UNIVERSITI TEKNOLOGI MARA

COMPARATIVE ANALYSIS OF INDUSTRIAL AND CASTOR OIL BASED POLYURETHANE: PHYSICAL, MECHANICAL, MORPHOLOGICAL PROPERTIES AND STRESS CRACKING RESISTANCE FOR GROUTING APPLICATION

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of the requirements for the degree of
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ABSTRACT

Polyurethane (PU) is widely used in the grouting industry for its high compressive strength and durability. However, the reliance on petroleum-based polyols and ozone-depleting hydrochlorofluorocarbons (HCFCs) as blowing agents raises significant environmental concerns. This study evaluates sustainable alternatives by developing a green PU using castor oil-based polyol and water (H_2O) as a blowing agent. The research was conducted in three stages. First, optimizing the polyol-to-isocyanate ratio using industrial polyurethane (PI). The 1:1 ratio demonstrated superior performance, exhibiting moisture absorption below 12%, lower viscosity, reduced weight, and a uniform, densely packed cellular morphology. Secondly, synthesize and compare the properties of green PU with different blowing agents (HCFC 141b and H2O). All formulations met grouting standards, with cream times of 10–20 s and rise times exceeding 48 s. The water-blown castor oil polyurethane (PCW) showed the lowest apparent density while maintaining compressive strength (30–150 kPa) and modulus (150–500 kPa) within standard requirements. FTIR analysis confirmed that all ratios are completely cured. Lastly, performing a comparative analysis of the environmental stress cracking resistance (ESCR) and antibacterial testing. The durability assessments through soil burial and chemical immersion revealed that PCW maintained moisture absorption below 12% and exhibited a 15–20% increase in compressive strength over 90 days. Furthermore, PCW displayed intrinsic antimicrobial activity against B. subtilis and E. coli. These findings demonstrate PCW's strong potential as a sustainable grouting material with reduced environmental impact.

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LIST OF SYMBOLS

Symbols	
CO_2	Carbon dioxide
HCFC	Hydrochlorofluorocarbons
HCFC 141b	1,1-dichloro-1-fluoroethane
N=C=O	Isocyanate
-O-H	Hydroxyl
PCH	Castor oil –HCFC 141b polyurethane
PCW	Castor oil- Water polyurethane
PI	Polyurethane Industry
PU	Polyurethane

LIST OF ABBREVIATIONS

Abbreviations

CFC	Chlorofluorocarbons
ESCR	Environmental Stress Cracking Resistance
FRT	Foam Reaction Time
FTIR	Fourier Transform Infrared
GWP	Global Warming Potential
HCl	Hydrochloride acid
HFC	Hydrofluorocarbons
HFO	Hydrofluoroolefins
KOH	Potassium Hydroxide
LCA	Life Cycle Assessment
MDI	Methylene diphenyl diisocyanate
NaOH	Sodium Hydroxide
ODP	Ozone Depletion Potential
SEM	Scanning Electron Microscopy
TDI	Toluene diisocyanate
UTM	Universal Testing Machine

LIST OF NOMENCLATURES

Nomenclatures

wt%	Weight percent
cm	Centimetre
g	Gram
K	Kilo
Kg	Kilogram
kN	Kilo-kewton
kPa	kilo-pascal
mg	Micro-gram
mm	millimeter
MPa	Mega-pascal
μm	micron/micrometre

CHAPTER 1
INTRODUCTION

1.1 Research Background

Cement always use as grouting material. Almost 10-70% cement were used in the repairing the building cracks, potholes and overlay or resurfacing of pavements. Nowadays, polyurethane is utilized as a grouting material since it satisfies all the necessary criteria for this application. Polyurethane is composed of polyols, isocyanates, catalysts, and other additives. Polyurethane can be produces as both thermoset and thermoplastic forms (Wang et al., 2020). Grouting is the process that injects materials into cracks or cavities in concrete, masonry structures, soil, or rocks, same both PU grout Polyurethane grouting includes injecting expanding as for polyurethane to fill and seal fractures, voids, and re-level slabs (Thomas et al., 2018).

The industrial polyurethanes are synthesized from petrochemical resources, which, raise concerns due to their environmental impact, particularly their non-renewable nature and potential for pollution. In response to these issues, green polyurethanes, which are derived from renewable resources such as castor oil, have been developed. These green alternatives aim to reduce the carbon footprint of polyurethane production while maintaining the material's performance in various applications, including grouting.

Castor oil is a vegetable oil derived from the seeds of the Ricinus communis plant. Known for its unique chemical composition, it is rich in ricinoleic acid, a rare hydroxyl fatty acid that gives castor oil its distinctive properties, including high viscosity, stability, and moisture retention. The use of castor oil in the construction industry has become increasingly significant due to its sustainable and functional benefits, particularly in the production of protective coating, and sealants.

Blowing agents are crucial substances utilized in the manufacturing of polyurethane (PU) foams. The main role is to produce gas throughout the polymerization process, which becomes encapsulated within the polymer matrix. This process results in the formation of a cellular, foam-like structure. The inclusion of blowing agents is essential as they influence various key characteristics of the foam,

such as density, thermal insulation performance, mechanical strength, and the overall lightweight design.

Blowing agents play a crucial role in the production of polyurethane foam, fulfilling several key functions. Initially, they facilitate the growth of the polymer matrix by creating internal pressure, which leads to the development of foam. Furthermore, they regulate the density of the foam, which influences both its weight and performance attributes. Thirdly, the thermal insulation properties of the foam are significantly enhanced through the formation of closed cell structures that effectively trap gas. Ultimately, blowing agents enhance material efficiency by decreasing the necessary solid content, resulting in a final product that is both lighter and more economical.

HCFC 141b is a commonly utilized blowing agent in the production of industrial polyurethane. This compound emerged in the 1990s as an alternative to fully ozone depleting chlorofluorocarbons (CFCs), owing to its comparatively lower potential for ozone depletion. HCFC 141b is recognized for its ability to produce foams that exhibit outstanding mechanical strength, a fine and uniform cellular structure, and effective thermal insulation performance. Moreover, it demonstrates excellent compatibility with conventional polyurethane formulations and provides notable dimensional stability, particularly in the context of rigid foam applications.

In light of these benefits, it is important to note that HCFC 141b poses significant environmental hazards. While it is not as detrimental to the ozone layer as its CFC predecessors, it still has an ozone depletion potential (ODP) of 0.11. Furthermore, HCFC 141b possesses a significant global warming potential (GWP), playing a role in climate change upon its release into the atmosphere. Consequently, its production and utilization have been regulated on an international scale through the Montreal Protocol and the Kigali Amendment, with various countries implementing phase out timelines.

To overcome this, a research of using two types blowing agents were utilized, using1,1-Dichloro-1-fluoroethane (HCFC 141b) and water. A blowing agent is a chemical substance utilized in the production of polyurethane foam to produce gas, which forms the cellular structure of the foam. The gas expansion process occurs via physical or chemical mechanisms. Volatile liquids, like HCFC 141b, serve as physical blowing agents that vaporize when heated, resulting in the expansion of the polymer

matrix. In contrast, chemical blowing agents are substances, such as water, that, when they undergo chemical reactions, generate gases like CO_2.

Polyurethane foam is a lightweight substance that provides outstanding insulation, boasts a significant expansion rate, and features a rapid response time. Polyurethane foam serves as a highly effective solution for pipeline repairs, leak fixes, foundation restoration, and the elevation of sunken concrete slabs (Mohamed Jais, 2017). Polyurethane grouting is a widely employed technique for the purpose of filling and sealing cracks, voids, and relevelling slabs (Ruiz, 2021). This technology plays a crucial role in infrastructure repair, particularly for economic reasons, as it effectively seals any damage or holes in concrete bridges and structures.

A variety of companies in Malaysia provide grouting and waterproofing services. These enterprises focus on the distribution of polyurethane grouts, machinery, accessories, polyurethane blanket grouting, polyurethane slab jacking, marine floats, spray foam insulation and equipment, cement plaster bonding glue, and various bonding agents. Nonetheless, Malaysia is deficient in studies and dependable data concerning the application of polyurethane as a grouting material, which hinders its ability to address any potential issues that may arise.

Historically, conventional grouting materials comprised sand, cement, and water, which exhibited slow setting times, shrinkage, and limitations on application below 32°F (Gopinathan et al., 2017). To achieve cost effective and efficient maintenance, polyurethane can be utilized as a grouting material instead of cement grouting. This can be accomplished by determining the optimal ratio of polyol and isocyanate formulation to produce polyurethane with desirable compression and expansion properties. Polyurethane grouting offers enhanced hygiene, expedited application, and reduced disruption compared to cement grouting. Polyurethane grouting offers a cost effective, rapid, and efficient solution for addressing settlement and instability issues.

The grouting materials provide exceptional mechanical strength, durability, and resistance to environmental factors like moisture and temperature variations. This investigation contrasts industrial and green polyurethanes according to these criteria, emphasizing their physical, mechanical, and morphological characteristics. This study aims to thoroughly assess these properties to enhance our understanding of how green polyurethanes might serve as alternatives or supplements to conventional polyurethanes in the grouting sector.

This study seeks to address challenges encountered by the construction sector and to minimize carbon emissions to ensure the sustainability of pavement and roadway infrastructure. There are numerous construction companies that utilize polyurethane as a grouting material; however, there are limitations and drawbacks that stem from insufficient research and understanding. This study may contribute to addressing the challenges within the construction sector, particularly in the use of polyurethane as grouting materials.

1.2 Problem Statement

The use of cement as grouting has been worldwide and as alternative, polyurethane can be used in order to preplace the cement as grouting materials. Malaysia's construction industry is primarily challenged by the rigidity of polyurethane production. The difficulty in determining the optimal formulation of polyol and isocyanate to produce rigid polyurethane that can fill a specific void or volume contributes to an unresolved void issue (Amran et al., 2021). Due to the uneven ratio of polyol to isocyanate, complications such as polyurethane shrinkage, soften and do not cure appropriately. It has become a major concern for researcher and construction industry’s workers as a result of endless research. Improperly formulated polyurethane production results brittle polyurethane, which can reduce the polyurethane's durability and toughness. These complications become unsettle when subjected to repeated loads (Sidek et al., 2017). This concern arises due to the rapid movement of the heavy transportation on the highways and pavements.

On the other hand, there is concern regarding whether polyurethane possesses waterproofing properties. Waterproofing makes an object resistant to continued water infiltration under specific conditions. As cement is not waterproof, polyurethane must be waterproof for grouting purposes (Keith, 2020). Cement is a highly porous material with a propensity to absorb water from precipitation as well as ambient humidity. This issue is related to highway and pavement cracks when polyurethane is injected to fill a void when underground water has accumulated or is flowing (Wang et al., 2022).

The properties of polyurethane are the most important reason why Malaysian industry has replaced cement with polyurethane as grouting material. Cement grouting has been widely utilised for decades. Nevertheless, the porous nature of cement based grout products is a major drawback of cement grouting. The problem with this is that

the pores allow dirt and other outside elements to embed themselves within the grout. This causes discoloration and even permits mould and mildew to embed in the grout. The cement not only acts as a conduit for stains, but it also has insufficient followability to fill voids, which can be messy (Khan et al., 2022).

In addition, cement is high time consuming and heavier than polyurethane. The production cost of cement grouting is comparable to that of urethane. However, as demand rises due to incorrect formulation, the price of polyurethane derived from petrochemicals is high due to the rise in petrochemicals and crude oil prices (Abrar et al., 2021). The nature polyurethane foam, initially in resin, turns into rigid foam makes it an advantage in grouting applications as it can expand quickly, fill the void or crack, and cut the material cost as less material is needed due to the developing ability of polyurethane foam (Saleh et al., 2019).

This have become initiative in order to solve this problems, three different ratios of polyol and isocyanate; 0.5:1, 1:1 and 1:0.5 will be investigate on which formulation produce rigid and hard properties that provides the greatest strength of polyurethane that can solves problems faces in the Malaysia's industry. In addition, it may help with the research to determine which formulations have brittle properties and enhance expansion. So, it become the main focus of this research to established the best composition of polyol and isocyanate in order to produce hard and rigid polyurethane suitable for grouting applications.

Polyurethane (PU) grouting is extensively utilized in construction and infrastructure due to its superior mechanical strength, adhesion, and expansion characteristics. Conventional industrial polyurethane is predominantly sourced from petroleum based polyols, resulting in significant reliance on fossil resources and raising environmental issues, including elevated carbon emissions and lack of biodegradability (Karuf et al., 2025). Also the potential use of blowing agent as to create a cellular structure, reducing density while increasing insulation and stiffness. The selection of blowing agents, including hydrochlorofluorocarbon (HCFC) presents environmental and health hazards attributable to its ozone depletion potential and associated regulatory constraints (Jaf et al., 2023).

In addressing these challenges, bio based polyurethanes, particularly those sourced from castor oil, have emerged as viable alternatives owing to their renewable characteristics, biodegradability, and similar mechanical properties (Sardon et al., 2021). Castor oil based polyurethane (Green PU) shows promise in multiple

applications; however, additional research is necessary to assess its viability as a sustainable alternative to industrial polyurethane in grouting applications.

The type of blowing agent employed is a crucial toward the production of polyurethane foam performance, impacting their morphology, density, mechanical strength, and durability (Jaf et al., 2023). Conventional industrial polyurethane foams utilized 1,1-dichloro-1-fluoroethane (HCFC 141b) as the physical blowing agent. In contrast, water based chemical blowing agents present a more environmentally sustainable option. The influence of these blowing agents on the physical, mechanical, and morphological characteristics of castor oil based polyurethane in comparison to industrial polyurethane is not well understood.

This research aims to conduct a comparative study evaluating the physical, mechanical, and morphological properties of industrial and green polyurethane foams utilizing different blowing agents (HCFC 141b and water). This study evaluates key parameters including density, compressive strength, thermal stability, cell morphology, and water absorption to ascertain the viability of castor oil based polyurethane as a sustainable alternative for grouting applications while maintaining performance standards.

1.3 Research Question

i) What are the optimal composition of polyol and isocyanate (0.5:1, 1:1, and 1:0.5) for synthesizing polyurethane with desirable properties?

ii) What is the efficiency of castor oil based polyurethane synthesis using HCFC 141b and water as blowing agents in achieving the best industrial polyurethane composition?

iii) How do the physical, mechanical, morphological, environmental stress cracking resistance (ESCR), and antimicrobial properties of castor oil based polyurethane vary with different blowing agents at a 1:1 polyol-to-isocyanate ratio?

1.4 Research Objective

This research embarks on these objectives

a) To determine the best compositions of polyol and isocyanate ratios through physical, mechanical and morphological properties of industrial polyurethane.

b) To evaluate the efficiency of water as blowing agents through the physical, mechanical and morphological of castor oil polyurethane with different type blowing agent using the best composition polyol and isocyanate

c) To compare physical, mechanical, morphological with Environment Stress Cracking Resistance (ESCR) and antibacterial properties of polyurethane industry and castor oil with water as blowing agent.

1.5 Significance of Study

This study will focus on synthesizing polyurethane with different ratio of polyol and isocyanate order to find a suitable formulation can be used to act as polyurethane grouting in order to replace cement grouting. By utilizing the optimum polyol and isocyanate formulation to produce suitable expanding ability polyurethane with good physical, mechanical, and morphological properties. Polyurethane grouting are lightweight material, water resistant ability and have high penetration.

Also, this research provides valuable insights into the comparative performance of industrial and castor oil based polyurethane (green polyurethane) for grouting applications, with a focus on different blowing agents (HCFC 141b and water). This study contributes to the development of eco-friendly polyurethane materials by evaluating castor oil-# based polyurethane as a potential alternative to petroleum based polyurethane. This research supports the reduction of reliance on fossil derived polyols, helping to minimize carbon footprint and environmental impact. By exploring the effects of using water as a blowing agent, the study aligns with global initiatives to phase out ozone depleting substances like HCFC 141b. The study provides critical performance data for engineers and manufacturers considering bio based polyurethane for grouting applications in construction, tunnelling, and soil stabilization. Evaluating parameters such as compressive strength, density, expansion ratio, and durability

ensures that the proposed alternative can be effectively used in structural and infrastructure projects. The study helps industries transition toward greener materials by providing comparative data on cost effectiveness, performance, and processing feasibility of castor oil based polyurethane. Exploring alternative blowing agents like water may reduce dependency on expensive or environmentally hazardous chemicals, leading to potential cost savings and regulatory compliance.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

This literature review aims to explore and compare the characteristics of industrial and green polyurethane foams formulated with different types of blowing agents. The study will examine mechanical strength, durability, thermal stability, and environmental impact, providing insights into the advantages and limitations of each type for grouting applications. Through this comparative analysis, the review seeks to contribute to the development of more sustainable and high performance polyurethane grouting materials for future applications.

