configuration. It should be noted that such a test specimen is ideal for testing very soft polymers (e.g. rubber) and it is used when comparing soft and more stiff polymers. To evaluate the mechanical properties of five different samples of solid propellant, a tensile test is performed under the atmospheric pressure and speed of 2 mm/min. The test is carried out using the tensile test machine from Shimadzu with capacity of 50 kN as depicted in Plate 3.3.

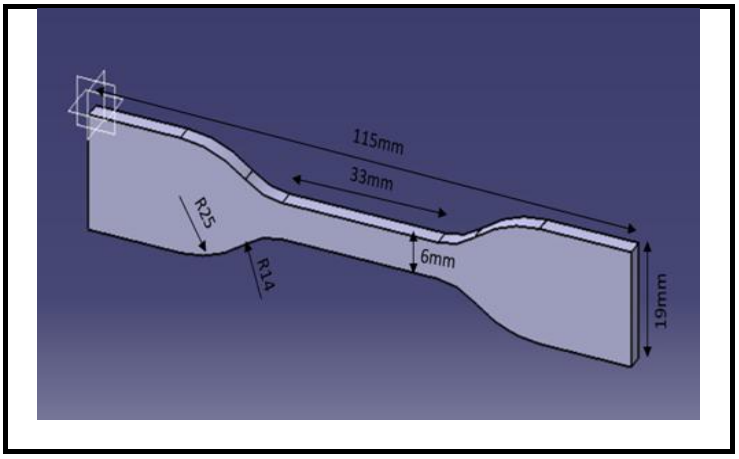


Figure 3.5 Dimension Of The Test Specimen Used For AP/Sorbitol Solid Propellant

Plate 3.3 Example Depiction Of The Solid Propellant Sample Under Uniaxial Tensile Test

3.3.1.2 Hardness Test

Shore A durometers can measure hardness on a scale from 0 to 100, with higher values indicating harder materials [249] . This scale has been frequently employed in the polymer industry for selecting materials, maintaining consistent product quality, and facilitating comparisons of material hardness. In this study, the durometer is used for the hardness test. Shore D hardness testing was performed on the fractured halves of the tensile specimens immediately after tensile failure. This approach ensured that the hardness measurements were taken from the same batch, curing history, and material conditions as the tensile samples, while also fulfilling the minimum flat-surface and thickness requirements specified by ASTM D2240.

3.3.2 Thermal Gravimetric Analysis (TGA)

TGA technique is one of the thermal analysis methods to characterize sample. It measured mass change of substance as function of temperature. This technique used to characterize thermal stability of material, melting point, and decomposition temperatures. TGA can be used using two experimental approaches [250], which are isothermal and non-isothermal (dynamic). In this study, a non-isothermal analysis method was used involving the rapid heating of material sample to a specific temperature and monitoring the mass change as a function of time while maintaining

the temperature. TGA measurements were performed at multiple heating rates of 5, 10, 15, and 20 °C/min to obtain kinetic parameters using standard iso-conversional approaches.

Thermogravimetric (TG) analysis was conducted using a Thermogravimetric Analyzer model Q500 plate 3.4. The DSC and TGA instrument were calibrated one month before the experiment following the procedure specified in the manufacturer's technical guide Sample is extracted from the minimum signature solid propellant with a mass 10 mg. The tests are run in nitrogen atmosphere from ambient temperature to 500˚C with a heating rate of 5,10,15 and 20˚C min. The result from different heating rate is used as variable to calculate kinetic parameter at high temperature decomposition.

Plate 3.4 Thermal Gravimetric Analayzer

3.3.3 Differential Scanning Calorimeter (DSC)

Differential Scanning Calorimetry (DSC) was employed to characterise the thermal behaviour of the propellant samples by recording heat-flow changes during controlled heating. The method provided quantitative values for melting, phase transitions, and decomposition onset, which are essential indicators of thermal stability. In this study, DSC was used strictly as an analytical tool to identify the temperatures at

which structural or chemical changes occur within the AP/sorbitol/metal formulations, thereby supporting the evaluation of propellant safety and performance.

Approximately 10 mg of each propellant sample was weighed and placed in an aluminium crucible. The crucible was sealed and positioned inside the DSC sample holder, which was then tightly closed to ensure consistent thermal contact and prevent atmospheric interference. The DSC analysis was conducted using a constant heating rate of 5 °C/min, under a steady flow of high-purity nitrogen gas at 20 mL/min, which served as an inert purge to prevent unwanted oxidation during the test. The choice of 5 °C/min heating rate was based on recommendations from prior research [219][251], which suggests that slower heating rates offer better resolution of thermal events, reduce thermal lag, and provide more accurate onset temperatures for energetic materials. For energetic systems, including AP-based composite propellants, such controlled rates are necessary to distinguish overlapping exothermic events and minimize thermal runaway risks. Nitrogen was selected as the purge gas to create an inert environment, thereby ensuring that the observed thermal responses were intrinsic to the material rather than influenced by external oxygen.

In this research, DSC was conducted at a single heating rate of 5 °C/min, while TGA was performed using multiple heating rates (5, 10, 15, and 20 °C/min). This difference reflects the distinct analytical purposes of the two techniques. DSC was used mainly to identify thermal transitions such as melting and the onset of AP and sorbitol decomposition. A single low heating rate is standard for energetic materials because it provides well-resolved peaks, minimises thermal lag, and ensures safe operation, as higher heating rates can shift or distort exothermic transitions in AP-based propellants. In contrast, TGA was used to obtain kinetic parameters, which require multiple heating rates for iso-conversional models (e.g., Ozawa–Flynn–Wall). The varying rates allow activation energy to be calculated reliably across different degrees of conversion. Therefore, the use of one rate for DSC and multiple rates for TGA is intentional and methodologically consistent with the objectives of each thermal analysis technique. This setup allowed for precise determination of ignition temperature, LTD, and HTD points, which were later correlated with mass loss profiles from TGA and used in kinetic modelling.

3.3.4 Kinetic Analysis

The kinetic studies of AP/sorbitol solid propellant have been examined by two different methods via Kissinger and Flyn-Wall-Ozawa with different heating rates based on (3.8) and (3.9). Under isothermal conditions, kinetic data are normally determined by measuring the rate of propellant breakdown [252]. In thermo-kinetics, activation energy and pre-exponential factors values of Kissinger methods are lower than Ozawa method. The Ozawa method states that both the activation energy and the pre-exponential factor are functions of the degree of conversion, indicating that they remain constant throughout the reaction. The underlying assumption of iso-conversional techniques is that the reaction rate at a fixed conversion level is only a function of temperature. This approach requires no knowledge of the reaction rate equation. These parameters could be achieved from the dependence of exothermic peak temperature as a function of heating rate. The Kissinger correlation can be utilized to define the relationship among decomposition temperature and heating rate [253].

The kinetics of heterogeneous decomposition of a solid-state substance can be investigated through the TGA [254], [255] at high temperature decomposition (HTD). HTD was chosen as the reference temperature because it represents the final and most energetic stage of decomposition, where complete breakdown and maximum heat release occur. This provides a reliable point for comparing combustion energy and thermal performance across formulations, as it reflects the true energetic potential of the propellant. HTD is also less influenced by early-stage fluctuations, making it suitable for consistent kinetic evaluation. Four different heating rates were used for the 5, 10, 15 and 20°C/min [256]. The activation energy was calculated using two different methods, including Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS) [256], [257].

The iso-conversional approach, originally proposed by Flynn, Wall, and Ozawa using the Doyle approximation for the temperature integral, provides a practical alternative to the Kissinger method for kinetic analysis. One of the main advantages of this technique is that it does not require prior knowledge of the reaction rate equation. In solid composite propellants, thermal decomposition kinetics are typically described using a single-step kinetic model. To accurately analyse activation energy, specific decomposition temperatures must be identified across different heating rates, as these temperatures correspond to consistent extents of reaction. This requirement forms the

basis for the so-called "iso-conversional methods" [258]. The fundamental assumption of iso-conversional analysis is that the reaction rate, at a fixed degree of conversion, is solely temperature-dependent. In the Ozawa method, it is further assumed that both the activation energy and the pre-exponential factor remain constant throughout the reaction. Experimentally determined activation energy values are then used to select the most suitable kinetic model. Moreover, the iso-conversional method enables the evaluation of how activation energy varies with the degree of conversion under different heating rates at the respective peak temperatures [259].

The activation energy was determined by analyzing the slope of a graph of ln=β for Flynn-Wall-Ozawa (FWO) and ln=(β/T^2) for Kissinger-Akahira-Sunose (KAS) against different heating rates β, with T_m representing the temperature in kelvin at maximum degradation rate of sample [260] as shown in equation (3.8) and (3.9) The activation energy (Ea) is obtained from the slope value of the plot, while the exponential factor (A) can be approximated from the intercept of the corresponding plots.

$$ln(\beta) = ln\left(\frac{AE_a}{R}\right) - 5.331 - 1.0516\frac{E_a}{RT_m} \tag{3.8}$$

$$ln\left(\frac{\beta}{T_m^2}\right) = ln\left(\frac{AR}{E_a}\right) - \frac{E_a}{RT_m} \tag{3.9}$$

In this study, the Kissinger and Flynn–Wall–Ozawa (FWO) methods will be compared by evaluating the activation energy values obtained from each technique and examining their consistency across the decomposition process. Kissinger provides a activation energy based on peak temperature, while FWO provides conversion-dependent activation energy across the reaction range. If differences arise, FWO results will be prioritised because they capture the multi-step and non-linear decomposition behaviour typical of AP-based propellants, while Kissinger serves as a verification benchmark for overall thermal stability.

3.3.5 Scanning Electron Microscope (SEM) & Energy Dispersive X-Ray (EDX) Analysis

Morphology, composition, microstructure, and dispersion between AP, Sorbitol, and additive metals were characterized using a scanning electron microscope

(HITACHI SU3500) instrument with 200-500 times of magnification. Double sided conductive adhesive tape was attached on the SEM specimen stub, 12.5 mm diameter. Solid powder sprinkled on the double conductive adhesive tape. The purpose of analysing the powder was strictly to verify elemental composition, confirm the presence of AP, sorbitol, Mg, and Fe_2O_3, and identify their characteristic spectra before processing. This procedure was performed solely to confirm the elemental composition of the raw constituents and to obtain clear spectra without interference from the binder matrix before solid propellant was Mold. Sputter coating with platinum thin layer at 10 nm is necessary for the samples to obtain high SEM image quality [38] and Energy dispersive X-ray (EDX) is used to identify the chemical elements, estimation purity and the position of each material content in solid propellant [40].

To accurately characterize the internal structure of the final solid propellant, a fractured segment from the tensile dog-bone specimen was used after tensile testing as mentioned in section 3.3.1. This cured cross-section allowed proper assessment of particle dispersion, oxidizer–fuel interaction, void formation, and microstructural uniformity within the binder matrix.

Calibration of SEM equipment was done 3 months before experimentation show in plate 3.5.

Plate 3.5 Scanning Electron Microscpoe And EDX Equipments

3.3.6 X-Ray Diffraction

X-ray diffraction (XRD) analysis in this study was performed exclusively on the raw, unburned propellant powders to identify the crystalline phases present in the initial formulations. Combustion residues were not examined, as the primary objective was to characterise the solid-state structure before ignition and to evaluate how the incorporation of metal additives influences phase composition. SP5 was excluded from XRD analysis because its formulation is nearly identical to SP4, differing only by a small increase in AP content and introducing no new additives or catalytic species. Since XRD results for SP4 already captured the crystalline phases of the AP/sorbitol system, SP5 was expected to produce an essentially identical diffraction pattern, offering no additional structural information. To avoid redundant data and focus on samples where compositional changes could influence crystal structure or residue formation, XRD analysis was limited to formulations containing metal additives (SP6–SP8).

The Rigaku X-ray Diffraction (XRD) machine, equipped with a Cu Kα radiation source (λ = 1.5406 Å), was utilized to collect Powder X-ray Diffraction (XRD) patterns of the solid propellant samples. This instrument operates by directing monochromatic X-rays onto the powdered sample, allowing for the identification of crystalline phases based on their unique diffraction patterns. The measurements were conducted within a scanning angle range of 5° to 90° (2θ), ensuring comprehensive phase detection and structural analysis. A scan rate of 8° per minute was applied to optimize data resolution while maintaining efficient data acquisition. This setup provides critical insights into the crystallographic properties of the propellant components, enabling the evaluation of phase composition, crystallinity, and possible structural transformations resulting from formulation modifications or thermal exposure [261].

3.3.7 CHNO Elemental Analysis.

The Carbon-Hydrogen-Nitrogen-Sulfur-Oxygen (CHNS-O) analyzer, specifically the Flash EA-1112 model, was employed to determine the elemental composition of the AP/Sorbitol/Mg propellant. This technique provides quantitative data on the percentage content of carbon (C), hydrogen (H), nitrogen (N), and oxygen (O), which are essential for evaluating the purity and stoichiometric balance of the

synthesized compounds. CHNS-O analysis is particularly useful in assessing the consistency of the formulation and detecting any unintended variations in composition that may affect combustion performance. Reproducibility was verified by ensuring that the replicate measurements for each element varied by less than 2% relative deviation, confirming the reliability of the analytical procedure For this study, 10 mg of each of the five propellant samples was accurately weighed and prepared for analysis, ensuring reliable and reproducible results [262]. To minimise sorbitol's hygroscopic effects, samples were pre-dried and weighed quickly in a low-humidity environment to prevent moisture uptake during CHNS-O analysis.

3.3.8 Fourier Infrared Transform Analysis

FTIR was employed to ascertain the chemical species and chemical composition of five samples. FTIR is a technique used to obtain infrared spectra of transmission and absorption of solid fuel samples to investigation of chemical bonds.

In this study FTIR technique used to ascertain the chemical composition and sample crystal structure. It can identify chemical bonds between functional groups by the absorption of infrared radiation which excites vibrational modes in the bond. The detection depth is 1-2 micrometres deep. A milligram of solid propellant sample was set at a pressure of 69-10^3 MPa. The scanning range of FTIR was between 4000-500 cm^{-1} with resolution of 4 cm^{-1} [263].

3.4 Measuring the Thrust, Burn Rate and Concentration Smoke of Solid Propellant

During development of a minimum signature solid propellant, it is tested extensively and characterized. This includes the testing of the burn rate under different pressure and temperature, impurities, and conditions [114]. A solid rocket motor nozzle is a specifically designed front section of the thrust chamber that regulates the expansion of the exhaust products to effectively transfer the energy forms produced in the combustion chamber to kinetic energy, providing thrust to the vehicle[157]. This is accomplished by creating the required nozzle geometric profile. Under high temperatures, this nozzle is subjected to extremely high pressures and rapid dense gas flow. Extreme heat transfer occurs in the nozzle's throat area, which reduces thrust and severely reduces the propulsion system's performance. The burning rate of propellant in a motor is a function of many parameters and at any instant governs the mass flow rate $\dot{m}$ of hot gases generated and flowing from the motor in stable combustion.

3.4.1 Measuring Thrust of Solid Propellant

Thrust measurement is a critical evaluation parameter in assessing the performance of solid propellants. It provides direct insight into the propellant's ability to generate force, which directly correlates to its energy output and efficiency. In this study, the thrust produced by AP/Sorbitol and AP/Sorbitol/Mg solid propellant formulations was measured using a static test setup Digital Load Cell Weight Sensor HX711 AD Converter Breakout Module 5Kg Electronic Scale. Prior to testing, the load cell used for thrust measurement was calibrated using a certified 5 kg standard weight to ensure accuracy across the expected measurement range. This calibration step allowed verification of the sensor's linearity and sensitivity. Similarly, the digital balance for residue mass measurement was checked against the same standard weight to confirm proper zeroing and scale accuracy. These procedures ensured that measurement errors were minimized and that the recorded thrust and residue values were reliable

In this section, only AP/Sorbitol and AP/Sorbitol/Mg formulations were selected for thrust performance evaluation, as the primary focus of this research is to investigate the effect of magnesium as a chloride scavenger and, metal fuel additive,

rather than the influence of ferric oxide catalytic materials. The AP/Sorbitol formulation was included as a baseline reference propellant to provide a clear comparison of thrust enhancement resulting from the addition of magnesium. The result then was compared with common Potassium Nitrate/Sorbitol (KNSB) impulse to see the effectiveness of oxidizing agent and metal additive. This strategic selection allows for a more direct assessment of how metal additives influence combustion energy and thrust characteristics without interference from other modifiers This setup allows for controlled ignition and continuous monitoring of thrust over the burn duration. By analysing the resulting thrust-time curves, important performance metrics such as peak thrust, total impulse, and specific impulse can be derived. The thrust data not only validate the thermochemical predictions but also serve to compare the effectiveness of metal additives in enhancing propellant output. The details of mechanical drawing with throat diameter for rocket motor class-D is shown in appendix 1.

3.4.2 Nozzle Design

The performance of a solid rocket motor is highly dependent on the relationship between the propellant geometry and the nozzle configuration. In this sub-section, the focus is placed on designing the appropriate sizing of the nozzle and estimating the expected performance based on standard motor classifications. The initial design is guided by the performance target of a class D rocket motor, which typically delivers a total impulse ranging from 5-10 Ns. For this reason, schedule 80 PVC pipe was used as the test chamber, imposing a thrust limit of below 3 kg. Accordingly, the internal pressure was constrained to about 120 psi to ensure structural safety This range serves as the benchmark for determining the required propellant mass, burn time, and grain geometry to achieve stable and efficient thrust within the defined classification.

Few vital variables are set and fixed at certain value so that nozzle design is aligned with these values: this is crucial to ensure safe and optimal performance and certain safety during combustion:

Chamber pressure (P_c) = 120 Psi

Chamber Temperature (T_c) = 1800 k

Specific Ratio (k)= $\frac{C_p}{C_v}$ = 1.157

Ideal gas constant (R)= 321.50 JKg^{-1} K^{-1}

Target force = 6 N

Target Burning duration= 3s-5s

Grain weight = 20g

To calculate the throat velocity, first must need to calculate temperature at throat use (3.10). Hence throat velocity can be calculated using equation (3.11)

$$T_t = T_c(\frac{2}{K+1}) \tag{3.10}$$

$$V_t = \sqrt{kR}T_C \tag{3.11}$$

Equation (3.12) used to determine the velocity at nozzle exist, it is crucial to get the mass flowrate in relation with the force that has been set in equation (3.13).

$$v_{exist} = \sqrt{\frac{2k}{k-1}}\sqrt{RT_C}\sqrt{1-\left[\frac{P_e}{P_c}\right]^{\frac{k-1}{k}}} \tag{3.12}$$

$$\dot{M} = \frac{F}{V_e} \tag{3.13}$$

(T_e) exists temperature is calculated from the equation (3.14) to get the important value of Specific volume in equation (3.15) for nozzle design.

$$T_e = T_c\left[\frac{P_e}{P_c}\right]^{\frac{k-1}{k}} \tag{3.14}$$

$$V_{specific} = \frac{RT_C}{P_C} \tag{3.15}$$

Eventually area of throat (A_t) and exit (A_e) for nozzle can be determined using (3.16) with all gathered information. In addition, the exit (d_e) and throat diameter (d_t) is determined by classic formula of circle (3.17).

$$A_{t,e} = \frac{V_{t,e}\dot{m}}{v_{t,e}} \tag{3.16}$$

$$A_{t,e} = \frac{\pi d_{t,e}^2}{4} \tag{3.17}$$

3.4.3 Measuring& Evaluate Smoke /Plume Concentration.

The evaluation of smoke opacity from solid propellants was conducted in accordance with the AGARD PEP WG-21 standard, which outlines procedures for characterizing visible smoke from rocket motor plumes. This method is critical in assessing the minimum signature capability of propellant formulations by quantifying the extent of visual obscuration caused by combustion products. In this study, propellant samples ranging from 5 to 10 grams were ignited in an open environment under controlled ambient conditions. A light transmittance meter (LS116 Digital Transmittance Meter) was positioned behind a translucent acrylic screen to measure the degree of light blocked by the smoke plume. The device projects a parallelled light beam through the smoke column, and the reduction in light intensity is used to calculate smoke opacity in terms of percentage light blockage (%).

Measurements were taken at a fixed distance and angle relative to the plume to ensure repeatability, following the AGARD recommendation on optical path alignment. Each formulation was tested in three times to ensure data consistency. The recorded data include the maximum opacity, average transmittance, and duration of obscuration, which together provide a comprehensive smoke profile for each propellant type. The relative humidity (R.H) and ambient temperature were recorded using the SKY2000-M multi-gas detector, as the formation of condensed gases is highly influenced by surrounding humidity and temperature conditions [264]. These results were analysed to determine the effectiveness of metal additives such as magnesium and ferric oxide in reducing visible smoke, supporting the development of low-signature propellant systems.

In addition to the primary formulations based on AP/sorbitol with and without metal additives, several benchmark solid propellants were examined. These included AP/HTPB/Al, a standard high-performance composite propellant widely used in tactical missile applications; KNSB (Potassium Nitrate/Sorbitol) and KNSU (Potassium Nitrate/Sucrose), which represent typical sugar-based propellants known for their simplicity and clean combustion; and Starch/AP, a binder-oxidizer system of interest for its cost-efficiency and minimal processing requirements. These comparative samples were subjected to the same smoke opacity measurement protocol, adhering to the AGARD PEP WG-21 standard, to assess their visual exhaust signature relative to the AP/Sorbitol/Mg-based formulations as illustrated in Figure 3.6.

Smoke formation is strongly influenced by relative humidity and the temperature surrounding; therefore, it is essential to determine these parameters during testing. The standards provided by AGARD offer a reliable reference framework, serving as a blueprint to predict the smoke classification of solid propellants based on their combustion behaviour under defined environmental condition. From Figure 3.6, the classification along the down the rows are determined by the measured light opacity, which is used as an indicator of primary smoke. As the light opacity increases, the primary smoke intensity increases, corresponding to a downward progression in the classification matrix. In contrast, secondary smoke is governed by gas-phase condensation effects and is influenced by ambient conditions, particularly relative humidity, which promotes saturation and visible plume formation. The addition of these references serves to contextualize the performance of the developed minimum signature propellants in terms of combustion cleanliness and visibility, enabling a clearer understanding of how formulation chemistry influences smoke production.

	AGARD Ambient R.H to Produce Saturation Gas			
AGARD OBSTRUCTION		1.0	0.90	0.55 0.0
	0.35	AA	AB	AC
	0.90	BA	BB	BC
	1.00	CA	CB	CC

Figure 3.6 AGARD PEP WG-21 Proposed Primary And Secondary Smoke Classification Scheme [118]

Light transmission through the smoke generated from the combustion of five AP/Sorbitol-based solid propellant samples was measured and compared against the

light transmission of other commonly used propellants, namely KNO_3/Sorbitol, AP/Starch, and AP/HTPB/Al, under a controlled Malaysian relative humidity of 74%. The resulting light transmission data were used to calculate smoke opacity. The solid residues from the AP/Sorbitol-based propellant combustion were collected using a filter, and the captured particles were further analysed for their morphology and elemental composition.

The combustion process was conducted in an open area to minimize contamination from hydrogen chloride (HCl) gas. The concentration of HCl released into the surrounding atmosphere during combustion was monitored using a SKY2000-M multi-gas detector, in accordance with the standards set by the Occupational Safety and Health Administration (OSHA)) [265] which stipulates a permissible exposure limit not exceeding 5 parts per million (ppm).

The solid propellant of AP/HTPB/Al , KNO_3/Sorbitol and AP/Starch was prepared accordingly with previous literature [58], [266]. The details on solid propellant formulation can be referred to Table 3.1.

Table 3.1
Composition Of Solid Propellant

Sample Name	Formulation Wt. %	METAL ADDITIVE
SP4	Ap 77% Sorbitol 23 %	-
SP5	Ap 80% Sorbitol 20%	-
SP6	Ap 77% Sorbitol 23 %	Mg 1%
SP7	Ap 77% Sorbitol 23 %	Ferric oxide 0.05%
SP8	Ap 77% Sorbitol 23 %	Mg1%, Ferric oxide 0.05%
SP9	KNO3 65% Sorbitol 35%	-
SP10	AP 77% HTPB 20%	Al 10%
SP11	AP 80% Starch 23%	-

3.4.3.1 Smoke Opacity

Smoke opacity was measured using a photometric opacity meter, which quantifies the reduction in light transmission caused by suspended particles in the exhaust plume. The instrument detects the percentage of light blocked by smoke, providing a direct measure of exhaust opacity for each propellant sample. For the

smoke-opacity measurements, each propellant formulation was tested three times to ensure repeatability. The mean opacity value was used for analysis, while the variation between repeated burns was monitored to confirm experimental consistency. The solid propellants were burned at 3 different weights: 0.2 g, 0.3 g, and 0.4 g in chamber. The reading on light transmittance was recorded. Measured light transmittance was used to calculate the smoke opacity shown in Figure 3.8. The experiment was set up for measuring the light transmittance pass through the smoke release from the combustion show in Figure 3.7. The device LS116 used to measure light transmittance has light wavelength 380-760nm, which confirms to the International Commission on Illumination (CIE) bright vision function standard. The LS116 Light Transmittance Meter is a digital device used to measure the percentage of visible light passing through a material. It operates using a parallel light source and a receiver, providing accurate readings of light attenuation. In this study, the LS116 was used to evaluate smoke opacity by comparing light transmission before and after combustion of solid propellant samples. The LS116 light transmittance meter is originally designed to measure the opacity of materials and smoke emissions, particularly from vehicle exhaust systems. It calculates smoke opacity using the formula equation (3.18) [267] making it suitable for assessing the optical density of smoke from combustion processes.

$$Opacity\ (\%) = 100 - Light\ Transmittance \tag{3.18}$$

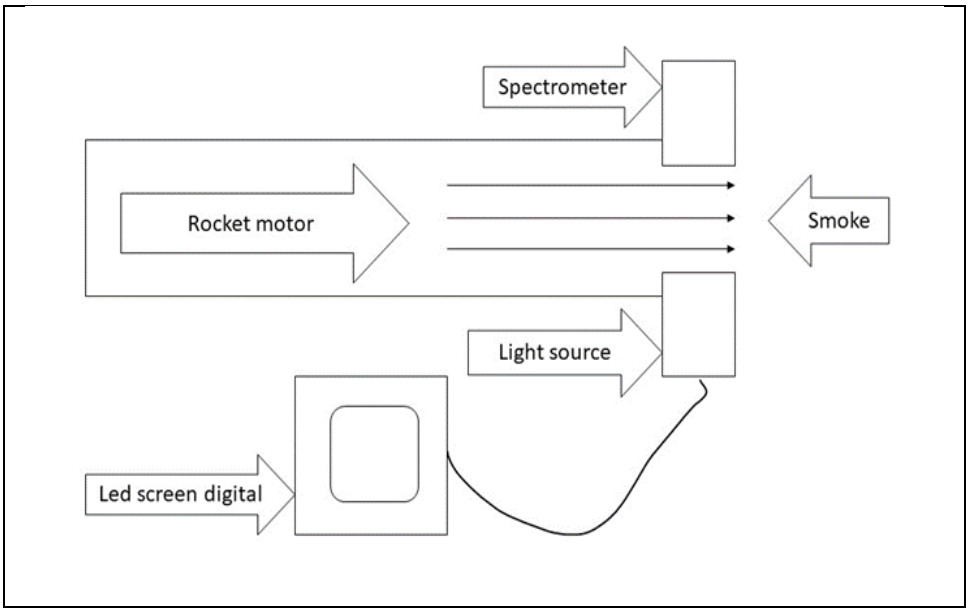


Figure 3.7 Smoke Opacity Measurement Setup Based on AGARD Standard

3.4.3.2 Smoke Particle

Propellant samples with a known mass of 0.3 g were combusted inside a test chamber fitted with an acrylic cylinder containing a glass wool filter. The glass wool, pre-weighed before combustion and reweighed after, was used to quantify the mass of solid particulates (primary smoke) captured during the burn [268], as illustrated in Figure 3.8. To enhance the efficiency of smoke flow through the filter, a fan was employed to actively draw the smoke across the glass wool medium.

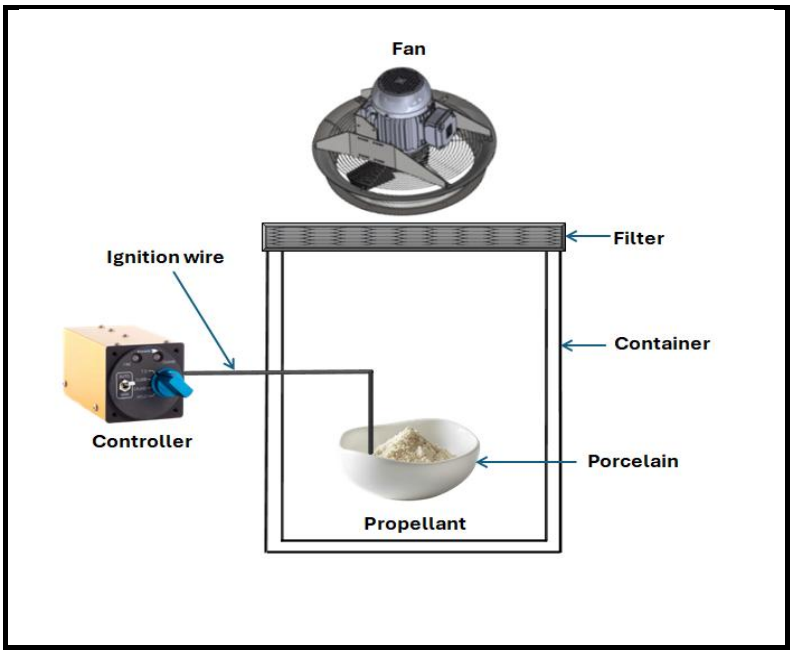


Figure 3.8 Smoke Particle Collector

3.4.3.3 Morphology and Composition of Combustion Product

The solid residual products resulting from the combustion of the solid propellant samples were collected and characterized using a Scanning Electron Microscope (SEM) (HITACHI SU3500) to examine the surface morphology and elemental composition of the combustion products [269]. Remarkably SEM/EDX analysis was conducted only on SP7 and SP8 because these formulations produced measurable solid residues after combustion. The remaining samples underwent near complete combustion, leaving insufficient residue for meaningful microstructural or elemental analysis Specific points on the sample surface were magnified at 100 μm to obtain more detailed information on particle deposition and distribution [270].

CHAPTER 4
RESULTS & ANALYSIS

4.1 Introduction

This chapter presents the results of the development of minimum signature solid propellant from AP-based sorbitol with metal additives. This chapter is divided into three sections. Section 4.2 focusses on the optimum ration which contributes to high energy performance with less combustion product. Section 4.3 characterized and evaluate kinetic properties of solid propellant. Section 4.4 focuses on testing and evaluating the performance of solid propellants, as well as measuring the opacity of the smoke produced by each formulation.

4.2 Formulation of Minimum Signature Solid Propellant

The performance of AP/sorbitol-based solid propellants is governed by thermochemical properties, oxygen balance, elemental stoichiometry, and heat energy release. Parametric analysis shows that optimizing the oxidizer-to-fuel ratio is critical for achieving efficient combustion and acceptable specific impulse while avoiding excess oxidizer or unburned fuel residues. Thermochemical predictions indicate that formulations within the 77–80 wt.% AP range provide a favourable balance between energy release and combustion stability.

To examine this behaviour, five minimum-signature propellant formulations were evaluated, with AP/sorbitol/Mg as the primary system and additional samples introduced to assess the influence of AP loading and catalytic effects. Increasing AP content within the optimized range enhanced theoretical energy output, while the presence of metal additives influenced combustion efficiency. Experimental bomb calorimetry generally followed the same trend predicted by thermochemical calculations, although small deviations were observed due to heat losses and combustion inefficiencies. Overall, the combined computational and experimental analysis confirms that careful control of oxygen balance and formulation stoichiometry is essential for optimizing the thermal performance of AP/sorbitol-based minimum-signature solid propellants.

Total five formulations of minimum-signature composite solid propellant samples were examined and analysed in this study. The AP/Sorbitol/Mg sample serves as the primary material in this study, while four additional samples were prepared for comparison in terms of AP content and the catalytic effects on the solid propellant. The solid powder of ammonium perchlorate loading was raised from 77% to 80%, and a metal additive was added as shown in Table 4.2. The samples were prepared based on the previously calculated optimized ratio and used to determine their heat of combustion, which was then compared with the computed values.

4.2.1 Parametric Analysis

The percentage by mass of mixtures was determined using (3.1) and (3.2) considering the weight of ammonium perchlorate and sorbitol present in Table 4.1, Between the range of 77 to 80% amount of AP, the formulation of solid propellant mixtures has optimum oxygen balance and elemental stoichiometric coefficients. As the oxidizer content increases, the carbon constituents in the mixture slightly decrease, despite the total oxygen remaining constant. This indicates that the mixture becomes oxidizer dominant. Referring to the oxygen balance value, a positive trend is observed as the oxidizer concentration increases. Furthermore, the elemental stoichiometric coefficient exceeds 1 when the AP composition surpasses 77%, signifying an oxidizer-rich mixture.

Table 4.1 show the stoichiometric formulation, oxygen balance (O/B), and elemental stoichiometric coefficient for different AP-to-Sorbitol weight ratios provide critical insights into the combustion behaviour and efficiency of the propellant. As the oxidizer (AP) content increases from 60% to 85%, several key trends emerge regarding combustion efficiency, fuel-to-oxidizer balance, and thermal stability. A notable trend is the shift in oxygen balance as the AP concentration increases. A negative oxygen balance (O/B < 0) signifies a fuel-rich mixture, where incomplete combustion may occur due to insufficient oxygen availability, leading to the formation of carbon monoxide (CO), soot, or unburned fuel residues. At 60% AP, the oxygen balance is -29.10%, indicating a highly fuel-rich condition. As the AP concentration increases, the oxygen balance progressively shifts toward zero, reaching -0.8% at 80% AP and eventually becoming positive (6.8%) at 85% AP, indicating an oxidizer-rich environment. While a positive oxygen balance promotes complete combustion,

excessive oxidizer content can result in higher flame temperatures, which may contribute to propellant instability or excessive chamber material erosion.

Another key observation is the increase in the elemental stoichiometric coefficient, which represents the reaction balance between oxidizer and fuel. At 60% AP, the coefficient is 0.689, confirming a highly fuel-rich system. As the AP content increases, this coefficient gradually rises, reaching 1.069 at 80% AP and further increasing to 1.23 at 85% AP, demonstrating a shift toward oxidizer dominance at higher AP concentrations. This shift has significant implications for combustion efficiency, as higher oxidizer content improves reaction completeness and thermal efficiency but may also lead to increased chamber pressure and potential over-oxidation.

The impact of these variations in AP content extends to combustion stability and efficiency. Fuel-rich compositions (AP $\leq$ 70%) typically result in lower flame temperatures, which can reduce overall thrust performance and lead to incomplete combustion. On the other hand, balanced compositions (AP 75%-80%) approach stoichiometric conditions (O/B $\approx$ 0), ensuring optimal combustion energy with minimal excess reactants, making them ideal for controlled and efficient energy release. However, oxidizer-rich formulations (AP $\geq$ 85%) promote complete combustion but may introduce challenges such as increased thermal stress, higher combustion temperatures, and greater risk of material degradation.

