

Characterization of New Minimum Signature Solid Propellant Based on Ammonium Perchlorate Synergetic with Sorbitol and Magnesium Metal

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ABSTRACT

Since the introduction of the concept of insensitive weapons, rocket motor designers must now ensure that their vulnerability is as low as possible. One of the inherent characteristics of a sugar-based propellant is the obvious plume coming out from the nozzle as a result of chemical reactions. The present work characterises a novel minimum signature composite solid propellant based on Ammonium perchlorate/Sorbitol with magnesium metal as a fuel and chlorine scavenger. The elemental stoichiometric coefficient and oxygen balance for the propellant mixture were calculated to find the optimum ratio between the oxidizer and fuel. The solid propellant of AP was prepared by mixing it with melted sorbitol and casting it into the cylindrical hollow tube using the freestanding method. The burning rate for five samples was measured at atmospheric pressure. The morphology was characterized by a scanning electron microscope. Calculation results reveal that ammonium perchlorate at 0.77% weight produced the best elemental stoichiometric coefficient and oxygen balance. The sample with the additive metal showed a significant increase in the burning rate. In all formulation samples of magnesium and ferrous oxide, the maximum burning rate at 0.126 cm/s showed that the metal additive and the propellant worked synergistically with each other. Results from SEM indicate that ammonium perchlorate spherical particles were uniformly dispersed within sorbitol. The addition of ferrous

oxide increases the interaction between the component, and at the same time it gives the oxidizer the ability to interact more efficiently with the fuel and improve the physical strength of the solid propellant. This research is a minor step for Malaysia to become a top competitor in the aerospace industry in the future.

Keywords: *Minimum Signature Solid Propellant; Ammonium Perchlorate; Sorbitol; Elemental Stoichiometric Coefficient; Oxygen Balance*

Introduction

Solid and liquid rocket motors are the two main propulsions employed in rocket launch systems. A rocket motor is a rocket engine that produces thrust by breaking the chemical energy bond between the fuel and the oxidizer during the combustion process [1]. They convert chemical energy into gas pressure in combustion chambers, and the gas flows out of the chamber, creating thrust that propels the rocket [2]. Thrust is created as the high thermal energy of the combustion gases is transformed into kinetic energy in the exhaust [3]. Thus, the resulting gas does not only produce thrust but also produces smoke that causes problems for the defense system, obstructs vision, and affects human health.

Developing a minimum-signature (smokeless) propellant is important because it is non-polluting, acid-free, solid particulate-free, and smoke-free [4]. These smokeless propellants remove the possibility of exposing the locations from which the missiles are launched because they do not produce the bright flame and thick smoke trail that aluminizes propellants when they burn [5]. This minimum signature propellant relates to energetic compositions and propellant formulations that improve the performance of rocket propulsion. Together with the high oxygen balance of propellant, it will contribute to improving the burning rate, increasing molecular energy and kinetic reactivity, and making them good oxidizers [6]-[7]. Moreover, it is necessary for the development of propellant to comply with insensitive munition (IM) that effectively meets their performance, readiness, and operational requirements on demand while minimizing the severity of a reaction and anything resulting from unintentional damages [8]-[9].

Many composite propellants in use today are composed of polyurethane binder based on hydroxyl-terminated polybutadiene (HTPB), ammonium perchlorate (AP) as an oxidizer, and aluminum powder (Al) as metallic fuel [10]. The presence of 20 percent Hydrogen chloride (HCl) in the exhaust products is the main drawback of AP-based commercial solid propellants [11]. There are several ways to lower the amount of HCl in the exhaust from propellant products. They can be divided into three different categories: neutralized, scavenging chlorine ions using alkali metals and using non

chlorinated oxidizers [12]-[13]. Each category has its advantages and difficulties, depending on the chemical and physical properties of the materials used. Halophilic substances, such as alkali metals, are often utilized in scavenged propellants to scavenge chlorine ions during combustion and produce alkali metal chlorides [14]. Magnesium is used as a scavenger to reduce HCl gas in the combustion of AP (ammonium Perchlorate) based solid propellant due to its ability to reduce 55% HCL gas, increase burning rate by 20%, and reduce ignition temperature for solid propellant [15]. Magnesium is converted during the combustion of the propellant into magnesium oxide (MgO), which upon contact with water transforms into magnesium hydroxide (Mg(OH)₂), a strong base that combines with HCl to produce magnesium chloride [10]. In comparison, non-chlorinated strong oxidizing agents are used in the production of minimal signature propellants such as FOX-7, ADN, HNF, CL-20, HMX, and RDX. However, there are many obstacles and shortcomings to using these oxidizers, such as raw material availability, complex production processes, high cost of production, compatibility with others, and high sensitivity [16]-[17].