2.2 Polyurethane Grouting

Polyurethane is widely utilized in grouting applications owing to its exceptional physical and chemical properties. A key characteristic of polyurethane is its capacity for rapid volumetric expansion upon injection, enabling it to efficiently fill fractures, gaps, and voids, even in intricate or irregular geometries (Caou et al.,2024). This expansion forms a secure seal and aids in preventing additional infiltration of water or other fluids, which is very beneficial in waterproofing and structural restoration.

Furthermore, polyurethane grouts demonstrate superior adherence to various construction materials, such as concrete, brick, and steel. This robust adhesion capability improves the mechanical integrity of the restored structures and promotes long term durability (Chen et al., 2018). The material can be customized to display either hard or flexible properties based on the formulation. Rigid polyurethane is typically employed for soil stabilization and void filling, while flexible polyurethane is better appropriate for sealing dynamic joints that experience structural movement (Wang et al., 2024).

Polyurethane offers exceptional resistance to dampness, a range of chemicals, and microbiological invasion. These characteristics render it particularly appropriate for application in hostile environments, including sewage systems, basements, tunnels, and subterranean infrastructure (Sahin, 2025). Moreover, its rapid curing period facilitates building and repair activities with less interruption and decreased downtime.

Due to its advantageous characteristics such as elevated expansion ratios, excellent substrate adhesion, chemical resistance, and formulation versatility of polyol and isocyanate to produce polyurethane continues to be the material of choice for grouting applications in civil engineering, geotechnical projects, and waterproofing systems (Wang et a., 2022).

2.3 Structure of Polyurethane

Polyurethane is a polymer made up of organic units linked together by urethane (AlMaadeed et al., 2020). Depending on its application, it can be characterized as thermoset and thermoplastic. They are made by combining a polyol with a diisocyanate in the presence of catalysts and additives.

Both polyol and diisocyanate are available in various variations and ranges, allowing for a wider range of applications for the materials produced. Polyurethane was first discovered in the 1930s and is currently utilized in many items, including coatings, plastic components, seat cushions, and more (Furukawa, 2019).

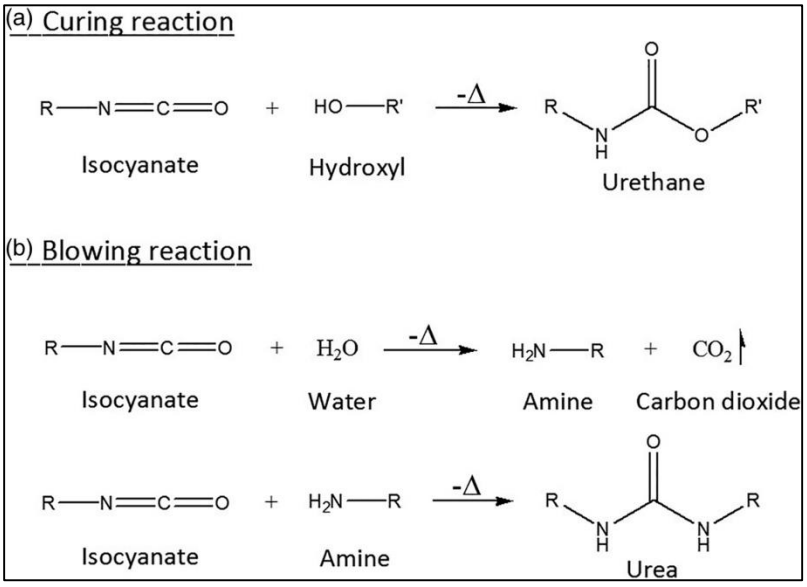


Figure 2.1 Main Chemical Involves in The Production of Polyurethane Foam (a) Curing Reaction Resulting Urethane Linkage (b) Blowing Reaction Resulting Rising of The Foam (Maillard et al., 2020)

The phase segregation in polyurethane has hard and soft segments. Choosing the suitable polyurethane composition is critical because polyurethane can be used in

various applications. A polyol provides elasticity in the supple areas of the material (Jiang et al., 2018). Chain extenders and isocyanate are used in the hard segment to give strength and rigidity at cross linking points.

The production of urethane bonds is crucial to polyurethane's unique chemistry (Rajesh et al, 2019). This reaction takes place between an isocyanate and a hydroxyl, as shown in Figure 2.1 (a). This highly exothermic reaction is typically catalysed by the deprotonation of the hydroxyl by a tertiary amine (triethylenediamine for example). A three dimensional network forms when a polyol with three or more functionalities is used, such as a thermoset polymer. The majority of diisocyanates used in the industry have an aromatic basis, which allows for good reactivity with polyols, an unusual phase separation behaviour during the synthesis of polyurethane, and good mechanical and chemical resilience (Maillard et al., 2021).

The major components that influence the characteristics are polyols, which can either be polyester or polyether based. Flexible structures are produced by higher molecular weight (Mw) polyols (2000 g/mol and beyond) with low functionality. Rigid structures are produced by lower Mw polyols (around 500 g/mol) with high functionalities (Kosmela et al, 2016). A secondary reaction between isocyanate and water, as shown in Figure 2.1 (b), is particularly important in the chemistry of polyurethane since moisture is frequently present in the raw materials and the air. In this process, carbon dioxide and a primary amine are produced. The main amine then forms urea groups by a reaction with an additional isocyanate. Since the isocyanates used to make polyurethane are frequently bifunctional, this reaction does not result in crosslinking; instead, the creation of carbon dioxide encourages the formation of chemical foam (Santamaria-Echart et al., 2021).

In the last few decades, polyurethane has experienced numerous improvements, all of which may be attributed to the material's good character and innumerable advantages. They may be easily produced and modified during sample preparation to manage their parameters. They have excellent bio stability properties, high abrasion resistance, adjustable mechanical strength, superior chemical resistance, and are biocompatible and biodegradable (Zhu et al., 2022).

Polyurethane can be synthesized without using blowing agents, yielding non-foamed, thick polyurethane materials. Non blowing agent polyurethanes are generally utilized in applications necessitating solid or compact materials, including coatings,

adhesives, sealants, elastomers (together known as CASE applications), and rigid structural components (Randall et al., 2020).

In the absence of a blowing agent, the reaction between polyol and isocyanate yields a thick, cross linked polymer network without producing internal gas. This produces a uniform, solid substance characterized by superior mechanical strength, dimensional stability, and exceptional chemical resistance (Pascault et al., 2018). In contrast to polyurethane foams, which are intended for lightweight insulation and cushioning, non-blown polyurethanes are generally employed in applications where strength, abrasion resistance, and load bearing capacity are essential.

Non blowing polyurethane elastomers are extensively utilized in wheels, rollers, industrial gaskets, and protective coatings owing to their exceptional wear resistance and durability under mechanical stress (Hepburn, 2018). In adhesive and sealant applications, the lack of a blowing agent enhances substrate adhesion, increases adhesive strength, and improves durability in adverse conditions (Ionescu, 2023).

Non blowing polyurethanes, without volatile blowing agents also often favoured in situations where minimal emissions, less environmental effect, or worker safety are paramount (Zia et al., 2022). Moreover, these systems are more manageable during processing, as they do not release any harmful gas, but might hinder curing and dimensional precision.

2.3.1 Polyol

Polyols are hydroxyl functionalized esters with a molecular weight ranging from 300 to 9000 g/mol. Flexible polyols, like glycerine and dipropylene glycol, yield low modulus polyurethane, whereas branched and high functionality polyols such as sucrose, sorbitol, and Mannich bases result in more stiff polyurethane systems. Comparable molecules, differing in molecular weight, are routinely amalgamated to create polyol combinations. The number of –OH groups in their molecules varies. Consequently, the hydroxyl content of polyols is characterized by average functionality (Ratna et al., 2020).

Polyols are a fundamental component in the synthesis of polyurethane, acting as the reactive source of hydroxyl (-OH) groups that interact with isocyanates to form urethane linkages. In general, polyols can be classified into two main categories: petroleum based (conventional) polyols and bio based (green) polyols. Conventional

polyols are usually polyether or polyester types derived from fossil fuels, while green polyols are synthesized from renewable resources such as castor oil, soybean oil, palm oil, or other plant based sources (Patel et al., 2020; Salimon et al., 2021).

Figure 2.2 shows the chemical chain of polyester polyol. In this case polyester polyol will be use for the production of polyurethane. Polyesters and polyurethanes rely heavily on polyols as constituents. As chain extenders, they exert a significant amount of control over the mechanical, thermal, and physical properties and can reduce the overall price. For instance, oligomeric polyether polyols are known to significantly improve the flexibility and toughness of polyesters and polyurethanes, whereas monomeric polyols produce more rigid polymers due to their low equivalent weights (de Souza et al., 2021). High molecular weight polyols yield flexible polyurethanes, while low molecular weight polyols yield rigid polyurethane.

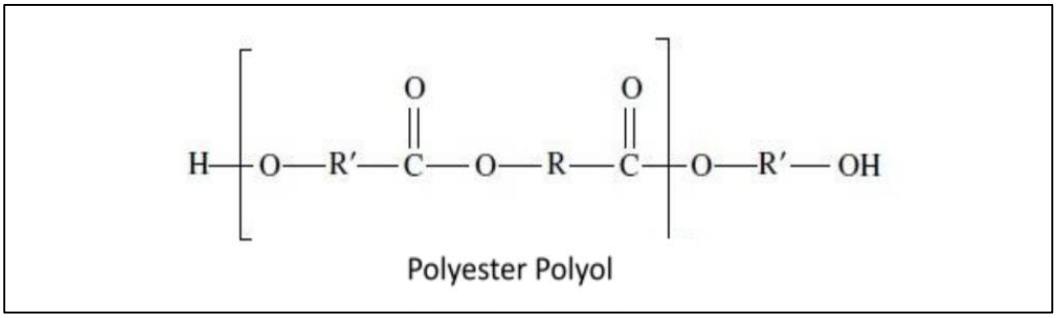


Figure 2.2 Chemical Chain of Polyester Polyol (Peakment, 2021)

In terms of backbone structure, polyols may be broken down into three main types: ether polyols, polyester polyols, and specialty polyols. Ether polyols are the most common type of polyol. Polyols are utilised in numerous products, such as elastomers, fibres, flexible foam, adhesives, sealants, inks, paints, and coatings. They are frequently copolymerized with other monomers to produce polyurethane and polyester elastomers when used in engineering plastics which will be used in this study (Kaur et al., 2022).

Polyols are primarily synthesized from petroleum residue (starter) (Dutta, 2018). Alkaline oxide is used as a catalyst in the chemical reaction that produces polyester polyols from petroleum. A dicarboxylic acid and functional alcohol react to form polyether polyol, which is the end product of this reaction. Hydrolytic stability, cost, viscosity, and flexibility are advantages of polyether polyols, while oxidative stability, modulus/strength, thermal instability, and flammability are negatives (Ratna et al., 2021).

2.3.2 Isocyanate

Two or more isocyanate groups are on each molecule of isocyanates used to create polyurethane. Aromatic diisocyanates, such as TDI and methylene diphenyl diisocyanate (MDI), are the most extensively utilized isocyanates (MDI) (Larson et al., 2019). Figure 2.3 shows the chemical structure of Methlene Diphenyl Diisocynate (MDI). Numerous household items are produced using TDI and MDI. They combine with other substances to produce a variety of polyurethanes. These polyurethanes are used to produce foam for furniture cushions, carpet padding, and waterproof sealant (Polyurethanes & Diisocyanates, 2022). More reactive than the aliphatic isocyanates are these aromatic ones. An estimated 5 million tons of MDI are produced each year, making it the most commonly utilized isocyanate for polyurethane manufacture worldwide. Thermoset polyurethanes by using MDI, which comes in pure and polymeric forms. When producing MDI, aniline, and formaldehyde are reacted in the presence of an acid catalyst, such as hydrochloric acid, and then the product is exposed to phosgene (Ajay et al., 2017).

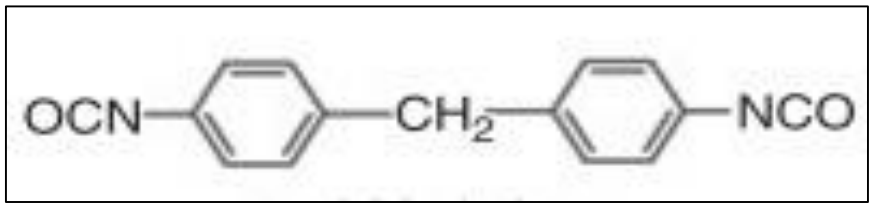


Figure 2.3 Chemical Structure of Methylene Diphenyl Diisocyanate (MDI) (Choi et al., 2019)

MDI polymers have also been included in the formulations of polyurethane. Carbodiimide modified urethane structured MDI exhibit excellent durability, hydrolysis resistance, and mechanical properties. In addition, it is used to increase the low temperature safety of polyurethanes because it improves the comparability of free NCO and polyol during prepolymer production (Liu et al., 2020). The thermal and mechanical properties of polyurethane have been studied Johannes et al, 2013) studied the glass transition temperature, the elastic modulus, and the Charpy impact strength drop when adding plant oil based polyol to the MDI.

On the other hand, static fracture toughness improves only slightly compared to the industry polyurethane. The fracture is due to the additional cross linking caused by

the MDI moieties. Glass transition temperature rises dramatically as a result, whereas elastic modulus falls dramatically. This shows that the polyol type must be suitable for the isocyanate to produce good properties of polyurethanes.

2.4 Green Polyurethane

Green polyurethane (Green PU) denotes polyurethane materials that are wholly or partially sourced from renewable, nontoxic, or eco-friendly resources, as opposed to conventional petroleum based chemicals. Green PU seeks to mitigate environmental effect over the product's life cycle encompassing synthesis, usage, and degradation while preserving performance levels akin to traditional PU. This transition is predominantly accomplished by substituting traditional polyols with bio based, recycled, or CO_2 derived polyols, also known as green polyols. These green polyols are derived from natural oils, sugars, lignin, CO_2, or recycled plastics, functioning as the soft section of the polyurethane backbone. Green building and sustainable construction initiatives (Gama et al., 2018). Table 2.1 shows the difference between types of green polyol with its functions in polyurethane.

Table 2.1
Types of Green Polyol

Type of Green Polyol	Example	Function in PU	Reference
Vegetable Oil Based Polyols	Castor oil, Soybean oil	Act as a primary polyol; provide flexibility, biodegradability, and low toxicity.	Ghasemlou et al. (2019)
Lignin Based Polyols	Kraft lignin, organosolv lignin	Provide rigidity, flame resistance, and improved thermal stability.	Komisarz et al. (2022)
Sugar Based Polyols	Sorbitol, Sucrose polyols	Act as crosslinks; increase foam hardness and reduce cell size.	Shin and Lee (2025)
CO_2 Based Polyols	Cardyon® (Covestro)	Reduce carbon footprint; offer similar reactivity to petroleum-based polyols.	Covestro AG. (2020).
Recycled Polyols	PET-based polyol	Derived from recycled plastics; provide structural strength and reduce waste.	Fang, W.et al. (2020)
Cellulose Based Polyols	Microcrystalline cellulose	Enhance thermal stability and mechanical strength in rigid foams.	Węgrzyk et al. (2023)

Green polyols vary significantly in their chemical structure and hydroxyl value depending on their origin, processing method, and the degree of functionalization. For instance, castor oil based polyols naturally contain hydroxyl groups and are widely studied due to their biodegradability and non-toxic nature. However, their hydroxyl value can differ based on modifications such as transesterification, epoxidation, or hydrolysis (Zhang et al., 2020). This is because these chemical modifications alter the number and reactivity of hydroxyl groups present in the molecular structure of the polyol (Ghasemlou et al., 2021).