In conclusion, the analysis indicates that an AP content of 75-80% provides the most balanced combustion conditions, where the oxygen balance is nearly neutral, and the elemental stoichiometric coefficient approaches unity. These formulations are expected to deliver optimal combustion performance, higher energy output, and reduced solid residues, making them more suitable for efficient propulsion applications. However, further experimental validation through bomb calorimetry, burn rate testing, and combustion stability assessments is necessary to refine the ideal mixture and optimize performance for specific operational conditions.

Table 4.1

Effect of Mass Ratio Solid Propellant on Oxygen Balance Mixtures.

Weight %		Stoichiometric Formula	Oxygen Balance	Elemental Stoichiometric Coefficient
AP	Sorbitol			
60	40	$C_{1.32}H_{5.11}N_{0.51}O_{3.36}Cl_{0.51}$	-29.10	0.689
65	35	$C_{1.152}H_{4.9}N_{0.53}O_{3.36}Cl_{0.55}$	-22.025	0.758
70	30	$C_{0.98}H_{4.67}N_{0.59}O_{3.36}Cl_{0.59}$	-14.95	0.840
75	25	$C_{0.82}H_{4.48}N_{0.64}O_{3.38}Cl_{0.64}$	-7.875	0.941
80	20	$C_{0.65}H_{4.25}N_{0.68}O_{3.37}Cl_{0.68}$	-0.8	1.069
85	15	$C_{0.49}H_{4.04}N_{0.72}O_{3.38}Cl_{0.72}$	6.8	1.23

Figure 4.1 illustrates the relationship between solid propellant formulation and elemental stoichiometric coefficients. At stoichiometric balance $\emptyset_e = \mathbf{1}$, the formulation of propellant is 0.775 weight percent. At a value 0.775 wt% and above, the elemental stoichiometric balance is fuel lean at $\emptyset_e > \mathbf{1}$. It indicates oxidizing agent dominance. The straightforward method presented for calculating elemental stoichiometric coefficients could be simply extended to multicomponent fuel-oxidizer systems. Because these characteristics are simple to calculate, they might be used to quickly calculate elemental stoichiometric coefficient at any given mixture composition.

Furthermore, to address the limitations associated with the elemental stoichiometric coefficient, the oxygen balance of the mixture was determined and plotted against the solid propellant composition. As shown in Figure 4.2, when the oxygen balance exceeds 0.8%, it becomes positive. Additionally, at approximately 0.775 wt.% in correlation with the elemental stoichiometric coefficient, the oxygen balance is nearly zero. This finding indicates that ammonium perchlorate serves as the primary oxygen provider for fuel combustion, significantly influencing the overall reaction efficiency and combustion behaviours.

Table 4.2 presents the finalized values for the solid propellant formulations. The compositions vary based on the AP-to-sorbitol ratio and the inclusion of metallic additives. SP4 and SP5 contain AP and sorbitol at weight ratios of 77:23 and 80:20, respectively, without any additives, serving as reference formulations. SP6, as the

primary developed solid propellant formulation, incorporates [121] 5% Mg as a metal fuel to evaluate its impact on combustion efficiency.

In conclusion by referring (4.1), the selection of 5% Mg in the AP/Sorbitol-based solid propellant formulation is justified by its ability to enhance burn rate, increase flame temperature, optimize oxygen balance, reduce smoke emissions, and maintain structural integrity. Beside that it produces thermochemical modelling, experimental validation, and literature benchmarks collectively support 5% Mg as an optimal additive concentration that balances high-energy output with minimal combustion by-products. Furthermore, magnesium is oxidised to magnesium oxide (MgO), indicating that Mg participates as an energetic metal fuel and as a precursor for chlorine mitigation. This formulation represents a high-performance, low-signature propellant design suitable for advanced propulsion applications. Additionally, SP7 includes 0.05% ferric oxide as a catalyst to function as a burn rate modifier, enhancing combustion characteristics since amount of catalyst used not affected the combustion reaction small amount is enough for the reaction [271]. Lastly, SP8 combines both 5% Mg and 0.05% ferric oxide to evaluate the synergistic effects of metal additives on combustion performance and burn characteristics. These formulations were systematically analysed to assess their thermal behaviour, energy release, and residue formation, providing valuable insights into their suitability for optimized solid propellant performance.

$$6NH_4ClO_4 + C_6H_{14}0_6 + 5Mg \rightarrow 3N_2 + 6HCl + 3CO_2 + 3CO + 16H_2O + 5MgO \quad (4.1)$$

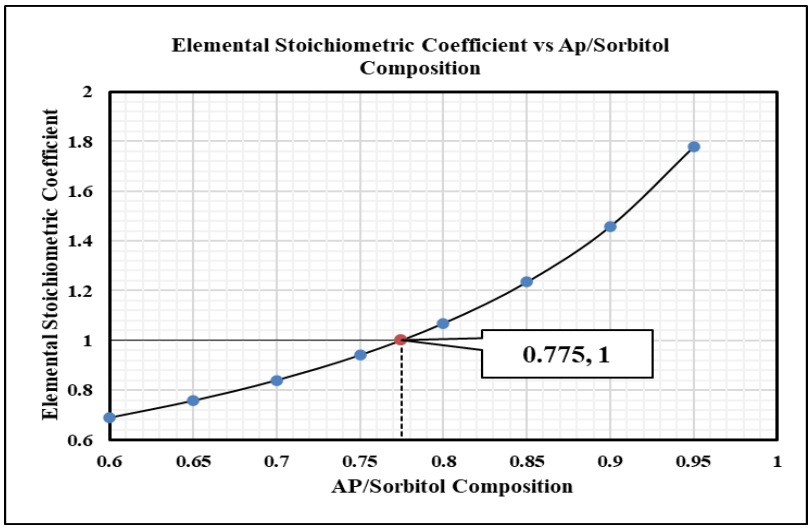
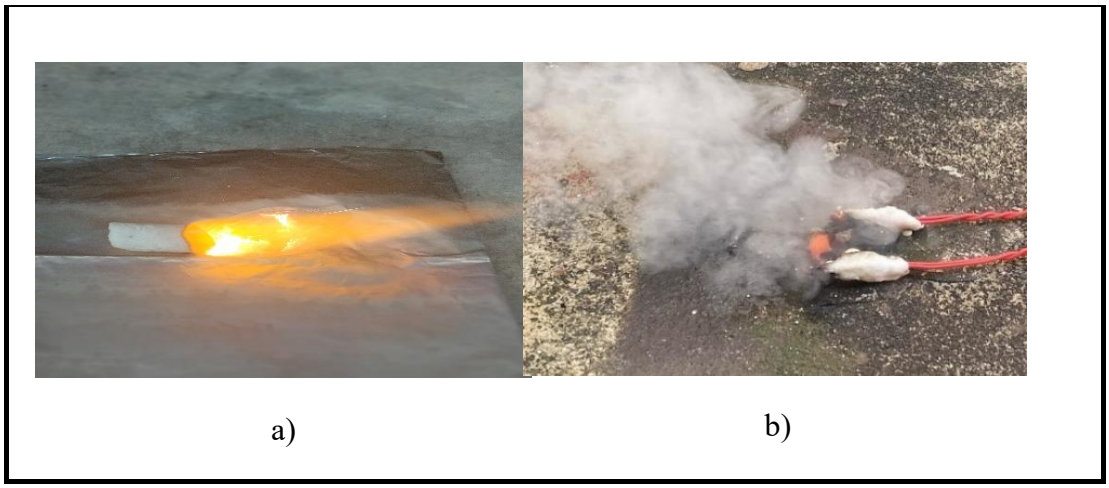


Figure 4.1 Elemental Stoichiometric Coefficient of Solid Propellant



a) b)

Plate 4.1 Combustion Of A) AP/Sorbitol/Mg Solid Propellant And B) KNSB Solid Propellant

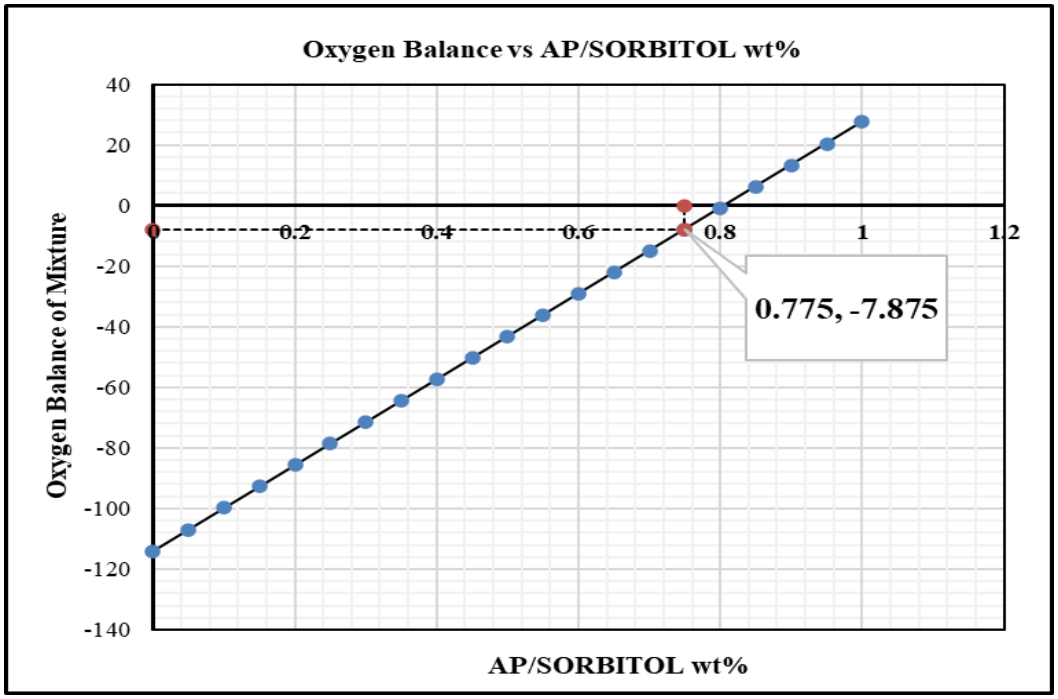


Figure 4.2 Oxygen Balance of the AP/Sorbitol Mixture

Moreover, oxygen balance value is aligned with elemental stoichiometric coefficient value since both parameters can also determine solid propellant performance. Hence it indicates at that 0.775 wt% weight percent, the mixture composition will have high performance and possess a high rate of reaction. plate 4.1 show the comparison of smoke release from combustion of AP/sorbitol/Mg and common solid propellant Potassium nitrate/sorbitol (KNSB) at atmospheric pressure and temperature. The thick smoke from the KNSB burning product mainly solid particles from incomplete combustion of fuel. This show that positive oxygen balance with strong oxidizing agent is capable to provide more oxygen to burn the fuel

completely. Moreover, it capable to burn Mg metal completely since no trace of solid gas particle observed in this combustion.

Table 4.2
Mass Fractions of The Components Used In Minimum Signature Solid Propellant

Sample Name	Formulation Wt. %	Additive
SP4	Ap 77% Sorbitol 23 %	-
SP5	Ap 80% Sorbitol 20%	-
SP6	Ap 77% Sorbitol 23 %	Mg 5%
SP7	Ap 77% Sorbitol 23 %	Ferric oxide 0.05%
SP8	Ap 77% Sorbitol 23 %	Mg5%, Ferric oxide 0.05%

4.2.2 Heat Combustion Analysis

Heat of combustion for the propellant mixture clearly dependent on the amount of oxygen balance. One of the crucial characteristics of highly energetic materials is oxygen balance. The oxygen balance of a substance is the proportion of excess or deficiency of oxygen needed for its complete oxidation of solid propellant to form carbon dioxide and water, thus can be utilized to optimize the composition of explosives or propellant formulations as needed [165]. An energetic compound should have zero and above OB requirements to have high heat of combustion, as this corresponds to the stoichiometric oxidation of all carbon convert to carbon dioxide and hydrogen to water. The high heat of combustion indicates that an energetic material has a positive oxygen balance. Approaching zero in this balance is preferable for achieving higher energy performance. Conversely, propellants with significantly high positive oxygen balances typically exhibit lower energy performance [173][13]. Figure 4.3 depicted the maximum value of the heat of combustion 4364.16 KJ/Kg happened at value approach zero and heat of combustion start to decrease as oxygen balance increase.

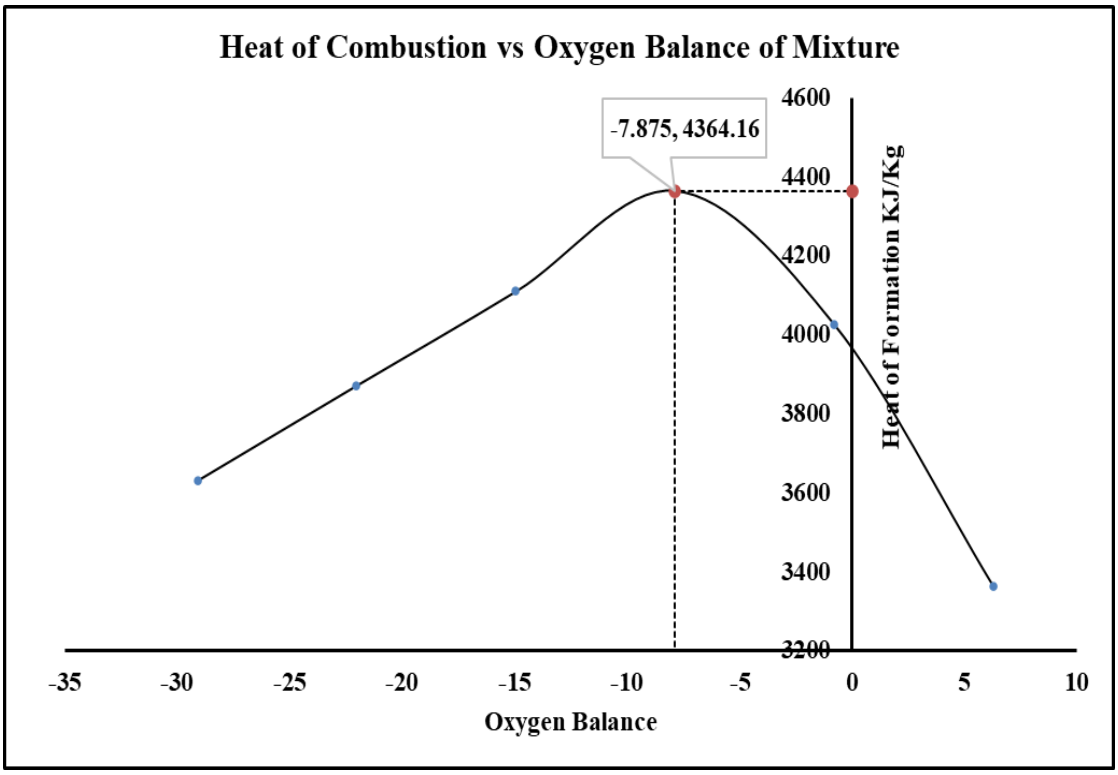


Figure 4.3 Effect of Oxygen Balance on Heat of Combustion of Ammonium Perchlorate/Sorbitol

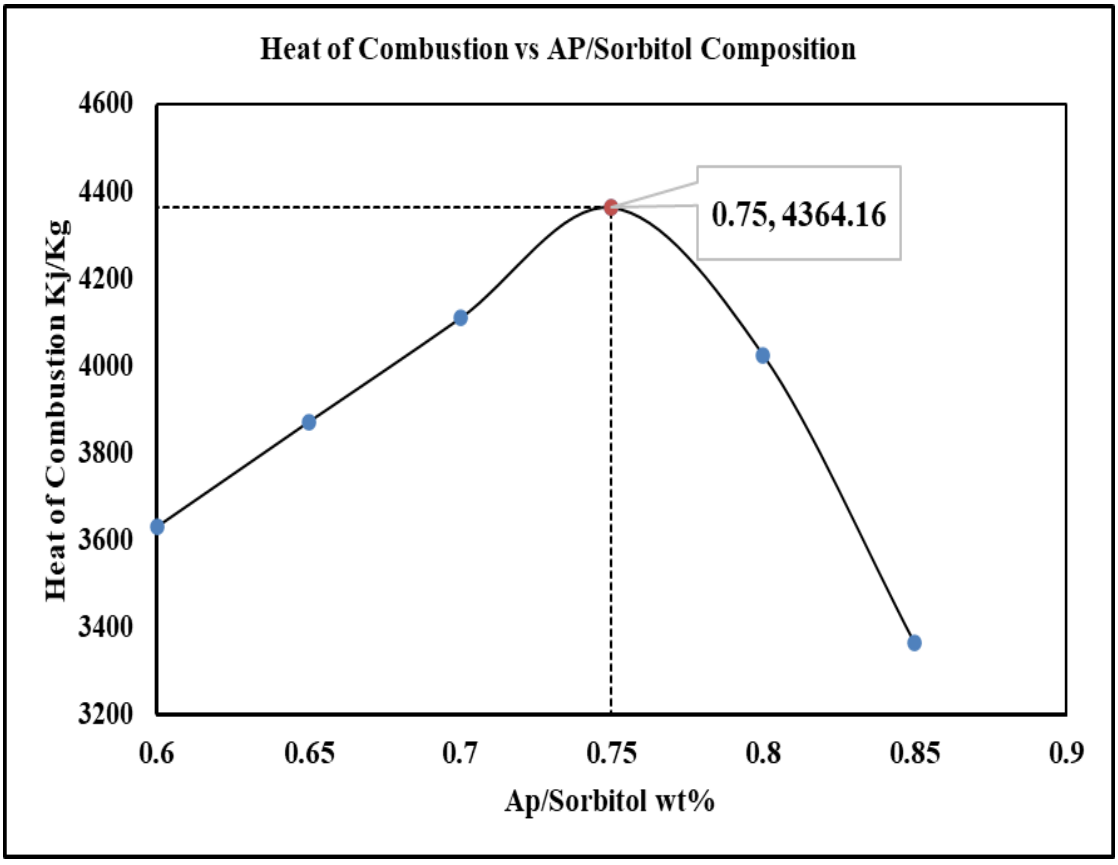


Figure 4.4 Heat of Combustion And Ammonium Perchlorate/ Sorbitol Mixture.

The oxygen balance clearly demonstrates an enhancement in positive value with an increase in the oxidizing agent's quantity. Figure 4.3 and Figure 4.4 reveal a strong relationship between Heat of combustion and Oxygen balance, particularly as the amount of the oxidizing agent increases. This calculation established the dependency relationship between the Heat of Combustion and the quantity of the oxidizing agent. The close connection between the intramolecular aspects of the oxidizer and fuel elements is indicated by the stoichiometry coefficient parameter proposed by Bakhman.et al. [239]. In addition to the choice of oxidizing agent, the interaction between the oxidizer and fuel significantly influences the prediction of heat generation and the formation of combustion gas products.

4.2.2.1 Bomb Calorimeter

The results of the calculations and experiments are illustrated in Table 4.3. The calorific value of the metalized propellant has shown a substantial increase compared to the baseline propellant. A noticeable observation from Table 4.3 is that the heat of combustion is lower for the sample SP5 which contain ammonium perchlorate-based propellants compared to the other sample which contains 77% AP-based propellants. However, the heat of combustion for Magnesium alone, SP6 was higher than that for iron oxide in SP7. The lower heat of combustion for SP5 AP-based propellants compared to SP4 AP-based propellant is attributed to the fact that excessive oxygen content can reduce the energy combustion same as reported in literature [172].

Table 4.3
Comparison Between Theoretical And Experimental Value of Energy Combustion

Samples	Heat of Combustion (Theoretical) Q KJ/mol	Heat of Combustion (Experiment) KJ/mol	Error Analysis (%)
SP4 (base line)	-4364.16	-2134.47	51.1
SP5	-4025.91	-1979.55	50.8
SP6	-6515.32	-4142.57	36.4
SP7	-5431.06	-3110.35	42.7
SP8	-7564.22	-5344.88	29.3

The samples contain magnesium metal have high heat of combustion, primarily due to the highly exothermic nature of the formation of two metal oxides and chloride

(MgCl and MgO), contributing to high heat of combustion values. The high heat of combustion of these sample can cause erosion on the nozzle throat which can reduce the specific impulse of solid rocket performance [269]. Additionally, sample with iron oxide which serves as a catalyst, lowering the activation energy and providing an easier path for the reaction to take place does not contribute in energy release. Moreover, the samples ignite more easily due to the lower ignition temperature effect of catalyst addition. From all the formulations propellant, it reveals that SP8 demonstrates the highest calorific value. This highlights the interaction between iron oxide, magnesium metal, and the AP/sorbitol propellant. Although iron oxide alone doesn't make a substantial contribution to the energy performance, its synergistic effect with AP/sorbitol/Mg is notably impressive.

The experimental heats of combustion obtained from bomb calorimetry were consistently lower than theoretical predictions, with percentage errors ranging from 29.3% to 51.1% shown in Table 4.3. The huge difference because of theoretical heat of combustion values is typically derived under idealized assumptions, including complete combustion, equilibrium product formation, and full oxidation of metallic additives. In contrast, bomb calorimeter measurements reflect real combustion behaviour, where incomplete oxidation of metal particles, formation of condensed combustion products, and residual unreacted material can significantly reduce the measured heat released. Additionally, unavoidable heat losses to the bomb calorimeter body, ignition system, and surrounding water jacket further contribute to lower experimental values. All these factors is effect of experiment environment on calorimetric value of solid propellant [272]. Therefore, the observed deviation represents the inherent limitation of translating ideal thermochemical predictions to practical calorimetric measurements, rather than weaknesses in experimental execution.

Although the percentage difference between the experimental and theoretical heats of combustion is large, the results remain internally consistent across the samples. Both datasets exhibit the same directional behaviour, indicating that the experimental measurements preserve the relative energy ranking rather than varying randomly. As shown in Figure 4.5, the Pearson correlation between theoretical and experimental heat of combustion is strongly positive ($R^2 = 0.994$), demonstrating an almost perfectly linear relationship between the two variables. This implies that, despite a systematic balanced in magnitude, increases in theoretical heat of combustion are accompanied by

proportional increases in the experimental values, confirming a stable and repeatable trend across the samples.

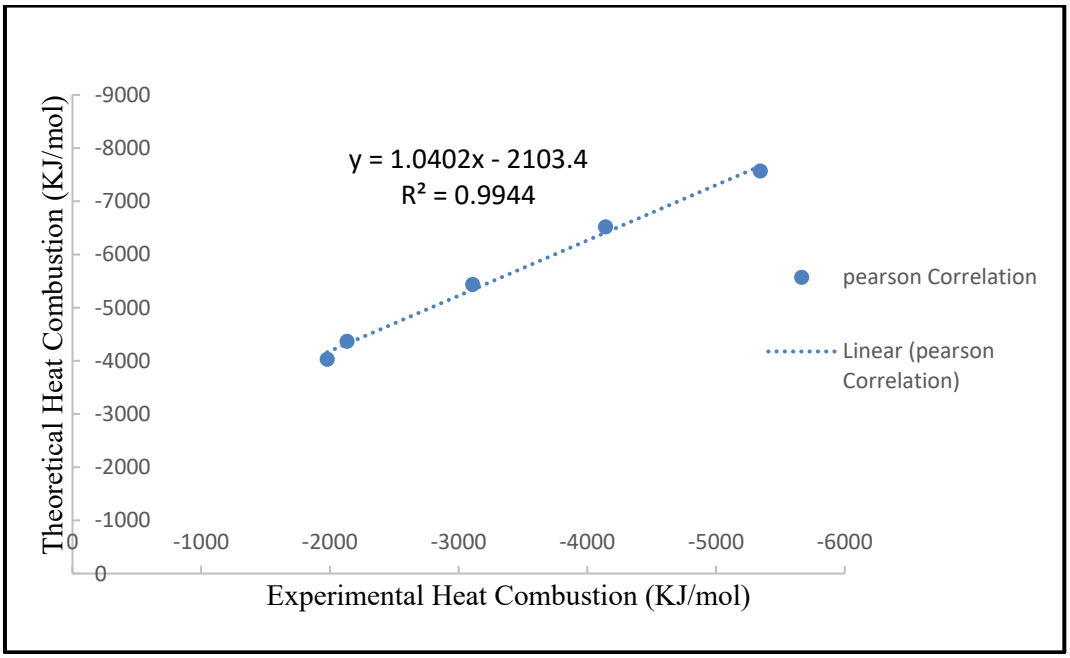


Figure 4.5 Correlation Between Theoretical And Experimental Value Heat of Combustion

4.2.2.2 Open Burning

The initial burn rate investigation was conducted at ambient pressure and room temperature as a preliminary step to gain a deeper understanding of the solid propellant's combustion behaviour. This approach ensures a controlled assessment before conducting burn rate measurements under varying pressure and temperature conditions, thereby minimizing the risk of unforeseen accidents.

The bar chart shown in Figure 4.6 clearly shows that magnesium and ferric oxide significantly increase the burning rate of solid propellant at 1 atm. The burning rate of propellants increased from 0.0107 to 0.126 cm/s. The burning rate of ammonium perchlorate/sorbitol solid propellant in this study was relatively low when compared to other ammonium perchlorate based solid propellants [273]. Magnesium functions as a solid propellant burning rate enhancer in addition to being a chloride scavenger. Because magnesium is an alkali earth metal, it works as a fuel for oxidation while performs as a chlorine scavenger. SP8 has a high burning rate, and it possesses the highest burning rate which implies that both magnesium and ferric oxide have a synergistic effect. This depicts magnesium metal and ferrous oxide as efficient burning rate enhancer.

Figure 4.7 shows the burning process of the composite solid propellant for the 5 samples. It illustrates that the combustion of ammonium perchlorate mixed with sorbitol produces little visible smoke. It essentially indicates that the interaction between the oxidizer and the fuel has fully developed to achieve a complete combustion reaction. Apart from the absence of soot emission, there was no solid residual detection for samples SP4 and SP5. It indicates that the entire amount of carbon and hydrogen in the sorbitol is converted into carbon dioxide and water vapor. Nevertheless, despite the use of a powerful oxidizing agent, the burning rate of the solid propellant was slightly lower. Magnesium and ferric oxide were incorporated as metal additives in three solid propellant samples, SP6, SP7, and SP8, to enhance the burning rate and overall combustion performance. These metal additives play a crucial role in accelerating the combustion process by increasing thermal energy release. The combustion flame of SP6, SP7, and SP8 exhibited a vigorous and intensely bright yellow flame due to metal additive, with SP7 and SP8 demonstrating particularly explicit luminosity. Since additive metals is a key contributor to solid propellant performance, its presence significantly improves the heat of combustion, leading to higher energy output and more efficient fuel oxidation.

On the contrary, the reaction of magnesium metal with chloride gas causes the production of solid residual due to the reaction of magnesium with chloride gas. Plus, the addition of ferrous oxide as a catalyst to the formulation also contributes to the formation of solid products. This will contribute to the production of solid particles and solid residuals as unwanted products. This will be discussed in next section.

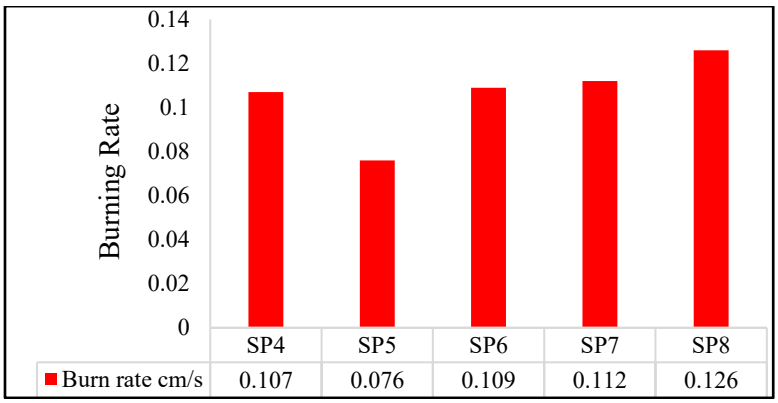


Figure 4.6 Burning Rate of Solid Propellant For Every Different Formulation

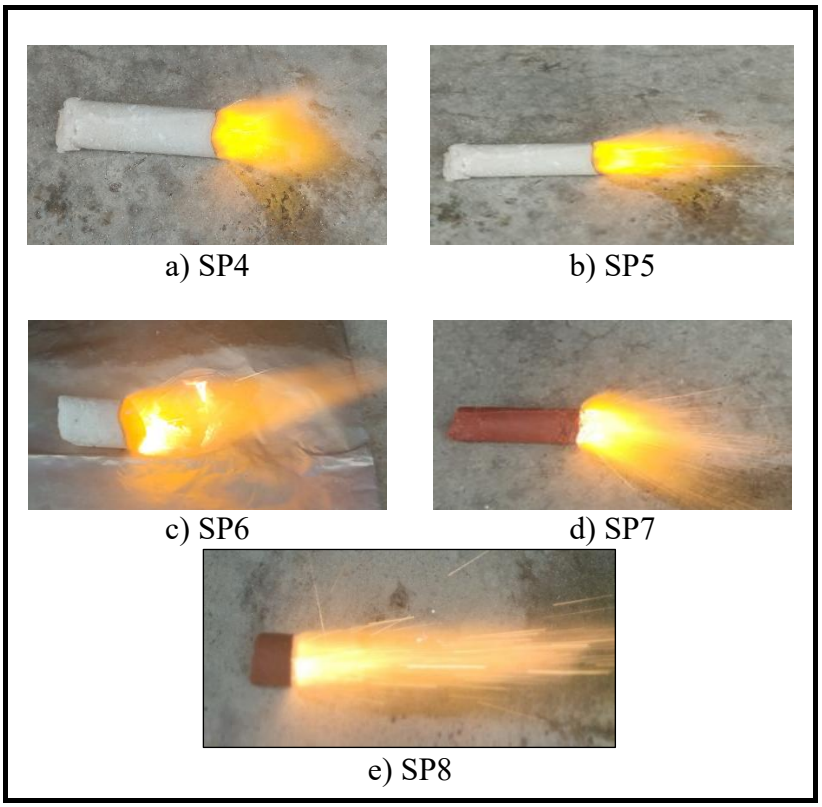


Figure 4.7 Combustion of Minimum Signature Composite Solid Propellant

In conclusion five compositions of a novel minimum-signature composite solid propellant using ammonium perchlorate and sorbitol were produced. The optimum ratio between ammonium perchlorate and sorbitol was calculated at 0.775 wt% using the elemental stoichiometric coefficient and oxygen balance. From the burning test of optimized formulation, ammonium perchlorate/sorbitol produces very minimum smoke and leaves no solid residual. It indicates that all carbon compounds in sorbitol have been completely reacted by oxygen from ammonium perchlorate. The interaction between ammonium perchlorate and sorbitol is improved by adding magnesium metal and ferrous oxide. Additive metals improve the burning rate of the solid propellant significantly. Magnesium metal and ferrous oxide show a good synergetic effect by increasing the burning rate extensively at 0.126 cm/s. To sum up, ammonium perchlorate/sorbitol propellant has a significant promise in replacing imported, expensive, minimal-signature solid propellant. Future work on the kinetic study, thermal decomposition, characterization, and burning rate effect of these propellants discussed on next section.

4.3 Characterization of Solid Propellant.

Solid propellants were characterized chemically and physically for understanding their chemical composition, physical properties, thermal stability, and combustion performance. After that kinetic analysis were done to evaluate propellants stability and ignition properties. A comprehensive evaluation of these properties ensures the formulation's reliability, efficiency, and safety in propulsion applications. This section focuses on the chemical and physical characterization of AP/Sorbitol-based solid propellants with and without metal additives, aiming to assess their impact on combustion behaviour, structural integrity, and energetic performance,

4.3.1 Mechanical Properties

4.3.1.1 Tensile Test

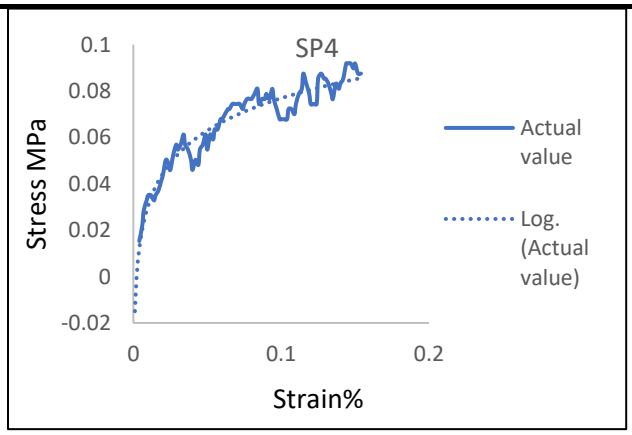
Table 4.4 summarizes the stress calculations for each of the samples based on the provided maximum force data and the geometric dimensions (thickness and width), along with their respective formulations and metal additives. Overall, the analysis results of the samples have shown significant variations in mechanical strength, which can be attributed to their respective formulations and the presence of the metal additives. Specifically, samples SP 6, SP 7 and SP 8 have demonstrated a clear trend of increasing strength with the addition of metal additives.

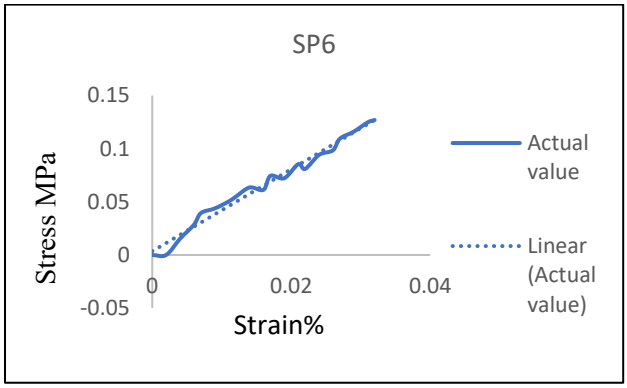
Table 4.4
Raw Data of Mechanical Properties of Samples With Different Formulation

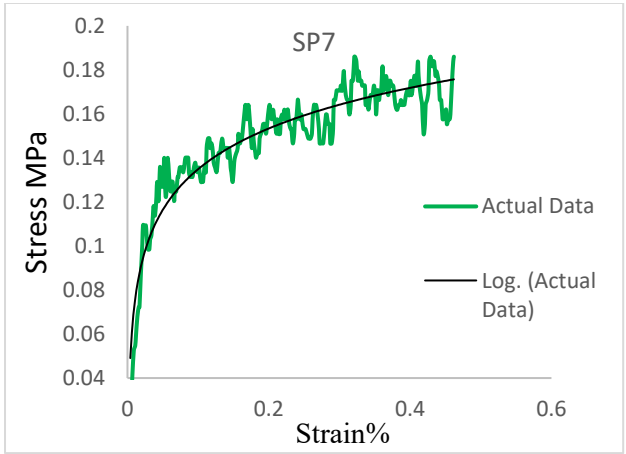
Sample	Thickness (mm)	Width (mm)	Max. Force (N)	Stress (N/mm2)
SP4	3.5	6	15.625	0.446
SP6	3.5	6	21.875	0.625
SP7	3.5	6	32.8125	0.938
SP8	3.5	6	39.0625	1.116

The samples containing metal additive show significantly increase in physical properties. SP7 and SP8 contain ferric oxide as catalyst improve strength significantly compared to SP6 which has mg metal only. Although mg metal did improve physical properties of solid propellant, with ferric oxide the interaction is better as shown in SP8. This means that metal catalysts not only increase burning rate but also increase solid

propellant physical properties by providing medium for oxidizer and fuel to well dispersed for interacting each other.