In recent studies, researchers enhanced the physical and chemical performance of ammonium perchlorate by studying the synergistic effect with other active materials such as HMX, carbon material, metal catalyst, and metal nanoparticles [18]-[21]. Knowing that AP (ammonium perchlorate) has many distinct features and synergistic effects with other materials, it has been discovered that including carbon materials in catalyst systems may significantly increase heat release at a lower HTD (high-temperature decomposition) temperature [22]. The synergistic capabilities of AP with different compound materials have become an advantage in enhancing its performance and reducing harmful gas emissions. To benefit from this, sorbitol is utilized to observe its synergistic effect with AP. Moreover, sorbitol is an organic sugar alcohol that possesses many remarkable features that make it easily available: it is inert, does not caramelize, has a better pot life, and has a lower thermal decomposition temperature [23]-[24]. It is commonly used with potassium nitrate as a sounding rocket for solid propellant, while 40 % of the exhaust products are liquid droplets of potassium carbonate, which reduces rocket performance [23], [25]. By combining sorbitol, sucrose, and carbon powder with the solid propellant, it was reported that the smoke produced is minimized compared to other common formulations of KNSB (potassium nitrate sorbitol) propellant [26]. Few research that combined perchlorate metal with sorbitol has been done recently [27]-[28]. By mixing potassium perchlorate with potassium nitrate/sorbitol condensed particles, it managed to reduce 25.1% from 43.6% of common KNSB solid propellant [29]. This happens because potassium perchlorate is a strong oxidizing agent with a high positive oxygen balance of 39% [30]. It has altered the oxygen balance of the mixture to provide more oxygen to ensure complete combustion takes place.

Thus, the goal of this research is to find a solution for materials that possess lower sensitivity, minimum smoke production, availability of raw material, and lower shock sensitivity while possessing compatibility that could be blended to create such a formulation. The main objective of this work is to conduct theoretical and practical research into various methods of chemical compositions of the prepared samples to reduce the smoke content emitted during burning. The investigation included the use of chemical compounds as a performance enhancement and a gas scavenger. Magnesium (Mg) powder and ferric oxide (Fe_2O_3) are two of the chemical additives included in this study. The main emphasis was placed on the formulation and synthetization of high-performance minimum-smoke propellants that might be able to act as a substitute for conventional double-base (DB) or elastomer modified composite double-base (EMCDB) propellants, which are currently in use for multiple tactical missile applications.

Elemental 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 have a linear relationship with their calorimetric values. This relationship has been effectively applied to determining the calorific value of fossil fuels. According to Bakhman [31], elemental stoichiometric coefficients ϕ_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 ϕ_e is defined in Equation (1).

$$\phi_e = \frac{\sum n \cdot v_o}{(-1) \sum n_r \cdot v_r} \quad (1)$$

where v 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 $\phi_e < 1$, fuel-lean if $\phi_e > 1$, and stoichiometrically balanced at $\phi_e = 1$, [31]–[33].

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 $C_aH_bN_cO_dCl_e$ combustion products in relation to the oxygen balance of AP/Sorbitol solid propellant [16], [34]. 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, controlling the oxidizing agent content in the propellant's mixture will change the value of the oxygen balance.

The oxygen balance of the solid propellant of ammonium perchlorate/sorbitol formulation was calculated using Equation (2) [35]-[36] to optimise the propellant composition to get an oxygen balance as close to zero due to the fact that the heat of explosion is the highest when the oxygen balance is zero. This is because it corresponds to the stoichiometric oxidation of carbon to carbon dioxide and hydrogen to water [37].

$$\Omega = \frac{[d - (2a) - (\frac{b-e}{2})] \times 1600}{M} \quad (2)$$

where Ω is oxygen balance is oxygen mole, a is carbon mole, b is hydrogen mole and e is chlorine mole. M is the relative atomic mass of oxygen.