The hydroxyl value expressed as the number of milligrams of potassium hydroxide (KOH) required to neutralize the acetic acid taken up by one gram of polyol is a critical parameter that determines the reactivity of the polyol with isocyanates (Myslicki et al., 2022). A higher hydroxyl value typically implies a greater number of reactive sites, which leads to increased crosslinking density and mechanical strength in the resulting polyurethane (Liu et al., 2019). On the other hand, polyols with a lower hydroxyl value may produce polyurethane with more flexibility but lower mechanical properties (Ramírez-Wong et al., 2020).

2.4.1 Castor oil Polyol

Green polyols are compounds derived from soy that are used to manufacture flexible foam for furnishings, mattresses, mattress toppers, pillows, carpet cushions, and vehicle seats (Müller, 2021). Castor oil is a common feedstock used to produce polyols for the production of polyurethane as shown in Figure 2.4. Castor oil exhibits biodegradability, low toxicity, excellent flexibility, enhanced hydrolytic stability, and excellent adhesion and abrasion. In addition, castor oil based polyurethane can provide a variety of desirable qualities that make it suitable for a wide range of applications, including those in which sustainability, flexibility, and abrasion resistance are essential (Da Silva Neto et al., 2024).

In green polyurethane systems, variability in hydroxyl value directly impacts the foam morphology, thermal stability, and mechanical properties of the final product. For example, a castor oil based polyol may have a hydroxyl value around 160 mg KOH/g, but after modification through ring opening reactions or addition of multifunctional alcohols, it can reach values exceeding 250 mg KOH/g, enhancing its compatibility with rigid foam applications (Zhou et al., 2023). Thus, controlling and tailoring the hydroxyl

value in green polyols is essential for designing bio-based polyurethane materials that can match or surpass the performance of conventional systems.



Figure 2.4 Castor oil (Zhou et al., 2023)

Castor oil is a widely recognized bio based polyol in polyurethane production due to its inherent hydroxyl functionality, renewability, and nontoxic nature. Unlike other vegetable oils that require extensive chemical modification to introduce hydroxyl groups, castor oil naturally contains hydroxyl groups in the form of ricinoleic acid, which constitutes approximately 90% of its fatty acid content (Zhang et al., 2020). These inherent hydroxyl groups make castor oil a highly reactive material for polyurethane synthesis, offering a more sustainable and less energy intensive alternative to petroleum based polyols (Salimon et al., 2021).

The hydroxyl value of castor oil typically ranges between 160–170 mg KOH/g, which directly influences its crosslinking ability with isocyanates (Liu et al., 2019). In the context of grouting materials, this hydroxyl value is critical because it determines the formation of a dense, interpenetrated polymer network, which affects mechanical strength, dimensional stability, and water resistance are key properties for structural applications like sealing cracks or voids in concrete (Ramírez-Wong et al., 2020). Additionally, the relatively high hydroxyl value of castor oil allows it to generate rigid polyurethane networks that exhibit strong adhesion to substrates and excellent compressive strength are the essential for effective load transfer and sealing performance in grouting applications (Zhou et al., 2023). When further modified

through processes like transesterification or incorporation of polyfunctional alcohols, the hydroxyl value can be increased to above 250 mg KOH/g, further enhancing the reactivity and strength of the resulting polyurethane matrix (Ghasemlou et al., 2021).

In addition to its great strength and flexibility, polyurethane derived from castor oil has a high tear strength, meaning it can endure tearing and other forms of mechanical stress. This makes it an appropriate material for applications involving high levels of stress or impact. It has also been discovered that polyurethane derived from castor oil has excellent chemical resistance, meaning it can resist harm from a variety of substances. This makes it an ideal material for situations in which it may be exposed to chemicals or other harsh substances.

As a grouting material, castor oil based polyurethane has the ability to flow and effectively fill cavities, especially in narrow spaces. This is because to its low viscosity, which enables it to penetrate small cracks and gaps before expanding and hardening to produce a strong and permanent bond. This makes it an ideal material for filling gaps and spaces in concrete, masonry, and metal structures.

Polyurethane derived from castor oil adheres well to a variety of surfaces, including concrete, metal, and wood. This assures that the grouting material will adhere firmly to the structure, hence giving long lasting stability and strength (Zhang et al., 2024). A significant and expanding use is the manufacture of flexible and stiff polyurethane foams utilized for insulation, packaging, and cushioning. These foams have mechanical qualities akin to petroleum based alternatives while delivering superior environmental performance (Araújo et al., 2020).

Moreover, castor oil based polyurethane exhibits good resistance to hydrolysis and chemical degradation, making it suitable for underground and aqueous environments where grouting materials often encounter water ingress and variable pH conditions (Myslicki et al., 2022). They are extensively utilized in protective coatings, wood finishes, sealants, and industrial adhesives, where their natural origin enhances value in green building and sustainable construction initiatives (Gama et al., 2018).

2.4.2 Blowing Agents

Blowing agents are chemical chemicals utilized to form a cellular structure in materials like polyurethane (PU) foams by producing gas bubbles during the polymerization process. These gasses become confined within the polymer matrix,

resulting in foam expansion. The use of the foam structure markedly enhances attributes including thermal insulation, cushioning, and lightweight qualities (Lima et al., 2022). Blowing agents are crucial in the production of flexible, rigid, and semi rigid polyurethane foams, classified into physical (volatile) and chemical (reactive) categories.

Table 2.2 shows the difference of the type blowing agents and the example of each type. There were two type of blowing agents which are physical and chemical blowing agent. Physical blowing agent are volatile liquids or gases that evaporate due to heat released during the polyurethane reaction. The resulting gas will form foam cell. Examples include hydrocarbons (e.g., pentane), hydrofluorocarbons (HFCs), hydrochlorofluorocarbons (HCFCs), and hydrofluoroolefins (HFOs) (Gama et al, 2018).

Chemical blowing agent generate gas through chemical reactions within the formulation. The most common chemical blowing agent in polyurethane systems is water, which reacts with isocyanate to form carbon dioxide (CO_2) gas (Ionescu, 2016).

Table 2.2
Different type of blowing agent

Type	Example	Mechanism	Common Applications	Reference
Physical	HCFC 141b	Evaporation from exothermic heat	Rigid insulation foams, structural foams	Grumo et al. (2019)
	Pentane (n-/iso-/cyclo)	Evaporation	Appliance insulation, construction panels	Ionescu (2016)
	HFC 245fa	Evaporation	Spray foam insulation	Gama et al. (2018)
	HFO 1233zd	Evaporation	Low GWP rigid foams	Gama et al. (2018)
Chemical	Water	Reacts with isocyanate → CO_2	Flexible foams, green PU foams	Al-Moameri et al. (2019) Ionescu (2016)
	Azodicarbonamide (ADC)	Decomposition → N_2, CO	Microcellular PU, plastics	Hanifarianty et., (2022)

Polyurethane foams are integral to various applications, including grouting, due to their excellent mechanical properties and adaptability. The selection of blowing agents significantly influences the foam's characteristics and the advantages. Table 2.3

shows the comparison between advantages and disadvantages of the types of blowing agent.

This review examines the use of HCFC 141b and water as blowing agents in the production of PU foams derived from castor oil based polyols, focusing on their implications for grouting applications.

Table 2.3
Advantages and Disadvantage Different Types of Blowing Agent

Type	Advantages	Disadvantages
HCFC 141b	Good foam quality; easy processing; low thermal conductivity	Ozone depleting; phased out under Montreal Protocol
HFC 245fa	Non-ozone depleting; good insulation	High global warming potential (GWP)
HFO 1233zd	Very low GWP; non-ozone depleting	Higher cost; limited availability
Pentane	Low cost; good insulation performance	Flammable; safety concerns in production
Water (chemical)	Environmentally friendly; renewable; cost-effective	Produces less expansion than physical agents; may increase foam density
ADC (chemical)	Fine cell structure; controllable decomposition	Generates toxic decomposition by products at high temperatures

Note: Jaf et al. (2023); Chen and Huang (2024); Jin et al. (2019) & Kmetty et al. (2018)

The 1,1-Dichloro-1-fluoroethane (HCFC 141b) has been widely used as a physical blowing agent in rigid polyurethane foams, particularly for insulation materials in building facades. Its application in PU foam formulations has been associated with low thermal conductivity and desirable insulating properties (Lingling, 2023).

However, environmental concerns have arisen due to HCFC 141b's ozone depletion potential (ODP) and global warming potential (GWP). These concerns have led to regulatory measures aimed at phasing out HCFC 141b in favour of more environmentally friendly alternatives. However, environmental concerns have arisen due to HCFC 141b's ozone depletion potential (ODP) and global warming potential (GWP). These concerns have led to regulatory measures aimed at phasing out HCFC 141b in favour of more environmentally friendly alternatives (Richard,2015). It is shows the foams produced with HCFC 141b exhibit low thermal conductivity and good mechanical strength, beneficial for insulation and structural support in grouting. However, environmental regulations necessitate the transition to alternative blowing agents.

Water can serve as a chemical blowing agent in polyurethane foam production (Al-Moameri et al., 2019). It reacts with isocyanate groups to generate carbon dioxide (CO_2), which acts as the blowing gas. While water blown foams are considered more environmentally benign due to the absence of Ozone Depletion Potential (ODP) and low Global Warming Potential (GWP), they present challenges such as higher foam density and increased thermal conductivity. These factors can affect the insulation performance and mechanical properties of the foam, which are critical in grouting applications (Torres-Rivas et al., 2018). Research shows water is environmentally friendly, water blown foams may have higher densities and thermal conductivities, potentially compromising insulation efficiency and increasing material usage. These factors must be balanced against environmental benefits.

A research explored the use of castor oil in the development of rigid polyurethane foams produced using water as a blowing agent. Their study highlighted significant improvements in mechanical properties, including an increase in foam density (77–117 kg/m³), Shore A hardness, a measurement scale used to determine the hardness of flexible materials like rubber and soft plastics The indentation resistance of a material when a spring loaded indenter is pressed against it. The higher the Shore A value, the harder the material. The water blown polyurethane shows the shore A hardness is between 50–69.5, and compression strength were between 1.9–3.3 MPa when compared to unmodified foams. This approach emphasizes the viability of castor oil as a sustainable alternative to petrochemical based polyols (Srinivasan et al. 2023). An examined water blown polyurethane adhesives made from castor oil and bamboo charcoal composites, achieving low density foams with enhanced water resistance and thermal stability. The study confirmed that incorporating 4 wt % water as a blowing agent produced foams with optimal mechanical performance, making them suitable for applications such as interior particleboards (Lee et al. 2019).

Researcher employed a statistical mixture design to optimize the formulation of bio based polyurethane foams derived from castor oil polyols. Their findings indicated that adjusting the composition allowed for tuneable physical and mechanical properties, supporting the feasibility of water blown systems for customizable foam production (Kakaide et al., 2024). It is shown the bio foams developed had densities ranging from 61 to 100 kg/m³, compressive moduli from 11 to 15 MPa, compressive strengths between 273 and 429 kPa, tensile moduli from 3.2 to 4.9 MPa, and tensile strengths between 370 and 500 kPa.

They are also investigated the thermal and mechanical properties of castor oil based polyurethane foams produced using water as a blowing agent. The study found that the water blown foams exhibited superior thermal insulation properties and mechanical resilience compared to conventional formulations, highlighting their potential for energy-efficient building applications (Bera et al., 2020).

In a comparative study, analysed the environmental impact of water blown castor oil based polyurethane foams using life cycle assessment (LCA). The results indicated a significant reduction in greenhouse gas emissions compared to traditional polyurethane foams, further emphasizing the environmental benefits of castor oil and water as sustainable raw materials (Zhang et al., 2021).

2.5 Grouting Material

Grouting is a method for repairing and strengthening matrices that have been damaged or loosened (Gullu et al., 2016). Polymers are now commonly utilized as grouting materials in concrete bridges and concrete buildings because they are cost effective and effective in sealing any cracks or holes that may emerge (Shen et al., 2020). In addition, it can be used to patch pipeline leaks, seal dam joints, anchor walls on construction sites, and stabilize the soil.

Historically, cement, also known as cement nous grout, was used as a grouting material (El-Reedy, 2020). Cementitious grouting is a process in which a fluid cementitious grout is pumped under pressure to fill forms, voids, and cracks as shown in Figure 2.5. It is applicable in a variety of contexts, including bridges, marine applications, dams, and rock anchors. In fact, 94% of American contractors use cement based grout for both commercial and residential projects.



Figure 2.5 Example of Cement Grouting (El-Reedy, 2020)

Cement based grout has a long history of performing its intended function. However, there are issues with cement based grout that make it unsuitable for certain applications (Renovations, 2019). Cement grouting is dirtier, slower, and more disruptive than polyurethane grouting. From commercial floors to interstates and everywhere in between, polyurethane grouting is a cost effective, quick, and efficient solution for addressing settlement and instability. Also, compared to cement grout, polyurethane grout is lighter, has a faster setting time, does not shrink, requires less equipment, and can be installed in any climate (Keith., 2020).

2.5.1 Polyurethane as Grouting Material

Polyurethane grouting involves the injection of expanding polyurethane to fill and seal cracks and voids and re level slabs. Polyurethane resin grouting steps out when it comes to improving the mechanical and sealing capabilities of soil and rock environments and constructions (Saleh et al., 2019). Under pressure, a liquid grout material is injected into the rock environment or construction as the basic premise of grouting technologies as shown in Figure 2.6. Filling the fissures and pores in the rock mass with grouting material causes the fragmented rock mass or loose grains of material

to solidify and join. In the mining business, polyurethane grouting technologies were first deployed in the 1980s (Bodi et al.,2016).

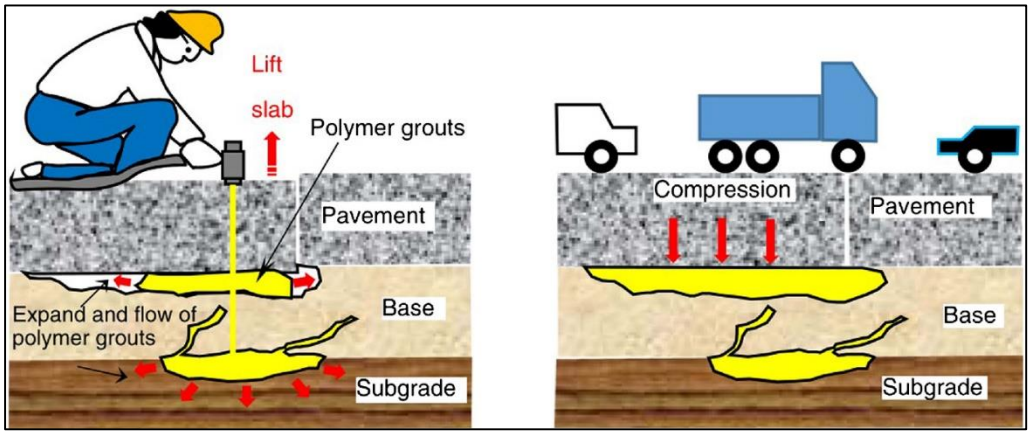


Figure 2.6 Example of Polyurethane Grouting (Wei et al., 2017)

There are two types of polyurethane grouting; single component and plural component. There are a variety of polyurethane grouting processes available, each with its density, reaction time, water reaction, expansion characteristic, and flexibility. It is critical to choose the suitable grouting for each project (Huang et al., 2022). Single component grouting has a low viscosity and can be injected into loose sandy soils, where they will react with groundwater (Ciardi et al., 2021). Figure 2.7 shows the example of the single component of polyurethane grouting. The term "single component" indicates that this epoxy grout does not require the mixing of two components. Technically, the grout is composed of urethane or acrylic silicone resin. These steps can be accelerated by adding additional catalysts. Repairing concrete pipes, walls, tunnels, and culverts with this grouting are frequent. Wet soils can be either curtain grouted or stabilized by injecting varying amounts of polyurethane into them, depending on the desired degree of subsurface water migration prevention.