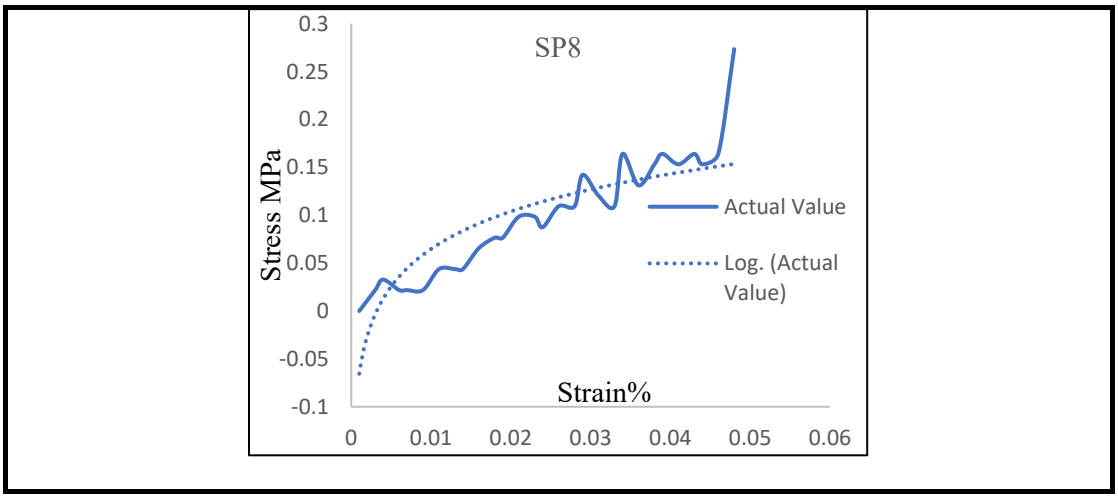


Figure 4.8 Stress Strain Curve for Samples SP4, SP6, SP7 And SP8

The study analysed the mechanical strength of solid propellant samples with various metal additives shown in graph stress-strain curve in Figure 4.8. The baseline sample (SP 4) without additives exhibited the lowest stress value of 0.446 N/mm² depicted in. Adding 5% magnesium (SP 6) increased the stress value to 0.625 N/mm². Incorporating 1% ferric oxide (SP 7) further enhanced the stress value to 0.938 N/mm². The combination of 5% magnesium and 1% ferric oxide (SP 8) resulted in the highest stress value of 1.116 N/mm². The findings demonstrate that metal additives significantly improve mechanical strength, with a progressive increase observed from SP 4 to SP 8. These results are crucial for material selection and processing in high-strength applications, warranting further investigation into the underlying factors.

Table 4.5
Mechanical Propeties of Solid Propellant AP/Sorbitol With Metal Additives

Samples	Young Modulus (GPa)	Ultimate Tensile Strength (Mpa)	Strain Break (%)
SP4	0.00362	0.112	0.154
SP6	0.00711	0.153	0.032
SP7	0.00422	0.229	0.462
SP8	0.01092	0.2735	0.048

Table 4.5 summarizes all the value mechanical parameters of solid propellant with and without metal additive. Metal additives including metal catalyst remarkably improve the mechanical strength of the propellant. SP 7 shows higher strain elongation

compared to all the samples, indicating it is more ductile and can absorb more energy during deformation. This demonstrates that ferric oxide not only functions as a burning enhancer but also facilitates physical interaction between the oxidizer and fuel. Hence, ferric oxide works synergistically with magnesium in SP 8 to improve the tensile properties of the solid propellant. The significantly lower strain at break values observed for SP6 and SP8 are primarily attributed to the incorporation of magnesium as a metal additive in the propellant formulation. The presence of Mg particles increases the stiffness of the AP/sorbitol matrix and restricts molecular mobility, resulting in a predominantly brittle mechanical response. Magnesium acts as a rigid filler that limits elastic deformation and promotes early crack initiation under tensile loading. Additionally, both AP and sorbitol are hygroscopic, which tends to reduce the mechanical properties over time. Plate 4.2 shows that samples with magnesium metal exhibit moisture at the break points, due to the hygroscopic nature of magnesium. Upon magnification under a microscope, the fracture patterns of the dog bone samples reveal that those containing magnesium break in a nice and uniform manner, whereas samples without magnesium exhibit irregular fracture patterns.

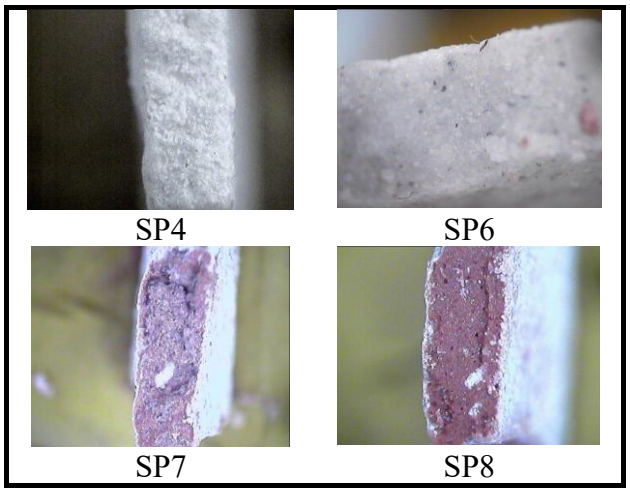


Plate 4.2 Crack Identification (Magnification: 40x –100x)

4.3.1.2 Hardness Test

The analysis Figure 4.9 revealed that SP 8 had the highest average Shore hardness value of 90.33, demonstrating the greatest resistance to indentation due to the combined addition of 5% magnesium and 1% ferric oxide. However, the AP/sorbitol-

based propellant remains brittle in nature. SP 7 and SP 6, which included 1% ferric oxide and 5% magnesium respectively, also showed significant increases in hardness, with SP 7 having a value of 86.67 and SP 6 having a value of 65.00. In contrast, the baseline sample SP 4, which contained no metal additives, had the lowest hardness value of 54.67. These results indicate that metal additives**,** particularly the combination of magnesium and ferric oxide, significantly enhance the hardness of the base formulation composed of AP and sorbitol, making them beneficial for applications requiring high hardness.

The findings clearly indicate that the addition of metal additives, such as magnesium and ferric oxide, significantly enhances the hardness of the base formulation composed of AP and sorbitol. The combination of magnesium and ferric oxide, as evidenced in SP 8, provides the greatest improvement in hardness, highlighting the potential for optimizing the material's formulation for applications that require high hardness.

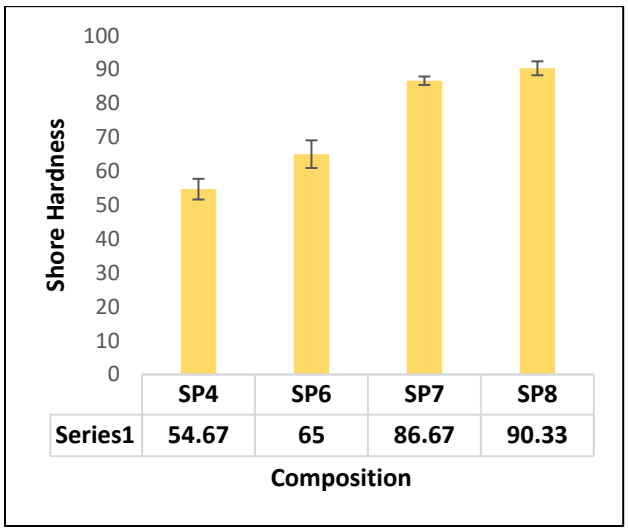


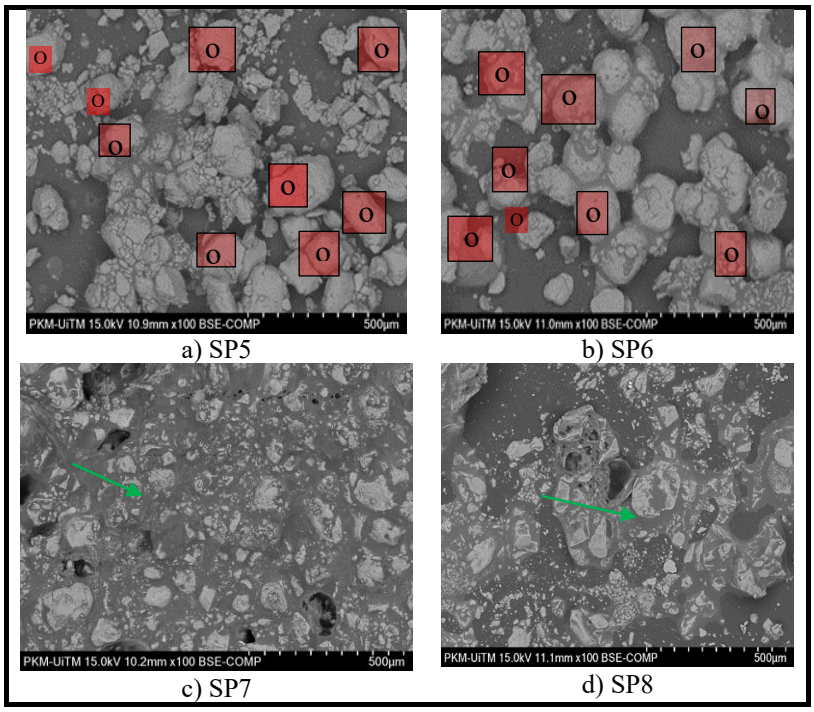
Figure 4.9 Shore A Hardnes With Different Composition Materials In Solid Propellant

Incorporating magnesium and ferric oxide into solid propellant compositions considerably enhances mechanical characteristics and toughness. These additives reduce moisture retention by scavenging absorbed water and promote a denser, more rigid microstructure within the AP/sorbitol matrix. The combined effect of reduced hygroscopicity and enhanced particle–matrix interaction increases surface stiffness, resulting in higher measured hardness values. This confirms that the elevated Shore A hardness reflects additive-induced reinforcement, rather than soft polymer behaviour. Hence by adding 5% magnesium improved these qualities, while adding 1% ferric oxide improved ductility and energy absorption even further. The combination of both

additives in SP 8 produced the highest results. The hygroscopic nature of the components influenced mechanical qualities over time, with moisture found around break points.

4.3.2 Scanning Electron Microscope &EDX

Morphology testing was also performed to investigate powder dispersion and propellant characteristics. The dispersion of particles was evaluated qualitatively based on SEM morphological observations at 100× magnification. No particle size distribution or quantitative image analysis was performed; therefore, the term “good dispersion” in this study refers solely to the absence of large agglomerates and macroscopic phase separation within the observed region, rather than to a quantitatively defined uniform particle distribution. SP4 was excluded from the characterization methods in this section, as its constituent materials are identical to those used in SP5, with the only difference being the proportion of AP and sorbitol. Since the variation lies solely in the weight ratio and does not introduce any new chemical components or additives, SP4 was not included in the detailed characterization procedures in this section. Cross-sectional were obtained for all propellant formulations to examine their internal morphology. As illustrated in Figure 4.10, the spherical AP particles, marked by red circles, are well dispersed within the propellant matrix. Distinct morphological differences were observed among the samples, which can be attributed to variations in their respective compositions. Notably, SP7 and SP8 contain ferric oxide as a catalytic additive. In addition to its catalytic function, ferric oxide appears to contribute to improved grain bonding, thereby slightly enhancing the physical strength of the propellant this proved in section 4.3.1.1. The presence of ferric oxide also facilitates the recrystallization of sorbitol, promoting better cohesion between components. The distribution of iron, originating from ferric oxide, confirms the effective incorporation and dispersion of the catalyst within the mixture. Green arrows in the micrographs highlight the uniform dispersion of melted sorbitol in SP7 and SP8, which is believed to play a role in reinforcing the structural integrity of the propellant grain as discussed in section 4.3.1.



a) SP5 b) SP6

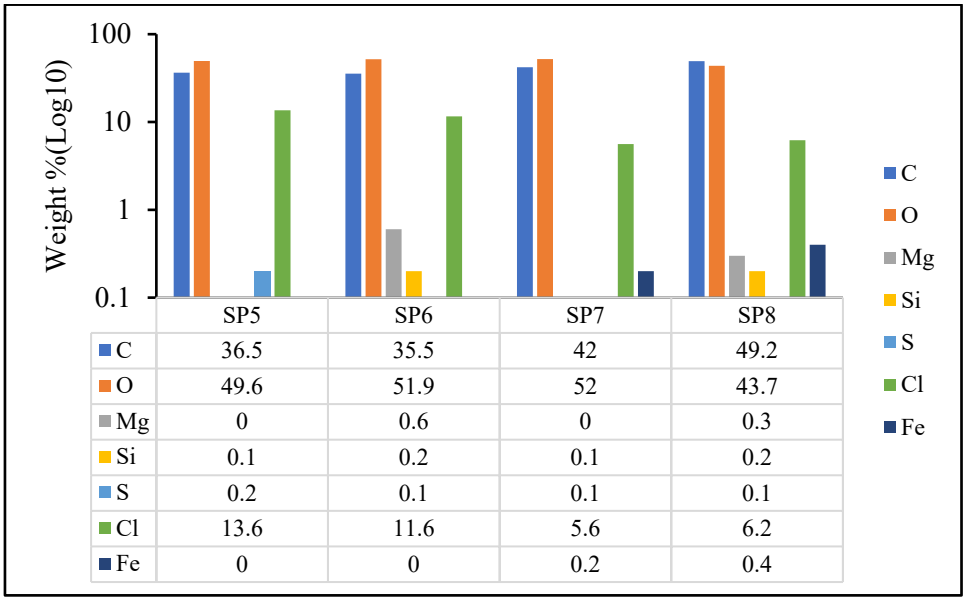
c) SP7 d) SP8

Figure 4.10 Solid Propellants Cross-Section Morphology

The morphology illustrated in Figure 4.10 shows good interaction and dispersion between fuel, oxidizer, and metal additive. In comparison to image (a), where ferric oxide was absent, the melted sorbitol exhibited poor physical interaction with the oxidizer particles, resulting in a less cohesive microstructure. In contrast, image (b) illustrates the formulation with added magnesium metal, where improved interaction between the oxidizer and melted sorbitol was observed. Although the sorbitol does not demonstrate perfectly uniform distribution, it shows a higher degree of contact and adhesion to the oxidizer particles. This suggests that the inclusion of metallic additives significantly enhances the interfacial bonding between sorbitol and the oxidizer, thereby improving the structural homogeneity and potential performance of the solid propellant.

Energy dispersive X-ray (EDX) is used to identify the chemical elements, estimation purity and the position of each material content in solid propellant as shown in Figure 4.11 [274]. The elemental analysis data indicate that the predominant elements in the solid propellant formulations are carbon, oxygen, and chlorine, which are primarily derived from the fuel (sorbitol) and the oxidizer (ammonium perchlorate). Magnesium is detected in samples SP6 and SP8, corresponding to its intentional inclusion as a metal additive, while iron is present in SP7 and SP8, confirming the incorporation of ferric oxide as a catalyst. Trace amounts of silicon were found across most samples, which are attributed to contamination from the glass sample holders used

during preparation. Additionally, the presence of sulphur is likely due to sample contamination during the preparation process.



	SP5	SP6	SP7	SP8
C	36.5	35.5	42	49.2
O	49.6	51.9	52	43.7
Mg	0	0.6	0	0.3
Si	0.1	0.2	0.1	0.2
S	0.2	0.1	0.1	0.1
Cl	13.6	11.6	5.6	6.2
Fe	0	0	0.2	0.4

Figure 4.11 EDX Analysis on Solid Propellant at Different Ratio of Additives

A comparison between samples SP4 and SP5 reveals a slight increase in oxygen content in SP5, supporting the observation that oxygen balance increases with a higher proportion of ammonium perchlorate. It proves that oxygen balance increases with the amount of ammonium perchlorate as depicted in Figure 4.11. Alternatively, the ammonium perchlorate-to-sorbitol ratio was held constant for samples SP6 to SP8, maintaining a fixed baseline of carbon and oxygen content. This allows for a clearer evaluation of the effects introduced solely by the addition of metallic additives. The results distinctly demonstrate that magnesium and ferric oxide play crucial roles in influencing elemental interactions within the solid propellant system. Notably, a reduction in chlorine content was observed in samples containing these metal additives. This suggests that magnesium acts as a chlorine scavenger, likely forming stable magnesium chloride, while ferric oxide may contribute to the neutralization or binding of chlorine species during combustion. These findings reinforce the catalytic and reactive functions of metal additives in modifying the chemical behaviour and potentially reducing corrosive by-products in the propellant formulation.

4.3.3 X-RAY Diffraction (XRD)

The powder of solid propellant from four samples was analyzed for the crystal structure of Ammonium perchlorate, Sorbitol, ferrite oxide, and magnesium metal, as illustrated in Figure 4.12. As shown in Figure 4.12, for the AP component in all samples, the primary diffraction peaks are observed at 15.27°, 19.37°, 22.64°, 23.89°, 24.61°, 27.41°, 30.9°, and 40.89°, corresponding to the planes of (101), (011), (201), (002), (210), (211), (112), and (401) for orthorhombic AP crystals [43], respectively, this suggests the formation of AP crystals exhibiting a high level of crystallinity.

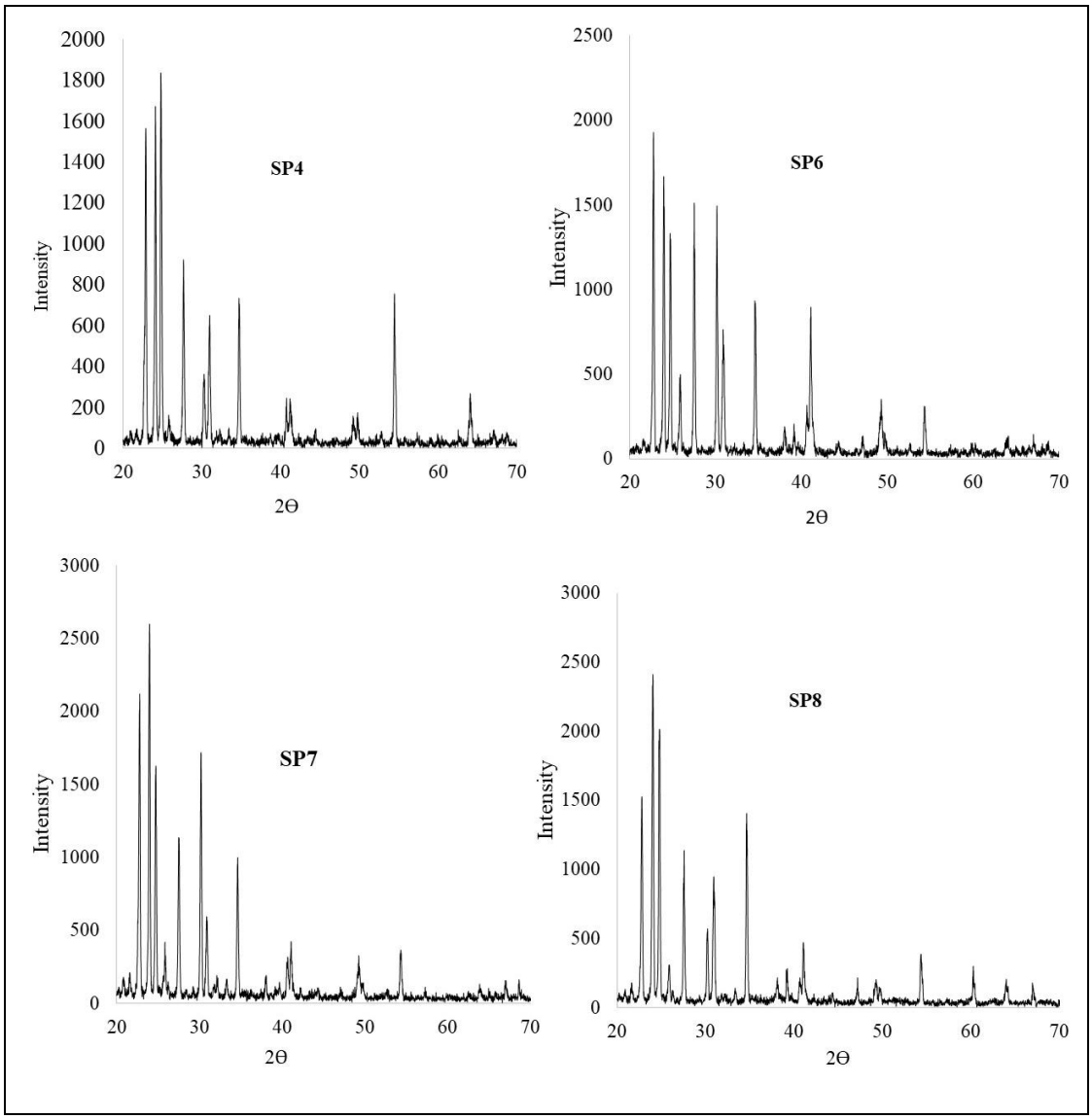


Figure 4.12 XRD Pattern Of Ammonium Perchlorate/ Sorbitol Solid Propellant

The X-ray diffraction (XRD) patterns of four solid propellant formulations SP4, SP6, SP7, and SP8 are presented across the 2θ range of 20° to 70°. Result for SP7 and

SP8 from Figure 4.12 show which contains ferrous oxide have many high peaks at angle 2Θ = 33.28°, 35.74°, 54.23° and 62.74°, which can be indexed to the (104), (110), (116) and (214) planes of Ferric oxide in a hexagonal phase, correspondingly. For SP6 and SP8 which contains magnesium metal shows a broad peak with high intensities of magnesium crystallites with a maximum peak from (024) and (221) crystal plane at 2 Θ of 38.29° and 39.05°. The diffraction features observed in the 2θ region around 38–39° are consistent with the presence of metallic magnesium. However, these reflections overlap with the dominant ammonium perchlorate peaks. SP4 and SP6 show several sharp peaks, indicating high crystallinity, while SP7 and SP8 exhibit even more intense peaks, especially between 20° and 35°, suggesting enhanced phase alignment or possibly greater crystallinity. Peak intensity in SP8 is slightly lower than in SP7 but still higher compared to SP4 and SP6. The main diffraction peaks for all samples occur in similar 2θ regions, signifying that the base crystalline phases are preserved across all formulations.

The addition of magnesium in SP6 and ferric oxide in SP7 and SP8 appears to have influenced the crystalline structure and diffraction intensity. SP4, the baseline without metal additives, shows lower peak intensities than SP6, indicating that magnesium may contribute to partial disruption or modification of the crystal lattice. SP7, which contains ferric oxide as a catalytic additive, exhibits the highest peak intensity overall, suggesting that ferric oxide not only integrates well but may also stabilize or promote crystallinity in the system. The slight decrease in SP8's intensity relative to SP7 may be due to the combined presence of both Mg and ferric oxide, which together introduce structural complexity or mild amorphization.

These results suggest that metal additives particularly ferric oxide influence the microstructural ordering of the solid propellant. The increased crystallinity in SP7 supports the hypothesis that ferric oxide acts not only as a burn rate catalyst but also affects the internal phase structure of the propellant supported from result section 4.3.1. The elevated peak intensity reflects improved phase organization or densification, which could correlate with better mechanical properties and combustion uniformity. On the other hand, SP6 shows a moderate reduction in peak sharpness and intensity, implying that magnesium's interaction with AP may create microstructural defects or a semi-amorphous state, which can enhance thermal reactivity. SP8, which combines both additives, displays a balance between enhanced crystallinity from ferric oxide and

slight lattice disruption from magnesium, suggesting a synergistic effect in the crystalline phase behaviour.

Previous studies [275] have shown that metal additives like Mg can disrupt crystal structures by integrating into the lattice or reacting with oxidizer particles, resulting in lower peak intensities and improved decomposition kinetics. In contrast, ferric oxide is known to promote phase stability and act as a structural binder at the microscopic level [276]. The current XRD results align with these findings showing reduced crystallinity in magnesium rich SP6 and enhanced peak intensity in ferric oxide containing SP7. The combined formulation in SP8 demonstrates an intermediate behaviour, supporting the idea that the interplay between oxidizer, fuel, and metallic additives significantly affects the microstructure, which in turn influences thermal and mechanical performance.

4.3.4 CHNS-O Element Analysis

In this study, CHNS-O analysis refers to a standard elemental characterization technique used to quantify the mass percentages of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) present in the solid propellant samples.

Table 4.6 presents the results of the CHNS-O analysis, indicating that the addition of additive metals significantly reduces the C/H ratio for SP6, SP7 and SP8. In contrast, the binder should possess a high C/H ratio in its backbone to ensure that the released exhaust gas has a lower average molecular weight and does not include heavy gases such as sulphur oxide [66]. In this study, sorbitol serves as a binder, fuel, and medium for dispersing the oxidizer with additive metals. As more metal is added, the C/H ratio significantly decreases compared to AP/Sorbitol alone, as indicated in sample SP8, which contains both magnesium metal and ferrite oxide as catalysts. This indicate that metal additives work with fuel binder because they lowered the C/H ratio hence reducing the formation of carbon monoxide gas formation while increasing the water vapor in rocket exhaust. Increased water vapor in the exhaust contributes to higher plume density, condensation, and secondary aerosol formation, which can increase exhaust visibility and infrared detectability. Therefore, although metal additives improve chemical efficiency, their influence on water vapour generation must be carefully balanced to avoid degradation of low-signature characteristics.

The CHNS analysis results show the elemental weight percentages of carbon (C), nitrogen (N), hydrogen (H), and sulphur (S), along with the carbon-to-hydrogen (C/H) ratio for five different propellant formulations (SP4–SP8). Although sulfur is not an intentional component of the AP/sorbitol-based formulations, trace amounts of sulfur were detected in all samples during CHNS-O analysis. These sulfur signals are attributed to unavoidable external contamination introduced during sample preparation and handling, such as contact with laboratory utensils, heating surfaces, or environmental exposure. Given that the detected sulfur content was consistently very low and did not vary significantly between samples, it was treated as background contamination rather than a formulation constituent. Consequently, sulfur values were excluded from formulation interpretation and combustion analysis, as they do not influence the comparative trends or conclusions of this study.

Table 4.6
Result of CHNS-O Analysis

Sample	C %	N%	H%	S%	C/H Ratio
SP4	15.87	7.73	9.98	2.59	1.59
SP5	11.88	8.12	10.13	2.57	1.17
SP6	7.52	6.96	9.30	2.55	0.80
SP7	8.29	7.59	10.78	2.46	0.77
SP8	13.93	11.14	21.92	2.52	0.63

SP4 contains the highest carbon content at 15.87%, while SP6 and SP7 exhibit the lowest, at 7.52% and 8.29%, respectively. The hydrogen content remains relatively consistent across most samples, ranging from 9.30% to 10.78%, except for SP8, which shows a substantial increase to 21.92%. The higher hydrogen content observed for SP8 is attributed to moisture absorption associated with the hygroscopic nature of sorbitol, magnesium and AP, leading to overestimation during CHNS-O analysis rather than a true increase in chemically bound hydrogen. SP8 also displays the highest nitrogen content at 11.14%. The Sulphur content, likely due to contamination, remains constant at 2.5% in all samples. The C/H ratio decreases progressively from SP4 (1.59) to SP8

(0.63), indicating increased hydrogen presence or reduced carbon content in later formulations.

The observed decrease in carbon content from SP4 to SP6–SP7 corresponds to the addition of metallic components (magnesium in SP6, ferric oxide in SP7 and SP8), which naturally dilute the organic fraction of the formulation. In contrast, the increase in nitrogen in SP8 suggests a higher proportion of AP, as AP is the main nitrogen source. The remarkably high hydrogen content in SP8 (21.92%) may indicate incomplete combustion, trapped moisture. The C/H ratio, often used to assess combustion potential and stoichiometric efficiency, shows a clear downward trend, suggesting a more hydrogen-rich and potentially more reactive formulation in SP8, enhancing flame temperature but risking instability.

The elemental composition confirms the influence of metal additives on the fuel-oxidizer balance. SP4 and SP5, which lack additives, maintain higher carbon levels due to their larger proportion of organic fuel (sorbitol). In contrast, SP6 and SP7 show reduced carbon and C/H ratios, indicating a shift in combustion dynamics, where magnesium and ferric oxide alter the oxidation pathway and thermal behaviour. The unusually high hydrogen content observed in SP8 may be attributed to the synergistic interaction between magnesium and ferric oxide, which could enhance moisture retention within the formulation or alter the thermal decomposition pathway, leading to the release of additional hydrogen-containing species, the increased nitrogen in SP8 aligns with its higher AP content, reinforcing earlier findings on oxygen balance and combustion efficiency. These changes imply that the presence of metal additives not only affects phase and morphology as shown in XRD but also alters the elemental distribution and energy release characteristics of the propellant.

The influence of metallic additives on elemental composition is well-supported by literature. For instance,[277] reported that the inclusion of metal oxides in composite propellants leads to a reduction in carbon content and C/H ratio, which correlates with higher burning efficiency and improved thermal conductivity. Similarly, research by [275] found that magnesium inclusion shifts the combustion equilibrium toward faster and cleaner burning, as excess hydrogen and oxygen become more readily available. These findings are consistent with the trends observed in SP6–SP8, where the decrease in carbon and C/H ratio suggests a leaner, more oxidizer-dominant formulation, potentially improving burn rate and reducing soot formation.

4.3.5 Fourier Transform Infrared (FTIR)

The comparative FTIR spectra of AP/Sorbitol (SP4), AP/Sorbitol/Mg (SP6), and AP/Sorbitol/Mg with ferric oxide (SP7 and SP8) are presented in Figure 4.13. These spectra provide insight into the vibrational characteristics of the functional groups present in the propellant formulations, particularly those associated with carbohydrates, ammonium ions, and combustion by-products such as hydrogen chloride (HCl). All samples display broad absorption bands around 3200 cm^{-1} and 1400 cm^{-1}, attributed to the stretching vibrations of ammonium ions, a key component of AP.

In addition, a less intense band appears near 2800 cm^{-1} across all samples, which is indicative of HCl gas vibration, suggesting that AP has undergone decomposition into its constituent gases. This observation aligns with previous reports confirming that AP decomposes to produce HCl, with characteristic vibrations detected in the 3100–2600 cm^{-1} range at temperatures around 450 °C [60]. A broad absorption band between 1000 and 1074 cm^{-1} is present in all samples and is associated with carbohydrate functional group vibrations, while a notable band around 600 cm^{-1} corresponds to vibrations of other organic components in the matrix.

Although metal additives were introduced in SP6 (magnesium), SP7 (ferric oxide), and SP8 (both magnesium and ferric oxide), all spectra maintain a similar overall pattern. This consistency is explained by the fact that infrared spectroscopy does not detect elemental metals, and for metal oxides, FTIR primarily provides information about the surface nature rather than the bulk structure of the metal compounds [278]. Nevertheless, their incorporation may indirectly influence the FTIR response of the propellant system through changes in peak position, intensity, or band broadening of existing functional groups. In this study, no new FTIR bands attributable directly to metal additives were detected.

Closer examination reveals important spectral changes attributable to the presence of metal additives. The –OH stretching band near 3300 cm^{-1}, attributed to hydroxyl groups in sorbitol, is most prominent and well-defined in SP4 and SP5, which do not contain metal additives. This band becomes broader and less intense in SP6, SP7, and SP8, indicating that magnesium and ferric oxide may interact with sorbitol, possibly disrupting hydrogen bonding and modifying the overall molecular environment.

The C–H stretching bands around 2900 cm^{-1} are consistently observed in all samples but show a reduction in intensity in SP6–SP8, which may be due to a decrease

in organic content and changes in molecular symmetry because of metal inclusion. Similarly, the nitrate and perchlorate-related absorptions in the regions of 1380–1450 cm^{-1} and 1050–1150 cm^{-1} are slightly shifted in SP6–SP8, suggesting possible coordination interactions between AP and the metal ions. These shifts further indicate that magnesium and ferric oxide may alter the distribution and orientation of AP within the matrix, influencing its chemical reactivity.

The observed spectral changes imply that metal additives modify the chemical environment of the propellant, potentially improving combustion characteristics. In SP6, magnesium may interfere with sorbitol–AP interactions, resulting in broader –OH signals. SP7 and SP8, which include ferric oxide, may experience enhanced complexation or catalytic interactions, altering nitrate and hydroxyl group vibrations. The combined effects observed in SP8 where both Mg and Fe_2O_3 are present—suggest a synergistic interaction that leads to broader and slightly shifted peaks, which may reflect increased molecular interaction, amorphization, or structural rearrangement within the propellant.

These findings are supported by prior research. For example, [279] reported that the addition of metal oxides to AP-based propellants caused noticeable shifts and peak broadening in FTIR spectra, particularly within nitrate and hydroxyl regions, due to strong functional group interactions. Likewise, [202] demonstrated that magnesium and iron oxides can form coordination complexes with both oxidizers and binders, leading to alterations in vibrational frequencies and improved uniformity in combustion. The present results are consistent with these studies, confirming that metal additives significantly influence the bonding structure and molecular dynamics of the propellant matrix, which will enhance thermal decomposition, combustion efficiency, and stability.