Methodology

Material

All chemical was purchased at Bendosen. Ammonium perchlorate is used with a purity of 99.9% and a size of 90 microns. Sorbitol used in granule form is needed as fuel and it provides a matrix medium for ammonium perchlorate to disperse. Magnesium and ferric oxide were used as additives to improve performance which were in powder form. Sorbitol used is a food additive with the purity of 99.5% in granule form is needed as fuel and it provides a matrix medium for ammonium perchlorate to disperse. Magnesium powder size of 45 microns with 99% purity and ferric oxide powder with purity of 95% were used as additives to improve performance which were in powder form.

Preparation

Sorbitol and ammonium perchlorate powders with an average particle size of 90 μ were used as feedstock materials in this work. Equation (1) is applied to check whether the mixture of samples is fuel-lean or fuel-rich. The empirical formula of the solid propellant mixture $C_aH_bN_cO_dCl_e$ was calculated and applied to Equation (2) [36]. The optimized formulation ratio between AP and sorbitol was used as the main ingredient to produce samples for this study as illustrated in Table 1. Sorbitol and ammonium perchlorate were weighted

according to the ratio calculated with optimum oxygen balance using Equation (2). The result from the calculation is depicted in Table 2 and a graph is plotted to find the optimum ratio for the optimum oxygen balance and optimum elemental stoichiometry coefficient.

Table 1: Composition of the solid propellants

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%

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 with magnesium and ferric oxide as a power enhancing material. Sorbitol was first heated at a temperature of 200 °C until it melted. Immediately ammonium perchlorate was added slowly to avoid accidents because ammonium perchlorate auto ignition temperature was 240 °C and it was stirred evenly until it formed a slurry-like paste followed by additive metals. To ensure that ammonium perchlorate and sorbitol are evenly mixed, an SEM test is carried out. The propellant in a slurry place is formed into a cylinder and it is allowed to freeze for 30 minutes and placed in a dry cupboard.

Scanning electron microscope

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 magnification. Double-sided conductive adhesive tape was attached to the SEM specimen stub, 12.5 mm diameter. Solid powder sprinkled on the double conductive adhesive tape. Sputter coating with a platinum thin layer at 10 nm is necessary for the samples to obtain high SEM image quality [38].

Measuring the burning rate of the solid propellant

The cured samples were cut into smaller pieces with diameters of $1.0 \times 1.0 \times 4.0$ cm, and the burning rate was measured at ambient pressure, as depicted in Figure 1. This method was adopted from [39].

Results and Discussion

Five minimum signature composite solid propellant samples were examined and described in this paper. Five measurement samples were collected on average, as illustrated in Table 2. The first two potential solid propellants served as the basis for a comparison of smokeless solid propellant measurements with a composite solid propellant containing a metal additive. The solid powder of ammonium perchlorate loading was raised from 77% to 80%, and a metal additive was added as shown in Table 1. Additionally, the impact of metal addition on burning characteristics was well examined.



Figure 1: Solid propellant of testing sample

Parametric analysis

The percentage by mass of mixtures was determined using Equations (1) and (2), considering the weight of ammonium perchlorate and sorbitol present in Table 2. From Table 2, between the range of 75% to 80% amount of AP, the formulation of solid propellant mixtures has optimum oxygen balance and elemental stoichiometric coefficients. As the amount of oxidizer increases, the carbon constituents in the mixture slightly decrease although total oxygen is constant. It means that the mixture has become an oxidizer dominant. By referring to the oxygen balance value, appears to be positive as the oxidizer is increased. Moreover, the elemental stoichiometric coefficient for the mixture is greater than 1 at the composition of AP greater than 75%.

Figure 2 illustrates the relationship between solid propellant formulation and elemental stoichiometric coefficients. At stoichiometric balance $\phi_e = 1$, the formulation of propellant is 0.775 weight percent. At a value of 0.775 wt.% and above, the elemental stoichiometric balance is fuel lean at $\phi_e > 1$. 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 the elemental stoichiometric coefficient at any given mixture composition.