Figure 2.7 Example of Single Component Polyurethane (Sabri et al., 2021)

Plural component grouting is used to re level concrete slabs and fill huge voids. Figure 2.8 shows the example of plural component of polyurethane grouting which contain certain types polyol and isocyanate. This grouting is applied with the help of a proportioning apparatus, which heats the chemicals and pushes them through a network of pipes to an impingement gun. This polyurethane foam expands at varied rates depending on the formula when the elements are combined. A concrete slab can be moved with ease with this grouting (Sabri et al., 2021). Grouting made of polyurethane has several advantages, including being lightweight, reactive and curing, water repellent, and available in an array of densities, speeds, and strengths.



Figure 2.8 Example of Plural Component Polyurethane (Ali et al., 2016)

The polyols used to produce polyurethane can be either natural or petrochemical based. Currently, polyether polyols synthesized from petrochemical are widely used in the production of polyurethane due to their availability in the market compared to other polyols (Ali et al., 2016). Almost 90% of industrial polyurethane production uses petrochemical based polyols as its main component (Ghasemlou et al., 2019). Whereas the industry is gradually adopting more sustainable, bio-based feedstock to reduce the carbon footprint, the current manufacturing landscape remains deeply reliant on petroleum-based precursors.

2.6 Physical Properties of Polyurethane

Physical properties are characteristics of a substance that can be observed or measured without changing the substance's chemical identity. These properties describe the physical state and behaviour of matter. Examples of the physical properties of polyurethane are include foam reaction time, density, rheology for viscosity study and solubility.

The sample was foamed until it expanded entirely and lost its tackiness, at which point the foam reaction time, cream time, rise time, gel time, and tack free time were all measured consecutively. Cream time is the time for the mixing of polyol and isocyanate components until the mixture starts to turn creamy. It is helps to determine how quickly the material starts to expands (Zhou et al., 2023; Li et al., 2020). The standard value of polyurethane grouting is in the range between 10-20 seconds. Gel time is the time from the mixing until the material start to lose fluidity and become gel like properties. It is critical for injection grouting as it is the time available to pump the material before it thickens. The standard value of the gel time must be low as it could disturb the curing process (Ramírez-Wong et al., 2020). Rise time is the time for the foam become fully expand. It is importance for assessing when to stop injection (Liu et al., 2019). The standard value of polyurethane grouting for rise time is more than 40 seconds. Lastly, the tack free time is the time for the foam surface to become non sticky. It indicates the ends of the surface curing and when it safe to touch (Liu et al., 2019. The standard time of polyurethane grouting also must be low in seconds as to form a rigid polyurethane (Amran et al.,2021).

The apparent density is example of physical properties. The ASTM D1622 is a technique measured an apparent foam density. The apparent density of polyurethane

(PU) foams is a key parameter that governs their performance in structural, thermal, and chemical applications. Higher density foams typically demonstrate enhanced compressive strength, dimensional stability, and solvent resistance, while lower density foams provide lighter weight and improved thermal insulation but at the expense of mechanical stability (Zhang et al., 2010). Also, the choice of blowing agent has a significant influence on foam density. Physical blowing agents such as HCFC 141b and its alternatives generally produce foams with more uniform closed cell structures, resulting in higher apparent densities (Lima et al., 2022). In contrast, water as a chemical blowing agent generates CO_2 through its reaction with isocyanates, which often leads to the formation of larger, less uniform cells, thereby reducing the density (Prociak et al., 2019). The increasing use of bio based polyols, such as castor oil, also affects foam density due to their high viscosity and hydroxyl functionality, which can restrict bubble growth and alter cell morphology (Silva et al., 2013). Overall, apparent density serves as a fundamental indicator of foam morphology and directly correlates with the balance between mechanical reinforcement and insulation performance in polyurethane systems. The average apparent density of the polyurethane must in between 56-76 kg/m3 (Saleh, 2019).

In grouting research, the followability and inject ability of polyurethane are often evaluated using simplified indices rather than using rheometer, due to the rapid polymerization and foaming of polyurethane systems. Practical rheological indicates such as flow distance through a transparent hose or capillary per unit mass have been adopted as indirect viscosity measures, since greater travel distance corresponds to lower viscosity and higher mobility (Yoon et al., 2021). This type of approach aligns with field oriented tests where pump ability and spread are more relevant than precise shear stress curves (Li et al., 2020). Recent studies emphasize that an increased rheology index correlates with higher viscosity and reduced flow like ability, thereby directly affecting penetration in cracks and pores (Zhang et al., 2023). Such indices provide engineers with a simple way to link laboratory foam/grout characterization with onsite injection performance, especially for reactive systems where rheometer struggle to capture rapid viscosity rise (Wang et al., 2025). Consequently, rheology index methods based on distance–mass ratios are increasingly recognized as practical tools for assessing followability, complementing standard rheometer in the study of industrial and bio based polyurethane grouts. The rheology of the sample is studied to calculate the polyurethanes and reinforced polyurethane. The viscosity studies the flow and time

taken off the polyurethane sample. A lower rheology index with a short time taken indicates polyurethane has a high viscosity, which is suitable for grouting properties (Dutta et al., 2019).

Water absorption is the amount of water absorbed by a substance, measured as the weight of the absorbed water on the dry material. The water absorption behaviour of polyurethane is a critical property influencing its long term durability in grouting and construction applications. polyurethane generally exhibits low to moderate water uptake due to its hydrophobic backbone and closed cell morphology; however, the presence of urethane linkages and micro voids allows for limited moisture ingress (Zhang et al., 2019). Reported absorption levels typically range from 2% to 12% of weight gain depending on formulation, blowing agent, and exposure environment (Gama et al., 2022). In water saturated conditions, a maximum uptake near 12% has been documented, which corresponds with diffusion of water molecules into soft segments and hydrogen bonding interactions with polar urethane groups (Ma et al., 2021). For polyurethane grouting specifically, controlling water absorption is vital because excessive uptake can compromise dimensional stability and adhesion to the substrate. Thus, monitoring percentage weight gain during immersion provides a simple and effective durability indicator. The water absorption of the polyurethane should be at most 12% (Possart et al., 2017). Hydrophobic or repel water foam is helpful in grouting applications (Huang et al., 2022).

Moisture content is the quantity of water that can be removed from the sample without altering its chemical structure. The moisture content calculation uses the formula using the air oven method for a week of durations. Because of its closed cell structure, rigid polyurethane insulation is rarely influenced by water or moisture: it does not absorb or transport water. Thus there is no capillary action, so regular moisture in buildings does not enhance thermal conductivity (Ann, 2022). Several researchers have confirmed that the low moisture content of rigid polyurethane foams is primarily attributed to their closed cell morphology, which restricts vapour diffusion and eliminates capillary transport pathways (Li et al., 2020). As a result, rigid polyurethane materials generally retain their thermal insulation efficiency and dimensional stability even under fluctuating humidity, making them suitable for building envelopes and grouting works where long term exposure to moisture is inevitable (Gama et al., 2022). Furthermore, the oven dry method has been widely employed to quantify residual moisture in polyurethane samples, providing reliable data on their stability and

confirming that moisture uptake does not significantly alter the chemical structure of the polymer (Cangemi et al., 2023).

2.7 Chemical Properties

Chemical qualities of matter refer to its potential to experience a chemical change or reaction due to its makeup. Identify the elements, electrons, and bonds that could cause a chemical shift. An infrared spectrum of absorption or emission of a solid, liquid, or gas can be obtained using the Fourier transform infrared (FTIR) technique. FTIR is widely applied to confirm the chemical structure of polyurethane. The characteristic absorptions of polyurethane include the stretching of N–H groups (typically 3300–3400 cm^{-1}), C–H stretching of aliphatic chains (2850–2950 cm^{-1}), and the carbonyl group C=O (1700–1725 cm^{-1}), which is strongly associated with urethane linkages (Li et al., 2020). The carbonyl band is particularly sensitive to hydrogen bonding, and its position can indicate the degree of phase separation between hard and soft segments (Gama et al., 2022). The absence of peaks corresponding to the isocyanate group (–N=C=O, around 2270 cm^{-1}) in the final product confirms complete reaction during polymerization (Ma et al., 2021). Recent studies highlight FTIR as a reliable technique not only for structural identification, but also for correlating functional group intensities with mechanical, thermal, and moisture resistance properties of polyurethane foams (Wang et al., 2025). The summarize of FTIR spectrum of polyurethane was shown in Table 2.4.

Figure 2.9 shows the FTIR spectrum of various ratios of NCO/OH (Chee et al., 2012). The figure shows the presence of the amine (N-H), carbonyl urethane group (C=O), carbonate group (CN-H) and (C-O-C), indicating that there is a linkage of polyurethane. From the observation, the free NH band, hydrogen-bonded NH with oxygen (ether) and hydrogen bonded NH with oxygen (carbonyl) are all present in the urethane linkage.

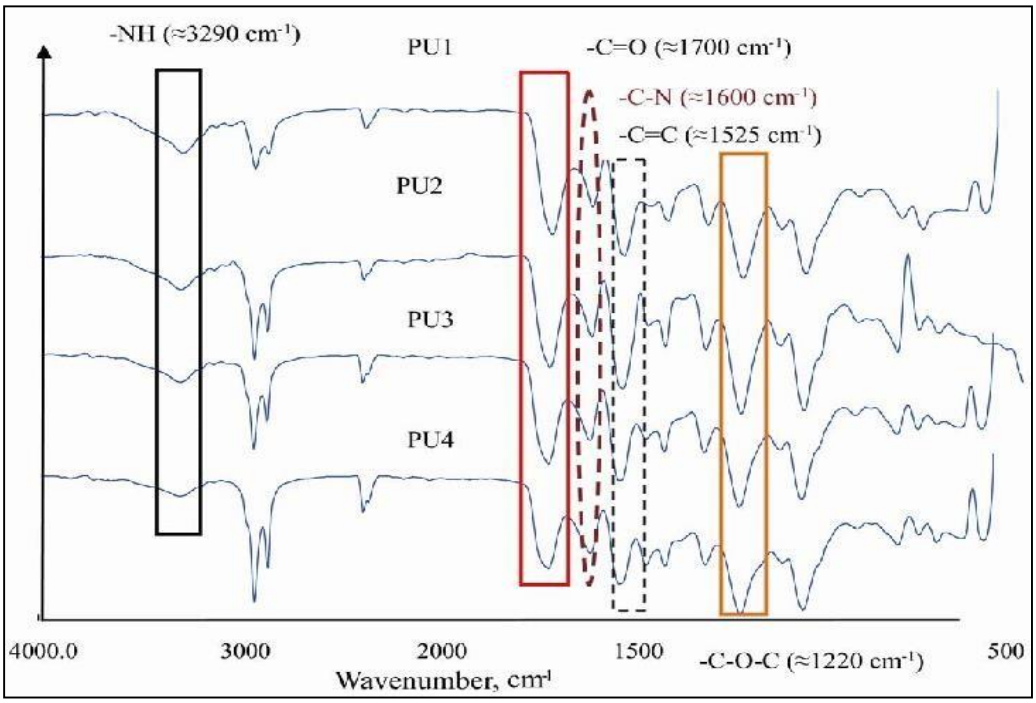


Figure 2.9 Example of The FTIR Spectrum Various Ratios of NCO/OH (Chee et al., 2012)

Table 2.4
FTIR Spectrum of polyurethane

Wavenumber (cm⁻¹)	**Functional Group**	Polyurethane **Structure**
3300–3400	N–H stretching	Urethane linkage (hydrogen bonded)
2850–2950	C–H stretching (aliphatic)	Polyol chains / aliphatic segments
1720–1700	C=O stretching	Urethane carbonyl (free or H-bonded)
1640–1610	C=O stretching (amide I region)	Hard segment hydrogen bonding
1530–1510	N–H bending + C–N stretching (amide II)	Urethane hard domains
1410–1380	C–H bending (methyl)	Aliphatic polyol groups
1220–1080	C–O–C stretching	Ester/ether linkages in polyol backbone
~2270	–N=C=O stretching (isocyanate group)	Disappears if fully reacted

Note: Dulyanska et al. 2024

2.8 Mechanical Properties

Mechanical properties can be defined as property that involves both stress and strain or a reaction toward an applied force. One example of mechanical properties is compression strength. Compression is one of the most crucial tests. This test is for compressive loads, such as platens or special equipment on a universal testing machine, which is used to evaluate the behaviour of a sample when subjected to crushing loads. Stress strain diagrams can evaluate features such as elastic limits and ratio limitations, failure sites, yield strength, and compressive strength in various materials.

The polyurethane's density can affect the polyurethane foam's mechanical characteristics (Manjun et al., 2020). The lower the density of the polyurethane, the more mechanical qualities can have little impact effect compared to the higher density. As structural materials, rigid polymer foams can be utilised due to their combination of high mechanical properties and low density. Whatever their application, their optimization requires an understanding of the microstructure mechanical properties relationship. In fact, the mechanical response of these materials is dependent upon their structure and the inherent properties of the polymer in the cell wall (Alsubari et al., 2021). Also, the size of the material plays an essential role in the mechanical properties. The thickness of the cell wall, the size distribution, and the shape of the cells determine the structure properties. Larger samples may be weaker than smaller ones but may also be stronger (Jiang et al., 2018).

The thickness or the diameter of the polyurethane sample also has a certain impact on the stress strain curve (Ju et al., 2015). Compression testing on pure polyurethane stated that the absorption energy level and the compression properties of polyurethane increase with thickness. The mechanical properties of polyurethane also affect the sample's formulation and morphology of the material. Based on (Gama et al., 2018) research on the mechanical performance of polyurethane foams, the mechanical properties of the polyurethane depend on the diameter of the cell and the arrangements of the cell in the sample. The linear cell arrangement indicates that the sample has a strong elastic response to the material. Also, the curve in the stress strain presents a plateau due to material deformation of the rupture of the cell walls.

Bio based polyols such as castor oil, lignin, vegetable oils, and chemically modified bio polyols were produce foams whose compressive strength and modulus can either approach or fall below those of industrial polyurethane depending on the

replacement level and any chemical modification or compatible applied. Several studies report that partial replacement (low–moderate wt%) with lignin or functionalized castor oil polyols can maintain or slightly enhance compressive stiffness due to increased hard segment content or forming fillers, while high replacement levels such as large fractions of unmodified castor oil tend to increase cell size, open the cell structure, and substantially lower compressive strength (Kim et al., 2024). Conversely, tailored modifications of castor oil which contains thiolene functionalization, increased hydroxyl value, or multifunctional polyols have produced bio polyurethane foams with compressive strengths reported as high as ~571 kPa and have markedly improved modulus, demonstrating that bio based polyurethane performance with optimum formulation dependent can match with the industrial polyurethane when polyol functionality, crosslink density, and cell control are optimized.

From these findings, bio based polyurethane can be act as a grouting material. The ideal ratio for polyurethane to act as grouting materials can be determined by examining the stress strain curve (modulus) of the sample, which is influenced by the density and thickness sample of the polyurethane (Li et al., 2020).

2.9 Morphology Properties

Morphology properties can be defined as the study of the size, shape, surface, and structural cell characteristics of living or un-living things. A scanning electron microscope (SEM) test can be used to study morphology. SEM uses an electron beam to magnify a picture of a sample for further analysis. The specimen's structure can be determined using ASTM E2089. Solid inorganic materials microanalysis and failure analysis can be carried out using SEM microscopy, also known as SEM analysis. High magnification electron microscopy yields sharp images with a fine resolution and can carefully examine the smallest characteristics and artefacts (Zhao et al., 2022).