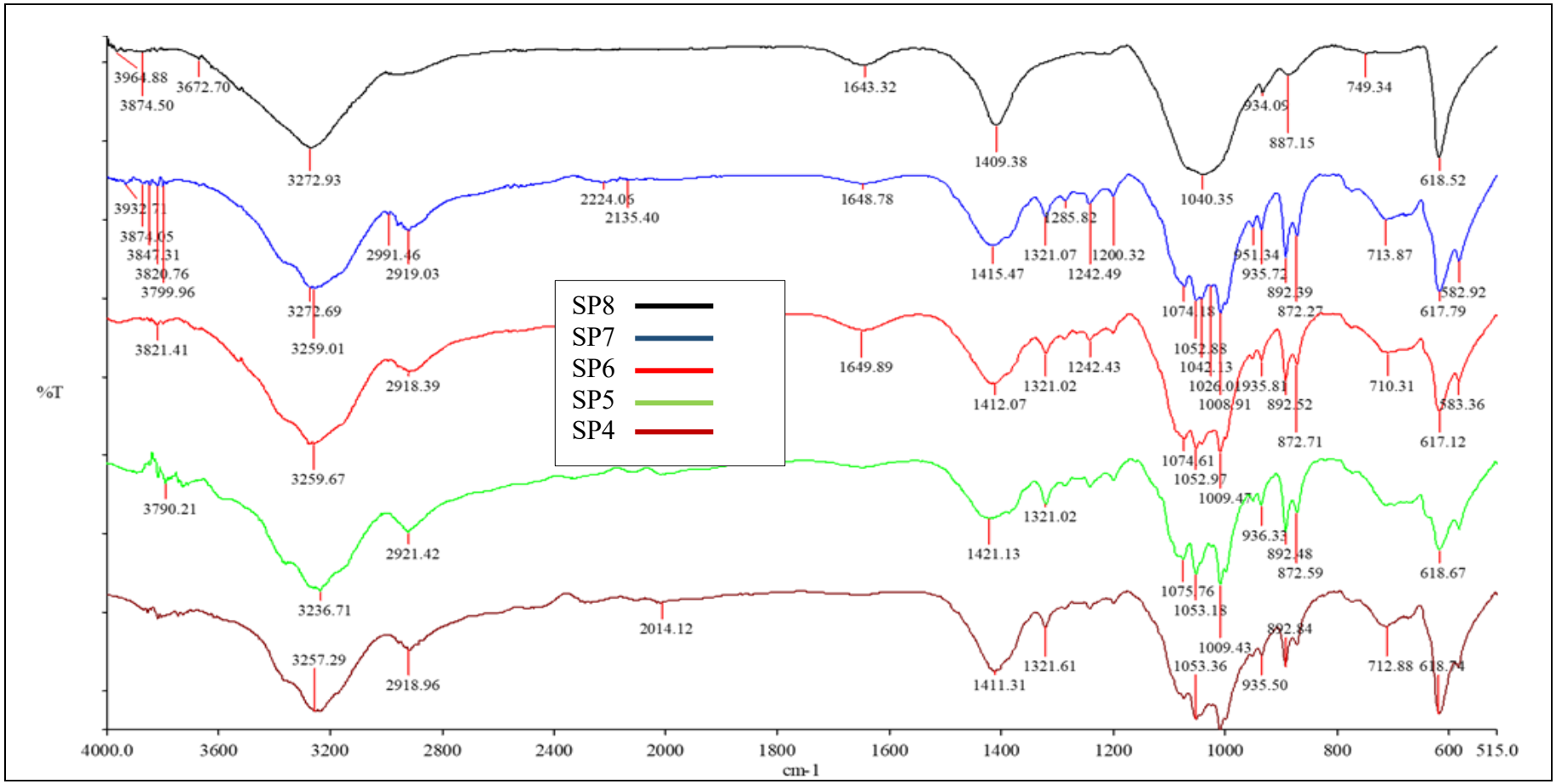


Figure 4.13 FTIR Spectra of Solid Propellants Of Ammonium Perchlorate/ Sorbitol /Magnesium Metal and With Catalyst

4.3.6 Differential Scanning Calorimeter (DSC) AND Thermal Gravimetric Analysis (TGA)

One of the most crucial characteristics of an energy material is thermal stability. The DSC analysis was utilised to investigate the thermal properties of the solid propellants. A decomposition temperature of >200 °C is typically required for energetic materials to be utilised in practical applications [176]. In this study, the thermal stability of the five samples Ammonium perchlorate/Sorbitol solid propellant with and without additives was evaluated using differential scanning calorimetry (DSC) and Thermogravimetric analysis. The result of the analysis at 5 °C/min for five samples illustrated in Figure 4.14, Figure 4.15 and Figure 4.16 .

The TGA of the AP/Sorbitol-based solid propellants (SP4–SP8) at a heating rate of 5 °C/min reveals significant insights into their thermal decomposition behaviour**.** As shown in Figure 4.14, all samples begin to degrade thermally beyond 200 °C, with major mass losses occurring between 250 °C and 400 °C. SP4 and SP5, which no metal additives, exhibit more gradual two-step decomposition with relatively higher residual mass, indicating greater thermal stability. In the comparative the TGA of the solid propellant samples SP4 to SP8 reveals distinct multi-step thermal degradation behaviour, with differences in total and stage-wise mass loss that correspond to the presence and type of metal additives in each formulation. SP4 and SP5, which do not contain any metal additives, exhibited moderate total mass losses of 7.3% and 7.6%, respectively. These decompositions occurred in three clear stages, with SP4 showing 1.0%, 2.5%, and 3.8% loss across the respective steps. This finding suggests that more sorbitol is decomposed, especially in SP4 because to its high sorbitol level. It implies that sorbitol has a synergistic impact on decreasing the thermal breakdown temperature of an ammonium perchlorate combination [280] [281]. As a result, the amount of oxidising agent and fuel in the formulation appears to have a significant influence on both the ignition temperature, thermal decomposition, and mass loss of solid propellant indicate that most of the decomposition occurred earlier, which aligns with its DSC exothermic response in Figure 4.15.

In contrast, SP6 which containing magnesium shows a steeper and earlier mass loss. Due the presence of magnesium in SP6 resulted in a higher overall mass loss of 8.4%, with a more pronounced drop during the second step 3.2%, indicating accelerated decomposition due to the catalytic activity of magnesium., while SP7 and especially

SP8 with ferric oxide and both metal additives respectively display accelerated degradation profiles, with SP8 experiencing the most rapid thermal breakdown suggesting that the combination of magnesium and ferric oxide significantly reduces thermal stability. Mass loss in SP7, which contains ferric oxide, followed a similar pattern to SP6 with slightly lower total mass loss 7.9%, suggesting that ferric oxide enhances decomposition but in a slightly more gradual manner. The combined use of magnesium and ferric oxide in SP8 produced the highest loss in the second stage 4.8%, contributing to a total mass loss of 8.4%. Interestingly, SP8 also showed a reduced mass loss in the final step 2.3%. These results collectively confirm that the inclusion of metal additives increases the reactivity of the formulation, shifts decomposition to lower temperatures, and alters the mass degradation pathway potentially improving combustion energyat the expense of reduced thermal stability TGA analysis of mass loss. These differences highlight that the inclusion of metal additives plays a catalytic role in enhancing decomposition.

The TGA of samples containing magnesium metal demonstrates that it initially improves the thermal stability of the propellant, shifting the first stage of decomposition to a higher temperature range. This is attributed to the ability of Mg to act as a chemical scavenger for initial decomposition gases, such as hydrogen chloride, and the formation of a protective magnesium hydroxide layer on the particle surface. However, once this threshold is exceeded, the reaction rate increases dramatically evidenced by the steep TGA slope representing a rapid condensed phase reaction between the metal fuel and the oxidizer. This behaviour correlates magnesium concentrates the energy release into a highly intense and rapid combustion event.

During the second decomposition stage, the presence of magnesium exhibits a clear catalytic effect on the thermal degradation behaviour of the propellant. As observed in the TGA curves, samples containing magnesium undergo a more rapid mass loss within the main decomposition region compared to formulations without magnesium. This accelerated decomposition indicates that magnesium promotes faster breakdown of the propellant. Notably, SP6 and SP8 do not exhibit a distinct third decomposition stage, suggesting that the catalytic action of magnesium facilitates a more complete and consolidated decomposition process within the second stage. Magnesium likely promotes faster degradation by facilitating heat transfer and acting as an energetic component, while ferric oxide accelerates the breakdown of ammonium perchlorate via its catalytic oxidation properties.

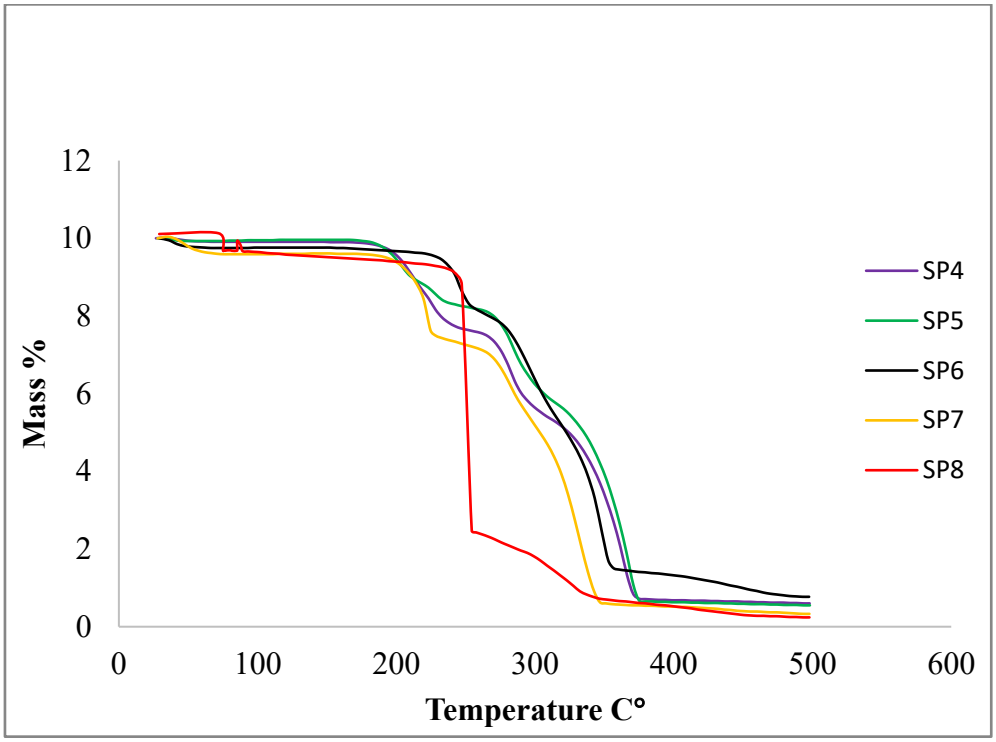


Figure 4.14 TGA Thermogram of Ammonium Perchlorate/Sorbitol with and Without Metal Additives at Heating Rate 5 C°/Min

The synergistic interaction in SP8 results in the lowest second decomposition onset and fastest mass loss, which, while indicating lower second thermal stability, may improve ignition and combustion efficiency. This is consistent with previous studies by Zhou et al. [282] and Tang et al. [283], which demonstrated that metal additives reduce the activation energy and enhance the thermal reactivity of AP-based propellants. Overall, the TGA data confirm that metallic additives, particularly in combination, significantly influence the thermal decomposition kinetics and must be carefully optimized to balance between performance and stability in solid propellant design.

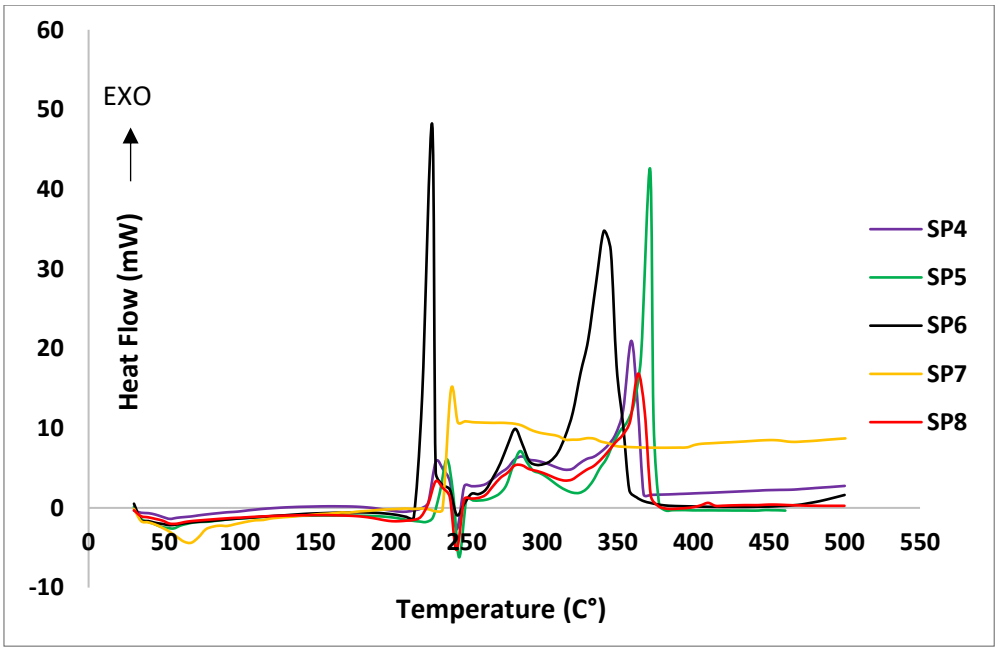


Figure 4.15 DSC Thermogram of Ammonium Perchlorate/Sorbitol with and Without Metal Additives at Heating Rate 5 C°/Min

The DSC thermograms of AP/Sorbitol-based solid propellants with and without metal additives (SP4–SP8), as shown in Figure 4.15, reveal significant differences in thermal behaviour and heat release profiles. Each formulation exhibits one or more exothermic peaks corresponding to decomposition stages, with notable variations in peak onset, intensity, and shape. The main exothermic peak for the AP/Sorbitol mixture is situated within the range of 280-371 C°, suggesting the exothermic decomposition of ammonium perchlorate. In contrast to the thermal breakdown of pure AP, the AP/Sorbitol combination exhibits a considerable reduction in the exothermic peaks at the first and second decomposition temperatures. Conversely, high temperature decomposition (HTD) takes place at a temperature that is far higher and represents the complete breakdown of AP. [284].

SP6 shows the most intense and sharp exothermic response, particularly around 240–290 °C and again at ~340 °C, indicating highly energetic decomposition by the presence of magnesium. In contrast, SP4 and SP5—formulations without metal additives display more stable and moderate thermal responses with major peaks occurring at higher temperatures (~280–370 °C), suggesting slower and more thermally resistant decomposition. SP7, which includes ferric oxide, demonstrates broader and lower-intensity exothermic activity, reflecting a more gradual decomposition process. SP8, incorporating both magnesium and ferric oxide, shows a combined behaviour earlier onset and moderately strong peaks pointing toward a synergistic catalytic effect

that promotes efficient but controlled combustion. These thermal trends imply that metal additives significantly modify the decomposition kinetics such as magnesium accelerates and intensifies the heat release, ferric oxide reduces the activation barrier, and their combination in SP8 balances reactivity with stability. This interpretation is supported by prior studies where metallic fuels such as magnesium are reported to enhance exothermicity through redox reactions, while ferric oxide acts as an AP decomposition catalyst [275], [285]. The DSC results confirm that additive selection and combination directly influence the ignition characteristics, energy output, and thermal safety of composite solid propellant systems.

Figure 4.16 illustrates the combination of TGA and DSC is essential for a comprehensive evaluation of the thermal behaviour of solid propellant formulations. By combining both techniques, it becomes possible to correlate mass loss events with specific thermal reactions, such as ignition, low-temperature decomposition (LTD), and high-temperature decomposition (HTD). This dual characterization not only identifies the decomposition stages but also quantifies the energy released, thereby offering a more accurate assessment of combustion reactivity, thermal stability, and potential hazards. The DSC–TGA thermograms of SP4–SP8 reveal distinct thermal decomposition stages, encompassing ignition onset, low-temperature decomposition (LTD), and high-temperature decomposition (HTD), with the inclusion of metal additives clearly influencing the thermal behaviour of the formulations as shown in Figure 4.16.

SP4 and SP5, which contain no additives, exhibit gradual decomposition with exothermic peaks at 210.38 °C, 280.57 °C, and 358.96 °C for SP4, and slightly higher temperatures of 223.39 °C, 285.40 °C, and 371.44 °C for SP5, suggesting improved thermal resistance due to increased AP content. In contrast, SP6–SP8, which incorporate magnesium and ferric oxide, demonstrate earlier and more aggressive thermal responses. SP6 begins decomposition at 243.59 °C with peaks at 282.21 °C and 340.83 °C, while SP7 shows the lowest LTD at 196.15 °C and an HTD at 334.40 °C, indicating that ferric oxide strongly enhances early-stage oxidizer breakdown. SP8, which combines both additives, ignites at 243.34 °C and peaks at 281.93 °C and 363.44 °C, showing a balance between catalytic acceleration and thermal control.

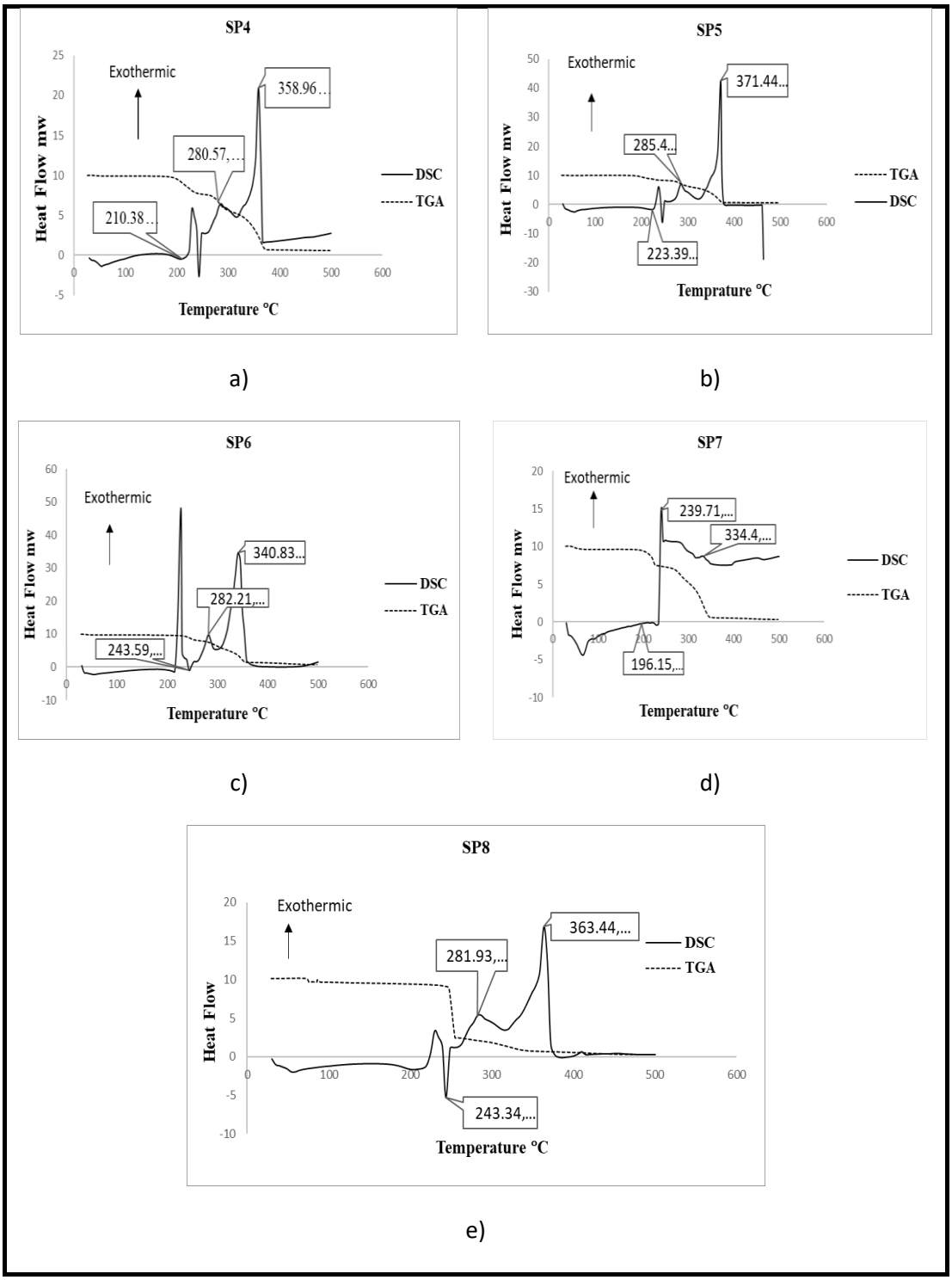


Figure 4.16 Integrating of DSC & TGA Thermogram of The Solid Propellant at Heating Rate 5 C°/Min.

The LTD and reduced HTDs in SP6–SP8, as confirmed by corresponding mass loss in TGA curves, underscore the catalytic effects of metal additives on decomposition kinetics. Magnesium enhances heat release and sorbitol oxidation, whereas ferric oxide promotes rapid AP degradation through redox activity. SP8 demonstrates a synergistic effect, improving combustion initiation while avoiding overly aggressive breakdown.

These results align with findings from Xu et al. [270] , who noted that metallic additives significantly reduce the activation energy of AP-based propellants and modify the thermal degradation pathway. While such modifications enhance ignition sensitivity and energy output, they also introduce thermal instability risks, highlighting the need for careful formulation balancing between performance and safety.

4.3.6.1 Ignition Temperature

To support the findings presented in Figure 4.14 and Figure 4.15, a summary of the thermal decomposition, ignition temperature and a comparison with pure base materials are provided in Table 4.7. The DSC trace for pure ammonium perchlorate (AP) shows that the initial endothermic decomposition begins at 242 °C [200], and completes around 452.8 °C, providing as a thermal decomposition benchmark for the formulated samples. Sorbitol, demonstrates thermal decomposition 200°C with maximum temperature at 350°C, with its melting point occurring at approximately 98.7 °C. In comparison, the formulated propellants from SP4 to SP8 exhibit significant variations in ignition temperature, LTD, and HTD, it influenced by both composition and the presence of metal additives. SP4 and SP5, composed solely of AP and sorbitol, display ignition temperatures near the AP baseline, with SP5 showing slightly higher thermal resistance due to its increased AP content. Notably, the DSC curves of these formulations reveal initial endothermic decomposition beginning as early as 210 °C, progressing to 218 °C as the AP content increases. This behaviour suggests a chemical interaction between sorbitol and AP, likely involving disruption of the AP ionic lattice, which facilitates energy transfer and enhances molecular mobility [286] . As a result, the decomposition of the mixture occurs rapidly after sorbitol's degradation, indicating a coordinated thermal response between oxidizer and fuel in the composite system.

The inclusion of metal additives in SP6, SP7, and SP8 causes noticeable changes. SP6 and SP8, both containing magnesium, have higher ignition points around 243 °C, indicating a delayed start to decomposition but more energetic combustion once triggered. SP7, which includes ferric oxide, has the lowest ignition temperature at 196 °C and the lowest LTD and HTD values, reflecting strong catalytic effects that promote early and rapid decomposition. SP8 shows a balance between the two effects, starting decomposition similarly to SP6 but achieving higher HTD than SP7, suggesting a synergistic influence of both magnesium and ferric oxide. These trends confirm that

metal additives can significantly shift thermal behaviour, helping to optimize the performance and stability of the solid propellant formulations.

Table 4.7
Summary DSC And TGA Result of Solid Propellants

Samples	**Transition temperature C°**		
	Ignition Temperature C°	**LTD C°**	**HTD C°**
AP [189] (base line)	242	297.8	452.8
Sorbitol [280] (baseline)	200-360	-	-
SP4	210.38	280.57	358.96
SP5	218.99	285.47	371.44
SP6	243.59	282.2	340.83
SP7	196.15	239.71	334.4
SP8	243.34	281.93	363.44

From Table 4.7, Sorbitol clearly reduces the peak temperature of AP's thermal decomposition compared to pure thermal decomposition AP, causing the AP High-HTD to be shifted to a lower temperature range for all samples. While the addition of ferrite as a catalyst to the propellant evidently shows a significant impact on its thermal decomposition. As observed in Figure 4.16 and Figure 4.14, SP7 exhibits the lowest ignition temperature and thermal decomposition temperature. A comparison between SP7 and SP4 reveals that the catalyst plays a crucial role in lowering the ignition temperature, thereby improving the burning rate and increase propellant sensitivity toward heat, whereas not utilizing an apparent influence on energy release as discussed in previous topic section in the heat of combustion. It is noticeable that not much energy is produced during this thermal breakdown since the catalyst promotes the right path of processes and reduces activation energy, which speeds up the reaction.

Given that the sample utilised in this study comprises 75% AP, it conforms to the practical application standard, that suggests the proportion of AP oxidizer by weight should be between 75% and 80%[189]. Hence the contribution of AP to thermal

breakdown becomes highly significant below 400°C, particularly at low heating rates. This means that sorbitol interacts compatibly with AP to improvise the ignition temperature of the mixture.

4.3.7 Kinetic Analysis

The TGA thermogram for the AP/sorbitol sample (SP4) was obtained at different heating rates shown in Figure 4.17 respectively. As the heating rate increases, the curve gradually shifts towards higher temperatures. This phenomenon will likely occur, given that the low heating rate creates a longer time interval needed to produce curves. DSC thermograph for AP/sorbitol/Mg (SP6) clearly depicts the phase change from orthorhombic to cubic occur at endothermic peak 243.49 C°. Three exothermic peaks appear as shown in Figure 4.17 and at a higher heating rate total mass decomposition happens at higher temperatures. The first exothermic peak is initial thermal decomposition happen at 234.41 C° mainly due to decomposition of sorbitol, second peak is Low thermal decomposition happen at 291.48 C° and third peak is high thermal decomposition occur at 358.96 C°, this could represent almost propellant has decomposed into gas and vaporized.

Refer to Table 4.8, the calculated value of activation energy for the AP/sorbitol is 128.151 KJ/mol and 132.608 KJ/mol by KAS and FWO. After increasing the amount of AP, the value of activation energy changes significantly. The result highlights that high amounts of AP will increase the decomposition rate greatly, hence the amount of AP in propellant formulation will substantially affect the thermal decomposition of solid propellant. It is apparent that both activation energy of SP4 and SP5 are lower than the reported for pure AP (223.5 kJ/mol) for the high temperature decomposition stage [191]. This is because sorbitol decomposes at lower temperatures and generates reactive intermediates that facilitate AP breakdown, resulting in a reduced apparent activation energy in the TGA measurements. It is suggested that sorbitol can alter the reaction pathways. Furthermore, differences in heating rate range, conversion interval selection, and the chosen kinetic model (Kissinger/FWO) can produce systematic variation in reported values. Therefore, the lower activation energies are attributed to formulation effects on sorbitol with AP decomposition and possible catalytic interactions between the AP/Sorbitol. This can also influence the pre-exponential factor,

it means that sorbitol has significant impact on kinetic decomposition process by lowering the activation energy.

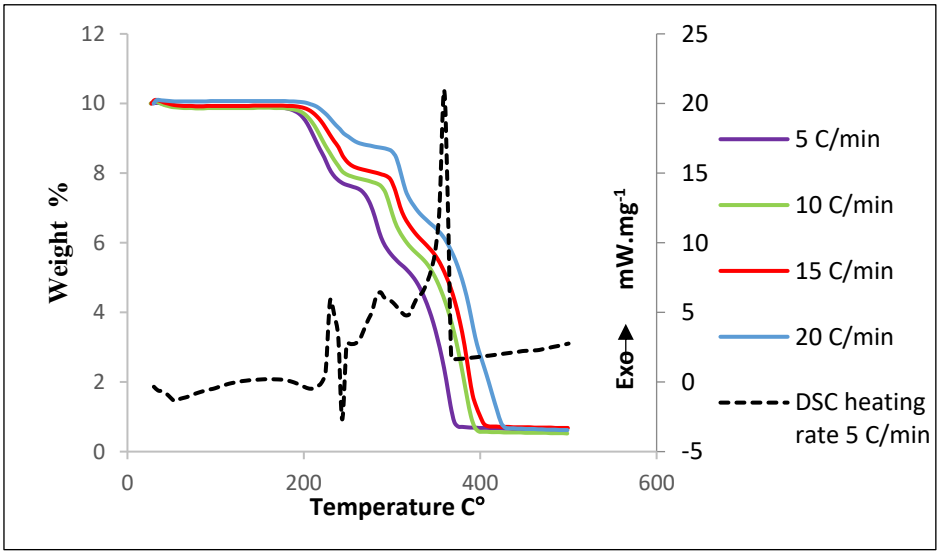


Figure 4.17 TGA Thermogram For AP/Sorbitol For Sample SP6 With Different Heating Rate And DSC Curves Of Thermal Decomposition Of AP/Sorbitol (SP6)

The inclusion of magnesium metal into formulation causes a considerable change in the values of activation energy and exponential factors compared to the AP/sorbitol propellant. The value of activation energy for SP6 is 156.6773 KJ/mol using KAS and 161.156 KJ/mol using FWO was highest compared to all formulation because it does not provide catalytic path but improves the combustion energy for the propellant. Mg metal interacts with the reactants in the propellant mixture, stabilizing the initial state or intermediates and thereby increasing the activation energy [287]. This stabilization necessitates a higher energy input to overcome. Additionally, Mg may alter the reaction pathway, introducing steps with higher energy barriers. During thermal decomposition, magnesium reacts with chloride to form magnesium chloride, which acts as a chlorine scavenger, enhancing the thermal stability of the AP/sorbitol mixture. Consequently, this increased stability requires higher temperatures and activation energy to initiate decomposition.

From the Table 4.8 shows the value of activation energy and pre-exponential factors decreasing with addition of ferric oxide for all formulation. The result points out that there is greatly catalytic proficiency in solid propellant especially for AP/sorbitol/catalyst which has the smallest activation and pre-exponential factors. Among all formulations, SP6, which incorporates magnesium, exhibited the highest activation energy (Ea), with values of 156.68 kJ/mol (KAS) and 161.16 kJ/mol (FWO),

indicating enhanced thermal stability and resistance to decomposition. SP8, containing both Mg and Fe_2O_3, also showed elevated Ea values, demonstrating a synergistic effect of the metal additives on the energy barrier. In contrast, SP5 formulated with a higher proportion of AP but without metal additives recorded the lowest Ea, suggesting easier ignition but reduced thermal control. SP4 (AP/sorbitol base) and SP7 (with only Fe_2O_3) fell within an intermediate range. The calculated pre-exponential factor (lnA) values were higher for SP6 and SP8, reflecting an increased reaction frequency and catalytic enhancement during decomposition.

This is further supported by the lower Ea/lnA ratios of SP8 and SP7, which suggest improved burn rates and catalytic response. Although natural logarithm pre-exponential factor and activation energy are two separate variables in theory but Gallagherh et al. [288] discovery of the kinetic compensating effect revealed a linear relationship between lnA and Ea. A lower Ea/lnA ratio indicates a more reactive system with a higher frequency of molecular collisions per unit energy barrier, typically associated with catalysed or enhanced decomposition pathways. For example, SP5, with the lowest Ea/lnA values (KAS 4.74), (FWO 3.89), demonstrates higher reactivity and faster decomposition, consistent with its high AP content and absence of metallic stabilizers. Conversely, SP4 and SP6 exhibit higher Ea/lnA ratios (KAS, 5.96 and 5.72 respectively), reflecting greater thermal resistance and a more controlled decomposition profile. Interestingly, SP8 which combines Mg and Fe_2O_3 balances both energy and reactivity, with moderate Ea/lnA values (5.68 KAS; 4.02 FWO), suggesting a synergistic kinetic behaviour.

These findings indicate that magnesium increases both the activation energy and the thermal reactivity of the propellant, while ferric oxide predominantly acts as a burn-rate modifier. Ferric oxide enhances the decomposition rate of AP and the combustion of sorbitol due to its large surface area and numerous reactive sites [289]. It absorbs gaseous reactants, facilitating exothermic decomposition and acting as a catalyst for both AP decomposition and fuel oxidation. As a semiconductor with hole conductivity, ferric oxide accelerates electron transfer during AP decomposition, providing alternative reaction pathways and reducing the energy barrier. This results in a higher frequency of successful collisions and a lower pre-exponential factor, ultimately improving the overall reaction efficiency. The consistency of results between the KAS and FWO models confirms the reliability of the thermal analysis as illustrated in Figure 4.18 and Figure 4.19. Overall, the integration of magnesium and Fe_2O_3, as in

SP8, offers an optimized balance between thermal stability and ignition responsiveness, making it a promising formulation for minimum signature solid propellant applications.

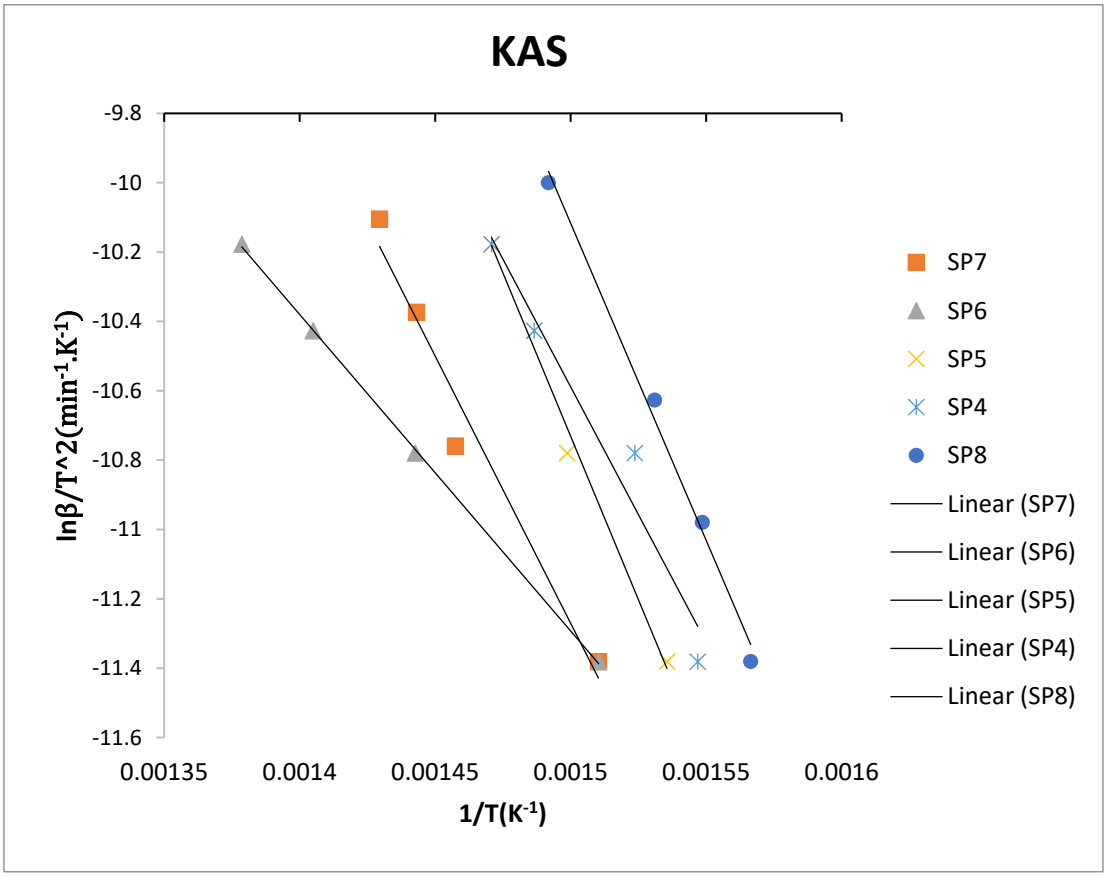


Figure 4.18 Kissinger Akahira Sunose (KAS) Linear Plots of Ln(B/T²) Versus 1/T For SP4–SP8 Solid Propellant Formulations

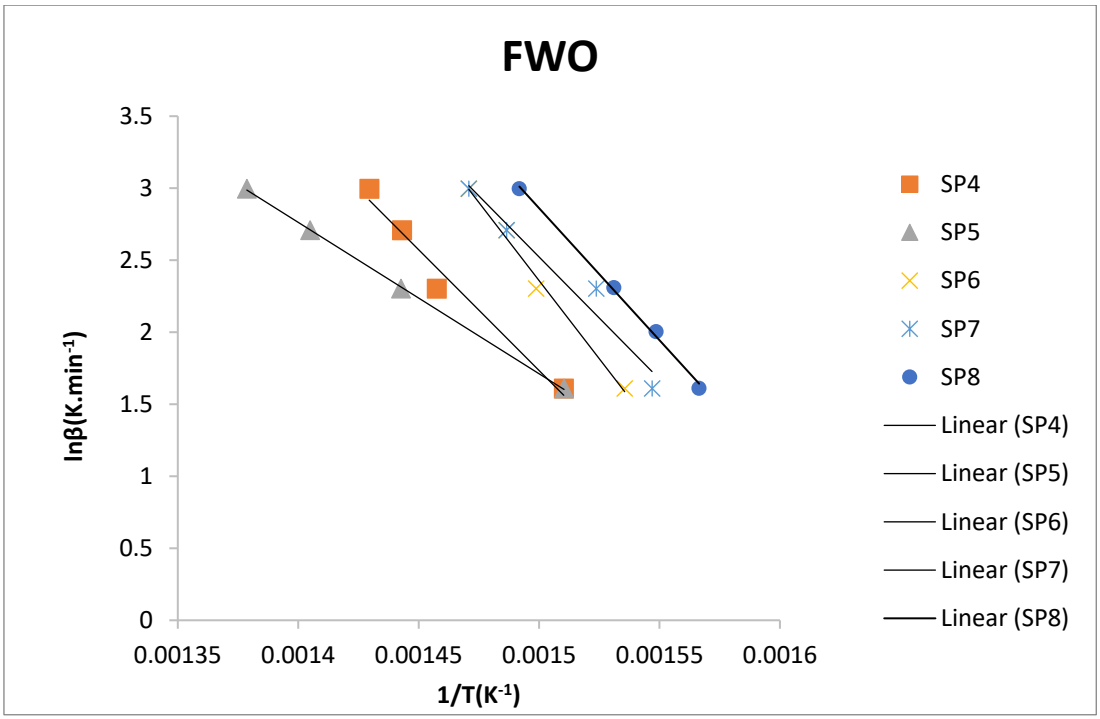


Figure 4.19 Flynn–Wall–Ozawa (FWO) Linear Plots of Ln B Versus 1/T For SP4–SP8 Solid Propellant Formulations

Table 4.8
Thermo-Kinetics Data of AP/Sorbitol And AP/Sorbitol/Mg With Catalyst Using KAS And FWO.