Table 2: Effect of mass ratio solid propellant mixtures on oxygen balance

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

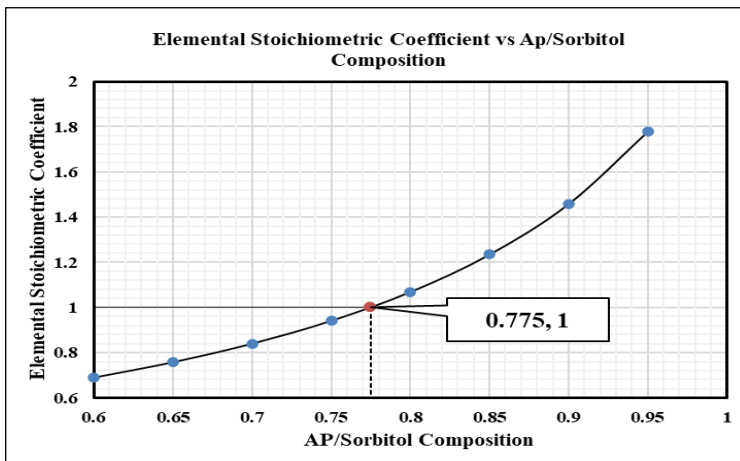


Figure 2: Elemental Stoichiometric coefficient of solid propellant

Furthermore, to overcome the limitation of the elemental stoichiometric coefficient, the oxygen balance mixture was determined and plotted against the composition of the solid propellant composition. Referring to Figure 3, the weight of 0.8% and above of oxygen balance became positive. From Figure 3, the mixture composition at 0.775 wt.% in correlation to the elemental stoichiometric coefficient shows that the oxygen balance was almost 0. It indicates that ammonium perchlorate plays the main role as an oxygen provider of fuel.

Moreover, the oxygen balance value is aligned with the elemental stoichiometric coefficient value since both parameters can also determine solid propellant performance. Hence it indicates that 0.775 wt.% weight percent,

the mixture composition will have high performance and possess a high rate of reaction.

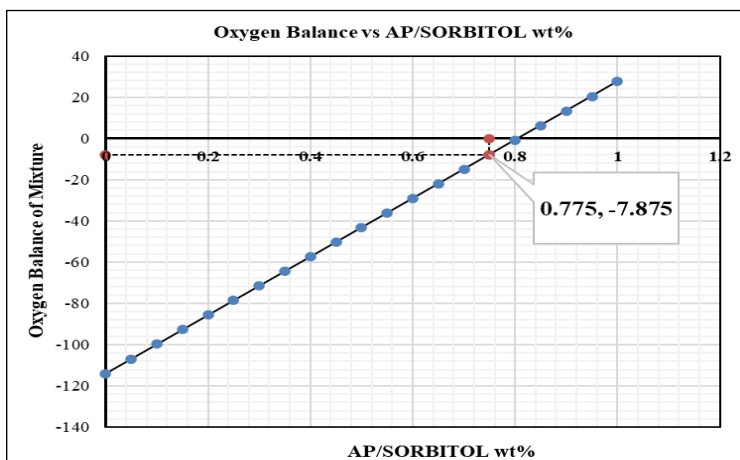


Figure 3: Oxygen balance of the AP/Sorbitol mixture

Scanning electron microscope

Morphology testing was also performed to investigate powder dispersion and propellant characteristics. For all formulations, a micro picture of the propellant cross-section was conducted. In Figure 4, the spherical ammonium perchlorate marked by “red o” was well dispersed among the other components. It is noticeable that the morphology of one propellant differs from another due to the differences in composition. SP7 and SP8 comprise ferric oxide as a catalyst. It also serves as a medium for propellant grain bonding, which slightly improves the physical strength. It also helps the sorbitol recrystallize and the components to connect with each other. Furthermore, the iron element is derived from ferrous oxide, showing that ferrous oxide material is drawn into the mixture and well disseminated. Green arrows show that the melted sorbitol is uniformly well dispersed for SP7 and SP8. This improves the strength of the solid propellant. The morphology illustrated below shows good interaction and dispersion between fuel, oxidizer, and metal additive.

Compared to Figure 4a, there was no ferric oxide in the formulation of the melted sorbitol which did not physically interact well with the oxidizer. For the sample in Figure 4b, magnesium metal was added to the formulation which showed that oxidizer and melted sorbitol had better interactions. Melted sorbitol interacts with most oxidizer particles although they do not uniformly distribute. Hence, additive metals play a vital role in the interaction between oxidizer and sorbitol.