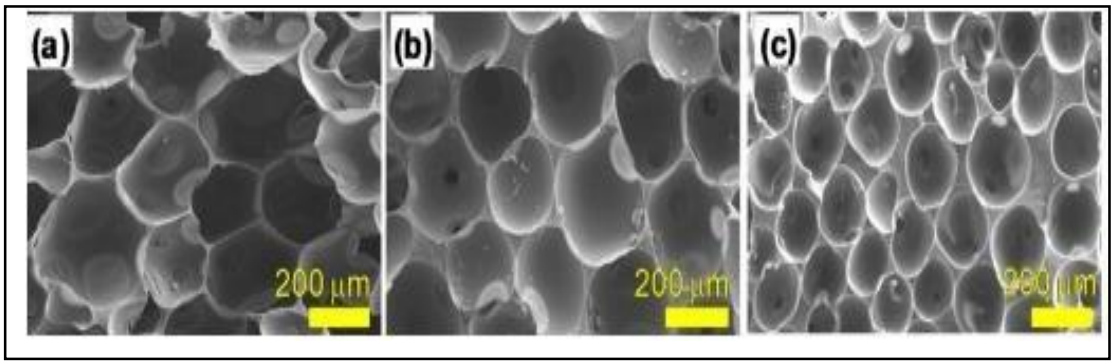


Figure2.10 SEM Photograph of Polyurethane with Different Densities (Kairytė et al., 2020)

Based on the SEM findings in Figure 2.10, polyurethane can be considered a foam matrix composed of multiple hollow foams. Each closed foam is carefully stacked on top of the next cell. These cellular structures have a more polygonal shape in samples with a low density, and the outside walls of individual cells are nearly completely in contact with one another. When the sample has a higher density, the foams resemble spheres, and the contacting area between each foam diminishes, suggesting that the specific surface area of the foams has decreased (Kairytė et al., 2020).

SEM studies consistently report that polyurethane cellular microstructures whose average cell size, cell openness (closed vs. open cells), and wall thickness depend strongly on formulation between polyol and isocyanate and other addictive and also on the incorporation of fillers or fibre reinforcements; these microstructural features correlate closely with foam density, compressive strength and impact behaviour (Mendes et al., 2024).

SEM analysis also been used to track effects of chemical modification of castor oil (e.g., transamidation, acetylation) or the use of bio polyol blends (lignin, PCL triols) on the microstructure: modified polyols tend to produce more uniform cell structures and reduced incidence of large voids or irregular cells, which is shown by narrower cell size distributions in SEM micrographs and aligns with improved mechanical and thermal behaviour reported in those works (Wloch et al., 2024)

From Figure 2.11 shows the SEM micrograph of castor oil based polyol polyurethane. It indicating the reduce amount of hydroxyl group in polyol contribute to the different size of the foam. Estimated the size of the cell wall foam is between 250 µm to 1000 µm (Hejna et al., 2016). Castor oil-based foams typically form larger cells than pure petroleum-based PU because castor oil has lower reactivity, which slows cell wall formation. It is also shows an open-cell cellular structure on SEM micrograph, especially when plasticizers are added to produce flexible foams (Kim et al., 2024).

These findings on the morphology of polyurethane foam, indicates also, if the foam shows stable cell structure and is smaller in size (uniform cell), it will give better strength and modulus of the polyurethane rather than foam with irregular and bigger cell size (Soberi et al., 2017).

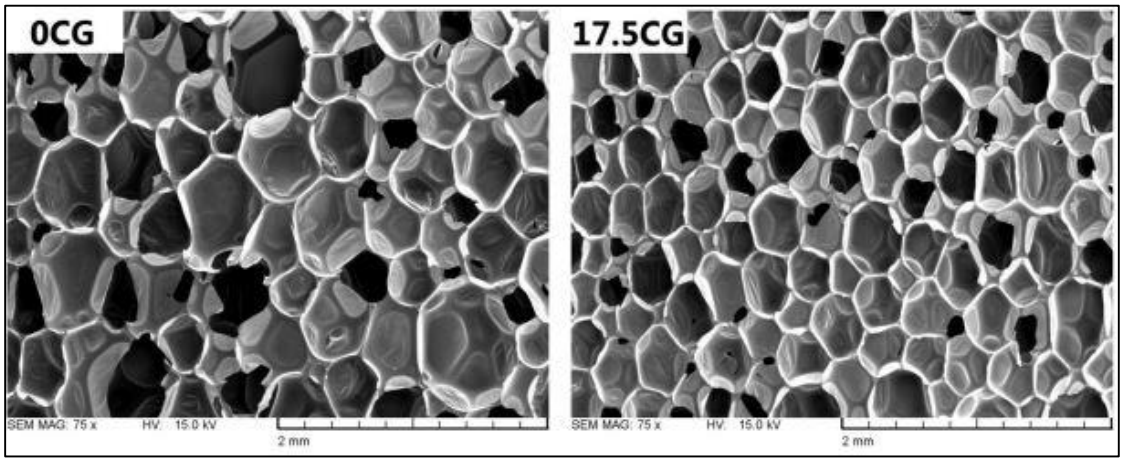


Figure 2.11 SEM photograph of castor oil based polyurethane (Hejna et al., 2016)

2.10 Environmental Stress Cracking Resistance (ESCR)

2.10.1 Soil burial testing

Soil burial tests are commonly used to evaluate the bio degradation and environmental degradation of polyurethane and other polymer materials (Zuliani et al., 2022). Environmental stress cracking is a type of material degradation caused by the simultaneous activity of two degradants: stress and the chemical effect. The chemical ingredients speed up the production of macroscopic brittle cracks but do not chemically degrade the polymer. Similarly, the applied stress is lower than the level that would ordinarily induce mechanical damage to the materials. However, when they work together, they produce material failure (Wypych, 2017).

During a soil burial test on pure polyurethane, the material is normally buried in soil for a specified amount of time, and then its physical and mechanical properties are examined. The test conditions, such as soil type, temperature, and moisture content, can impact the rate and amount of degradation. In comparison to other polymers, polyurethane is often resistant to biodegradation in soil due to its high crosslink density and chemical inertness (Skleničková et al., 2020). Soil burial is a standard method to evaluate polyurethane resistance to biologically-induced stress cracking, as microorganisms can trigger degradation that manifests as surface cracking and loss of mechanical integrity (Lopes et al., 2022).

A study was conducted to examine the polyurethane after a specified period of time buried in soil. According to the findings, a pure polyurethane film that was buried in soil for 120 days lost 35% of its weight, showed evidence of physical and chemical degradation, as well as microbial colonisation. Temperature, moisture content, and the presence of microorganisms were found to affect the breakdown of polyurethane in soil burial testing (Zuliani et al., 2022). Others investigations have revealed that pure polyurethane can remain in the environment for significantly longer durations. Polyurethane foam buried in soil for up to 35 years exhibited no substantial degradation or weight loss, according to the findings (Magnin et al., 2020). It also shows mechanism to failure. This is due to the microbial activity especially fungi like Geomyces pannorum in acidic soils leads to the formation of hydrophilic chain ends, increasing surface wettability and further encouraging microbial colonization. This often results in loss of tensile strength, discoloration, and macroscopic cracking (Tang et al., 2021).

Further study was make of the compressive properties. The pure polyurethane foam buried in soil for up to 720 days. After 180 days of burial, the compressive strength and modulus of the foam reduced by 20 to 50%. This was shows that the compression properties were decreasing over time (Zhang et al., 2022). The decrease in compression strength was caused by foam breakdown. The breakdown is most likely caused by soil microbes and enzymes, as well as physical and chemical forces such as erosion and hydrolysis (Wang et al., 2022).

These results highlight the complex and variable nature of polyurethane biodegradation in soil burial tests and suggest that the rate and extent of degradation may depend on a variety of factors, including the type of polyurethane, the test conditions, and the presence of environmental stressors or inhibitors.

2.10.2 Solubility Testing

Polyurethane is a type of polymer that can exhibit different levels of resistance to environmental stress cracking (ESC), depending on its formulation and the environmental circumstances to which it is subjected. ESC is a form of degradation that occurs when a material is subjected to tensile stress and particular environmental contaminants, such as certain chemicals, oils or solvents. The outcome is the formation of a network of surface fissures, which can damage the material's mechanical strengths and ultimately lead to its failure. Some types of polyurethane, for instance, may have strong water resistance yet are susceptible to breaking when exposed to specific chemicals. In certain circumstances, the polyurethane may be changed to improve its resistance to the specific chemicals (Xie et al., 2024).

A research conducted on the effect of salt water on the degradation of polyurethane foam (Huo et al., 2020). The foam was exposed to seawater for up to sixty days, and changes in its physical and mechanical properties were measured. The results revealed that exposure to salt water decreased the density and compression strength of the foam, indicating that there was degradation. These was because water breaks the polymer chains which have operated since the beginning of immersion. These processes weaken the adhesive irreversibly because they cause a binding failure and the loss of polymer fragments that pass into the seawater (Alia et al., 2013). It is shows the hydrolysis involves water molecule attacking ester or the urethane linkages causing chain breakage, reduced in molecular weight, decrease in mechanical properties to

failure. Hydrolysis is a chemical reaction in which water molecules break down the polyurethane's chemical bonds, causing the polyurethane to decompose into smaller molecules (Kemona et al, 2020). Acidic and alkaline generally accelerate hydrolysis compare to pure water (Li et al., 2020).

Furthermore, a study to examined the impact of acidic solution exposure on the mechanical characteristics of a polyurethane based adhesive. The adhesive was subjected to a solution of hydrochloric acid for up to 120 hours, and its tensile strength, elongation at break, and other mechanical properties were measured. Exposure to the acidic solution significantly decreased the adhesive's tensile strength and elongation at rupture, showing that it had degraded (Rudawska, 2020). This is due to the polyurethane undergoes hydrolysis. The presence of hydrogen ions (H+) in acidic liquids can accelerate this process, resulting in a more rapid breakdown of the polymer. The presence of other oxidizing agents, such as free radicals, which can react with the polymer and result in depolymerisation or crosslinking, might also contribute to the breakdown of polyurethane in acidic conditions (Speight, 2020). Acid environments can lead to reduction in functional groups, weight loss and loss of mechanical properties during prolonged exposure (Głowińska et al., 2024).

Alkaline solution can induce the braking down of ester linkages and greater swelling due to increased hydrophilicity. There were studies shows that alkaline degradation rates can be slower than acidic hydrolysis as base solution catalyse chain breakage and alteration in the morphological properties (Atchison, 2025). Weight analysis of polyurethane immersed in base solution shows initial of weight increased due to the water absorption followed by decreased when degradation starts. This pattern demonstrate that solubility and degradation were the combination of absorption and chemical breakdown (Alía et al., 2025). Hydrolytic degradation also disrupts hydrogen bonding and increase microphase separation in polyurethane, adversely affecting the mechanical performance (Wang et al., 2024).

2.11 Antibacterial Properties

In particular applications, the antibacterial properties of a polyurethane grouting material can be an essential factor. The usage of antibacterial polyurethane grouts can prevent the growth and spread of bacteria, fungi, and other microorganisms that can cause odours, discoloration, and degradation of the grout (Sienkiewicz et al, 2022).

Antibacterial polyurethane grouts have wide range of applications, including the construction and repair of buildings, tunnels, and other infrastructure. In these circumstances, the grout may be exposed to moisture, which can encourage the growth of germs. Antibacterial polyurethane grouts can assist prevent the growth of bacteria and preserve the structural integrity of a building (Qiu et al., 2020).

Escherichia coli (*E.coli*) is gram negative bacterium with thin layer peptidoglycan. The outer membrane containing lipopolysaccharide (LPS) and more susceptible to membrane disruptive agents (Padh et al., 2021). As for Bacillus subtilis (*B.subtilis*) is a gram negative bacterium with thick layer peptidoglycan layer. It has no outer membrane and generally more resistant to environmental stress (Sun et al., 2023). The difference in cell wall structure influence on how the polyurethane surfaces interact with both types of bacteria.

The example of antibacterial testing was shown as Figure 2.12 which study the inhibition zone of polyurethane against gram positive (*Staphylococcus aureus*) and gram negative (*Escherichia coli*). The results show no inhibition zone was observed for the polyurethane films on both bacteria. It also shows that the polyurethane film there were no bacteria growth even has been incubated after 48 hours (Zulkifli et al., 2017).

Generally limited studies have been conducted on the antibacterial characteristics of polyurethane for grouting applications. Nonetheless, some study has been undertaken in this area, and the findings indicate that antibacterial polyurethane grouts can effectively restrict the growth and spread of bacteria (Balouiri et al., 2016). The antibacterial effects of a grout containing silver have been investigated. The grout inhibited the growth of multiple bacterial species, including *Escherichia coli*, *Staphylococcus aureus* and *Pseudomonas aeruginosa*, according to laboratory tests. The research also found that the polyurethane grout containing silver might be used in applications where inhibiting the growth of bacteria and other microbes is crucial (Sehmi et al., 2016).

A study tested the antibacterial properties of a polyurethane coating formulated with castor oil and treated with a quaternary ammonium compound. The coated coating inhibited the development of *Staphylococcus aureus* and *Escherichia coli* and showed promise for usage in applications such as food packaging where controlling the growth of microbes is crucial (Wang et al., 2023). Based on the results, it shows there was inhibit zone on both bacteria. This is proven that bio based polyol such as castor oil has effective against common pathogens. Overall, additional study on the antibacterial

characteristics of castor oil based polyurethane for grouting applications is essential. Further research could assist determine the best effective antibacterial additives and formulations for use in polyurethane grouts containing castor oil.

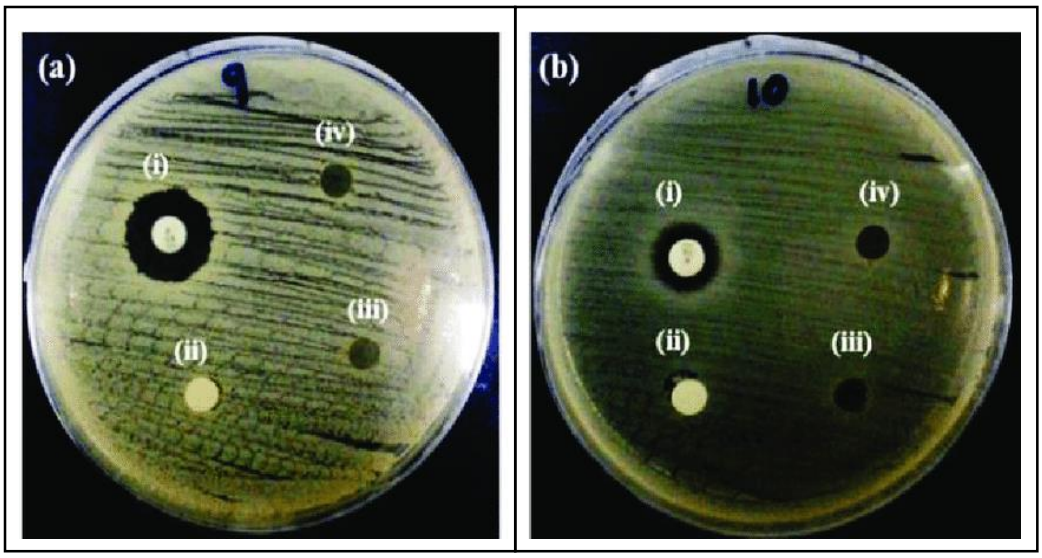


Figure 2.12 Antibacterial activity of polyurethane films against (a)*Staphylococcus aureus* (b) *Escherichia coli* after incubated for 24 h (Zulkifli et al., 2017)

2.12 Summary of Literature Review

Polyurethane (PU) is widely recognized as an effective grouting material due to its high strength, adhesion, and durability; however, industrial PU production commonly relies on petroleum based polyols and HCFC 141b blowing agents, which present significant environmental concerns. Castor oil based polyols offer a renewable alternative, and water as a chemical blowing agent has been shown to reduce environmental impact, though potential trade-offs in density, morphology, and mechanical properties remain. While prior studies have characterized the physical, mechanical, and morphological properties of various PU formulations, limited research has evaluated their long term durability under conditions relevant to grouting, such as soil burial and chemical immersion, or investigated their microbial resistance in geotechnical environments. These gaps underscore the need for comprehensive evaluation of green PU systems, comparing the influence of polyol type and blowing agent on performance, environmental stress cracking resistance, and biodegradation resistance.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 Introduction

This methodology has 3 stages. First part is the determine the polyol and isocyanate formulation by using industrial polyurethane exhibits balance stoichiometry and good properties of polyurethane grouting. Second part of these methods are to compare the industrial polyurethane with green polyurethane by using castor oil polyol. Then a comparison of castor oil with different blowing agents (HCFC 141b and water). Then the last stages to study the durability of all polyurethanes by conducted and compare environmental stress cracking and antimicrobial testing. Further steps of characterization such as physical, mechanical, morphological, will be discuss in this chapter**.** The findings from all the testing will be addressed in Sections 4 and 5 of this thesis.