	KAS			FWO		
	Ea (KJ/mol)	**lnA (s^{-1})**	**Ea/lnA ($KJ/mols^{-1}$)**	**Ea (KJ/mol)**	**lnA (s^{-1})**	**Ea/lnA ($KJ/mols^{-1}$)**
SP4 (AP/SORB)	128.151	21.49	5.963	132.608	29.45	4.51
SP5 (AP/SORB)	75.89019	16.01	4.75	80.1004	20.54	3.89
SP6 (AP/SORB/MG)	156.677	27.38	5.722	161.156	37.28	4.32
SP7 (AP/SORB/FE)	122.473	21.10	5.803	124.26	29.87	4.16
SP8 (AP/SORB/FE/Mg)	125.6495	22.12	5.681	127.565	31.71	4.02

4.3.7.1 Mechanism

The interaction between sorbitol and ammonium perchlorate (AP) plays a key role in determining the ignition and combustion behavior of sugar-based solid propellants. Sorbitol serves as the fuel, while AP acts as the oxidizer, supplying the oxygen needed for combustion. When heated, both components undergo thermal decomposition in a sequence of steps that lead to gas generation and heat release. Understanding this step-by-step mechanism helps explain how ignition occurs and how the reaction sustains itself, which is important for designing reliable and efficient propellants

Step 1: Sorbitol Melting (~94°C)

Sorbitol melts, improving thermal contact with solid ammonium perchlorate (AP). No major decomposition occurs yet.

Step 2: Sorbitol Decomposition (200–250°C) in equation (4.2)

Sorbitol starts to dehydrate and decompose, producing volatile fuel gases and char.

$$C_6H_{14}O_6 \rightarrow CH_2O + CH_3OH + CO + CO_2 + H_2 + H_2O + C\ (char) \quad (4.2)$$

Step 3: AP Endothermic Decomposition (~242°C) shown in (4.3)

$$NH_4ClO_4 \rightarrow NH_3 + HClO_4 \quad (4.3)$$

AP begins endothermic decomposition, releasing ammonia and perchloric acid.

Step 4: Exothermic Interaction (~297.8°C) shown in (4.4) and (4.5)

$$HClO_4 + CO/H_2/CH_3OH \rightarrow CO_2 + H_2O + Cl_2\ Heat \quad (4.4)$$

$$NH_3 + O_2 \rightarrow NO + H_2O \quad (4.5)$$

Oxidation of fuel gases from sorbitol by AP's decomposition products releases significant heat. Ignition occurs here.

Step 5: Final AP Decomposition (~452.8°C) shown in (4.6)

$$2NH_4ClO_4 \rightarrow N_2 + Cl_2 + O_2 + 4H_2O \quad (4.6)$$

Complete decomposition of AP releases oxygen and sustains combustion of any remaining fuel.

Step 6: Combustion of Char and Residual Fuel shown in (4.7) and (4.8)

$$C + O_2 \rightarrow CO_2 \quad (4.7)$$

$$C + \tfrac{1}{2}O_2 \rightarrow CO \text{ (limited } O_2) \quad (4.8)$$

Remaining solid carbon from sorbitol combusts using oxygen from AP or atmosphere.

Overall Combustion Reaction (Theoretical) shown in (4.9)

$$C_6H_{14}O_6 + NH_4ClO_4 + O_2 \text{ (from AP)} \rightarrow CO_2 + H_2O + N_2 + Cl_2 + \text{Heat} \quad (4.9)$$

A simplified representation showing sorbitol as fuel and AP as oxidizer in a highly exothermic propellant reaction.

4.4 Testing and Measure of Solid Propellant Performance

Minimum Solid rocket propellant was tested to determine its performance such as burn rate and thrust. For section 4.4.1 only one solid propellant which is Ap/Sorbitol/mg was tested as it aims propellant for this work same goes to section 4.4.2 only AP/sorbitol/mg was tested and compared with common KNSB solid propellant.

4.4.1 Thrust of Solid Propellant

The Table 4.9 above summarizes the maximum thrust, total impulse, and specific impulse for KNSB, AP/Sorbitol, and AP/Sorbitol/Mg propellants.

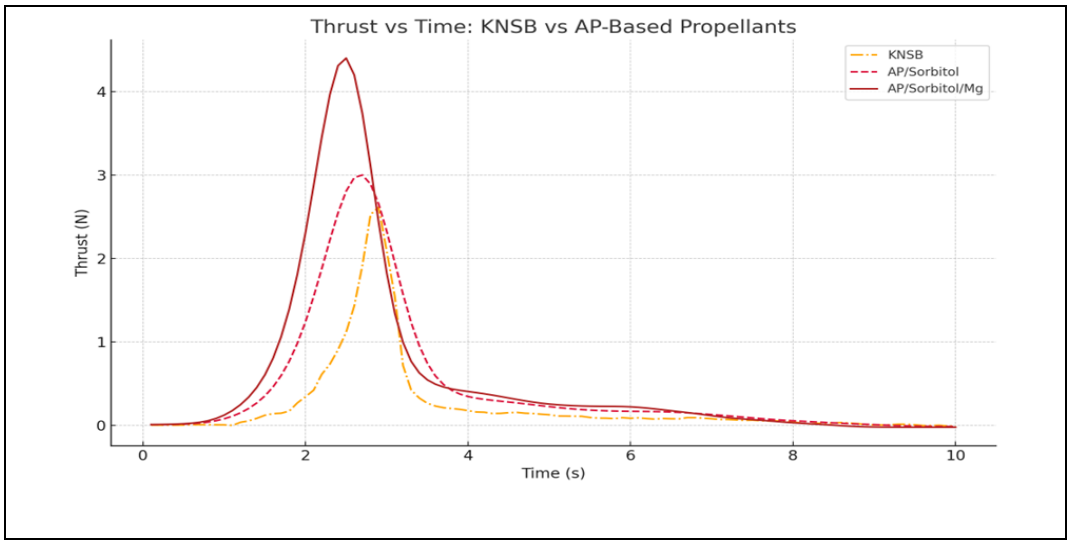


Figure 4.20 Thrust Profile for KNSB And AP Based Rocket Class D

Table 4.9
Summary Of Thrust And Impulse Characteristics For Solid Propellants

Propellant	Max Thrust (N)	Total Impulse (Ns)	Specific Impulse (s)
KNSB	2.61	2.42	12.33
AP/Sorbitol	3.00	4.61	23.52
AP/Sorbitol/Mg	4.40	5.81	29.60

From Figure 4.20 thrust clearly increases from KNSB to AP-based propellants. AP/Sorbitol/Mg demonstrates the highest peak thrust. Total impulse nearly doubles when switching from KNSB to AP/Sorbitol and further increases with the addition of Mg. Specific impulse, a key measure of efficiency, also significantly improves. The data confirms that AP-based propellants are significantly more energetic than KNSB. AP/Sorbitol strikes a balance between safety and performance, while AP/Sorbitol/Mg offers even greater performance but at the cost of higher thermal. These characteristics make AP-based formulations ideal for applications requiring high thrust-to-weight ratios. This analysis is consistent with known propellant chemistry and supports the use of AP-based mixtures in advanced amateur and academic rocketry.

The relatively low specific impulse obtained in this study compared with literature values can be attributed mainly to the low chamber pressure 120 psi and the laboratory scale motor class D. Most reported specific impulse data for AP-based propellants are measured under high chamber pressures with optimized nozzle expansion ratios, where exhaust flow approaches ideal isentropic conditions. In the present work, the low operating pressure, near-ambient exhaust conditions, and simplified nozzle design resulted in significant heat losses, reduced exhaust velocity, and non-ideal expansion. In addition, the formulations were developed with an emphasis on low-signature combustion rather than maximum energetic performance.

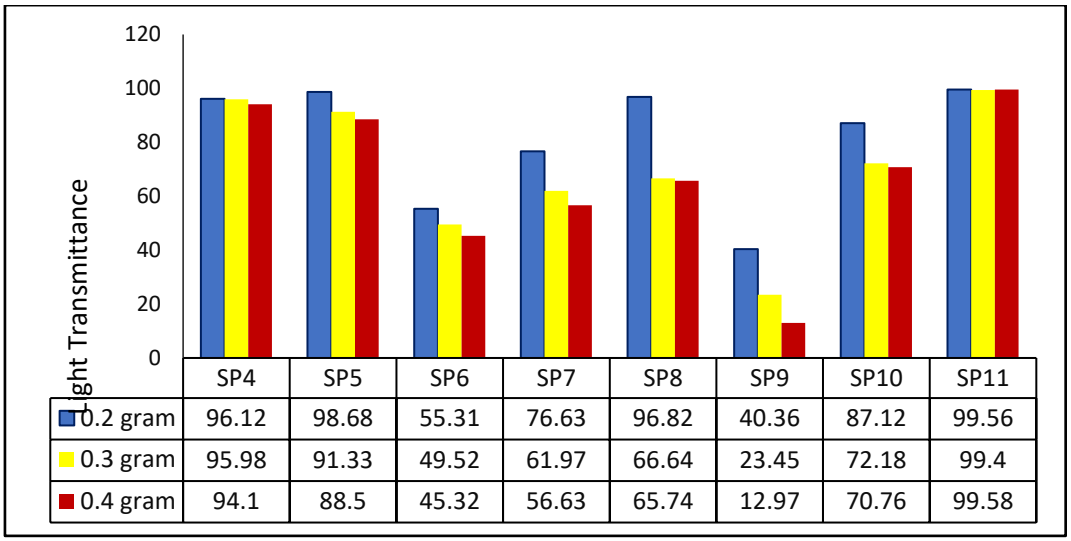
4.4.2 Smoke Opacity and Concentration of Solid Particle

Figure 4.21 above shows the light transmittance % of various solid propellant formulations from SP4 to SP11 at three different sample weights: 0.2 g, 0.3 g, and 0.4 g. SP4, SP5, and SP11 exhibit consistently high light transmittance values across all weights, exceeding 88%, with SP11 maintaining above 99% transmittance. In contrast, SP6, SP7, SP8, SP9, and SP10 demonstrate significantly lower transmittance, especially as weight increases. Notably, SP9 exhibits the lowest transmittance, dropping sharply

from 40.36% at 0.2 g to just 12.97% at 0.4 g. The data show a general trend of decreasing light transmittance with increasing sample weight, suggesting denser smoke production as propellant mass increases. SP4, SP5, and SP11 remain stable with only a slight drop, implying minimal smoke generation regardless of weight. Conversely, SP6, SP7, SP8, and SP9 display a significant reduction in transmittance as weight increases, indicating higher smoke opacity. SP8 shows better performance compared to SP6 and SP7, despite containing both magnesium and ferric oxide, which points to potential synergistic behaviour between the additives in reducing smoke formation.

These results suggest that propellants without metal additives (SP4, SP5, and SP11) promote more complete combustion with fewer solid combustion by-products, resulting in higher light transmittance. In contrast, the presence of metal additives in SP6 to SP10, particularly magnesium and ferric oxide, contributes to greater smoke formation due to the production of metal oxides and chlorides that scatter light. The case of SP8 is especially interesting despite containing both additives, it maintains higher transmittance than SP6 and SP7 individually, suggesting that the combined interaction may improve the combustion pathway or reduce particulate agglomeration. SP9, with the lowest readings, likely represents a poorly optimized formulation with inefficient combustion or excessive condensed-phase products.

These findings align with earlier research indicating that clean burning solid propellants particularly those free from metal fuels demonstrate superior light transmittance due to reduced particulate emission during combustion. In previous study [290] have shown that the inclusion of metal additives tends to increase primary smoke production due to incomplete vaporization and oxidation, contributing to lower light transmittance. Moreover, this trend supports the use of transmittance-based opacity measurement as a valid indicator of combustion energy and smoke signature of various propellant formulations. This result was used to determine smoke opacity to study detail about smoke particle from burning of propellant.



	SP4	SP5	SP6	SP7	SP8	SP9	SP10	SP11
0.2 gram	96.12	98.68	55.31	76.63	96.82	40.36	87.12	99.56
0.3 gram	95.98	91.33	49.52	61.97	66.64	23.45	72.18	99.4
0.4 gram	94.1	88.5	45.32	56.63	65.74	12.97	70.76	99.58

Figure 4.21 Light Transmittance Across Different Propellant Weights

4.4.2.1 Smoke Opacity

The Figure 4.22 presents the smoke opacity of various solid propellant formulations (SP4 to SP11) at three different sample weights: 0.2 g, 0.3 g, and 0.4 g. It is observed that SP4, SP5, and SP11 maintain very low smoke opacity across all weights, indicating clearer combustion. In contrast, SP6, SP7, SP8, SP9, and SP10 show significantly higher opacity values that increase with mass. SP9 recorded the highest opacity at 0.4 g, followed by SP6 and SP8.

An upward trend in smoke opacity is evident with increasing propellant weight, especially in formulations containing metal additives. SP4, SP5, and SP11 remain consistently below 20% opacity, regardless of weight, suggesting efficient combustion and minimal particulate formation. SP6 and SP8 show moderate to high opacity, which increases steadily with added weight. Notably, SP9 exhibits a sharp spike, especially at 0.4 g, indicating that its formulation results in a high concentration of smoke particles, potentially due to incomplete combustion or excessive condensed phase products.

The increase in smoke opacity with weight supports the hypothesis that higher fuel loading leads to greater particulate production and denser smoke, especially in propellants with metallic content. SP6 (with magnesium) and SP7 (with ferric oxide) produce moderate levels of smoke due to the formation of metal oxides and chlorides. This is Supported by previous studies [291]. SP8, which contains both Mg and Fe_2O_3, shows relatively lower opacity compared to SP6 and SP9, suggesting improved

combustion synergy. SP9 has high opacity indicates poor burn efficiency, possibly due to excessive metal additives or poor oxidizer-to-fuel balance. Meanwhile, SP11, which consists of non-metallic AP-based fuel, produces negligible smoke, aligning with the goal of minimum signature propellants.

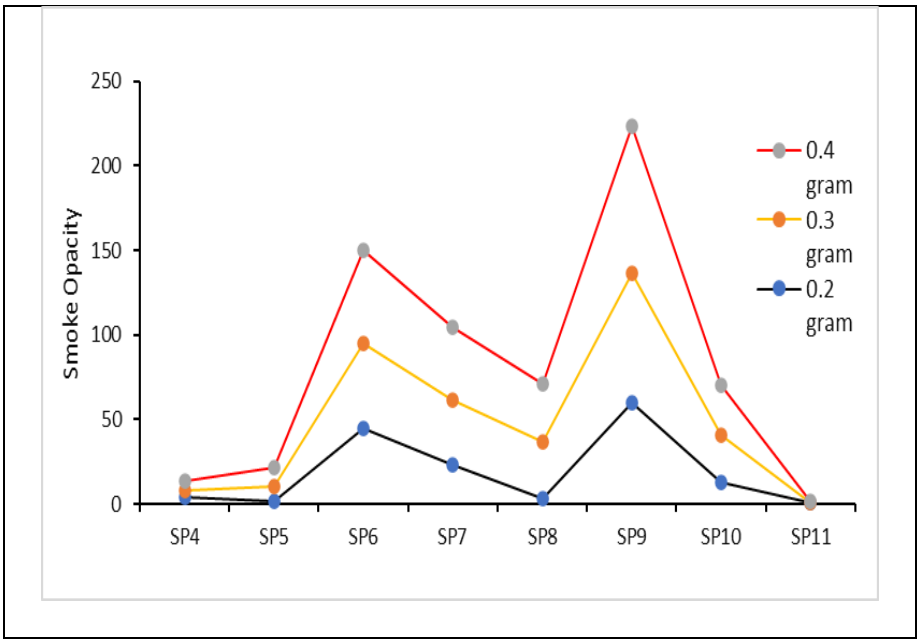


Figure 4.22 Smoke Opacity At Different Solid Propellant Weights

The optical signature of the exhaust is governed primarily by formulation type and secondarily by propellant mass. Increasing propellant mass from 0.2 to 0.4 g generally decreases light transmittance and increases smoke opacity, indicating that larger mass containing metal additive generates a higher concentration of light-scattering products in the plume. This mass effect is strongest for metal containing formulations (SP6–SP10), where smoke opacity rises sharply with mass, consistent with increased formation of condensed phase particulates (e.g., metal oxides and soot) that dominate light transmittance decrease. In contrast, SP4 and SP11 maintain consistently high light transmittance and near-zero smoke opacity across all masses, demonstrating that non-metalized formulations produce minimal condensed particles and remain relatively insensitive to mass changes in terms of visible signature. Overall, these results confirm that minimizing metal-derived condensed products is critical for achieving low-signature behaviour, and they support the use of light transmittance and smoke opacity as a practical screening metric to distinguish low-smoke formulations from highly smoky ones under controlled test conditions.

4.4.2.2 Smoke particle

SP9, SP10, and SP11 were excluded from the smoke particle residue collection test as they fell outside the primary scope of the study and were subject to experimental limitations. This decision was made to maintain methodological consistency and reduce experimental variability, ensuring that comparisons remained focused on the core objective evaluating the influence of metal additives within the AP/Sorbitol propellant system.

Figure 4.23 presents the mass of solid smoke particles collected from the combustion of various AP/sorbitol-based solid propellants. SP4 and SP5, which contain no metal additives, produced the lowest amounts of particulate residue at 0.036 g and 0.033 g, respectively. In contrast, SP6, SP7, and SP8 formulations incorporating magnesium, ferric oxide, or both exhibited higher levels of solid particulate emissions. SP6 yielded 0.043 g of residue, while SP7, containing only ferric oxide, generated the highest amount at 0.056 g. SP8, with both Mg and Fe_2O_3, produced 0.046 g, slightly less than SP7, suggesting a synergistic interaction that may enhance combustion energyand reduce unburned particulate emissions.

The trend indicates that metal additives increase the solid residue content due to incomplete combustion and the formation of metal oxides and chlorides, which remain as condensed phase products. However, the slightly reduced particle mass in SP8 compared to SP7 suggests that magnesium may react with chlorine-based combustion products, forming volatile compounds or reducing particle agglomeration. This aligns with findings from previous studies [292], where magnesium has been reported to improve combustion completeness and reduce smoke density when used with oxidizers like AP.

These results demonstrate a clear relationship between the type of metal additive used and the quantity of solid combustion residue formed. They also support the hypothesis that combining metal fuels and catalysts, such as in SP8, can potentially optimize combustion behaviour while minimizing particulate emissions. Overall, the particle mass data validate findings from light transmittance and opacity measurements, confirming the influence of formulation on smoke characteristics and combustion cleanliness.

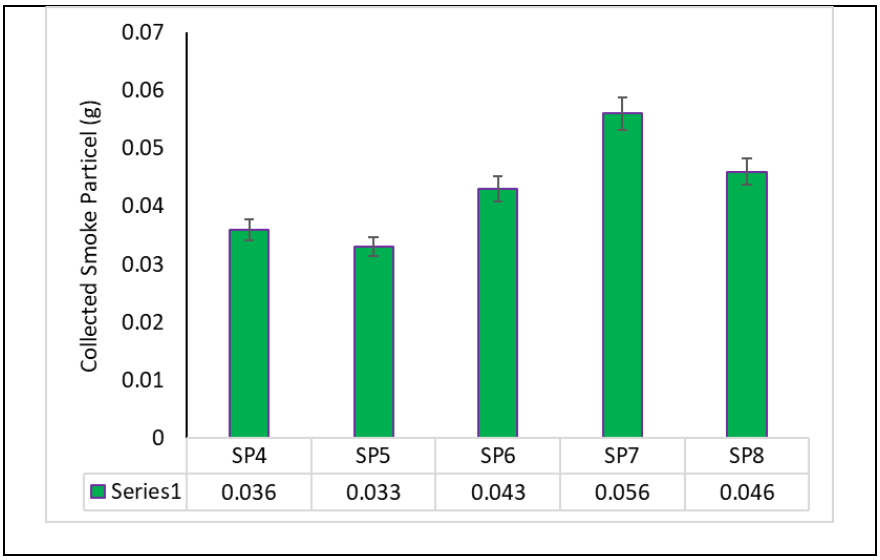


Figure 4.23 Mass of Solid Residue Collected from the Combustion of Solid Propellant Samples

4.4.2.3 Morphology and Elemental Composition of Combustion Residue

Solid Propellants SP7 and SP8 were selected for this analysis primarily because it left solid combustion product. The SEM image of SP7, presented in Figure 4.24, reveals a porous and rough surface morphology, indicative of a high-energy, catalysed combustion process accompanied by substantial gas evolution. The presence of ferric oxide round by red colour likely played a significant role in this structure, contributing to an increased burn rate and resulting in an open, sponge-like residue. This irregular morphology may also suggest a degree of incomplete combustion, with unreacted or residual iron oxides contributing to the rough surface texture.

In comparison, SP8, as shown in Figure 4.25, exhibits a similarly porous structure with numerous cavities and voids, signifying intense gas release during combustion consistent with the observations in SP7. However, the surface texture of SP8 appears comparatively smoother and less aggressively rough. This difference is likely attributed to the combined effect of magnesium (green colour) and ferric oxide (Fe_2O_3) within the formulation. Magnesium may facilitate a more energetic and uniform burn, which partially melts the residue surface, leading to smoother and less irregular features. This morphology is consistent with a system in which both energetic fuel (Mg) and a catalytic agent (Fe_2O_3) are present, producing a more intense yet controlled combustion. The appearance of rounded or globular features may represent re-solidified metal oxides, such as MgO, magnesium chloride and mixed oxides involving Ferric

oxide. The dual-metal additive system in SP8 appears to enhance combustion energy and modifies the post-combustion residue morphology, resulting in a cleaner and more uniform surface compared to SP7.

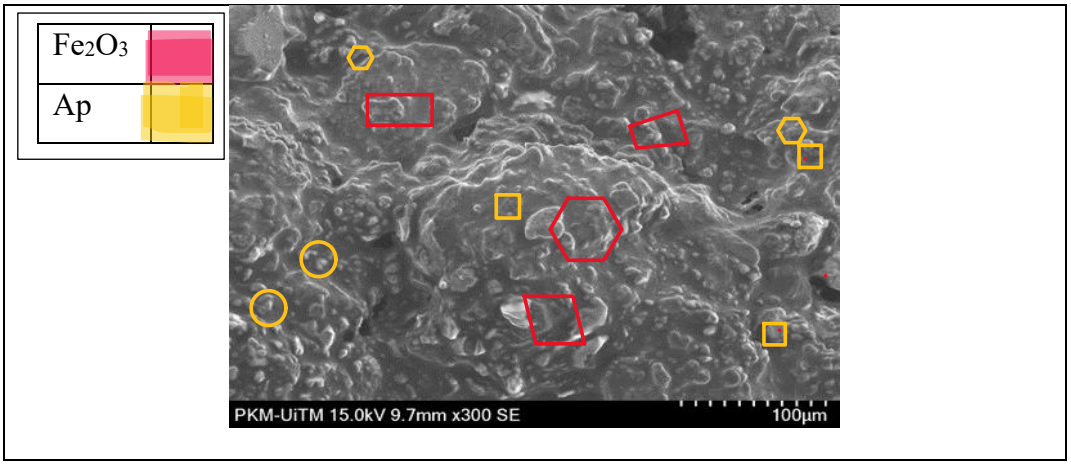


Figure 4.24 SEM Image For Solid Residual of SP7

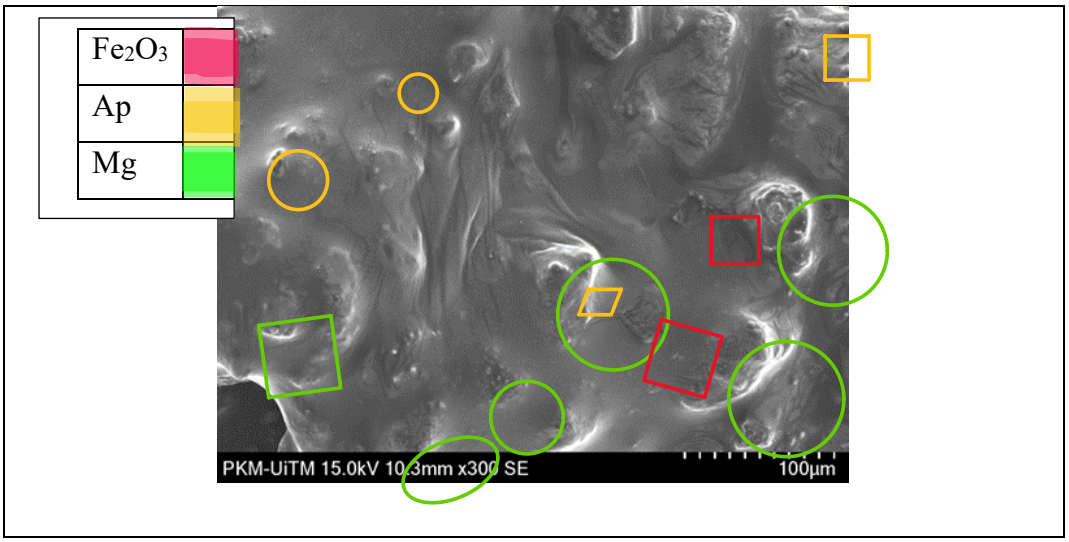


Figure 4.25 SEM Image For Solid Residual Of SP8

The EDS in Figure 4.26 depict the results for SP7 support the morphological observations obtained from SEM analysis. A significant presence of oxygen (45.07 wt%) and chlorine (45.78 wt%) was detected, primarily originating from the decomposition of ammonium perchlorate AP (yellow), the main oxidizer. A smaller proportion of iron (1.41 wt%) is attributed to the ferric oxide (Fe_2O_3) catalyst, while the presence of carbon (7.74 wt%) is likely derived from the sorbitol fuel component. The high oxygen content reflects an intense oxidation environment during combustion, while the elevated chlorine level indicates a strong contribution from AP

decomposition. Although the iron content is relatively low, its presence confirms the catalytic role of ferric oxide, which enhances the thermal decomposition of AP without being fully consumed in the reaction. The residual iron likely remains in the form of unreacted iron oxide byproducts. The observed carbon content suggests incomplete combustion or the presence of carbonaceous residues, consistent with the rough, porous texture seen in the SEM image of SP7.

In the case of SP8, the EDS analysis reveals a combustion residue rich in oxygen (57.10 wt%) and carbon (23.39 wt%), indicating that oxidation played a dominant role during combustion. Magnesium, detected at 2.67 wt%, reflects the inclusion of Mg as a metal additive. Its presence in the residue suggests that magnesium did not completely vaporize but instead contributed to the formation of condensed products, such as magnesium oxide (MgO) and magnesium chloride ($MgCl_2$). Acting as a chlorine scavenger, magnesium likely reacted with chlorine gas released during AP decomposition, which may explain the smoother globular features observed in the SEM image. Chlorine content was measured at 15.51 wt%, confirming AP's role in the oxidizing process which react with magnesium metas to form solidified magnesium chloride. Iron was also detected at a low level (1.33 wt%), representing ferric oxide used as a catalytic additive. Although present in small quantities, Fe_2O_3 likely enhanced the burn rate by accelerating AP decomposition. Its low residue content supports the conclusion that ferric oxide served primarily as a catalyst rather than a major component of the combustion byproducts.

In conclusion, The results in Figure 4.26 show that SP7, with ferric oxide alone, catalyzes the decomposition of AP but doesn't suppress chlorine release, which explains its high Cl concentration. The low carbon content implies relatively complete combustion. In contrast, SP8 demonstrates a balanced combustion effect due to the synergistic interaction between magnesium and ferric oxide. Magnesium scavenges chlorine to produce magnesium chloride in solid form and gas product. Which contributes to smoother post-combustion residues (as confirmed by SEM), while ferric oxide enhances the decomposition rate. The increased oxygen and carbon content in SP8 suggest higher reactivity, possibly with the formation of metal oxides and other stable residue.

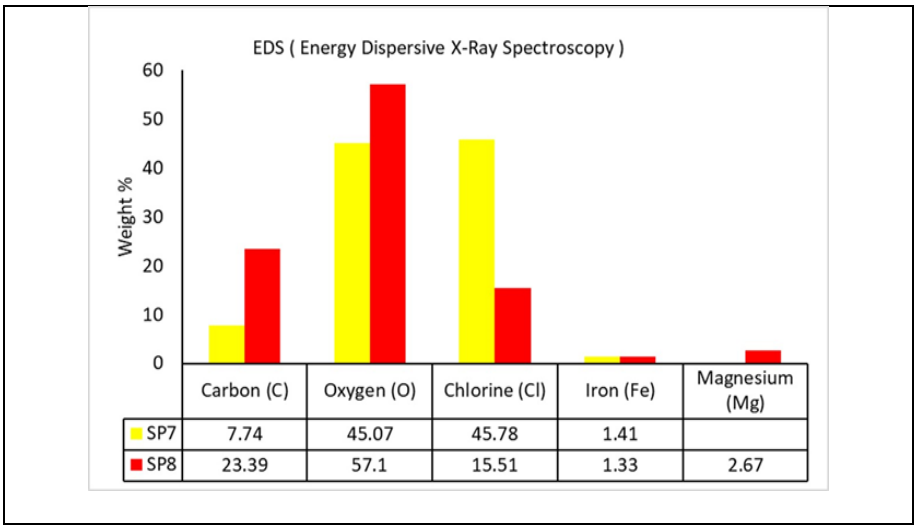


Figure 4.26 Energy-Dispersive X-Ray Spectroscopy For Sp7 And Sp8

4.4.2.4 Mechanism of Combustion Solid Propellant

Initial conclusion the comparison of smoke density from the combustion of AP/sorbitol-based solid propellants with other conventional formulations such as AP/HTPB/Al and KNO_3/sorbitol reveals that the AP/sorbitol system produces significantly less smoke. This reduced smoke generation can be attributed to the efficient combustion mechanism that occurs between ammonium perchlorate (AP), a powerful oxidizing agent, and biodegradable organic polymers such as sorbitol and starch. The strong oxidizing capability of AP ensures complete oxidation of the organic fuel, minimizing the formation of carbonaceous soot. The simplified combustion reaction for the AP/sorbitol-based propellant is as follows in (4.10).

$$C_6H_{14}O_6 + 6NH_4ClO_4 \rightarrow 6CO_2 + 16H_2O + 3N_2 + 6HCl + O_2 \quad (4.10)$$

In this reaction, sorbitol undergoes oxidation in the presence of AP, producing carbon dioxide, water, nitrogen, and hydrogen chloride (HCl) gas. While the absence of soot formation contributes to a cleaner combustion process, the production of HCl remains a concern due to its corrosive and toxic nature. To address this issue, metal additives are incorporated into the propellant formulation to function as chlorine scavengers, effectively reducing HCl emissions. For instance, magnesium (Mg) reacts with HCl through the following reaction (4.11).

$$Mg + 2HCl \rightarrow MgCl_2 + H_2 \quad (4.11)$$

This reaction results in the formation of magnesium chloride ($MgCl_2$), a condensed-phase product, thereby capturing chlorine in solid form and preventing its release as a gaseous pollutant. Similarly, ferric oxide (Fe_2O_3) reacts with HCl as follows (4.12).

$$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O \quad (4.12)$$

This acid-base reaction produces iron (III) chloride ($FeCl_3$) and water, further contributing to the reduction of free HCl gas in the exhaust. These metal additives thus play a critical role in enhancing the environmental performance of AP-based propellants by facilitating cleaner combustion and mitigating the release of harmful chlorine-containing gases.

4.4.3 AGARD Classification

The AGARD classification in Table 4.10 for SP4 to SP8 reveals distinct trends in smoke signature based on formulation composition. SP4 and SP5, without metal additives, exhibited the highest light transmittance (~94–98%) and lowest smoke residue masses (0.036 g and 0.033 g), making them AGARD Class A of STANAG 6016, indicative of clean combustion and minimal visual obstruction for primary smoke production.

Table 4.10
Agard Smoke Classification

Sample	AGARD Smoke Class
SP4	AB
SP5	AB
SP6	BB
SP7	BB / BC
SP8	BB

In contrast, SP6 and SP7, containing magnesium and ferric oxide respectively, demonstrated reduced light transmittance (~45–76%) and higher smoke particle mass

(up to 0.056 g), placing them in Class BB/BC due to denser smoke production from metal oxide and chloride residues. SP8, incorporating both Mg and Fe_2O_3, showed an intermediate performance with a moderate smoke profile (classified as BB), suggesting a synergistic effect that balances thrust enhancement with reduced particulate output.