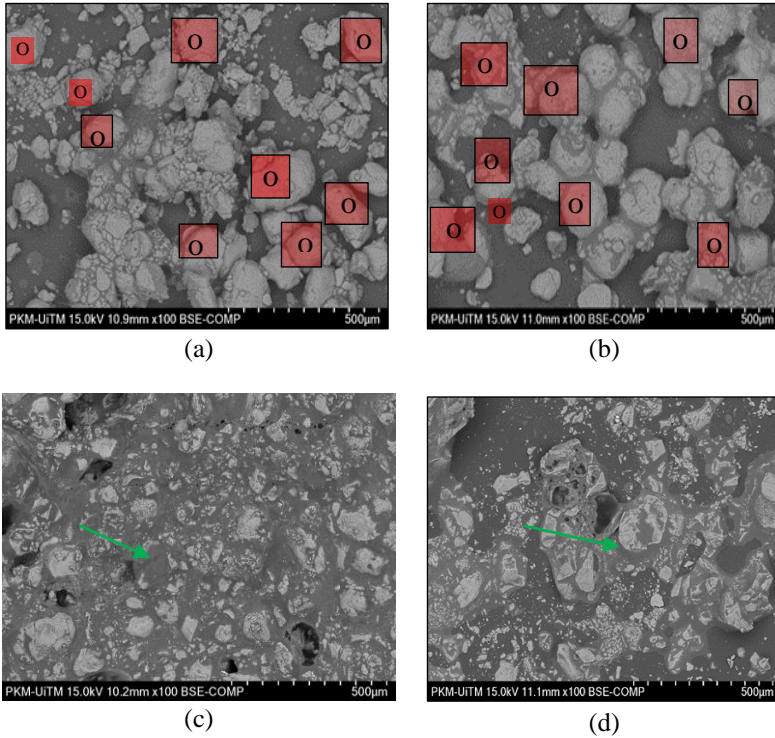


Figure 4: Solid propellants cross-section morphology; (a) SP5, (b) SP6, (c) SP7, and (d) SP8

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 5 [40]. The data show that the significant elements in solid propellants are carbon, oxygen, and chlorine. The magnesium metal is present in samples SP6 and SP8, while iron oxide is present in SP7 and SP8. Silicon is the impurities found in most samples due to the glass XRD sample holder. Sulphur is caused by contamination during the preparation of samples. A comparison between two samples SP4 and SP5 shows that oxygen content slightly increases. It proves that oxygen balance increases with the amount of ammonium perchlorate as depicted in Figure 5. Alternatively, the amount of ammonium perchlorate/sorbitol ratio is fixed as the quantity of carbon and oxygen for the SP6 to SP8. It clearly indicates that magnesium metal and iron oxide play vital roles in the interaction of elements in solid propellant systems. Moreover, chlorine quantity is reduced as magnesium metal and iron oxide are added to the samples which tells us that chloride is scavenged by magnesium and neutralized by iron oxide.

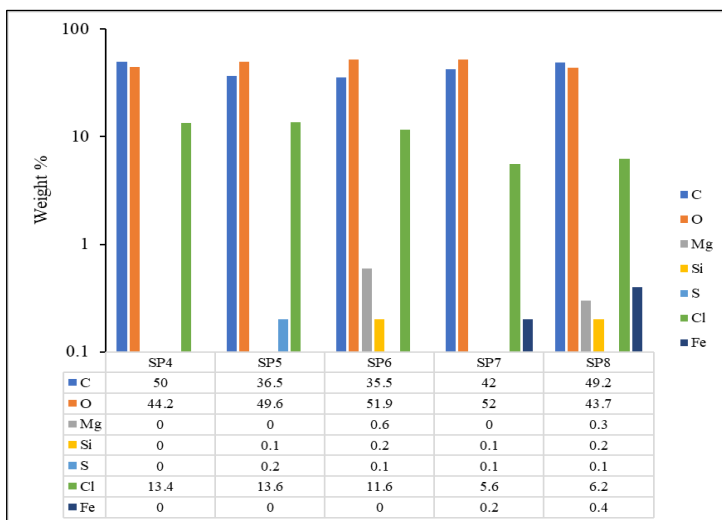


Figure 5: EDX analysis on Solid propellant at different ratios of additives

Burning rate of composite solid propellant

The graph shown in Figure 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 [41]. 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 performing 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 enhancers.