3.2 Materials

Table 3.1 presents the materials utilized in the production of the industrial industry (PI) and green polyurethane derived from castor oil based polyol (PC). NCL LG108GI Polyol Resin was utilized as a polyol, while NCL LG108 GI Hardener served as an isocyanate, comparable to 4,4-methyl diphenyl diisocyanate, (MDI) in the production of industrial polyurethane. The manufacturing of both products were by NCL Chemical & Equipment Sdn Bhd at Puchong Selangor. Both materials were provided by our industrial collaborator, Al Fazance Resources.

The green polyurethane synthesized by using different blowing agents utilizes castor oil based polyol, which has a hydroxyl value of 161.89 g/mol. Isocyanate (4,4-methylenediphenyl diisocyanate) has an NCO content of 31% and a molecular weight of 360. Pentamethyldipropylenatriamine copolymer (PMDETA) serves as a blowing catalyst, while dimethylcetylhexamine (DMCHA) acts as a gelling catalyst, with both catalysts provided by Teguh Sainstek Sdn Bhd. Water (H_2O) and 1,1-Dichloro-1-fluoroethane (HCFC 141b) will serve as the blowing agents.

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Table 3.1
Materials used in Production of Industry and Green Polyurethane

Material	Appearance	Equivalent Weight (g/mol)	Comments
NCL LG108 GI Polyol	Yellowish	-	OH value : 199 mg KOH/g
Castor oil	Pale yellow viscous	933.45	OH value : 161.68 mg KOH/g
NCL LG108 GI Isocyanate	Dark brown	-	Viscosity : 200
4,4-methylenediphenyl diisocyanate	Dark brown	360	NCO content : 31%
HCFC 141b	Clear colourless	-	Physical lowing agent
Distilled water	Clear colourless	18.02	Chemical blowing agent

3.3 Formulation to Produce Industrial Polyurethane

Table 3.2 represents the three ratios of polyol and isocyanate utilized for the production of industrial polyurethane. This formulation requires the identification of three types of ratios to determine the optimal proportions of polyol and polyurethane. The isocyanate (MDI) was mixed into the polyol within a plastic cup and subsequently stirred for 5 seconds at 3000 rpm utilizing a mechanical stirrer. The sample was left to rest for 24 hours at room temperature for foaming process takes places (Suresh, 2017). Figure 3.1 shows he schematic diagrams of the methods involved.

The ideal polyurethane formulation that yields superior grouting material characteristics will be chosen based on its physical, chemical, mechanical, and morphological properties. A premeneliry study had been conducted to study the mixing speed and time suitable producing polyurethane. Based on the study, speed of the mechanical stirrer was 3000 rpm and stir for 5 seconds were chosen as it was enough to produced polyurethane within the physical properties of standard of grouting applications (Ostapiuk, 2023).

Table 3.2
Composition of Polyol and Isocyanate for Polyurethane Industry

NCL LG108 GI Polyol	NCL LG108 GI Isocyanate
0.5	1
1	1
1	0.5

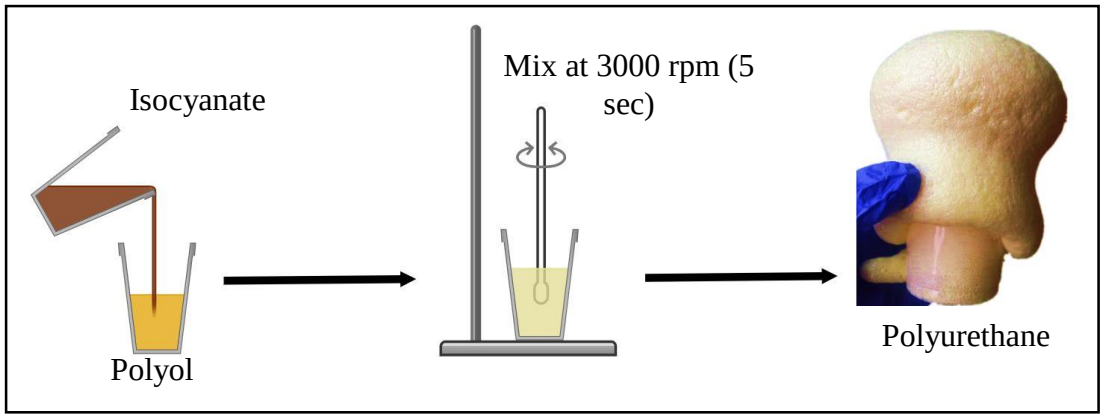


Figure 3.1 Graphical flow methods involved in the production of Polyurethane Industry

3.4 Physical Properties

3.4.1 Foam Reaction Time (FRT)

ASTM D7487 is the standard approach for analysing the foam reaction time of polyurethane with different composition of polyol and isocyanate (Pérez-Tamarit et al., 2019). Using a stopwatch, the characteristics of time, such as cream time, gel time, rise time, and tack free time, were observed and recorded during the foaming process, beginning with the formation of the white precipitate and continuing until the foam completely expanded and lost its stickiness as shown in Figure 3.2. The recording sequence times were as stated in Table 3.3. This technique can also determine the reaction rate of each constituent compound.

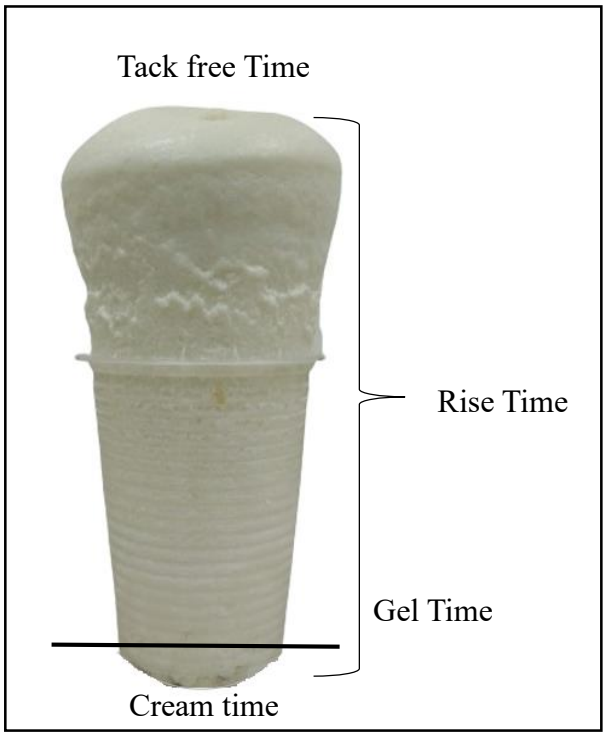


Figure 3.2 Steps to calculate Characterization time

Table 3.3
Definition of each Characterization Time

Characterization of time	Definition
Cream time	Time taken for the reaction to take place after the reaction takes control
Gel time	Time taken for the rising foam to begin to polymerize or gel
Rise time	Time taken for the polyurethane sample to rise and stop
Tack free time	Time taken for the polyurethane sample to lose its stickiness

Note: Amundarain et al., 2022

3.4.2 Apparent Density

$$\frac{[\boldsymbol{weight\ of\ polyurethane - weight\ of\ cup}] \times \mathbf{1000}}{\boldsymbol{volume\ of\ cup}} \quad (3.1)$$

The definition of apparent density is the overall density of the foam sample. Density can also be related to the polyurethane's mechanical performance. The polyurethane's density was determined using technique ASTM D1622 as shown on the Figure 3.3 (Khan et al., 2019). Equation (3.1) illustrates the calculation required to get the apparent density of a polyurethane sample. Three sample were used to calculate the average point of the apparent density.

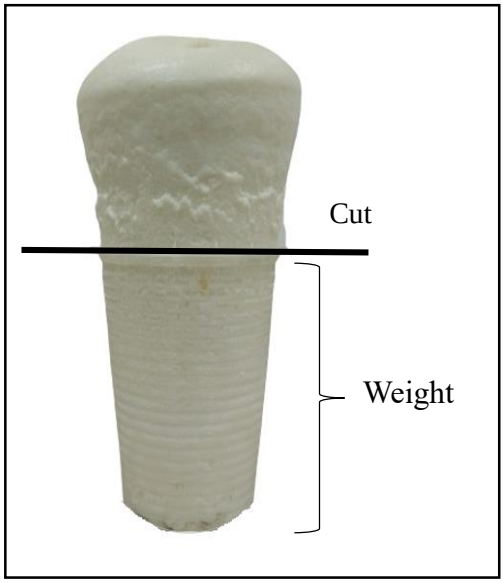


Figure 3.3 Steps required to conducted the apparent density testing

3.4.3 Rheology Index

The rheology test is analysis to determine the polyurethane's potential to expand and flow Additionally, the viscosity of each sample ratio may be determined. This experiment was conducted using transparent hose. The isocyanate was poured into the polyol and stirred for five seconds. The polyurethane solution pours into the end of hose. The chemical should then be poured into the transparent pipe in the opposite direction of the earth's gravity (Gatto et al., 2021). The clear pipe was kept upright so that polyurethane could flow freely and expand until it stopped altogether as shown in

the Figure 3.4. The rheology index was calculated by using Equation (3.2).



Figure 3.4 Example of rheology testing

$$\frac{\boldsymbol{Distance\ of\ sample\ flow}}{[\boldsymbol{weight\ of\ polyol}] + [\boldsymbol{weight\ of\ isocyanate}]} \quad (3.2)$$

3.4.4 Water Absorption Testing

ASTM D570 methods were used for the water absorption procedure. Absorption of water is used to determine the quantity of water absorbed under specified conditions. The information illuminates the performance of the materials in water or humid environments (Verma, 2025). As shown in Figure 3.5, the collected sample with 5 cm x 5 cm x 5 cm dimension were submerged in distilled water at room temperature for 24 hours under specified conditions. After immersion, samples are extracted, dried with a lint free cloth, and weighed. Using a Vernier calliper, the dimension after immersion is also measured (Dong et al., 2024).

The procedure occurs after two hours of immersion, at which time the specimen may exhibit a significant weight gain. Then, complete 24 hours of immersion. The process of measurement was repeated until constant weight and equilibrium were reached. Employing Equation (3.3), water absorption was calculated based on the weight gain percentage.

$$\frac{\boldsymbol{Wd - Wo}}{\boldsymbol{Wo}} \times \mathbf{100} \quad (3.3)$$

Wd: Weight of sample the after immersed

Wo: wet weight of the sample

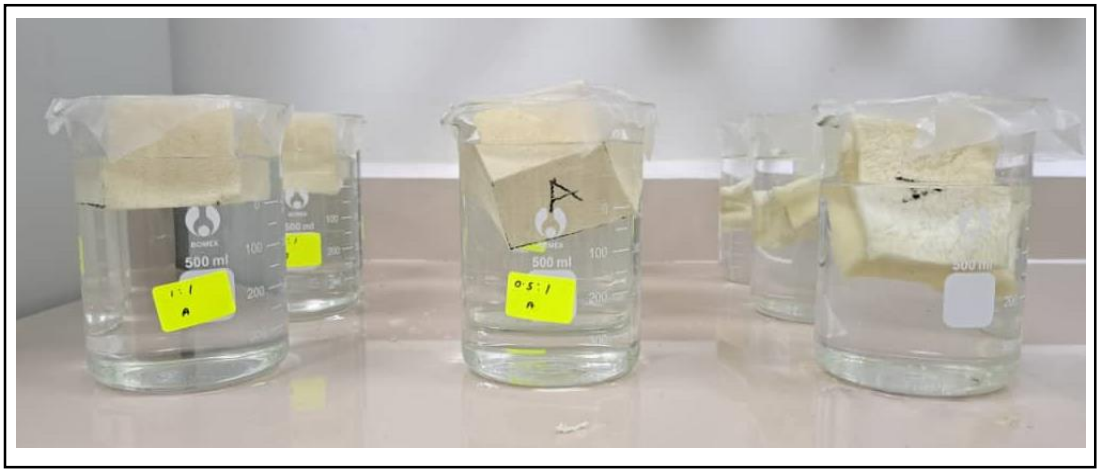


Figure 3.5 Water absorption testing

3.4.5 Moisture Content Testing

Using a drying oven, the moisture content of polyurethane was determined. The drying oven method is a thermogravimetric method (loss on drying) in which the sample is dried for a predetermined amount of time at a constant temperature, in this case 60°C for 24 hours (Sozcu et al., 2024). The sample measuring 5 cm × 5 cm × 5 cm was weighed prior to being placed in the oven as shown in Figure 3.6. It was determined by weighing the sample before and after drying, calculating the difference, and repeating the process until a constant weight was reached. The percentage of the sample's weight loss due to its moisture content was calculated using Equation (3.4).

$$\frac{\boldsymbol{Wd - Wo}}{\boldsymbol{Wo}} \times \mathbf{100} \qquad (3.4)$$

Wd: Weight of sample the after removed

Wo: wet weight of the sample

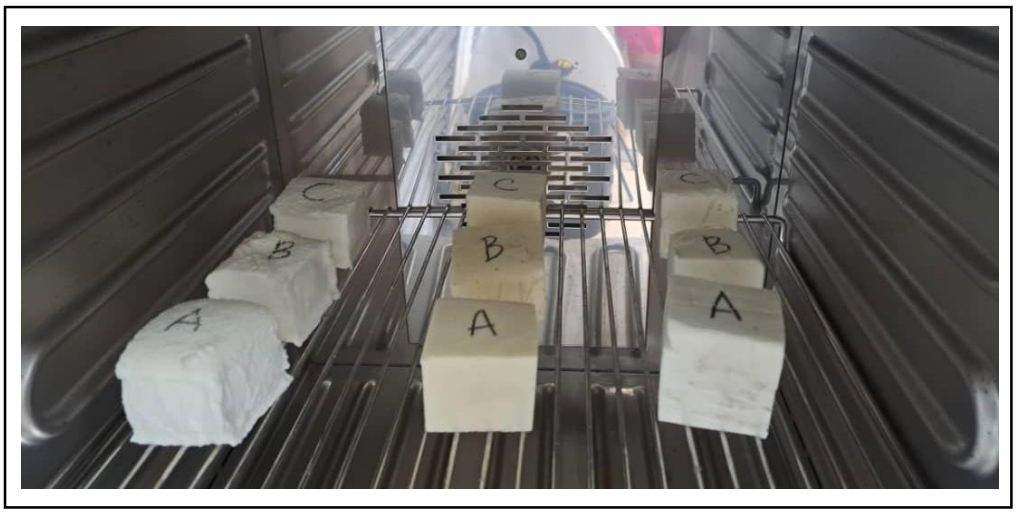


Figure 3.6 Oven drying method

3.5 Chemical Testing: Fourier Transmittance Infrared Spectroscopy (FTIR)

All polyurethane samples containing different proportions of polyol and isocyanate are subjected to Fourier Transmittance Infrared Spectroscopy (FTIR) by using Shimadzu IR Affinity-1 instrument. The FTIR analysis was using the ASTM E1252 method (Naghshbandi et al., 2019). This is as a result of examining the existence of functional groups within the sample.



Figure 3.7 FTIR Machine

Using FTIR machine as shown in Figure 3.7, the presence of hydroxyl and -NCO groups in polyol and isocyanate was determined. The presence of the urethane and amine functional groups, which are the essential functional groups of polyurethane, indicates that the polyurethane synthesis was successful. The FTIR spectroscopy utilised

approximately 1 mg of each sample and composition. The device was configured to perform 16 scans with a resolution of 4 cm^{-1}. The obtained spectrum ranged from 4000 cm^{-1} to 500 cm^{-1}.

3.6 Mechanical Testing: Compression Testing

The purpose of the compression test was to determine which compositions could withstand crushing loads and their behaviour under the loads. Compression tests were conducted using a Universal Testing Machine (UTM) with 50 kN and a cross-head speed of 5 mm/min. The dimensions of the sample were 5 cm × 5 cm × 5 cm. The polyurethane samples were subjected to compression testing in accordance with ASTM D1621 (Sukumar et al., 2021). Figure 3.8 shown the condition during compression testing of the polyurethane foam.