These results confirm that while metal additives improve energy release and combustion energy, they also introduce condensed phase byproducts that increase visual signature. The observed classifications align with AGARD PEP WG-21 criteria and findings from previous studies, supporting the conclusion that formulation tuning directly influences both performance and visual detectability in solid propellants.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Chapter Overview

This chapter concludes the contribution of the study involving the development of minimum signature solid propellant. The conclusion involves determination of formulation, production and experimental work. The main objective of this work is developed and determine the magnitude of combustion product was also summarized. Further work arising from study have been identified and highlighted in the final section of this chapter.

5.2 Conclusion

The synthesis of AP/sorbitol-based propellants with varied compositions was successfully achieved and yields several findings. The optimum ratio between ammonium perchlorate and sorbitol was calculated to be 77 wt.% based on elemental stoichiometric coefficients and oxygen balance analysis. Open burning tests of the optimum formulation showed that the ammonium perchlorate/sorbitol propellant produced no visible smoke and left no solid residue. This indicates that all carbon compounds in sorbitol were completely oxidized by the oxygen supplied by ammonium perchlorate. The addition of metal additives significantly improves the burning rate of the solid propellant. 5 wt.% magnesium metal and ferrous oxide exhibit a strong synergistic effect, increasing the burning rate to 0.126 cm/s. Furthermore, the visual inspection during open burning trials revealed clear distinctions in flame colour and intensity between additive-free and additive-enhanced formulations

The investigation of the AP/sorbitol solid propellant revealed that an ammonium perchlorate content of 77 wt.% represents the optimal condition in terms of energy release. It was observed that increasing the oxidizer content beyond this level reduces the heat of combustion and, consequently, degrades solid propellant performance. Conversely, higher oxidizer content enhances the thermal stability and increases the ignition temperature of the propellant. The addition of 5 wt.% magnesium metal

significantly enhances both heat energy release at 4142.57 kJ/mol and high specific impulse at 183.56 s, indicating that its role extends beyond that of a chlorine scavenger. Furthermore, the presence of magnesium markedly increases the thermal stability of the AP/sorbitol mixture by promoting stable high ignition temperature 243.59 ˚C. In contrast, 0.05 wt.% ferric oxide, which functions primarily as a catalyst, does not directly contribute to energy release or propellant performance. However, it effectively reduces the ignition temperature and thermal decomposition temperature, although it does not significantly affect overall performance. Finally, the combined use of magnesium and ferrite additives demonstrates a synergistic effect with the AP/sorbitol solid propellant, leading to improved performance and increased energy release.

Mechanical properties of solid propellant significantly affected by the inclusion of metal additives. Incorporating magnesium (Mg) and ferric oxide into solid propellant compositions has been shown to significantly enhance the mechanical properties and toughness of the propellant. As indicated by the results, the baseline sample without metal additives exhibits the lowest stress and Shore hardness values. The addition of 5 wt.% Mg improves these mechanical properties, while the inclusion of 1 wt.% ferric oxide further enhances ductility and energy absorption. The combined addition of both Mg and ferric oxide in SP8 yields the highest overall stress at 1.116 N/mm^2.

Fourier Transform Infrared Spectroscopy (FTIR) analysis demonstrated clear molecular interactions between additives and the binder-oxidizer system, such as changes in –OH and NO_3 vibrational modes, supporting evidence of catalytic influence and structural modification. X-ray Diffraction (XRD) analysis further revealed the presence of crystalline phases such as MgO, $FeCl_3$, and residual AP, confirming the formation of stable combustion byproducts and partial additive retention in the post-combustion matrix. Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Spectroscopy (EDS) highlighted distinct morphological differences between the samples. SP7 and SP8 displayed porous and sponge-like microstructures with embedded particulate residues, while EDS confirmed the presence of Mg, Fe, Cl, and O, indicating partial reactions and metal oxide formation. These structural changes are consistent with accelerated decomposition and increased reactivity due to the presence of additives. Additionally, CHNS-O elemental analysis revealed variations in carbon, oxygen, and chlorine content across formulations, supporting evidence of combustion completeness and additive interaction.

The kinetic evaluation using Flynn–Wall–Ozawa (FWO) and Kissinger–Akahira–Sunose (KAS) methods showed that the activation energy (Ea) varied between 75–161 kJ/mol across formulations, with SP6 exhibiting the highest Ea at 156.677KJ/mol using KAS and 161.156KJ/mol using FWO, due to its energy-dense combustion reaction pathway. The presence of magnesium increases the ignition barrier due to metal oxidizer interactions, while contributing high heat release once combustion occurs. Magnesium therefore contributes to higher energy release during combustion, despite increasing the measured activation energy in the thermal analysis stage. Conversely, ferric oxide exhibits a catalytic effect by lowering the activation energy at value 122.473 KJ/mol using KAS and 124.26 KJ/mol using FWO, facilitating proton and electron transfer during AP decomposition and promoting earlier breakdown of the oxidizer. The combined kinetic trends confirm that sorbitol content, metal fuel addition, and catalytic additives collectively govern the thermal decomposition behaviour and combustion efficiency of the AP/sorbitol-based propellants. The result of kinetic parameters evaluated from KAS is used as final value, due to accuracy as mentioned in literature [293].

The static firing tests demonstrate that the incorporation of magnesium significantly enhances propellant performance, producing a peak thrust of 4.40 N and a specific impulse of 29.60 s. Formulations employing a weaker oxidizer generated a lower thrust of 2.61 N compared to 3.00 N for formulations with a stronger oxidizer, despite using the same fuel composition. These results clearly indicate that oxidizer strength plays a decisive role in, thrust generation. Overall, the findings confirm that both metal additives and oxidizer selection are critical parameters in optimizing the thrust and specific impulse of solid propellants.

The light transmittance of a solid propellant increases when fewer or no metal additives are present in the formulation. For example, SP4 at a mass of 0.4 g exhibits a light transmittance of 94.1%, which is higher than that of samples containing metal additives at the same mass. AP-based solid propellants incorporating biodegradable natural polymers as fuel show the highest light transmittance, reaching up to 99% across all tested masses, indicating significantly reduced smoke intensity during combustion. This observation confirms that metal inclusion plays a crucial role in the formation of primary smoke. Meanwhile, samples formulated with a weak oxidizer exhibit the lowest light transmittance value of 12.97%, confirming that oxidizer strength plays a critical role in achieving clean and smoke-free combustion.

The combination of environmentally friendly biodegradable organic polymers with a strong oxidizer substantially reduces smoke and soot formation, thereby mitigating the adverse effects typically associated with AP combustion. Experimental results using starch instead of sorbitol as the fuel demonstrate that the use of natural polymers in AP-based formulations produces no solid residue after combustion, confirming that biodegradable fuels promote cleaner combustion.

Overall, the combustion of AP/sorbitol-based solid propellants under Malaysian ambient conditions (73–80% relative humidity) resulted in minimal smoke and no observable solid residues, confirming near-complete combustion driven by the synergistic interaction between sorbitol and ammonium perchlorate. However, the results also demonstrate that ambient temperature and relative humidity significantly influence smoke visibility, indicating that environmental conditions must be considered when evaluating the exhaust signature and operational performance of minimum-signature solid propellants.

SP4 and SP5 remain the cleanest-burning options, while SP8 emerges as the most promised formulation for tactical applications requiring both performance in term of specific impulse and energy release. The AGARD smoke classification applied in this study provides a structured framework for evaluating the exhaust signature of AP/sorbitol-based solid propellants by considering both primary and secondary smoke characteristics. Primary smoke was assessed through measured light transmittance and smoke opacity, reflecting the concentration of condensed-phase particles, while secondary smoke was evaluated based on visible exhaust persistence influenced by gaseous products and ambient conditions. By integrating experimental opacity measurements with AGARD-defined qualitative criteria, the classification reliably differentiates propellant formulations according to their smoke behaviour, supporting its suitability for minimum-signature performance assessment in this research.

The flow diagram outlining the development process of the minimum-signature solid propellant for this study is presented in Appendix 2. It begins with the classical approach of balancing chemical equations to establish a clear understanding of the appropriate ratio between oxidizing and reducing agents. This is followed by determining the optimal amount of oxygen and elemental stoichiometric equation required to achieve complete combustion, with the goal of producing a low-smoke exhaust that meets the STANAG 6016 Class A standard as defined in AGARD PEP WG-21 for minimum-signature solid propellants.

5.3 Recommendations

The objectives of this research were successfully met; however, certain limitations and scope constraints restricted broader exploration of its application in rocketry. To advance the current findings, several extensions can be undertaken. The following recommendations are proposed for future work to further enhance the understanding of minimum-signature solid propellants:

i) The use of strong oxidizing agents in combination with biodegradable fuels has been shown to produce cleaner combustion. In addition to sorbitol, fuels such as starch and sucrose also exhibit minimum-signature characteristics. These observations suggest a notable relationship between strong oxidizers and biodegradable fuels in promoting low-smoke, environmentally friendly combustion behaviour.

ii) The primary formulation investigated in this study AP/Sorbitol is highly hygroscopic, resulting in a short shelf life due to its tendency to absorb moisture from the surrounding environment. While for magnesium it surfaces layer expose to moist air will form a magnesium oxide which is hygroscopic. This moisture uptake negatively impacts key performance parameters, including ignition temperature, energy release, mechanical strength, and smoke generation, all of which deteriorate over time. To address this issue, effective strategies must be developed to reduce or mitigate the hygroscopicity of the formulation, as all three components AP, sorbitol, and magnesium are inherently moisture sensitive.

iii) Another interesting incidental finding in this study is that replacing sorbitol with starch significantly mitigates the issue of hygroscopicity. Starch, a natural biodegradable polymer, demonstrated clean combustion with no visible smoke or soot, along with a notably high burning rate. To utilize starch as a fuel, thermal processing was carried out by cooking it with the oxidizing agent in water until polymerization occurred, followed by cooling and curing for approximately one week. Under these processing conditions, the resulting solid propellant was observed to exhibit promising characteristics, including relatively high mechanical strength, low

hygroscopic behaviour, rapid combustion, and a soot-free exhaust. These encouraging attributes suggest that biodegradable polymer-based propellants warrant further detailed investigation to fully explore their potential in environmentally friendly, high-performance solid propulsion systems.

iv) The addition of ferric oxide as a catalyst effectively lowers the ignition temperature and exhibits synergistic behaviour with other propellant formulations. Moreover, it maybe reduces the hygroscopicity of the mixture which need to do a further work on this and enhances the mechanical strength of each composition. However, its use can lead to unstable and vigorous combustion, which in some cases may result in explosive reactions. Therefore, the development and investigation of alternative catalysts are necessary those that offer advantages in both physical and chemical properties to ensure improved performance and enhanced safety.

v) Furthermore, the formulation of AP/Sorbitol/Mg with the addition of ferric oxide was observed to produce an explosive output surpassing that of ammonium nitrate-based explosives. Hypothetically, this composition possesses high energy density and can detonate when triggered by a smaller initiating explosion, indicating a potential for high explosive power. While it does not self-detonate under normal conditions, the requirement of a primary detonation to initiate a secondary, more powerful reaction suggests a controlled yet highly energetic behaviour. This provide a gap need to be investigated about explosive behaviour of these substances. This accidental discovery deserves thorough investigation to evaluate its viability as a high energy density explosive for defence applications, while ensuring compliance with safety standards and regulatory requirements.

vi) The use of the Ballistic Evaluation Motor (BEM) with end burning method to determine the burning rate is not recommended since this way produce small nozzle diameter and cause ignition difficulties often lead to several issues, including unexpected explosions and cracking of the solid propellant grain. Instead, a hollow-tube grain configuration is advised, as it promotes more stable ignition and controlled combustion [294]. For better accuracy and safety, it is recommended to position a pressure sensor at the end of the

grain and employ a free-standing setup to minimize mechanical stress on the propellant. This approach improves test reliability while reducing the risk of structural failure during combustion

REFERENCES

[1] M. Málek, “CZECH TECHNICAL UNIVERSITY IN PRAGUE Design and characterization of small solid-propellant rocket engine,” CZECH TECHNICAL UNIVERSITY IN PRAGUE, 2019.

[2] A. Y. Alemayehu, “Design of a Solid Rocket Propulsion System,” *Int. J. Aeronaut. Sci. Aerosp. Res.*, no. July, pp. 224–229, 2020, doi: 10.19070/2470-4415-2000027.

[3] Ahmet Hakan Demir, “Istanbul technical university faculty of aeronautics and astronautics,” 2018.

[4] Ang Kiang Long, “Design, Fabrication and Testing of a Solid Propellant Rocket Motor,” 2011.

[5] M. Zaki *et al.*, “Propellant development for small rockets.”

[6] A. coma Busquets, “Specification, design and development of a solid rocket engine for suborbital sounding missions,” 2020.

[7] R. A. Chandru, N. Balasubramanian, C. Oommen, and B. N. Raghunandan, “Additive manufacturing of solid rocket propellant grains,” *J. Propuls. Power*, vol. 34, no. 4, pp. 1090–1093, 2018, doi: 10.2514/1.B36734.

[8] K. Lysien, A. Stolarczyk, and T. Jarosz, “Solid propellant formulations: A review of recent progress and utilized components,” *Materials (Basel).*, vol. 14, no. 21, 2021, doi: 10.3390/ma14216657.

[9] Dr Km Pandey and Dr Probhas Bose, “Development of Green Propellants for Short Range Solid Rocket : an Overview,” *Int. J. Eng. Res.*, vol. V5, no. 09, pp. 596–599, 2016, doi: 10.17577/ijertv5is090475.

[10] J. Yang, X. Gong, H. Mei, T. Li, J. Zhang, and M. Gozin, “Design of Zero Oxygen Balance Energetic Materials on the Basis of Diels-Alder Chemistry,” *J. Org. Chem.*, vol. 83, no. 23, pp. 14698–14702, 2018, doi: 10.1021/acs.joc.8b02000.

[11] Jacqueline Akhavan, “Thermochemistry of Explosives,” in *The chemistry of explosives*, 3rd ed., Cambridge: Royal Society of Chemistry, 2011, ch. Oxygen Bal, pp. 85–112.

[12] S. L. Chen *et al.*, “Optimizing the oxygen balance by changing the A-site cations in molecular perovskite high-energetic materials,” *CrystEngComm*, vol. 20, no. 46, pp. 7458–7463, 2018, doi: 10.1039/c8ce01350k.

[13] Jacqueline Akhavan, “Effect of Oxygen Balance,” in *The chemistry of explosives*, 3rd ed., Cambridge, 2011, ch. Thermochem, p. 100.

[14] F. yao Chen *et al.*, “A review on the high energy oxidizer ammonium dinitramide: Its synthesis, thermal decomposition, hygroscopicity, and application in energetic materials,” *Def. Technol.*, no. xxxx, 2022, doi: 10.1016/j.dt.2022.04.006.

[15] E. Leader, “in Propulsion Systems for Missiles,” 2021.

[16] B. P. Mason and C. M. Roland, “Solid propellants,” *Rubber Chem. Technol.*, vol. 92, no. 1, pp. 1–24, 2019, doi: 10.5254/rct.19.80456.

[17] M. Guven Gok and O. Cihan, “Energetic Materials and Metal Borides for Solid Propellant Rocket Engines,” *Int. J. Mater. Eng. Technol.*, vol. 003, pp. 109–119, 2020, [Online]. Available: http://dergipark.gov.tr/tijmet

[18] I. Trofimov, S. Boichenko, and S. Shamanskyi, “Analysis of rocket fuels and problems of their application on the example of Ukraine,” *Technol. Audit Prod. Reserv.*, vol. 6, no. 1(56), pp. 19–27, 2020, doi: 10.15587/2706-5448.2020.218358.

[19] R. S. van den Hurk *et al.*, “Characterization and comparison of smokeless powders by on-line two-dimensional liquid chromatography,” *J. Chromatogr. A*, vol. 1672, p. 463072, 2022, doi: 10.1016/j.chroma.2022.463072.

[20] A. Dey, A. K. Sikder, M. B. Talawar, and S. Chottopadhyay, “Towards new directions in oxidizers/energetic fillers for composite propellants: An overview,” *Cent. Eur. J. Energ. Mater.*, vol. 12, no. 2, pp. 377–399, 2015.

[21] M. Abd-Elghany, A. Elbeih, T. M. Klapötke, and M. Abdelhafiz, “Performance of advanced composite solid rocket propellants based on novel oxidizers,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 610, no. 1, 2019, doi: 10.1088/1757-899X/610/1/012002.

[22] D. Trache, T. M. Klapötke, L. Maiz, M. Abd-Elghany, and L. T. DeLuca, “Recent advances in new oxidizers for solid rocket propulsion,” *Green Chem.*, vol. 19, no. 20, pp. 4711–4736, 2017, doi: 10.1039/c7gc01928a.

[23] B. C. Terry, T. R. Sippel, M. A. Pfeil, I. E. Gunduz, and S. F. Son, “Removing hydrochloric acid exhaust products from high performance solid rocket propellant using aluminum-lithium alloy,” *J. Hazard. Mater.*, vol. 317, pp. 259–266, 2016, doi: 10.1016/j.jhazmat.2016.05.067.

[24] S. K. Pradhan, V. Kedia, and P. Kour, “Review on different materials and their characterization as rocket propellant,” *Mater. Today Proc.*, vol. 33, no. xxxx, pp. 5269–5272, 2020, doi: 10.1016/j.matpr.2020.02.960.

[25] M. Anniyappan, M. B. Talawar, R. K. Sinha, and K. P. S. Murthy, “Review on Advanced Energetic Materials for Insensitive Munition Formulations,” *Combust. Explos. Shock Waves*, vol. 56, no. 5, pp. 495–519, 2020, doi: 10.1134/S0010508220050019.

[26] I. B. I. Isrul, F. M. Sakri, P. M. B. P. Z. Sham, H. B. Sa’aid, M. T. Ahmad, and R. Abidin, “Low-cost Analytic Analysis for Civilian Rocket Flare using Solid Rocket Propellant (SRP) Composition,” *J. Aeronaut. Astronaut. Aviat.* , vol. 56, no. 1, pp. 229–242, 2024, doi: 10.6125/JoAAA.202403_56(1S).13.

[27] Z. hua Xue, Z. kangping Wang, R. xuan Xu, X. xue Zhang, and Q. long Yan, “Thermal interactions between hybrid HMX/ANPyO cocrystals and commonly used propellant ingredients,” *Energ. Mater. Front.*, no. xxxx, 2022, doi: 10.1016/j.enmf.2022.01.005.

[28] L. Jiang *et al.*, “Catalytic Effect of Ferrocenyl Energetic Catalysts for N-Guanylurea Dinitramide (GUDN or FOX-12) Decomposition,” *ACS Omega*, vol. 7, no. 29, pp. 25732–25740, 2022, doi: 10.1021/acsomega.2c03026.

[29] W. Zhang, X. Fan, H. Wei, and J. Li, “Application of nitramines coated with nitrocellulose in minimum signature isocyanate-cured propellants,” *Propellants, Explos. Pyrotech.*, vol. 33, no. 4, pp. 279–285, 2008, doi: 10.1002/prep.200800220.

[30] W. Xie *et al.*, “Sensitivity and Stability Improvements of NEPE Propellants by Inclusion of FOX-7,” *Propellants, Explos. Pyrotech.*, vol. 43, no. 3, pp. 308–314, 2018, doi: 10.1002/prep.201700251.

[31] Florin-Marian Dîrloman*)Traian Rotariu*Tudor-Viorel Țigănescu**Mihai Ionuț Ungureanu*, “Dîrloman_Rotariu_Țigănescu_Ungureanu.pdf,” Ilfov.

[32] M. Comet, C. Schwartz, F. Oudot, F. Schnell, and D. Spitzer, “Hazardous Properties of Molten Ammonium Dinitramide,” *Propellants, Explos. Pyrotech.*, vol. 45, no. 10, pp. 1600–1606, 2020, doi: 10.1002/prep.202000037.

[33] L. Wu, X. Li, and C. U. Ro, “Hygroscopic behavior of ammonium sulfate, ammonium nitrate, and their mixture particles,” *Asian J. Atmos. Environ.*, vol. 13, no. 3, pp. 196–211, 2019, doi: 10.5572/ajae.2019.13.3.196.

[34] A. Mr, Ž. Andrić, and A. Ž. Andrić, “Crystallization of Ammonium-Perchlorate from Solution of Electrolytically Produced Sodium-Perchlorate in a Crystallization of Ammonium-Perchlorate from Solution of Electrolytically Produced Sodium-Perchlorate in a Pilot-Scale Plant,” 2007. [Online]. Available: https://pdfs.semanticscholar.org/3d5f/28e380e040ede8f9ffb6692160c31058280d.pdf?_ga=2.212594836.181398010.1574557236-343990255.1574557236

[35] R. L. Dotson, “A novel electrochemical process for the production of ammonium perchlorate,” *J. Appl. Electrochem.*, vol. 23, no. 9, pp. 897–904, 1993, doi: 10.1007/BF00251024.

[36] L. T. DeLuca, “Overview of Al-based nanoenergetic ingredients for solid rocket propulsion,” *Def. Technol.*, vol. 14, no. 5, pp. 357–365, 2018, doi: 10.1016/j.dt.2018.06.005.

[37] G. N. and F. Kadhum, “Properties and Manufacture of Ammonium Perchlorate,” 2020. doi: 10.13140/RG.2.2.15030.11843.

[38] Y. Shim, S. Ha, E. Yoo, H. Jang, and R. Kim, “Recovery of Ammonium Perchlorate from Obsolete Ammunition and Its Application in Synthesis of Lithium Perchlorate,” *Propellants, Explos. Pyrotech.*, vol. 46, pp. 612–617, 2021, doi: 10.1002/prep.202000235.

[39] T. Deschner, E. A. Løkke, T. E. Kristensen, T. L. Jensen, and E. Unneberg, “Insensitive Minimum Smoke Propellants for Tactical Missiles,” *Ndiastorage.Blob.Core ...*, pp. 1–10, 2018, [Online]. Available: https://ndiastorage.blob.core.usgovcloudapi.net/ndia/2018/imem/20264_Deschner_Paper.pdf

[40] W. S. Setiani, R. H. Triharjanto, M. Ayu, E. Hafizah, and A. Navalino, “Study of The Potency of Reducing Smoke on Composite Propellant with High Specific Impulse,” *UIJRT*, vol. 03, no. 07, pp. 42–45, 2022.

[41] J. Kanazirev, Vladislav; Gorawara, “Halide scavengers for high temperature applications,” 2005 [Online]. Available: https://patents.google.com/patent/US20070116620A1/en

[42] Aderibigbe, “Effect of Process Parameters on Pot Life of Solid Propellant Composite Based on Hydroxyl- Terminated Polybutadiene: A Short Review Abbas,” *Energies*, vol. 6, no. 1, pp. 1–8, 2018, [Online]. Available: http://journals.sagepub.com/doi/10.1177/1120700020921110%0Ahttps://doi.org/10.1016/j.reuma.2018.06.001%0Ahttps://doi.org/10.1016/j.arth.2018.03.044

%0Ahttps://reader.elsevier.com/reader/sd/pii/S1063458420300078?token=C039B8B13922A2079230DC9AF11A333E295FCD8

[43] J. Chen *et al.*, "Highly space-confined ammonium perchlorate in three-dimensional hierarchically ordered porous carbon with improved thermal decomposition properties," *Appl. Surf. Sci.*, vol. 457, no. June, pp. 508–515, 2018, doi: 10.1016/j.apsusc.2018.06.301.

[44] J. Ashish, G. Swaroop, and K. Balasubramanian, Effect of Ammonium Perchlorate Particle Size on Flow , Ballistic , and Mechanical Properties of Composite Propellant. Elsevier Inc., 2019. doi: 10.1016/B978-0-12-813908-0.00008-3.

[45] P. Deng, H. Wang, X. Yang, H. Ren, and Q. Jiao, "Thermal decomposition and combustion performance of high-energy ammonium perchlorate-based molecular perovskite," *J. Alloys Compd.*, vol. 827, p. 154257, 2020, doi: 10.1016/j.jallcom.2020.154257.

[46] P. A. Figueiredo and F. M. Brójo, "Theoretical analysis of ammonium-perchlorate based composite propellants containing small size particles of boron," *Energy Procedia*, vol. 136, no. November, pp. 202–207, 2017, doi: 10.1016/j.egypro.2017.10.320.

[47] R. Nakka, "Effect of chamber pressure on burning rate for the potassium nitrate - dextrose and potassium nitrate - sorbitol rocket propellants," 1999.

[48] M. C. Olde, "Potassium Nitrate Sorbitol Propellant :Experimental Investigation of Solid Propellant Characteristics," 2019. [Online]. Available: http://repository.tudelft.nl/

[49] C. Américo, D. F. Moro, C. H. Marchi, and F. Aguiar, "Development of a Ballistic Evaluation Motor for Knsu Burn Rate Measurements," in *18 th Brazilian Congress of Thermal Sciences and Engineering*, 2020. doi: 10.26678/abcm.encit2020.cit20-0174.

[50] F. W. Gateru, S. K. Oina, J. Mboya, I. Kibandi, B. Owiti, and S. Aoki, "Development of Solid Propellant Motor for Low Altitude Model Rockets," *Proc. Sustain. Res. Innov. Conf. JKUAT Main Campus, Kenya*, pp. 1–8, 2021.

[51] Oyedeko K.F.K, "OPTIMISATION OF THE FORMULATION OF A DOUBLE – BASED SOLID PROPELLANT OPTIMISATION OF THE FORMULATION OF A DOUBLE – BASED SOLID," *IJESRT*, vol. 7, no. 8, 2018.

[52] M. C. Olde, “Potassium Nitrate Sorbitol Propellant - Experimental Investigation of Solid Propellant Characteristics,” 2019. [Online]. Available: http://repository.tudelft.nl/.

[53] Oyedeko K.F.K, “International journal of engineering sciences & research technology effect of propellant formulation on propellant properties *1,” *Int. J. Eng. Sci. Res. Technol.*, vol. 7, no. 8, pp. 305–313, 2018.

[54] E. D. Tolmachoff, L. C. Baldwin, J. T. Essel, T. D. Hedman, S. E. Kalman, and J. Kalman, “Effects of Select Metal Oxides on the Onset, Rate and Extent of Low Temperature Ammonium Perchlorate Decomposition,” *J. Energ. Mater.*, vol. 40, no. 4, pp. 429–444, 2022, doi: 10.1080/07370652.2021.1895914.

[55] T. Knop *et al.*, “Sorbitol-based hybrid fuel studies with nitrous oxide for the stratos ii sounding rocket,” *49th AIAA/ASME/SAE/ASEE Jt. Propuls. Conf.*, vol. 1 PartF, pp. 1–13, 2013, doi: 10.2514/6.2013-4049.

[56] K. Moody, A. Walsh, A. Ngo, S. Whyte, A. Stottlemyre, and K. Rouser, “Development of sorbitol-based solid rocket motors for propulsion education,” *AIAA Scitech 2020 Forum*, no. January, pp. 1–19, 2020, doi: 10.2514/6.2020-0067.

[57] M. T. Migliorino *et al.*, “Student firing tests and launches with commercial and self-made solid rocket motors,” *Acta Astronaut.*, vol. 197, no. April, pp. 23–34, 2022, doi: 10.1016/j.actaastro.2022.04.025.

[58] G. O. Adeniyi, I. Nkere, L. M. Adetoro, and O. S. Sholiyi, “Performance Analysis of a Dual-Fuel Sugar Based Solid Rocket Propellant,” *Eur. J. Eng. Technol. Res.*, vol. 6, no. 2, pp. 34–41, 2021, doi: 10.24018/ejers.2021.6.2.2347.

[59] H. Ajamein and M. Haghighi, “Effect of sorbitol/oxidizer ratio on microwave assisted solution combustion synthesis of copper based nanocatalyst for fuel cell grade hydrogen production Article Information,” *Iran. J. Hydrog. Fuel Cell*, vol. 4, no. 2015, pp. 227–240, 2015, [Online]. Available: http://dx.doi.org/10.22104/ijhfc.2015.265

[60] B. C. Terry, T. R. Sippel, M. A. Pfeil, I. E. Gunduz, and S. F. Son, “Removing hydrochloric acid exhaust products from high performance solid rocket propellant using aluminum-lithium alloy,” *J. Hazard. Mater.*, vol. 317, pp. 259–266, 2016, doi: 10.1016/j.jhazmat.2016.05.067.

[61] V. . . K. T.L Varghese, “Characteristic Various Oxidizer,” in *The Chemistry and Technology of Solid Rocket Propellants(A Treatise on Solid Propellants)*, New Dehli: Allied Publisher Pvt. LTD, 2017, p. 174.

[62] M. Nazri and M. Jaafar, “Development of Solid Rocket Propulsion,” *J. Mek.*, vol. 18, pp. 111–121, 2004, [Online]. Available: http://www.fkm.utm.my/~mekanika/Issue 18/development(nazri-II-jilid18).pdf

[63] S. R. Jain, “Solid propellant binders,” *J. Sci. Ind. Res. (India).*, vol. 61, no. 11, pp. 899–911, 2002.

[64] G. P. Sutton, “Rocket Propulsion Elements,” 6th ed., Carlifonia: Wiley, 1992, ch. Solid Prop, p. 417.

[65] P. Madya and F. Kejuruteraan Mekanikal, “Mengkaji Prestasi Pottasium Nitrat Sebagai Bahan Dorong Pepejal Untuk Enjin Roket. Md.Nizam Bin Dahalan (Ketua),” 2004.

[66] T.L Varghese, “Salient Features of Binders,” in *The Chemistry and Technology of Solid Rocket Propellants(A Treatise on Solid Propellants)*, New Dehli: Allied Publisher Pvt. LTD, 2017, ch. Composite, p. 26.

[67] T. Chai *et al.*, “Rheokinetic analysis on the curing process of HTPB-DOA- MDI binder system,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 137, no. 1, 2016, doi: 10.1088/1757-899X/137/1/012069.

[68] J. Fleming, W. Rousseau, M. Zebregs, and C. van Driel, “Solventless Extruded Double-Base (Edb) Propellant Charges—a Review of the Properties, Technology, and Applications,” *Int. J. Energ. Mater. Chem. Propuls.*, vol. 21, no. 3, pp. 13–46, 2022, doi: 10.1615/IntJEnergeticMaterialsChemProp.v21.i3.20.

[69] A. H. A. Hamid *et al.*, “Transient Thermal Analysis of Solid Propellant Rocket Motor,” *J. Appl. Eng. Des. Simul.*, vol. 2, no. 1, pp. 1–8, 2022, doi: 10.24191/xxxxxxx.

[70] W. Zhang *et al.*, “Challenging the limits of the oxygen balance of a pyrazole ring,” *Chem. Eng. J.*, vol. 451, no. P1, p. 138609, 2023, doi: 10.1016/j.cej.2022.138609.

[71] I. J. Powell, “Reduced vulnerability minimum smoke propellants for tactical rocket motors,” *41st AIAA/ASME/SAE/ASEE Jt. Propuls. Conf. Exhib.*, no. July, pp. 1–9, 2005, doi: 10.2514/6.2005-3615.

[72] T.L Varghese, “Advanced Solid Propellants,” in *The Chemistry and Technology of Solid Rocket Propellants(A Treatise on Solid Propellants)*, New Dehli: Allied Publisher Pvt. LTD, 2017, ch. Advanced O, pp. 172–196.

[73] A. M. Helmy and T. Mccormick, “AI AA-84- 1434 Investigation of New Energetic Ingredients for Minimum Signature Propellants AIAA / SAE / ASME 20th Joint Propulsion Conl2rence,” *20th Jt. Propuls. Conf.*, 1984.

[74] R. L. Bivin, J. T. Johnson, L. Markovitch, A. K. Mehrotra, L. E. Promenade, and A. I. Aa--, “AlAA 92-3724 Development of a Class 1 . 3 Minimum Smoke Propellant,” 1992.

[75] F. Hodge, K., T. Crofoot, and S. Nelson, “Gelled Propellants for Tactical Missile Applications,” 1999.

[76] K. Menke, T. Heintz, W. Schweikert, T. Keicher, and H. Krause, “Formulation and Properties of ADN/GAP propellants,” *Propellants, Explos. Pyrotech.*, vol. 34, no. 3, pp. 218–230, 2009, doi: 10.1002/prep.200900013.

[77] M. Y. Nagamachi, J. I. S. Oliveira, A. M. Kawamoto, and R. de Cássia L Dutra, “ADN - The new oxidizer around the corner for an environmentally friendly smokeless propellant,” *J. Aerosp. Technol. Manag.*, vol. 1, no. 2, pp. 153–160, 2009, doi: 10.5028/jatm.2009.0102153160.

[78] K. Menke, S. Eisele, M. Bohn, and P. Gerber, “Minimum Smoke Propellants with High Burning Rates and Thermodynamic Performance,” *Rto-Mp-091*, no. 13, pp. 1–20, 2002.

[79] G. Cican and A. D. Mitrache, “Rocket solid propellant alternative based on ammonium dinitramide,” *INCAS Bull.*, vol. 9, no. 1, pp. 17–24, 2017, doi: 10.13111/2066-8201.2017.9.1.2.

[80] D. M. K. Dennis, M. R. Williams, and M. E. Sigman, “Investigative probabilistic inferences of smokeless powder manufacturers utilizing a Bayesian network,” *Forensic Chem.*, vol. 3, pp. 41–51, 2017, doi: 10.1016/j.forc.2016.12.001.

[81] E. Landsem, T. L. Jensen, F. K. Hansen, E. Unneberg, and T. E. Kristensen, “Neutral polymeric bonding agents (NPBA) and their use in smokeless composite rocket propellants based on HMX-GAP-BuNENA,” *Propellants, Explos. Pyrotech.*, vol. 37, no. 5, pp. 581–591, 2012, doi: 10.1002/prep.201100136.

[82] M. Abd-Elghany, T. M. Klapötke, and A. Elbeih, “Environmentally safe (chlorine-free): New green propellant formulation based on 2,2,2-Trinitroethyl-formate and HTPB,” *RSC Adv.*, vol. 8, no. 21, pp. 11771–11777, 2018, doi: 10.1039/c8ra01515e.

[83] E. M. Youssef, T. Elshenawy, H. E. Mostafa, M. Radwan, and A. Elbeih, “Optimization of low signature base bleed propellant formulations,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 610, no. 1, pp. 0–10, 2019, doi: 10.1088/1757-899X/610/1/012003.

[84] K. F. O. & A. Onyieagho2, “OPTIMISATION OF THE FORMULATION OF A DOUBLE – BASED SOLID PROPELLANT OPTIMISATION OF THE FORMULATION OF A DOUBLE – BASED SOLID,” *Int. J. Eng. Sci. Res. Technol.*, vol. 7, no. 8, p. 10, 2018.

[85] J. D. Hunley, “The history of solid-propellant rocketry: What we do and do not know,” *35th Jt. Propuls. Conf. Exhib.*, 1999, doi: 10.2514/6.1999-2925.

[86] H. Yaman, V. Çelik, and E. Degirmenci, “Experimental investigation of the factors affecting the burning rate of solid rocket propellants,” *Fuel*, vol. 115, pp. 794–803, 2014, doi: 10.1016/j.fuel.2013.05.033.