Figure 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 no smoke. It essentially indicates that the intimacy 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 ferrous oxide metal additives were added to the solid propellant of the three samples, SP6, SP7, and SP8. These metals play a vital role in enhancing

the burning rate of solid propellants. Furthermore, the flame during the combustion of SP6, SP7, and SP8 shows a vigorous and bright yellow burning flame. Since magnesium is a major factor in enhancing solid propellant performance, it improves the heat of combustion. 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.

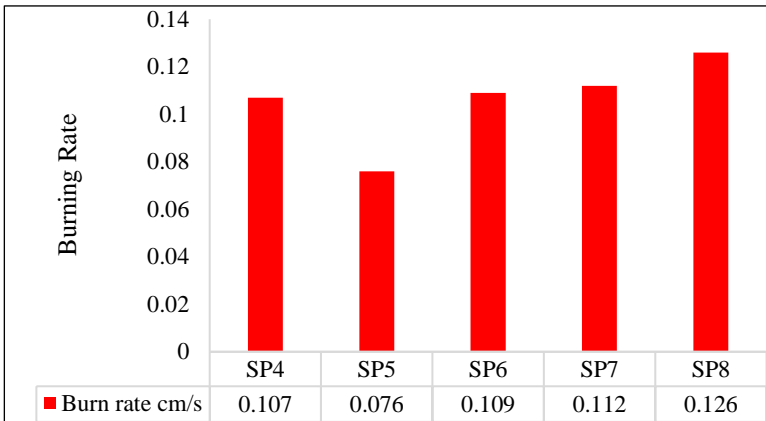


Figure 6: Burning rate of solid propellant for every different formulation

Conclusion

A total of five compositions of a novel minimum-signature composite solid propellant using ammonium perchlorate and sorbitol were characterized. 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 optimum formulation, ammonium perchlorate/sorbitol produces no 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. This

research is a minor step for Malaysia to become a top competitor in the aerospace industry in the future. Future work on the kinetic study, thermal decomposition, characterization, and burning rate effect of these propellants on high pressure needs to be conducted.

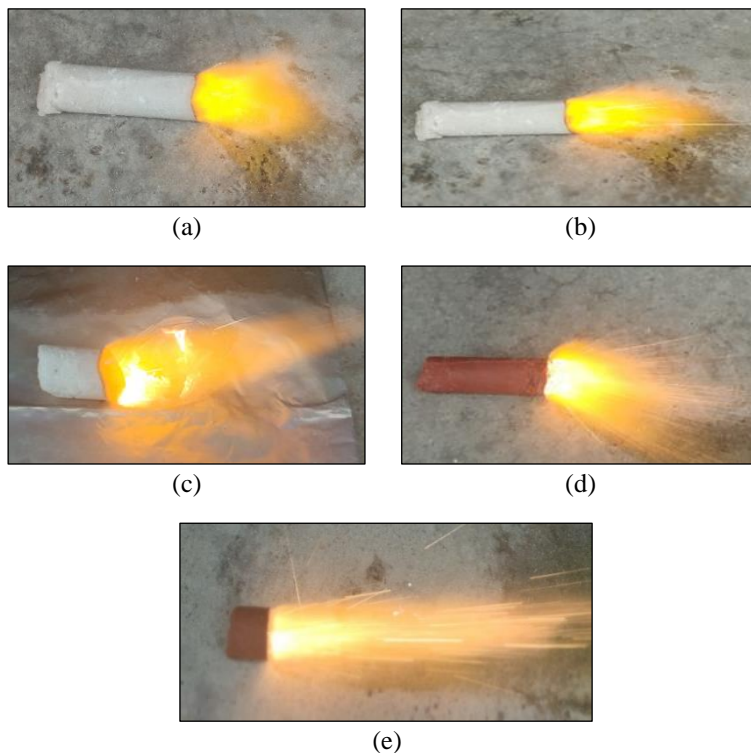


Figure 7: Combustion of minimum signature composite solid propellant; (a) SP4, (b) SP5, (c) SP6, (d) SP7, and (e) SP8

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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Conflict of Interests

All authors declare that they have no conflicts of interest.

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