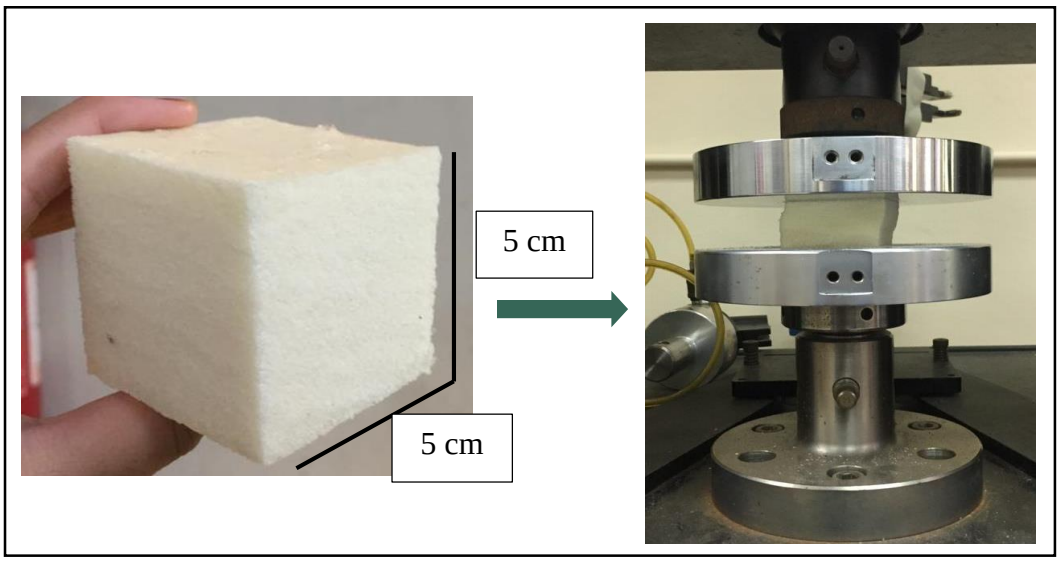


Figure 3.8 Compression Testing Procedure

3.7 Morphology Analysis: Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) generating signals that contain information about the sample's surface topography and composition. The model used for SEM JEOL JSM 6460. To observe the surface morphology of polyurethane foam, a thin slice of each of the different composition specimens measuring 10 mm by 10 mm was cut from the sample (Kosmela et al., 2020). To prevent surface charging and improves image quality on non-conductive sample, a thin layer of 3-10 nm of gold

coating was applied to the surface of the sample. The process involves thoroughly drying the specimen, placing it in a sputter coater under vacuum and using plasma to deposit a uniform gold layer as shown on Figure 3.9 (Schwab, 2025). The structure of the cell foam will have observed and compare the different type of ratio. The morphology of the cell foam is an important factor in determining the mechanical properties of polyurethane. performed in accordance with ASTM E 2089 using 80X magnification.



Figure 3.9 Sputter Coater

3.8 Fabrication of The Green Polyurethane using Different Blowing Agents

For the production of green polyurethane (PU), the ratio 1:1 of polyol and isocyanate was used for the formulation as same as industrial polyurethane as it has achieved optimization. The production of green Pu will use ratio 1:1. The castor oil will be using different blowing agent. The castor oil based polyol with the mixture blowing agent will mix using the mechanical stirrer for 2 minutes at 3000 rpm. Then, hardener (MDI) was poured into the mixture and stirred for 5 seconds at the same speed. The sample will then rest for 24 hours at room temperature for the complete curing.

Table 3.4 shows the chemical composition castor oil based polyol with HCFC 141b and water as blowing agent. It is shows the formulation for the castor oil with HCFC 141b as blowing agent (PCH) was 1:1 :0.25 whereas castor oil with water as blowing agent (PCW) was 1:1:0.015. The synthesis was repeated three times for each types of polyurethane. Figure 3.10 shows the schematic diagram for the methods of synthesizing of the castor oil polyurethane.

Based on Table 3.4, there were difference amount for the blowing agents. This was due to the castor oil which contains fatty acid chains, that can alter the cell structure and foam stability. To obtain the required closed cell structure and sufficient mechanical

strength, less HCFC 141b is required. As for PCW, the isocyanate and water highly react, possibly devouring them in the process. The stability of the polymer network can be jeopardized by an increased water ratio, which could reduce the efficiency of isocyanates in urethane synthesis.

Table 3.4
Formulation of Green Polyurethane with Different Blowing Agent

	Castor oil (HCFC 141b) (PCH)	**Castor oil (Water) (PCW)**
Chemical composition	1:1:0.25	1:1:0.015

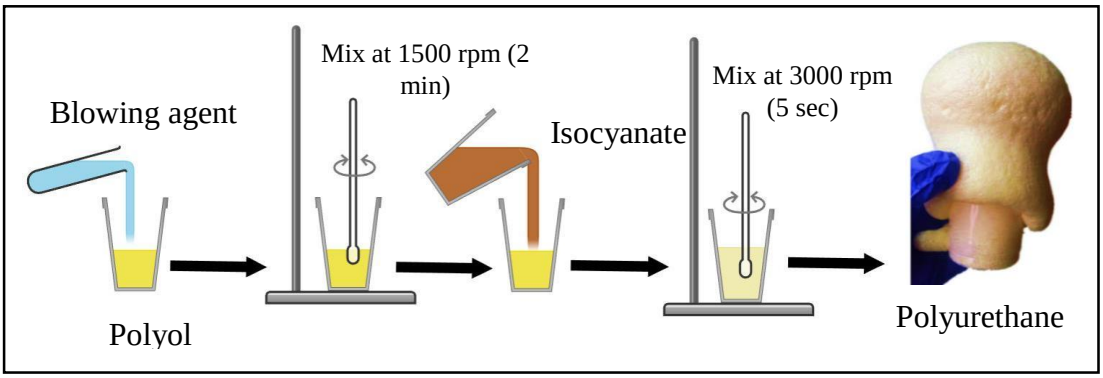


Figure 3.10 Graphical flow method for synthesized castor oil based polyol polyurethane using HCFC 141b and Water as blowing agents

Then, all the PCH and PCW sample were carried out the characterization which undergoes physical testing (foam reaction time, apparent density and rheology testing), FTIR analysis and morphological analysis. The steps of the characterization were followed according to their ASTM standards same as on characterization of the polyurethane industry (PI) as shown in section 3.4 to 3.7.

3.9 Environmental Stress Cracking Resistance (ESCR) Testing

3.9.1 Soil Burial Test

Environmental Stress Cracking can be explained when plastic resin degrades under stress due to the action of a chemical agent, resulting in the failure of a plastic component. The soil burial test must be conducted to determine the impact of burial on the mechanical characteristics of polyurethane after it has been buried in the soil. The

sample preparation conforms to ASTM G160 for all types of polyurethanes (Zuliani et al., 2022). The dimensions of the sample were 5 cm × 5 cm × 5 cm. The samples were buried roughly 25 cm deep in garden soil in an outdoor location with five different locations as shown in Figure 3.11. The samples were buried for 10,20, 30,60 and 90 days respectively. On each consecutive day, the samples were weighted and undergoes mechanical testing. The findings were recorded and documented. Figure 3.12 shows the graphical steps involved for the charactarization of the soi burial testing.



Figure 3.11 Soil burial testing

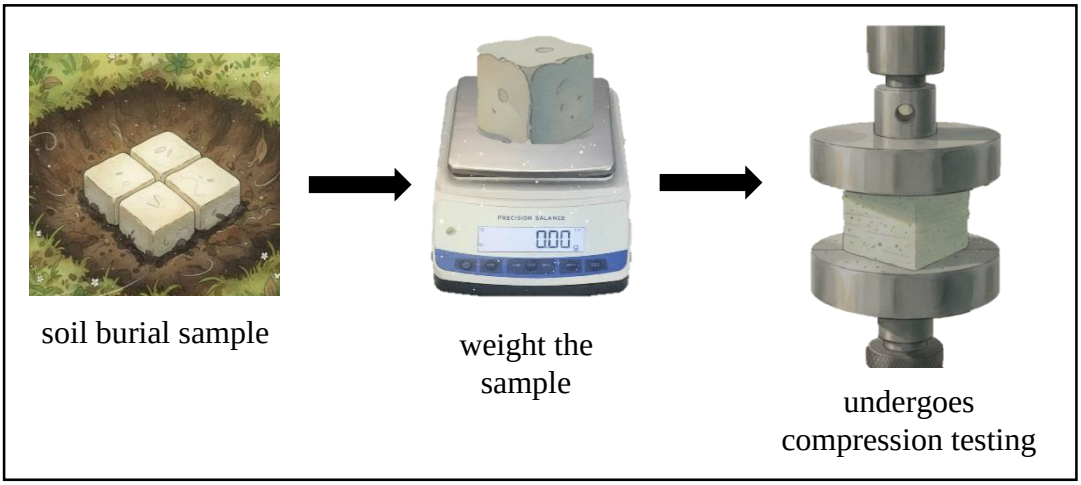


Figure 3.12 Graphical flow of characterization involved for soil burial test

3.9.2 Solubility Testing

The sample is prepared according to ASTM E1148. The dimensions of the four samples are 5 cm × 5 cm × 5 cm. The samples are immersed in dilute acidic and alkaline solutions. The dilute acid solution is a mixture between water and concentrated hydrochloride acid (HCL) in a 10:1 ratio, which means the pH value of the solution is between 6-7. The alkaline solution needs to be prepared by dissolving 8.5 g of sodium hydroxide in water and diluting it with water to 100 ml. The solution of Sodium Hydroxide (NaOH) has a pH of 9 (Ali et al., 2024). Figure 3.13 shows the conditions of the polyurethane foam when submerged in the acid and alkaline solution.

Each sample will be immersed in both solutions for 0, 10, 20, and 30 days. Every sample were weighted of the gain percentage for every consecutive days and undergoes compression testing. The compression properties of the polyurethane samples determined according to ASTM D1621. After being immersed in the acidic and base solution, the compression properties of polyurethane conducted using the Universal Testing Machine with the specification of 50 kN, and the crosshead speed is 50mm/min and morphological analysis by using SEM. The graphical flow of the characterization involved was shown on the Figure 3.14.

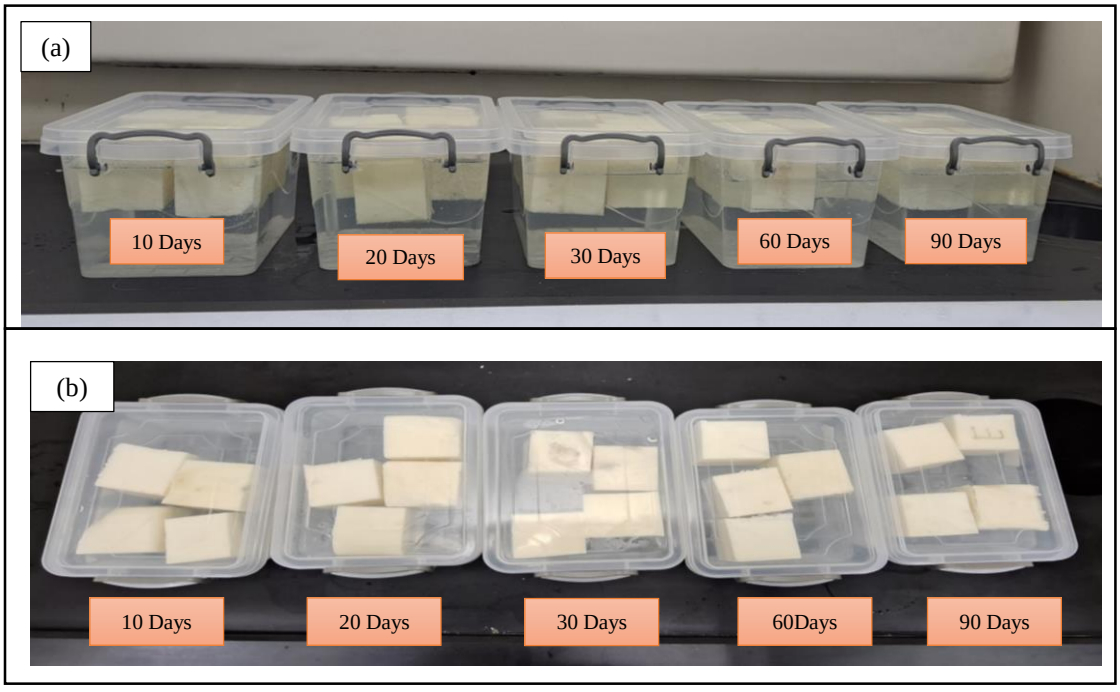


Figure 3.13 Conditions of the polyurethane sample when conducted solubility testing (a) Acidic solution (b) Alkaline solution

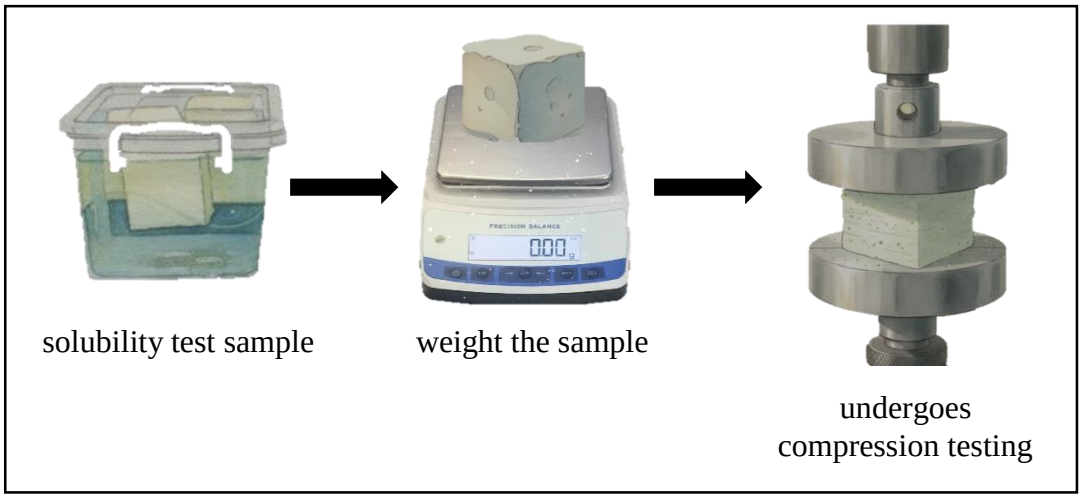


Figure 3.14 Graphical flow of characterization involved for the solubility test

3.10 Antibacterial Testing

Antimicrobial testing refers to a set of laboratory procedures used to evaluate the ability of a material or substance to inhibit the growth of, or kill, microorganisms such as bacteria, fungi, or viruses. In the context of polyurethane materials, antimicrobial testing is often conducted to determine whether the surface resists microbial colonization, inhibits biofilm formation, or demonstrates bactericidal or bacteriostatic activity (Bo et al., 2024). Depending on the application and types of bacteria of interest, there are a variety of antimicrobial testing methods that can be performed. In this research, a disc diffusion assay was carried out to analyse the antibacterial properties for both types of polyurethanes.

3.10.1 Preparation of Mueller Hilton agar plate

Mueller Hilton agar was used in for assessing antibiotic susceptibility. In order to prepare MH agar, 3.8 g of MH agar powder were dissolved in one litre of distilled water. The liquid was then heated and vigorously stirred to completely dissolve every component. The agar broth was then sterilized by autoclaving at 120 ° C for 15 minutes. On agar the plates, sterile MH agar was poured and allowed to settle at room temperature. The agar plates as shown in Figure 3.15 were stored at 4 ℃ (Aryal, 2022).