[87] R. STEINBERGER and P. D. DRECHSEL, “Manufacture of Cast Double-Base Propellant,” pp. 1–28, 1969, doi: 10.1021/ba-1969-0088.ch001.

[88] F. A. William, “Combustion of Solid Propellants,” *Combust. Theory*, no. May, pp. 299–264, 2019, doi: 10.1201/9780429494055-7.

[89] D. A. Reese, L. J. Groven, and S. F. Son, “Formulation and characterization of a new nitroglycerin-free double base propellant,” *Propellants, Explos. Pyrotech.*, vol. 39, no. 2, pp. 205–210, 2014, doi: 10.1002/prep.201300105.

[90] W. MacCrehan and M. Bedner, “Investigating guns, bombs, and rockets: A new MIST reference material for smokeless powder measurements,” 2006.

[91] D. B. Kirchner, J. C. Gaydos, and M. C. Battigelli, “Chapter 10 Combustion Products of Propellants and Ammunition History of Propellants,” *Occup. Heal. Soldier Ind. Base*, pp. 359–396, 1993.

[92] Akerlof, “Formulation of Composite Propellant which has minimum signature(smokeless) Combustion Stability,” 1970 [Online]. Available: https://www.quickcompany.in/patents/formulation-of-composite-propellant-which-has-minimum-signature-smokeless-combustion-stability

[93] L. T. Deluca, “Innovative solid formulations for rocket propulsion,” *Eurasian Chem. J.*, vol. 18, no. 3, pp. 181–196, 2016, doi: 10.18321/ectj424.

[94] P.Kudryavtsev, “Production technology development and creation of production of additives used in solid rocket propellants,” *Sci. Isr. Technol. Advantages*, vol. 16, no. January, 2017.

[95] V. P. Sinditskii, V. Y. Egorshev, D. Tomasi, and L. T. Deluca, “Combustion mechanism of ammonium-nitrate-based propellants,” *J. Propuls. Power*, vol. 24, no. 5, pp. 1068–1078, 2008, doi: 10.2514/1.35233.

[96] K. W. Naumann, C. M. Rienäcker, A. Weigand, B. Gmbh, P. O. Box, and A. Inn, “Solid Rocket Motors with Particle-Free Composite Propellant at Solid Rocket Motors with Particle-Free Composite Propellant at Bayern-Chemie Outline • Introduction Particle-Free APC Composite Solid Propellants Particle-Free AN Composite Solid Propellants P,” 2015.

[97] C. Oommen and S. R. Jain, “Ammonium nitrate: A promising rocket propellant oxidizer,” *J. Hazard. Mater.*, vol. 67, no. 3, pp. 253–281, 1999, doi: 10.1016/S0304-3894(99)00039-4.

[98] S. N. Asthana, P. G. Shrotri, and H. Singh, “Studies on minimum signature nitramine based high energy propellant,” *J. Energ. Mater.*, vol. 10, no. 1, pp. 1–16, 1992, doi: 10.1080/07370659208018632.

[99] A. Korotkikh, O. Glotov, and I. Sorokin, “Effect of Metal Additives on the Combustion Characteristics of High-Energy Materials,” *MATEC Web Conf.*, vol. 72, 2016, doi: 10.1051/matecconf/20167201048.

[100] K. Bharadwajan and S. Vathsal, “Smokeless solid propellants performance in various grain configurations for a missile application,” *Int. J. Res. Eng. Appl. Sci. (IMPACT FACTOR – Int. J. Res. Eng. Appl. Sci.*, vol. 6573, no. 6, pp. 2249–3905, 2016, [Online]. Available: http://www.euroasiapub.org

[101] B. Selvaraj, N. V. Masthiraj, and S. R. D. Kumar, “Minimum-Signature (Smokeless) Propellant,” vol. 2, no. 4, pp. 599–604, 2012.

[102] A. Bolonkin, “New Small Cheap Rocket for Small Payload,” 2018.

[103] E. Landsem, T. L. Jensen, F. K. Hansen, E. Unneberg, and T. E. Kristensen, “Mechanical properties of smokeless composite rocket propellants based on prilled ammonium dinitramide,” *Propellants, Explos. Pyrotech.*, vol. 37, no. 6, pp. 691–698, 2012, doi: 10.1002/prep.201200004.

[104] Anon, *Smokeless Propellants.*, no. 391. 1986. doi: 10.2514/5.9781600865671.0841.0884.

[105] F. Lillo, B. D'Andrea, G. Marcelli, and A. Brotzu, "Burning Rate Assessment of Minimum Smoke Solid Propellants," *Rto-Mp-091*, no. September, pp. 37.1 – 37.9, 2002.

[106] V. Gettwert, M. Bohn, and V. Weiser, "Performance of ADN/GAP Propellants Compared to Al/AP/HTPB," *Insensitive Munitions Energ. Mater. Technol. Symp.*, no. May, pp. 18–21, 2015, [Online]. Available: http://www.imemg.org/wp-content/uploads/2015/06/4A1-17186-Performance-of-ADN-GAP-Propellants-Compared-to-Al-AP-HTPB.pdf

[107] H. Lips and K. Menke, "FOX7/GAP rocket propellants for a shoulder launched projectile," *Proc. - 27th Int. Symp. Ballist. Ballist. 2013*, vol. 1, pp. 59–69, 2013.

[108] T. L. Jensen, E. Unneberg, and T. E. Kristensen, "Smokeless GAP-RDX Composite Rocket Propellants Containing Diaminodinitroethylene (FOX-7)," *Propellants, Explos. Pyrotech.*, vol. 42, no. 4, pp. 381–385, 2017, doi: 10.1002/prep.201600278.

[109] Y. J. Yim *et al.*, "Infrared irradiance reduction in minimum smoke propellants by addition of potassium salt," *Propellants, Explos. Pyrotech.*, vol. 40, no. 1, pp. 74–80, 2015, doi: 10.1002/prep.201400172.

[110] M. C. Olde, F. A. Kuhnert, and J. Van Slingerland, "Laser Ignition and Combustion Study of KNO3-Sorbitol based Solid Propellant," *8Th Eur. Conf. Aeronaut. Aerosp. Sci.*, pp. 1–11, 2019, doi: 10.13009/EUCASS2019-583.

[111] C. J. Whelchel and E. T. Foster, "Smokeless propellant for Rocket Propulsion and Gas Generators".

[112] S. Touidjine *et al.*, "Preparation of ammonium nitrate-based solid composite propellants supplemented with polyurethane/nitrocellulose blends binder and their thermal decomposition behavior," *Def. Technol.*, no. xxxx, 2022, doi: 10.1016/j.dt.2021.12.013.

[113] K. Menke, "AN/PolyGLYN propellant formulation-approaches to less sensitive rocket propellants," 2006, pp. 1–22.

[114] G. P. Sutton, "Rocket Propulsion Elements," 6 th., Carlifonia: Wiley, 1992, ch. Rocket Exh, p. 549.

[115] Jacqueline Akhavan, “Combustion, Deflagration and Detonation,” in *The chemistry of explosives*, 3rd ed., Cambridge: Royal Society of Chemistry, 2011, ch. Deflagrati, pp. 60–72.

[116] S.Venugopalan, “Two faces of Explosion: Deflagration and Detonation,” in *Demystifying Explosives: Concept in High Energy Materials*, R.Sivabalan, Ed., Pune, India: Elsevier inc, 2015, ch. Deflagrati, pp. 52–69.

[117] H. J. Li *et al.*, “Influence of afterburning suppression on the combustion of solid propellants: Recent progress and outlook,” *J. Phys. Conf. Ser.*, vol. 1507, no. 2, 2020, doi: 10.1088/1742-6596/1507/2/022020.

[118] AGARD Advisory Report, “Terminology and Assessment Methods of Solid Propellants, Rocket Exhaust Signatures,” 1993, pp. 1–6.

[119] S.Venugopalan, “Energetics of Eenergetic Materials,” in *Demystifying Explosives: Concept in High Energy Materials*, R.Sivabalan, Ed., Pune, India: Elsevier inc, 2015, ch. Compostion, pp. 19–49.

[120] O. S. Olaoye and O. A. Abdulhafeez, “Design and Performance Characteristics of a Rocket Using Potassium Nitrate and Sucrose as Propellants,” *Int. J. Sci. Res.*, vol. 3, no. 8, pp. 1892–1897, 2014.

[121] Oyedeko K.F.K and Egwenu S. O, “Effect of magnesium metal in the characteristics performance of a sucrose-based solid rocket propellant,” *Glob. J. Eng. Technol. Adv.*, vol. 6, no. 2, pp. 051–060, 2021, doi: 10.30574/gjeta.2021.6.2.0016.

[122] L. Galfetti *et al.*, “Nanoparticles for solid rocket propulsion,” *J. Phys. Condens. Matter*, vol. 18, no. 33, 2006, doi: 10.1088/0953-8984/18/33/S15.

[123] Y. Bayat, F. Jozi, and T. Khanlari, “Synthesis and characterization of novel energetic plasticizers based on poly glycidyl nitrate,” *J. Energ. Mater.*, vol. 40, no. 2, pp. 153–169, 2022, doi: 10.1080/07370652.2020.1845874.

[124] Y. ru Li, H. Ren, X. zhou Wu, H. xin Wang, and X. long Yu, “Estimating energy release performance of oxidizer-activated aluminum fuel particles under ultrafast stimulus,” *Def. Technol.*, no. xxxx, 2022, doi: 10.1016/j.dt.2022.01.001.

[125] M. man Li, R. Hu, M. hui Xu, Q. lin Wang, and W. tao Yang, “Burning characteristics of high density foamed GAP/CL-20 propellants,” *Def. Technol.*, no. xxxx, 2021, doi: 10.1016/j.dt.2021.08.006.

[126] L. Zhang, Q. Lin, B. Cheng, P. Wang, M. Lu, and Q. Lin, “Effect of hexanitroethane (HNE) and hydrazinium nitroformate (HNF) on energy characteristics of composite solid propellants,” *FirePhysChem*, vol. 1, no. 2, pp. 116–122, 2021, doi: 10.1016/j.fpc.2021.05.003.

[127] G. M. H. J. L. Gadiot, J. M. Mul, J. J. Meulenbrugge, P. A. O. G. Korting, A. J. Schnorkh, and H. F. R. Schöyer, “New solid propellants based on energetic binders and HNF,” *Acta Astronaut.*, vol. 29, no. 10–11, pp. 771–779, 1993, doi: 10.1016/0094-5765(93)90158-S.

[128] D. E. G. Jones, Q. S. M. Kwok, M. Vachon, C. Badeen, and W. Ridley, “Characterization of ADN and ADN-based propellants,” *Propellants, Explos. Pyrotech.*, vol. 30, no. 2, pp. 140–147, 2005, doi: 10.1002/prep.200400096.

[129] J. de Flon, S. Andreasson, M. Liljedahl, C. Oscarson, M. Wanhatalo, and N. Wingborg, “Solid propellants based on ADN and HTPB,” *47th AIAA/ASME/SAE/ASEE Jt. Propuls. Conf. Exhib. 2011*, no. August, pp. 1–12, 2011, doi: 10.2514/6.2011-6136.

[130] Q. Lu *et al.*, “Advances in the molecular simulation and numerical calculations of the green high-energy oxidant ADN,” *Mater. Today Commun.*, vol. 31, no. May, p. 103699, 2022, doi: 10.1016/j.mtcomm.2022.103699.

[131] K. FUJISATO, H. HABU, H. SHIBAMOTO, X. YU, A. MIYAKE, and K. HORI, “Combustion Characteristics of ADN (Ammonium Dinitramide) Based Solid Propellants,” *Trans. Japan Soc. Aeronaut. Sp. Sci. Aerosp. Technol. Japan*, vol. 10, no. ists28, p. Pa_89-Pa_92, 2012, doi: 10.2322/tastj.10.pa_89.

[132] S.Venugopalan, “Ecofriendly Oxidizer,” in *Demystifying Explosives: Concept in High Energy Materials*, R.Sivabalan, Ed., Pune, India: Elsevier inc, 2015, ch. Trends and, pp. 202–203.

[133] J. de Oliveira Silva, K. P. Cardoso, J. R. Campos Silva, E. Y. Kawachi, M. Y. Nagamachi, and L. F. de Araujo Ferrão, “ADN Recrystallization and Microencapsulation with HTPB by Simple Coacervation,” *Propellants, Explos. Pyrotech.*, vol. 45, no. 5, pp. 705–713, 2020, doi: 10.1002/prep.201900392.

[134] T. Heintz and M. J. Herrmann, “Properties and Structure of ADN-Prills,” *Propellants, Explos. Pyrotech.*, vol. 44, no. 6, pp. 679–686, 2019, doi: 10.1002/prep.201800328.

[135] T. T. Vo, D. A. Parrish, and J. M. Shreeve, “Tetranitroacetimidic acid: A high oxygen oxidizer and potential replacement for ammonium perchlorate,” *J. Am. Chem. Soc.*, vol. 136, no. 34, pp. 11934–11937, 2014, doi: 10.1021/ja5074036.

[136] N. V. a. B. Latypov and U. B. Abraham Langlet, UIf Wellmar, “Synthesis and Reactions of of 1,1-Diamino-2,2-dinitroethylene,” *Heterocycles*, vol. 54, no. 98, pp. 1573–1582, 1998.

[137] H. Gao and J. M. Shreeve, “The many faces of FOX-7: A precursor to high-Performance energetic materials,” *Angew. Chemie - Int. Ed.*, vol. 54, no. 21, pp. 6335–6338, 2015, doi: 10.1002/anie.201501973.

[138] B. Florczak, “Investigation of an Aluminized Binder/AP Composite Propellant Containing FOX-7,” *Cent. Eur. J. Energ. Mater.*, vol. 5, pp. 3–4, 2008.

[139] B. Florczak, “A Comparison of Properties of Aluminized Composite Propellants Containing HMX and FOX-7,” *Cent. Eur. J. Energ. Mater.*, vol. 5, pp. 3–4, 2008.

[140] D. B. Lempert, E. M. Dorofeenko, and Y. Shu, “Energy potential of solid composite propellants based on 1,1-diamino-2,2-dinitroethylene,” *Russ. J. Phys. Chem. B*, vol. 10, no. 3, pp. 483–489, 2016, doi: 10.1134/S1990793116030258.

[141] W. A. Goddard, “Energetic materials,” *Springer Ser. Mater. Sci.*, vol. 284, pp. 1193–1201, 2021, doi: 10.1007/978-3-030-18778-1_60.

[142] J. J. Sabatini and E. C. Johnson, “A short review of nitric esters and their role in energetic materials,” *ACS Omega*, vol. 6, no. 18, pp. 11813–11821, 2021, doi: 10.1021/acsomega.1c01115.

[143] J. Wang, L. Yang, W. Zheng, and J. Zhang, “Study on Comparative Performance of CL-20/RDX-based CMDB Propellants,” *Propellants, Explos. Pyrotech.*, vol. 44, no. 9, pp. 1175–1182, 2019, doi: 10.1002/prep.201900029.

[144] T.L Varghese, “CL-20,” in *The Chemistry and Technology of Solid Rocket Propellants(A Treatise on Solid Propellants)*, New Dehli: Allied Publisher Pvt. LTD, 2017, ch. Advanced S, p. 177.

[145] M. D. Ruesch *et al.*, “Burning rate and flame structure of cocrystals of CL-20 and a polycrystalline composite crystal of HMX/AP,” *Combust. Flame*, vol. 219, pp. 129–135, 2020, doi: 10.1016/j.combustflame.2020.04.009.

[146] O. Kositsyna, K. Varlan, M. Dron, and O. Kulyk, “Determining Energetic Characteristics and Selecting Environmentally Friendly Components for Solid Rocket Propellants At the Early Stages of Design,” *Eastern-European J. Enterp.*

Technol., vol. 6, no. 6(114), pp. 6–14, 2021, doi: 10.15587/1729-4061.2021.247233.

[147] A. E. D. M. Van Der Heijden, H. L. J. Keizers, and W. H. . Welland-Veltsmans, "HNF : Oxidiser for High Performance , Low-Signature Propellants," *Adv. Rocket Perform. Life Disposal, 23-26 Sept.*, no. September, pp. 23–26, 2002.

[148] H. S. Jadhav, M. B. Talawar, D. D. Dhavale, S. N. Asthana, and V. N. Krishnamurthy, "Synthesis, characterization and thermal behavior of hydrazinium nitroformate (HNF) and its new N-alkyl substituted derivatives," *Indian J. Chem. Technol.*, vol. 12, no. 2, pp. 187–192, 2005.

[149] P. S. Dendage, D. B. Sarwade, A. B. Mandale, and S. N. Asthana, "Characterization and thermal analysis of hydrazinium nitroformate (hnf)," *J. Energ. Mater.*, vol. 21, no. 3, pp. 167–183, 2003, doi: 10.1080/716100384.

[150] J. Louwers, G. M. H. L. Gadiot, A. J. Landman, T. W. Peeters, T. H. Van Der Meer, and D. Roekaerts, "Combustion and flame structure of hnf sandwiches and propellants," *35th Jt. Propuls. Conf. Exhib.*, no. June, 1999, doi: 10.2514/6.1999-2359.

[151] A. E. D. M. van der Heijden and A. B. Leeuwenburgh, "HNF/HTPB propellants: Influence of HNF particle size on ballistic properties," *Combust. Flame*, vol. 156, no. 7, pp. 1359–1364, 2009, doi: 10.1016/j.combustflame.2009.03.007.

[152] J. Athar, M. Ghosh, P. S. Dendage, R. S. Damse, and A. K. Sikder, "Nanocomposites: An ideal coating material to reduce the sensitivity of hydrazinium nitroformate (HNF)," *Propellants, Explos. Pyrotech.*, vol. 35, no. 2, pp. 153–158, 2010, doi: 10.1002/prep.200900024.

[153] V. E. Zarko, V. N. Simonenko, G. I. Anisiforov, and A. V. Aparin, "Combustion characterization of hydrazinium nitrate/energetic binder/Alex based model propellants," *Aerosp. Sci. Technol.*, vol. 11, no. 1, pp. 13–17, 2007, doi: 10.1016/j.ast.2006.07.004.

[154] T. M. Klapötke and M. Suceska, "Theoretical evaluation of TKX-50 as an ingredient in rocket propellants," *Zeitschrift fur Anorg. und Allg. Chemie*, vol. 647, no. 5, pp. 572–574, 2021, doi: 10.1002/zaac.202100053.

[155] M. López-López, J. L. Ferrando, and C. García-Ruiz, "Comparative analysis of smokeless gunpowders by Fourier transform infrared and Raman spectroscopy," *Anal. Chim. Acta*, vol. 717, pp. 92–99, 2012, doi: 10.1016/j.aca.2011.12.022.

[156] H. Shekhar, “Parametric Studies on Star Port Propellant Grain For Ballistic Evaluation,” *DefenceScience J.*, vol. 55, no. 4, pp. 459–469, 2005.

[157] T. K, V. V. Rao, and Y. P. Setty, “Observation in Ballistic Evaluation Motor Static Firing: Cracking in Graphite Nozzle,” *Int. J. Sci. Eng. Res.*, vol. 8, no. 10, pp. 1441–1446, 2017, doi: 10.14299/ijser.2017.10.007.

[158] P. Thakre and V. Yang, “Chemical erosion of carbon-carbon/graphite nozzles in solid-propellant rocket motors,” *J. Propuls. Power*, vol. 24, no. 4, pp. 822–833, 2008, doi: 10.2514/1.34946.

[159] J. Terzic, B. Zecevic, A. Catovic, and S. Serdarevic-Kadic, “Prediction of Internal Ballistic Parameters of Solid Propellant Rocket Motors,” *Mech. Eng.*, vol. 4, no. January, pp. 6–8, 2010.

[160] K. Ramesh, S. N. Jawalkar, S. Sachdeva, Mehilal, and B. Bhattacharya, “Development of a composite propellant formulation with a high performance index using a pressure casting technique,” *Cent. Eur. J. Energ. Mater.*, vol. 9, no. 1, pp. 49–58, 2012.

[161] Y. R. Alexander, N. Rathi, and P. A. Ramakrishna, “Development of high burn rate propellant and testing in miniature rocket motor for control applications,” *FirePhysChem*, no. May, pp. 1–7, 2023, doi: 10.1016/j.fpc.2023.05.001.

[162] V. Gettwert and S. Fischer, “Small scale motor tests of ADN/GAP based propellants,” *Fraunhofer-Institut für Chem. Technol. (International Annu. Conf.* , 2015.

[163] C. He and J. M. Shreeve, “Potassium 4,5-Bis(dinitromethyl)furoxanate: A Green Primary Explosive with a Positive Oxygen Balance,” *Angew. Chemie*, vol. 128, no. 2, pp. 782–785, 2016, doi: 10.1002/ange.201509209.

[164] M. H. Keshavarz, Y. H. Abadi, K. Esmaeilpour, S. Damiri, and M. Oftadeh, “A Novel Class of Nitrogen-rich Explosives Containing High Oxygen Balance to Use as High Performance Oxidizers in Solid Propellants,” *Propellants, Explos. Pyrotech.*, vol. 42, no. 10, pp. 1155–1160, 2017, doi: 10.1002/prep.201700139.

[165] S.Venugopalan, “Oxygen Balance,” in *Demystifying Explosives: Concept in High Energy Materials*, R. Sivabalan, Ed., Pune, India, 2015, ch. Energetic, p. 31.

[166] T. Miyazaki and N. Kubota, “Energetics of BAMO,” *Propellants, Explos. Pyrotech.*, vol. 17, no. 1, pp. 5–9, 1992, doi: 10.1002/prep.19920170103.

[167] Y. Wada *et al.*, “Combustion mechanism of tetra-ol glycidyl azide polymer,” *Sci. Technol. Energ. Mater.*, vol. 69, no. 5–6, pp. 143–148, 2008.

[168] H. Muthurajan, R. Sivabalan, M. B. Talawar, M. Anniyappan, and S. Venugopalan, “Prediction of heat of formation and related parameters of high energy materials,” *J. Hazard. Mater.*, vol. 133, no. 1–3, pp. 30–45, 2006, doi: 10.1016/j.jhazmat.2005.10.009.

[169] P. Ravi, S. P. Tewari, and R. Ramaswamy, “A DFT study on the structures and energies of isomers of 4-amino-1,3-dinitro-1,2,4-triazol-5-one-2-oxide: New high energy density compounds,” *Propellants, Explos. Pyrotech.*, vol. 38, no. 3, pp. 425–432, 2013, doi: 10.1002/prep.201200175.

[170] T. Ecker, S. Karl, and K. Hannemann, “Combustion Modeling in Solid Rocket Motor Plumes,” *EUCASS 2019 Proc.*, pp. 5–9, 2019, doi: 10.13009/EUCASS2019-79.

[171] N. Wingborg, “Heat of Formation of ADN-Based Liquid Monopropellants,” *Propellants, Explos. Pyrotech.*, vol. 44, no. 9, pp. 1090–1095, 2019, doi: 10.1002/prep.201900142.

[172] H. Muthurajan, R. Sivabalan, M. B. Talawar, S. Venugopalan, and B. R. Gandhe, “Computer code for the optimization of performance parameters of mixed explosive formulations,” *J. Hazard. Mater.*, vol. 136, no. 3, pp. 475–481, 2006, doi: 10.1016/j.jhazmat.2006.01.066.

[173] A. Alipoor and K. Mazaheri, “The Optimization of Detonation Properties in Gaseous Mixtures and Mixed Explosive Materials,” *J. Energ. Mater.*, vol. 31, no. 2, pp. 115–126, 2013, doi: 10.1080/07370652.2011.616870.

[174] Q. Yu *et al.*, “A study of N-trinitroethyl-substituted aminofurazans: High detonation performance energetic compounds with good oxygen balance,” *J. Mater. Chem. A*, vol. 3, no. 15, pp. 8156–8164, 2015, doi: 10.1039/c4ta06974a.

[175] Q. Yu *et al.*, “Pushing the Limits of Oxygen Balance in 1,3,4-Oxadiazoles,” *J. Am. Chem. Soc.*, vol. 139, no. 26, pp. 8816–8819, 2017, doi: 10.1021/jacs.7b05158.

[176] Q. Sun, X. Li, Q. Lin, and M. Lu, “Dancing with 5-substituted monotetrazoles, oxygen-rich ions, and silver: Towards primary explosives with positive oxygen balance and excellent energetic performance,” *J. Mater. Chem. A*, vol. 7, no. 9, pp. 4611–4618, 2019, doi: 10.1039/c8ta12506f.

[177] H. J. Kim, B. H. Lee, S. G. Cho, and S. K. Lee, “Comparative analysis of detonation velocity in determining product composition for high energetic molecules using stoichiometric rules,” *Anal. Sci. Technol.*, vol. 30, no. 6, pp. 405–410, 2017.

[178] N.Javadi, “Estimation of Velocity and Detonation Pressure of some High Energy Compounds using Kamlet Method based on Kistiakowski-Wilson Rules,” *J. Energ. Mater.*, vol. 13, 2019.

[179] T. Paraschiv, T. V. Tiganescu, G. O. Iorga, R. E. Ginghina, and O. C. Grigoroiu, “Experimental and theoretical study on three combustion models for the determination of the performance parameters of nitrocellulose - based propellants,” *Rev. Chim.*, vol. 70, no. 9, pp. 87–97, 2020, doi: 10.37358/RC.20.9.8320.

[180] Y. Yu, J. Chen, R. Zhang, Y. Li, and S. Pang, “Novel [NF2O]+ and [N3NFO]+-based energetic oxidizers for solid propellants with super high specific impulse,” *RSC Adv.*, vol. 7, no. 38, pp. 23709–23713, 2017, doi: 10.1039/c7ra03304d.

[181] S. V. Bondarchuk, “Modeling of explosives: 1,4,2,3,5,6-dioxatetrazinane as a new green energetic material with enhanced performance,” *J. Phys. Chem. Solids*, vol. 142, no. March, p. 109458, 2020, doi: 10.1016/j.jpcs.2020.109458.

[182] P. Politzer and J. S. Murray, “The role of product composition in determining detonation velocity and detonation pressure,” *Cent. Eur. J. Energ. Mater.*, vol. 11, no. 4, pp. 459–474, 2014.

[183] W. Tomaszewski, K. Cieślak, and A. Zygmunt, “Influence of processing solvents on decomposition of nitrocellulose in smokeless powders studied by heat flow calorimetry,” *Polym. Degrad. Stab.*, vol. 111, pp. 169–175, 2015, doi: 10.1016/j.polymdegradstab.2014.11.010.

[184] V. V. Boldyrev, “Thermal decomposition of ammonium perchlorate,” *Thermochim. Acta*, vol. 443, no. 1, pp. 1–36, 2006, doi: 10.1016/j.tca.2005.11.038.

[185] M. Rodríguez-Pesina, J. García-Domínguez, F. García-Hernández, L. M. Flores-Vélez, and O. Domínguez, “The Thermal Decomposition of Ammonium Perchlorate-Aluminum Propellants in Presence of Metallic Zinc Particles,” *Mater. Sci. Appl.*, vol. 08, no. 06, pp. 436–447, 2017, doi: 10.4236/msa.2017.86030.

[186] Z. Chai, B. Jin, W. Gong, R. Peng, and S. Chu, “Synthesis and thermal performance study of C60-polyglycidyl nitrate (C60-PGN) maleic acid copolymer lead salts,” *Fullerenes Nanotub. Carbon Nanostructures*, vol. 26, no. 12, pp. 880–886, 2018, doi: 10.1080/1536383X.2018.1531284.

[187] Y. H. Wang, L. L. Liu, L. Y. Xiao, and Z. X. Wang, “Thermal decomposition of HTPB/AP and HTPB/HMX mixtures with low content of oxidizer,” *J. Therm. Anal. Calorim.*, vol. 119, no. 3, pp. 1673–1678, 2015, doi: 10.1007/s10973-014-4324-z.

[188] X. Xue, Y. Yu, and R. Ye, “Unsteady chemical kinetics behavior of AP/HTPB propellant with micro-scale model,” *Combust. Sci. Technol.*, vol. 190, no. 12, pp. 2164–2187, 2018, doi: 10.1080/00102202.2018.1494594.

[189] L. ke Zhang and X. yang Zheng, “Experimental study on thermal decomposition kinetics of natural ageing AP/HTPB base bleed composite propellant,” *Def. Technol.*, vol. 14, no. 5, pp. 422–425, 2018, doi: 10.1016/j.dt.2018.04.007.

[190] R. F. B. Gonçalves, R. P. Silva, J. A. F. F. Rocco, and K. Iha, “Thermal decomposition kinetics of aged solid propellant based on ammonium perchlorate - AP/HTPB binder,” in *44th AIAA/ASME/SAE/ASEE Joint Propulsion Conference and Exhibit*, 2008. doi: 10.5772/52109.

[191] G. Lei *et al.*, “New Energetic Complexes as Catalysts for Ammonium Perchlorate Thermal Decomposition,” *Chinese J. Chem.*, vol. 39, no. 5, pp. 1193–1198, 2021, doi: 10.1002/cjoc.202000672.

[192] P. W. M. Jacobs and H. M. Whitehead, “Decomposition and combustion of ammonium perchlorate,” *Chem. Rev.*, vol. 69, no. 4, pp. 551–590, 1969, doi: 10.1021/cr60260a005.

[193] S. Chaturvedi and P. N. Dave, “A review on the use of nanometals as catalysts for the thermal decomposition of ammonium perchlorate,” *J. Saudi Chem. Soc.*, vol. 17, no. 2, pp. 135–149, 2013, doi: 10.1016/j.jscs.2011.05.009.

[194] J. Wang *et al.*, “Enhanced thermal decomposition properties of ammonium perchlorate through addition of 3DOM core-shell Fe2O3/Co3O4 composite,” *J. Alloys Compd.*, vol. 724, pp. 720–727, 2017, doi: 10.1016/j.jallcom.2017.07.033.

[195] X. Han *et al.*, “Thermal decomposition of ammonium perchlorate based mixture with fullerenes,” *J. Therm. Anal. Calorim.*, vol. 91, no. 2, pp. 551–557, 2008, doi: 10.1007/s10973-007-8290-6.

[196] S. Chaturvedi, P. N. Dave, and N. N. Patel, “Thermal decomposition of AP/HTPB propellants in presence of Zn nanoalloys,” *Appl. Nanosci.*, vol. 5, no. 1, pp. 93–98, 2015, doi: 10.1007/s13204-014-0296-3.

[197] L. E. N. Almeida, A. F. Martins, S. R. Gomes, and F. A. Cunha, “Thermal decomposition kinetics studies of ammonium perchlorate-htpb /al/ap solid propellants formulated with iron oxide burning rate catalyst in nano and micro scale.,” *49th AIAA/ASME/SAE/ASEE Jt. Propuls. Conf.*, vol. 1 PartF, pp. 1–18, 2013, doi: 10.2514/6.2013-4088.

[198] E. D. Tolmachoff and J. T. Essel, “Evidence and modeling of heterogeneous reactions of low temperature ammonium perchlorate decomposition,” *Combust. Flame*, vol. 200, pp. 316–324, 2019, doi: 10.1016/j.combustflame.2018.11.030.

[199] I. P. S. Kapoor, P. Srivastava, and G. Singh, “Nanocrystalline transition metal oxides as catalysts in the thermal decomposition of ammonium perchlorate,” *Propellants, Explos. Pyrotech.*, vol. 34, no. 4, pp. 351–356, 2009, doi: 10.1002/prep.200800025.

[200] A. Manash and P. Kumar, “Comparison of burn rate and thermal decomposition of AP as oxidizer and PVC and HTPB as fuel binder based composite solid propellants,” *Def. Technol.*, vol. 15, no. 2, pp. 227–232, 2019, doi: 10.1016/j.dt.2018.08.010.

[201] P. Zhai, C. Shi, S. Zhao, W. Liu, W. Wang, and L. Yao, “Thermal decomposition of ammonium perchlorate-based molecular perovskite from TG-DSC-FTIR-MS and: Ab initio molecular dynamics,” *RSC Adv.*, vol. 11, no. 27, pp. 16388–16395, 2021, doi: 10.1039/d0ra10559g.

[202] Y. Wang, X. Song, and F. Li, “Thermal Behavior and Decomposition Mechanism of Ammonium Perchlorate and Ammonium Nitrate in the Presence of Nanometer Triaminoguanidine Nitrate,” *ACS Omega*, vol. 4, no. 1, pp. 214–225, 2019, doi: 10.1021/acsomega.8b02515.

[203] A. V. Sergienko *et al.*, “Burning Characteristics of the HMX/CL-20/AP/Polyvinyltetrazole Binder/Al Solid Propellants Loaded with Nanometals,” *Propellants, Explos. Pyrotech.*, vol. 44, no. 2, pp. 217–223, 2019, doi: 10.1002/prep.201800204.

[204] M. B. Padwal and M. Varma, “Thermal decomposition and combustion characteristics of HTPB-coarse AP composite solid propellants catalyzed with

Fe2O3," *Combust. Sci. Technol.*, vol. 190, no. 9, pp. 1614–1629, 2018, doi: 10.1080/00102202.2018.1460599.

[205] M. K. Bharti, L. Bansal, and S. Chalia, "Catalytic potential of microsized additives in enhancing the linear burn rate of potassium nitrate-sucrose based composite solid propellant strands," *Int. J. Energ. Mater. Chem. Propuls.*, vol. 20, no. 4, pp. 61–77, 2021, doi: 10.1615/IntJEnergeticMaterialsChemProp.2021038209.

[206] K. Gańczyk-Specjalska, R. Bogusz, R. Lewczuk, and P. Magnuszewska, "Estimation of Thermodynamic and Thermochemical Properties of Solid Heterogeneous Rocket Propellants Containing Scavengers," *Probl. Tech. Uzbroj.*, vol. 150, no. 2, pp. 59–70, 2019, doi: 10.5604/01.3001.0013.5897.

[207] J. A. Vara, P. N. Dave, and S. Chaturvedi, "The catalytic activity of transition metal oxide nanoparticles on thermal decomposition of ammonium perchlorate," *Def. Technol.*, vol. 15, no. 4, pp. 629–635, 2019, doi: 10.1016/j.dt.2019.04.002.

[208] P. Zhang, L. Sun, J. Yuan, and J. Deng, "Synthesis of multifunctional additives for solid propellants: Structure, properties and mechanism," *Def. Technol.*, no. xxxx, 2023, doi: 10.1016/j.dt.2023.06.009.

[209] V. Rodić, M. Bogosavljević, S. Brzić, and B. Fidanovski, "Possibility of aluminium/magnesium exchange in composite rocket propellants," *Sci. Tech. Rev.*, vol. 68, no. 1, pp. 40–49, 2018, doi: 10.5937/str1801040r.

[210] R. W. Armstrong and W. L. Elban, "Materials science and technology aspects of energetic (explosive) materials," *Mater. Sci. Technol.*, vol. 22, no. 4, pp. 381–395, 2006, doi: 10.1179/174328406X84049.

[211] A. Göçmez, C. Erişken, Ü. Yilmazer, F. Pekel, and S. Özkar, "Mechanical and burning properties of highly loaded composite propellants," *J. Appl. Polym. Sci.*, vol. 67, no. 8, pp. 1457–1464, 1998, doi: 10.1002/(sici)1097-4628(19980222)67:8<1457::aid-app11>3.3.co;2-i.

[212] T. Sun, J. J. Xiao, Q. Liu, F. Zhao, and H. M. Xiao, "Comparative study on structure, energetic and mechanical properties of a ε-CL-20/HMX cocrystal and its composite with molecular dynamics simulation," *J. Mater. Chem. A*, vol. 2, no. 34, pp. 13898–13904, 2014, doi: 10.1039/c4ta01150c.

[213] O. G. Glotov and V. E. Zarko, "Condensed combustion products of aluminized propellants," *Zhongguo Hangkong Taikong Xuehui Huikan/Transactions*

Aeronaut. Astronaut. Soc. Repub. China, vol. 34, no. 3, pp. 247–256, 2002, doi: 10.6124/TAASRC.200209.

[214] W. Li, J. Ma, S. Wu, J. Zhang, and J. Cheng, "The effect of hydrogen bond on the thermal and mechanical properties of furan epoxy resins: Molecular dynamics simulation study," *Polym. Test.*, vol. 101, no. June, p. 107275, 2021, doi: 10.1016/j.polymertesting.2021.107275.

[215] N. Rohaiza Izamuddin *et al.*, "The effect of different weight percentage of sabitol on the tensile properties of Tss," 2022.

[216] A. M. A. Elall and G. Lin, "Effect of bonding agent production materials on the mechanical properties of composite solid rocket propellant," *MATEC Web Conf.*, vol. 189, 2018, doi: 10.1051/matecconf/201818908001.

[217] A. Yadav, C. S. Pant, and S. Das, "Research Advances in Bonding Agents for Composite Propellants," *Propellants, Explos. Pyrotech.*, vol. 45, no. 5, pp. 695–704, 2020, doi: 10.1002/prep.201900329.

[218] D. A. Reese, "Combustion characteristics of SMX and SMX based propellants," 2014.

[219] D. Liang, J. Liu, J. Xiao, J. Xi, Y. Wang, and J. Zhou, "Effect of metal additives on the composition and combustion characteristics of primary combustion products of B-based propellants," *J. Therm. Anal. Calorim.*, vol. 122, no. 1, pp. 497–508, 2015, doi: 10.1007/s10973-015-4750-6.

[220] Q. NIU, Z. HE, and S. DONG, "IR radiation characteristics of rocket exhaust plumes under varying motor operating conditions," *Chinese J. Aeronaut.*, vol. 30, no. 3, pp. 1101–1114, 2017, doi: 10.1016/j.cja.2017.04.003.

[221] J. C. Thomas, A. R. Demko, T. E. Sammet, D. L. Reid, S. Seal, and E. L. Petersen, "Mechanical Properties of Composite AP/HTPB Propellants Containing Novel Titania Nanoparticles," *Propellants, Explos. Pyrotech.*, vol. 41, no. 5, pp. 822–834, 2016, doi: 10.1002/prep.201600090.

[222] A. Dejeaifve, A. Sarbach, B. Roduit, P. Folly, and R. Dobson, "Making Progress Towards »Green« Propellants – Part II," *Propellants, Explos. Pyrotech.*, vol. 45, no. 8, pp. 1185–1193, 2020, doi: 10.1002/prep.202000059.

[223] K. Wang *et al.*, "Molecular dynamic simulation of performance of modified BAMO / AMMO copolymers and their effects on mechanical properties of energetic materials," *Sci. Rep.*, pp. 1–18, 2020, doi: 10.1038/s41598-020-75146-x.

[224] H. Singh, “Survey of New Energetic and Eco-friendly,” in *Chemical Rocket Propulsion*, Berlin: Springer International Publishing, 2017, ch. A Comprehe, pp. 127-138. doi: 10.1007/978-3-319-27748-6.

[225] P. Kumar, M. Kumar, and R. Lakra, “Effect of catalysts on the burning rate of phase stabilized ammonium nitrate based composite propellants,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 455, no. 1, 2018, doi: 10.1088/1757-899X/455/1/012022.

[226] U. Vellaisamy and S. Biswas, “Effect of metal additives on neutralization and characteristics of AP/HTPB solid propellants,” *Combust. Flame*, vol. 221, pp. 326–337, 2020, doi: 10.1016/j.combustflame.2020.08.006.

[227] C. Dennis and B. Bojko, “On the combustion of heterogeneous AP/HTPB composite propellants: A review,” *Fuel*, vol. 254, no. April, p. 115646, 2019, doi: 10.1016/j.fuel.2019.115646.

[228] G. L. Pellett, D. I. Sebacher, R. J. Bendura, and D. E. Wornom, “HCI in rocket exhaust clouds: Atmospheric dispersion, acid aerosol characteristics, and acid rain deposition,” *J. Air Pollut. Control Assoc.*, vol. 33, no. 4, pp. 304–311, 1983, doi: 10.1080/00022470.1983.10465578.

[229] C. Paravan *et al.*, “Reduction of Solid Propellants Environmental Impact: Al-Mg Mechanically Activated Fuels and AP-PSAN Blends,” in *9TH EUROPEAN CONFERENCE FOR AERONAUTICS AND AEROSPACE SCIENCES (EUCASS)*, 2022.

[230] F. Chalghoum, D. Trache, M. Benziane, and A. Benhammada, “Effect of micro- and nano-CuO on the thermal decomposition kinetics of high-performance aluminized composite solid propellants containing complex metal hydrides,” *FirePhysChem*, vol. 2, no. 1, pp. 36–49, 2022, doi: 10.1016/j.fpc.2022.03.007.

[231] D. A.Flanigan, “INVESTIGATION OF THE MECHANISM OF SOLID PROPELLANT BURN RATE,” 1967.

[232] T. Sippel, “Experimental and theoretical investigation of organically-capped , nanoscale alkali metal hydride and aluminum particles as solid propellant additives by Adam Lawrence A thesis submitted to the graduate faculty in partial fulfillment of the requirements f,” 2018.

[233] F. Chalghoum, D. Trache, F. Maggi, and M. Benziane, “Effect of Complex Metal Hydrides on the Elimination of Hydrochloric Acid Exhaust Products from High-Performance Composite Solid Propellants: A Theoretical Analysis,”

Propellants, Explos. Pyrotech., vol. 45, no. 8, pp. 1204–1215, 2020, doi: 10.1002/prep.201900408.

[234] M. D. Ruesch, G. C. Mathews, R. J. Tancin, S. F. Son, and C. S. Goldenstein, "Wavelength-modulation spectroscopy in the mid-infrared for temperature and HCl measurements in aluminum-lithium composite-propellant flames," *Combust. Flame*, vol. 242, p. 112180, 2022, doi: 10.1016/j.combustflame.2022.112180.

[235] J. H. Hansen, "SOLID PROPELLANT FORMULATIONS PROUDCING AND NEUTRALIZING EXHAUST," 1994

[236] U. Vellaisamy and S. Biswas, "Effect of metal additives on neutralization and characteristics of AP / HTPB solid propellants," *Combust. Flame*, vol. 221, pp. 326–337, 2020, doi: 10.1016/j.combustflame.2020.08.006.

[237] E. M. Youssef, T. Elshenawy, H. E. Mostafa, M. Radwan, and A. Elbeih, "Optimization of low signature base bleed propellant formulations," in *IOP Conference Series: Materials Science and Engineering*, 2019. doi: 10.1088/1757-899X/610/1/012003.

[238] R. Nakka, "KNSB Propellant," https://www.nakka-rocketry.net/sorb.html#Mechanical. [Online]. Available: https://www.nakka-rocketry.net/sorb.html

[239] S. R. Jain, K. C. Adiga, and V. R. Pai Verneker, "A new approach to thermochemical calculations of condensed fuel-oxidizer mixtures," *Combust. Flame*, vol. 40, no. C, pp. 71–79, 1981, doi: 10.1016/0010-2180(81)90111-5.

[240] S. R. Jain, "Energetics of Propellants, Fuels and Explosives; a chemical valence approach," *Propellants, Explos. Pyrotech.*, vol. 12, no. 6, pp. 188–195, 1987, doi: 10.1002/prep.19870120603.

[241] T.L Varghese, "Composite Propellant-Ingredients and their Function," in *The Chemistry and Technology of Solid Rocket Propellants(A Treatise on Solid Propellants)*, New Dehli: Allied Publisher Pvt. LTD, 2017, ch. Oxidizer, pp. 20–55.

[242] Zachary T. Fondren, "Thermodynamics and Kinetics of Phase Changes in Energetic Materials," Texas Tech University, 2020. [Online]. Available: https://www.ptonline.com/articles/how-to-get-better-mfi-results

[243] I. Sochet, "Blast effects of external explosions To cite this version : HAL Id : hal-00629253," *Hal.Archives*, 2011.

[244] S. ying Li, Y. Li, Y. jun Ding, H. Liang, and Z. liang Xiao, “One-step green method to prepare progressive burning gun propellant through gradient denitration strategy,” *Def. Technol.*, vol. 22, pp. 135–143, 2023, doi: 10.1016/j.dt.2021.11.009.

[245] D. Frem, “Estimating the metal acceleration ability of high explosives,” *Def. Technol.*, vol. 16, no. 1, pp. 225–231, 2020, doi: 10.1016/j.dt.2019.07.002.

[246] S. Jain, G. Gupta, D. R. Kshirsagar, V. H. Khire, and B. Kandasubramanian, “Burning rate and other characteristics of strontium titanate (SrTiO3) supplemented AP/HTPB/Al composite propellants,” *Def. Technol.*, vol. 15, no. 3, pp. 313–318, 2019, doi: 10.1016/j.dt.2018.10.004.

[247] G. Singh and S. P. Felix, “Studies of energetic compounds, part 29: Effect of NTO and its salts on the combustion and condensed phase thermolysis of composite solid propellants, HTPB-AP,” *Combust. Flame*, vol. 132, no. 3, pp. 422–432, 2003, doi: 10.1016/S0010-2180(02)00479-0.

[248] M. Sharma, V. Sharma, and P. Kala, “Optimization of process variables to improve the mechanical properties of FDM structures,” *J. Phys. Conf. Ser.*, vol. 1240, no. 1, 2019, doi: 10.1088/1742-6596/1240/1/012061.

[249] W. Brostow, H. Fałtynowicz, O. Gencel, A. Grigoriev, H. E. Hagg Lobland, and D. Zhang, “Mechanical and Tribological Properties of Polymers and Polymer-Based Composites,” *Chem. Chem. Technol.*, vol. 14, no. 4, pp. 514–520, 2020, doi: 10.23939/chcht14.04.514.

[250] L. V. Kakumanu, N. Yadav, and S. Karmakar, “Combustion Study of Composite Solid Propellants Containing Metal Phthalocyanines,” *Int. J. Aerosp. Sci.*, vol. 3, no. 2, pp. 31–36, 2014, doi: 10.5923/j.aerospace.20140302.01.

[251] S. Chen *et al.*, “Innovative methods to enhance the combustion properties of solid fuels for hybrid rocket propulsion,” *Aerospace*, vol. 6, no. 4, 2019, doi: 10.3390/aerospace6040047.

[252] G. F. Adams and R. W. Shaw, “CHEMICAL REACTIONS IN ENERGETIC MATERIALS,” *Phys. Chem*, vol. 43, p. 311, 1992.

[253] J. A. Vara, P. N. Dave, and S. Chaturvedi, “Investigating Catalytic Properties of Nanoferrites for Both AP and Nano-AP Based Composite Solid Propellant,” *Combust. Sci. Technol.*, vol. 193, no. 13, pp. 2290–2304, 2021, doi: 10.1080/00102202.2020.1734582.

[254] M. Rajić and M. Sućeska, “Study of thermal decomposition kinetics of low-temperature reaction of ammonium perchlorate by isothermal TG,” *J. Therm. Anal. Calorim.*, vol. 63, no. 2, pp. 375–386, 2000, doi: 10.1023/A:1010136308310.

[255] Z. He, Z. Xia, J. Hu, L. Ma, and Y. Li, “Thermal decomposition and kinetics of electrically controlled solid propellant through thermogravimetric analysis,” *J. Therm. Anal. Calorim.*, vol. 139, no. 3, pp. 2187–2195, 2020, doi: 10.1007/s10973-019-08608-8.

[256] M. Fathollahi, B. Mohammadi, and J. Mohammadi, “Kinetic investigation on thermal decomposition of hexahydro-1,3,5-trinitro- 1,3,5-triazine (RDX) nanoparticles,” *Fuel*, vol. 104, pp. 95–100, 2013, doi: 10.1016/j.fuel.2012.09.075.

[257] D. Zhang, L. Jiang, S. Lu, C. Y. Cao, and H. P. Zhang, “Particle size effects on thermal kinetics and pyrolysis mechanisms of energetic 5-amino-1h-tetrazole,” *Fuel*, vol. 217, no. July 2017, pp. 553–560, 2018, doi: 10.1016/j.fuel.2017.12.052.

[258] M. J. Starink, “The determination of activation energy from linear heating rate experiments: A comparison of the accuracy of isoconversion methods,” *Thermochim. Acta*, vol. 404, no. 1–2, pp. 163–176, 2003, doi: 10.1016/S0040-6031(03)00144-8.

[259] J. Zhang, L. Chen, J. Yang, C. Bian, and W. He, “Thermal behaviors, thermal decomposition mechanism, kinetic model analysis and thermal hazard prediction of 3,6,7-triamino-7H-[1,2,4]triazolo[4,3-b][1,2,4]triazole (TATOT),” *Thermochim. Acta*, vol. 724, no. April, p. 179515, 2023, doi: 10.1016/j.tca.2023.179515.

[260] C. P. Lin, Y. M. Chang, J. P. Gupta, and C. M. Shu, “Comparisons of TGA and DSC approaches to evaluate nitrocellulose thermal degradation energy and stabilizer efficiencies,” *Process Saf. Environ. Prot.*, vol. 88, no. 6, pp. 413–419, 2010, doi: 10.1016/j.psep.2010.07.004.

[261] X. L. Liu, S. Q. Hu, L. L. Liu, and Y. Zhang, “Condensed Combustion Products Characteristics of HTPB/AP/Al Propellants under Solid Rocket Motor Conditions,” *Aerospace*, vol. 9, no. 11, 2022, doi: 10.3390/aerospace9110677.

[262] S. Carlotti and F. Maggi, “Experimental techniques for characterization of particles in plumes of sub-scale solid rocket motors,” *Acta Astronaut.*, vol. 186, no. June, pp. 496–507, 2021, doi: 10.1016/j.actaastro.2021.06.011.

[263] N. Rahoui, B. Jiang, H. T. Pan, and Y. D. Huang, “Spectroscopy strategy for solid propellants quality control,” *Appl. Spectrosc. Rev.*, vol. 51, no. 5, pp. 431–450, 2016, doi: 10.1080/05704928.2016.1145124.

[264] D. I. Sebacher, R. J. Bendura, and D. E. Wornom, “Hydrochloric acid aerosol and gaseous hydrogen chloride partitioning in a cloud contaminated by solid rocket exhaust,” *Atmos. Environ.*, vol. 14, no. 5, pp. 543–547, 1980, doi: 10.1016/0004-6981(80)90083-9.

[265] O. - DTSEM - Salt Lake Technical Center, “Hydrogen Chloride in Workplace Atmospheres,” UTAH, USA, 1986.

[266] S. Singh, S. Raveendran, D. R. Kshirsagar, M. Gupta, and C. Bhongale, “Studies on the High Performance Characteristics of an Aluminized Ammonium Perchlorate Composite Solid Propellant Based on Nitrile Butadiene Rubber,” *Cent. Eur. J. Energ. Mater.*, vol. 18, no. 4, pp. 492–511, 2021, doi: 10.22211/CEJEM/145030.

[267] P. Haller, A. Haller, A. Kaźmierczak, J. Matla, Z. Sroka, and R. Wróbel, “Impact of water content in fuel for smoke opacity,” *Combust. Engines*, vol. 195, no. 4, pp. 123–127, 2023, doi: 10.19206/CE-169804.

[268] A. Abd Elall, AM; Lin, G. Abd Elall, “Novel method for measuring primary and secondary smoke density of reduced HCl exhaust composite solid rocket propellant (CSRP) based on nitramine energetic material,” *Sci. Technol. Energ. Mater.*, vol. 80, no. 3–4, p. 121, 2019.

[269] Y. Pal, A. Raja, and K. Gopalakrishnan, “Theoretical and experimental heat of combustion analysis of paraffin-based fuels as preburn characterization for hybrid rocket,” *J. Aerosp. Technol. Manag.*, vol. 12, no. 1, pp. 1–18, 2020, doi: 10.5028/jatm.v12.1180.

[270] Y. Li *et al.*, “Experimental study on combustion characteristics of powder magnesium and carbon dioxide in rocket engine,” *Acta Astronaut.*, vol. 155, no. July 2018, pp. 334–349, 2019, doi: 10.1016/j.actaastro.2018.11.006.

[271] V. Bagalkote, D. Grinstein, and B. Natan, “Energetic Nanocomposites as Burn Rate Catalyst for Composite Solid Propellants,” *Propellants, Explos. Pyrotech.*, vol. 43, no. 2, pp. 136–143, 2018, doi: 10.1002/prep.201700095.

[272] L. S. Jawale, C. Dey, Mehilal, M. Gupta, and B. Bhattacharya, “Effect of experiment environment on calorimetric value of composite solid propellants,” *Def. Sci. J.*, vol. 63, no. 5, pp. 467–472, 2013, doi: 10.14429/dsj.63.2896.

[273] P. N. Dave, R. Sirach, and R. Thakkar, “Thermal decomposition and kinetic investigation of AP and AP based composite solid propellant in the presence of nickel ferrite additive,” *J. Mater. Res. Technol.*, vol. 19, pp. 4183–4196, 2022, doi: 10.1016/j.jmrt.2022.06.123.

[274] S. Carlotti and F. Maggi, “Experimental techniques for characterization of particles in plumes of sub-scale solid rocket motors,” *Acta Astronaut.*, vol. 186, no. May, pp. 496–507, 2021, doi: 10.1016/j.actaastro.2021.06.011.

[275] L. Liu, J. Li, L. Zhang, and S. Tian, “Effects of magnesium-based hydrogen storage materials on the thermal decomposition, burning rate, and explosive heat of ammonium perchlorate-based composite solid propellant,” *J. Hazard. Mater.*, vol. 342, pp. 477–481, 2018, doi: 10.1016/j.jhazmat.2017.08.055.

[276] Z. Ma, F. Li, and H. Bai, “Effect of Fe2O3 in Fe2O3/AP composite particles on thermal decomposition of AP and on burning rate of the composite propellant,” *Propellants, Explos. Pyrotech.*, vol. 31, no. 6, pp. 447–451, 2006, doi: 10.1002/prep.200600060.

[277] H. Liu *et al.*, “Experimental investigation on the condensed combustion products of aluminized GAP-based propellants,” *Aerosp. Sci. Technol.*, vol. 97, p. 105595, 2020, doi: 10.1016/j.ast.2019.105595.

[278] I. E. Wachs, “Infrared spectroscopy of supported metal oxide catalysts,” *Colloids Surfaces A Physicochem. Eng. Asp.*, vol. 105, no. 1, pp. 143–149, 1995, doi: 10.1016/0927-7757(95)03325-5.

[279] C. Chen *et al.*, “Catalytic activity of K2Ba[Ni(NO2)6] on the thermolysis and laser ignition of CL-20, FOX-7 and TKX-50,” *J. Phys. Chem. Solids*, vol. 161, no. 2, 2022, doi: 10.1016/j.jpcs.2021.110411.

[280] N. Birta, N. Doca, G. Vlase, and T. Vlase, “Kinetic of sorbitol decomposition under non-isothermal conditions,” *J. Therm. Anal. Calorim.*, vol. 92, no. 2, pp. 635–638, 2008, doi: 10.1007/s10973-007-7938-6.

[281] C. J. Lundgren, “Identification of Different Crystalline forms of Sorbitol by DSC,” *TA Instruments*, pp. 1–3, 2001.

[282] L. bo Li, X. Chen, O. Musa, C. sheng Zhou, and M. Zhu, “The effect of pressure and oxygen concentration on the ignition and combustion of aluminum–

magnesium fuel-rich propellant," *Aerosp. Sci. Technol.*, vol. 76, pp. 394–401, 2018, doi: 10.1016/j.ast.2017.12.032.

[283] S. Zhou, X. Zhou, G. Tang, X. Guo, and A. Pang, "Differences of thermal decomposition behaviors and combustion properties between CL-20-based propellants and HMX-based solid propellants," *J. Therm. Anal. Calorim.*, vol. 140, no. 5, pp. 2529–2540, 2020, doi: 10.1007/s10973-019-09004-y.

[284] P. Zhang, W. Tan, X. Zhang, J. Chen, J. Yuan, and J. Deng, "Chemical modification of hydroxyl-terminated polybutadiene and its application in composite propellants," *Ind. Eng. Chem. Res.*, vol. 60, no. 10, pp. 3819–3829, 2021, doi: 10.1021/acs.iecr.0c06172.

[285] B. H. Chen, J. Z. Liu, W. J. Yang, H. P. Li, and L. J. Du, "Effect of Ammonium Perchlorate Coating on the Ignition and Combustion Characteristics of Al/JP-10 Nanofluid Fuel," *Combust. Sci. Technol.*, vol. 192, no. 8, pp. 1567–1581, 2020, doi: 10.1080/00102202.2019.1613385.

[286] Y. Oyumi and T. B. Brill, "Thermal decomposition of energetic materials 5. High-rate, in situ, thermolysis of two nitrosamine derivatives of RDX by FTIR spectroscopy," *Combust. Flame*, vol. 62, no. 3, pp. 233–241, 1985, doi: 10.1016/0010-2180(85)90149-X.

[287] A. Babar Zaheer, Malik, "Thermal decomposition and kinetic evaluation of composite propellant material catalyzed with nano magnesium oxide," *NUST J. Eng. Sci.*, vol. 7, no. 1, pp. 5–14, 2015, [Online]. Available: http://journals.nust.edu.pk/index.php/njes/article/view/89

[288] P. K. Gallagher and D. W. Johnson, "Kinetics of the thermal decomposition of CaCo3 in Co2 and some observations on the kinetic compensation effect," *Thermochim. Acta*, vol. 14, no. 3, pp. 255–261, 1976, doi: 10.1016/0040-6031(76)85002-2.

[289] D. Sun, Y. Li, P. Liu, B. Chen, and W. Fan, "Numerical Investigation on the Effect of Ammonium Perchlorate Content and Position on the Combustion Characteristics of an Ammonium Perchlorate/Hydroxyl-Terminated Polybutadiene Propellant," *Aerospace*, vol. 10, no. 8, 2023, doi: 10.3390/aerospace10080692.

[290] D. A. Sugeng *et al.*, "Experimental comparison of smoke opacity and particulate matter emissions with the use of emulsion fuel," *Evergreen*, vol. 7, no. 3, pp. 452–457, 2020.

[291] A. P. Chouinard, A. J. Adams, A. M. Wright, and M. K. Hudson, “Multi-wavelength opacity study of a hybrid rocket plume II,” *34th AIAA/ASME/SAE/ASEE Jt. Propuls. Conf. Exhib.*, no. January, 1998, doi: 10.2514/6.1998-3992.

[292] D. DOLL and G. LUND, “Magnesium-neutralized clean propellant,” in *27th Joint Propulsion Conference*, 1991, pp. 1–10. doi: 10.2514/6.1991-2560.

[293] S. Vyazovkin, “Misinterpretation of Thermodynamic Parameters Evaluated from Activation Energy and Preexponential Factor Determined in Thermal Analysis Experiments,” *Thermo*, vol. 4, no. 3, pp. 373–381, 2024, doi: 10.3390/thermo4030019.

[294] D. Rohini, C. Sasikumar, P. Samiyappan, B. Dakshinamurthy, and N. Koppula, “Design & analysis of solid rocket using open rocket software,” *Mater. Today Proc.*, vol. 64, pp. 425–430, 2022, doi: 10.1016/j.matpr.2022.04.787.

APPENDICES

APPENDIX 1

ITEM NO.	PART NUMBER	DESCRIPTION	MASS (g)	QTY.
1	Motor Case	PVC	30.0	1
2	Nozzle	Clay	8.8	1
3	Propellant	KNSU	24.0	1
4	Bulkhead	Clay	7.5	1
5	Ejection	Black Powder	0.8	1

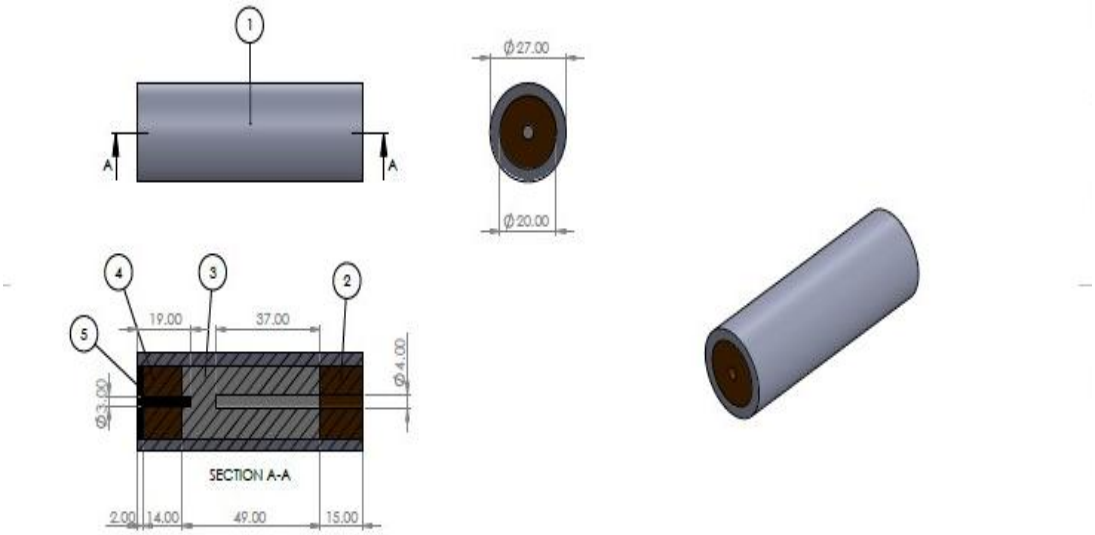
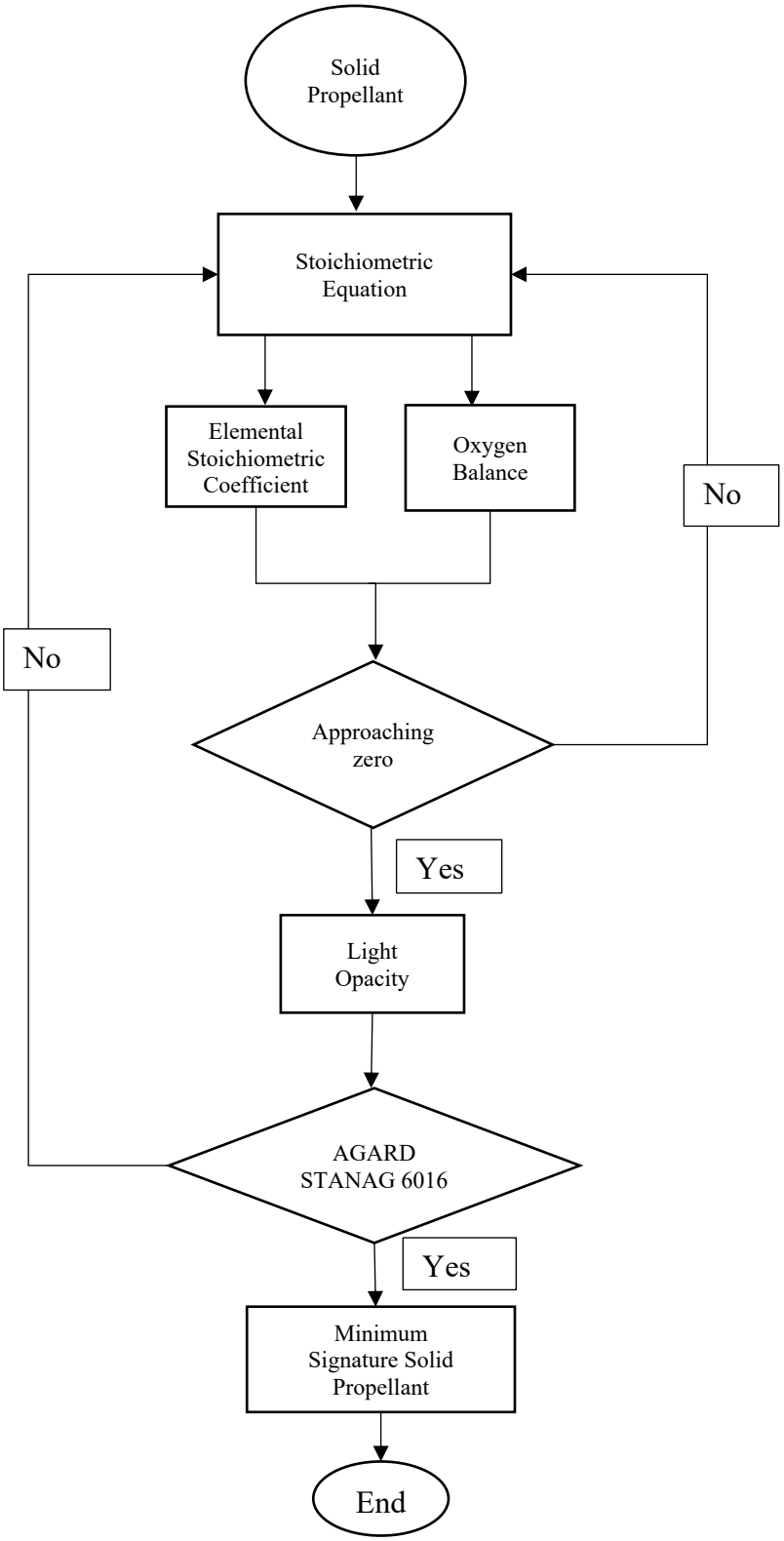


Diagram for Nozzle design

Example of calculation for stoichiometric coefficient

Empirical Formula	Weight%	Molecular Weight g/mol	Molar proportion in 1 g	Mol of atoms in 1 g of solid fuel mixture				
				C	H	N	O	Cl
NH_4ClO_4 (Ap)	60	117.49	0.511	-	2.044	0.511	2.044	0.511
$C_6H_{14}O_6$ (Sorbitol)	40	182.17	0.2196	1.32	3.074	-	1.318	-
			Total	1.32	5.11	0.511	0.360	0.511

APPENDIX 2


Solid Propellant
Stoichiometric Equation
Elemental Stoichiometric Coefficient
Oxygen Balance
No
No
Approaching zero
Yes
Light Opacity
AGARD STANAG 6016
Yes
Minimum Signature Solid Propellant
End

AUTHOR'S PROFILE

Muhammad Zakwan Bin Azizi obtained his degrees; Bachelor Degree of Chemical Engineering (Hons) in 2013, MSc in Material Engineering in 2016 both from University of Malaya (UM) and PhD in Mechanical Engineering in 2025 from University Technology Mara (UITM). His PhD thesis involve in development of solid rocket propellant and energetic material. Currently he is involved in production green energetic material focus on solid rocket propellant and design small scale rocket. He was also an environmental engineer and was UITM Postgraduate Teaching Assistant.

LIST OF PUBLICATION:

Azizi, Muhammad Zakwan. 2024. "Characterization of New Minimum Signature Solid Propellant Based on Ammonium Perchlorate Synergetic with Sorbitol and Magnesium Metal." *Article in Journal of Mechanical Engineering* 21 (2): 181–98. https://doi.org/10.24191/jmeche.v21i2.26262.

Azizi, Muhammad Zakwan, Ahmad Hussein Abdul Hamid, Zuraidah Salleh, Zulkifli Abdul Ghaffar, Alif Abni Adnan, Izzat Najmi Yaacob, Noraafiza Salleh, and Wilfredo Jr Kintanar Pardorla. 2024. "Heat of Combustion and Thermal Decomposition of Ammonium Perchlorate/Sorbitol Solid Propellant with Metal Additives." *Journal of Advanced Research in Experimental Fluid Mechanics and Heat Transfer* 18 (1): 94–105. https://doi.org/10.37934/arfmts.18.1.94105.

Azizi, Muhammad Zakwan, Hussein Abdul Hamid, Zuraidah Salleh, Jamaliah Mohd Said, Zulkifli Abdul Ghaffar, and Khairunnisa. 2025. "Investigating the Effects of Metal Additives on the Mechanical Properties of Ammonium

Perchlorate/Sorbitol Solid Rocket Propellants." *Journal of Aeronautics, Astronautics and Aviation* 57 (3): 363–68. https://doi.org/10.6125/JoAAA.202503_57(3S).08

Azizi, Muhammad Zakwan, Hussein Abdul Hamid, Zuraidah Salleh, Zulkifli Abdul Ghaffar, and Izzat Najmi. 2025. "Review on the Minimum Signature of Solid Propellant." *Journal of Aeronautics, Astronautics and Aviation* 57 (3): 369–88. https://doi.org/10.6125/JoAAA.202503_57(3S).09.

Fahizal, Aina Sofea, Zuraidah Salleh, Ahmad Hussein, Abdul Hamid, Lailatul Atila Alias, and Muhammad Zakwan Azizi. 2023. "Temperature Measurement of Ballistic Evaluation Motor Using K-Type Thermocouple." *Article in Journal of Mechanical Engineering* 13:301–13.

Abni, Alif, Bin Adnan, Ahmad Hussein Bin, Abdul Hamid, Zuraidah Salleh, Muhammad Zakwan Azizi, and Mohamad Amirul Muhammad. 2023. "Mathematical and Computational Fluid Dynamics Analysis of Low-Altitude Rocket Static Stabilityfor Various Fin Designs." *Tuijin Jishu/Journal of Propulsion Technology* 44 (4): 1001–4055.

Adnan, Alif Abni, Ahmad Hussein Abdul Hamid, Zuraidah Salleh, and Muhammad Zakwan Azizi. 2024. "Mathematical and Computational Fluid Dynamics Analysis of Low-Altitude Rocket Drag for Various Fin Configurations." *Journal of Aeronautics, Astronautics and Aviation* 56 (3): 781–90. https://doi.org/10.6125/JoAAA.202407_56(3).12.

Yaacob, Izzat Najmi, Ezanee Gires, Adi Azrif Basri, Kamarul Arifin Ahmad, Kamsani Kamal, Nor Afizah Salleh, Suraya Shahedi, et al. 2025. "Enhanced Thermal Performance and Hydrochloric Acid Gas (HCl) Emission Mitigation in Ammonium Perchlorate (AP)-Based Solid Propellants with Potassium Permanganate (KMnO4)/AP Dual Oxidisers." *Atmospheric Environment: X* 26 (May): 100329. https://doi.org/10.1016/j.aeaoa.2025.100329.