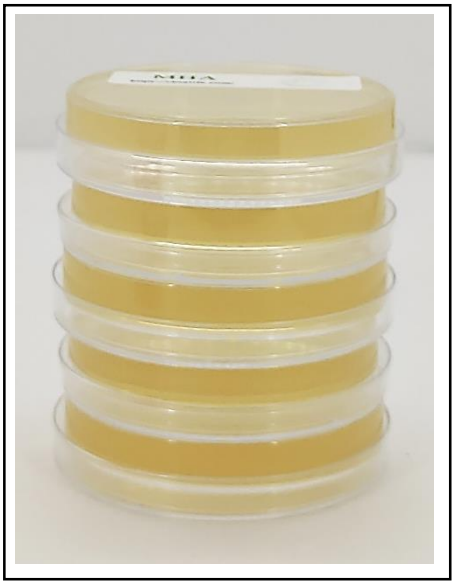


Figure 3.15 Mueller Hilton agar plate

3.10.2 Preparation of Inoculums

Antibacterial activity of polyurethane sheet was examined against Gram positive bacteria, including and Bacillus subtilis (*B. subtilis*), and Gram negative bacteria, including Escherichia coli (*E. coli*) and. The bacterial strains were pre cultured overnight at 37^{o}C in a rotary shaker with Muller Hinton broth which shows in Figure 3.16. Each strain was then adjusted to a concentration of 108 cells/mL with 0.5 McFarland turbidity standards to produce a bacterial suspension containing 1.5 108 cfu/mL (Bogale et al., 2022).

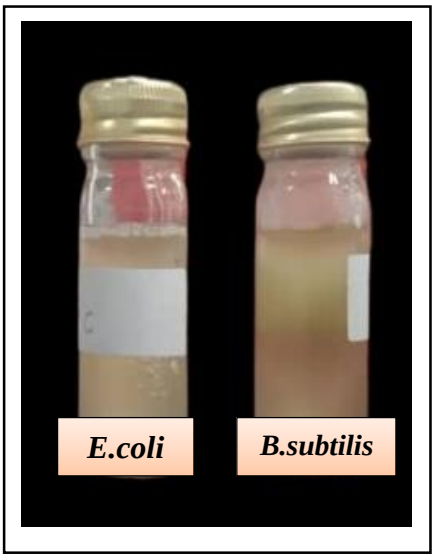


Figure 3.16 *E.coli* and *B.subtilis* in overnight culture

3.11 Antibacterial Procedure on Polyurethane

The antibacterial activity of polyurethane foam sheets was assessed using a disc diffusion assay method. Pathogenic Bacteria culture was swabbed onto molten, cooled Muller Hilton agar plates. Under a sterile condition, a thin layer with 5 cm by 5 cm dimension polyurethane was put on top of the Mueller Hilton agar medium and the plates were placed onto flat surface at room temperature for 24 hours. At the completion of a day, the inhibition zones around the disc were observed. The sample continue observed every day for 2 weeks and recorded changes of bacterial activity towards the polyurethane foam (Henderson, 2024). Figure 3.17 shows the example of the antibacterial testing of the polyurethane foam and Figure 3.18 shows the graphical flow of the antibacterial testing.

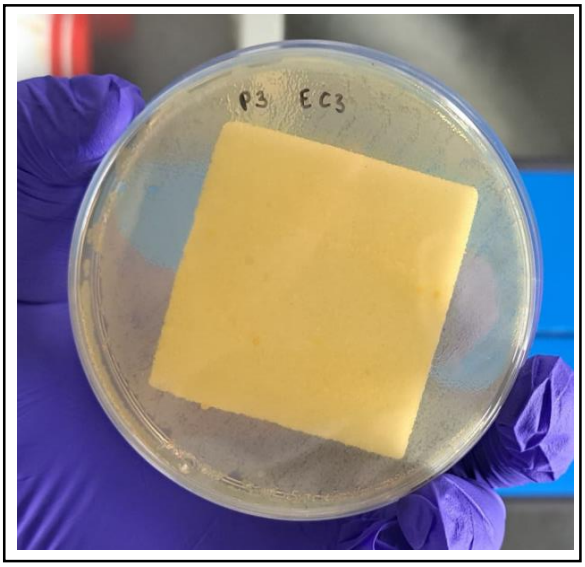


Figure 3.17 Example of Antibacterial Testing

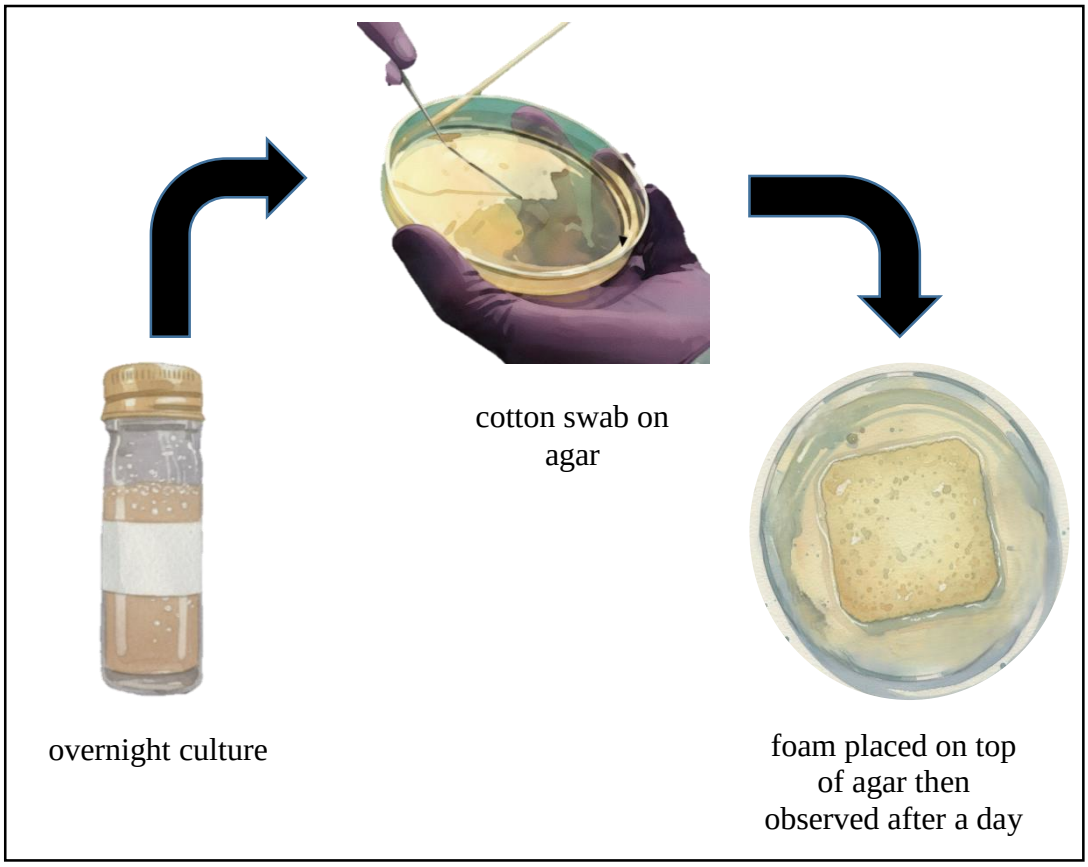


Figure 3.18 Graphical flow of the Antibacterial Testing

3.12 Flowchart Methodology

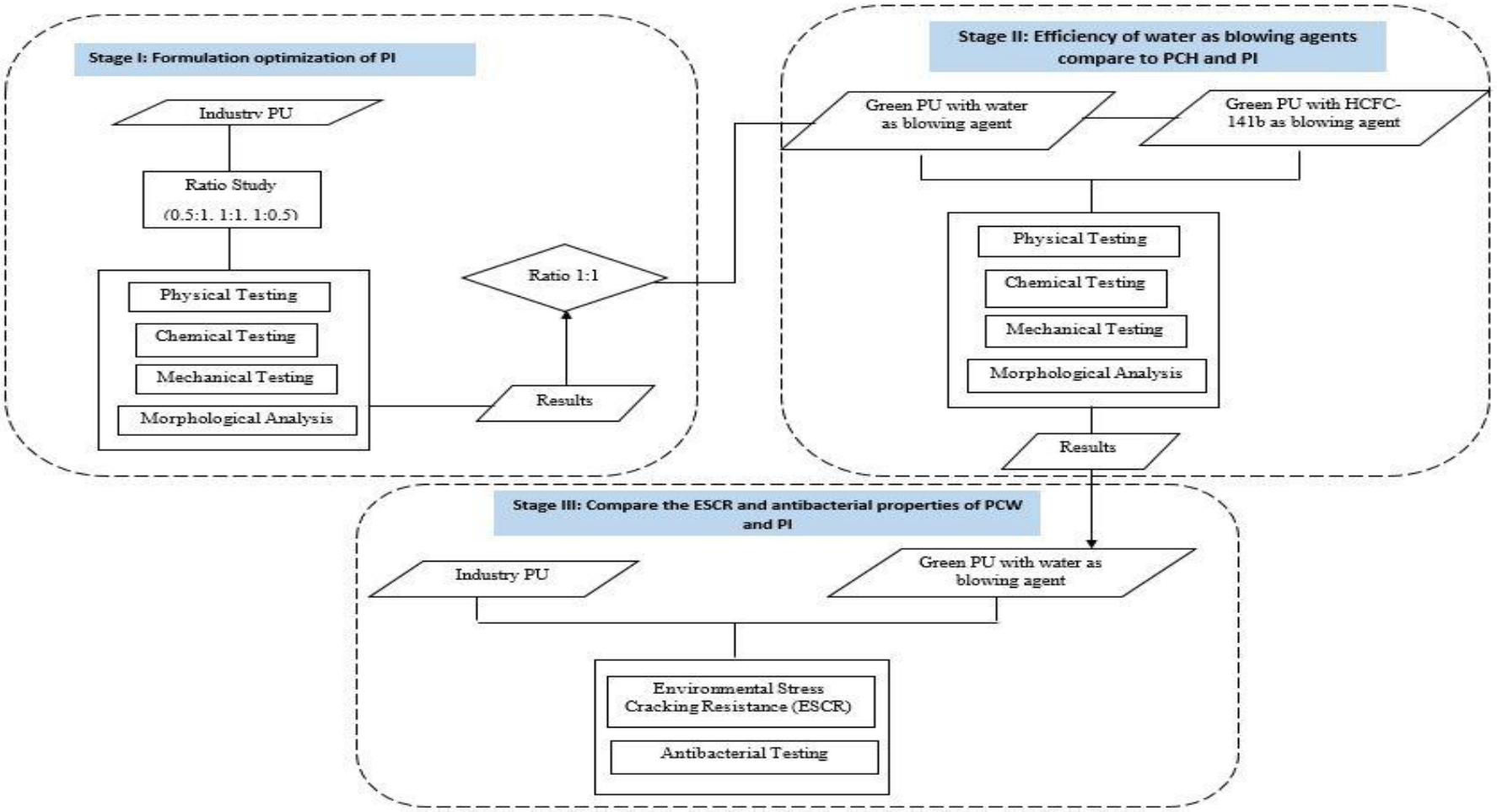


Figure 3.19 Research Roadmap of the methodology involved

CHAPTER 4
RATIO STUDY ON INDUSTRIAL POLYURETHANE

4.1 Introduction

This chapter explored the synthesis of various types ratio of polyol and isocyanate to identify the optimal formulation for producing desirable qualities in polyurethane grouting. The characterization of three ratio types [0.5:1, 1:1, and 1:0.5] will be discussed, along with a comparison of their qualities in terms of physical, chemical, mechanical, and morphological characteristics. This section has further explored the optimal composition for synthesizing polyurethane for grouting applications, thereby addressing issues prevalent in the polyurethane grouting business.

4.2 Preliminary Study on the Mixing Speed and Time

A preliminary study has been conducted in order to determine the methods of speed and time value in order to aim the suitable conditions in producing good properties of polyurethane. Table 4.1 shows the results of the preliminary study on the mixing speed and time. A proper stirring speed and time is crucial for minimising air contamination during the mixing of two solutions. It is a significant factor that can affect the quality of the product. A longer mixing time may result in the separation or separation of the bland ingredients (Le et al., 2019)

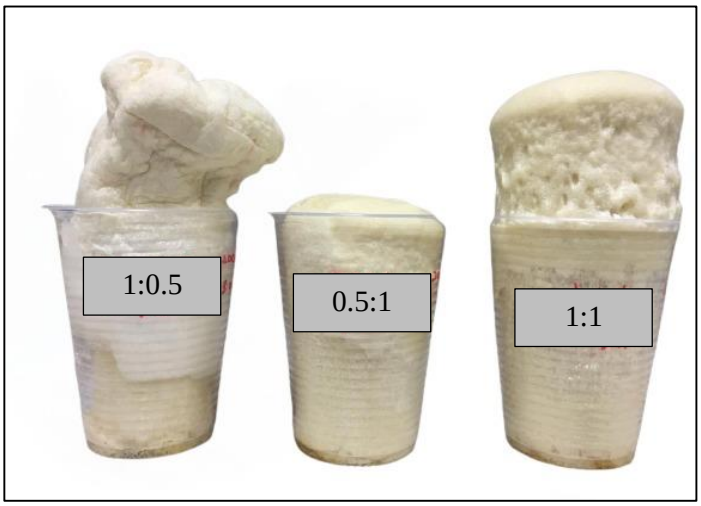


Figure 4.1 Polyurethane sample were mixed using speed of 2000 rpm for 5 seconds

Table 4.1
Preliminary Result On the Characterization Time to Study Mixing Ratio Time and Speed

	1000 rpm			**2000 rpm**			**3000 rpm**		
	3s	**5s**	**7s**	**3s**	**5s**	**7s**	**3s**	**5s**	**7s**
Cream Time	6.88	12.61	13.23	7.12	9.13	12.24	8.38	11.03	12.33
Gel time	4.21	2.58	1.75	4.07	2.69	3.52	1.52	1.05	1.31
Rise Time	50.31	43.78	47.59	49.90	54.45	52.48	47.23	58.35	54.84
Tack Free time	14.01	21.33	17.29	9.45	2.38	1.59	4.15	2.81	6.99
Texture after 24 hours	soft	soft	soft	soft	Hard but brittle	Hard but brittle	Hard but brittle	hard	soft

Three types of speed and time were choosing for polyurethane production. The polyurethane was generated at 1000 rpm, 2000 rpm and 3000 rpm for durations of 3, 5 and 7 seconds. The sample exhibited pre-existing voids for the speed of 1000 rpm and 2000 rpm as shown in Figure 4.1 and 4.2. The presence of uncured polyurethane at the base of the sample results in a soft bottom and a hard top. This indicates that polyol and isocyanate were not completely cross linked with one another even after a day. The cream time and rise time of the 3, 5and 7 second for 1000 rpm sample were below than 10 seconds. Only sample with 2000 rpm 7 seconds and 3000 rpm, the cream time is between 10-20 seconds indicate that the time required for the cream time confronts standards of polyurethane grouting as which state in section 2.4.1 (Amran et al, 2021).

This phenomenon can be attributed to the lack of homogeneity in the mixing of isocyanate and polyol, as certain regions may contain an excess of isocyanate that remains unreacted. This unreacted isocyanate can result in a sample exhibiting a brittle surface and a sticky texture.

Excessive polyol may result in the sample exhibiting tackiness and the presence of soft regions. The lower portion exhibits a soft consistency, whereas the upper portion is characterized by a hard texture, indicating inadequate vertical dispersion. The upper region may exhibit adequate mixing as a result of turbulence, whereas the lower region appears to have insufficient shear or mixing. Furthermore, the brief mixing durations of 5 and 10 seconds do not facilitate adequate microscale blending, given the viscous states of the polyol and isocyanate. Although the sample is physically in an appropriate mixture, the chemical reaction does not take place (Lapshin et al., 2025).

Polyurethane with stirring of 3000 rpm results show the for the 5 seconds stirring time sample had a rigid form and there were cure properly. But as for the 3and 7 seconds shows the sample become solid but brittle and soft to touch. Even though the 7 second is 12.33s which already qualified the cream time standard of polyurethane grouting, the mixed shows it has over shear the mix, introduced too much air or can start reaction prematurely when exothermic heating occurs. Longer mixing time may improve reactivity but can prolong rise time possibly due to overmixing or different cell structure formation (Rifathin et al., 2023).

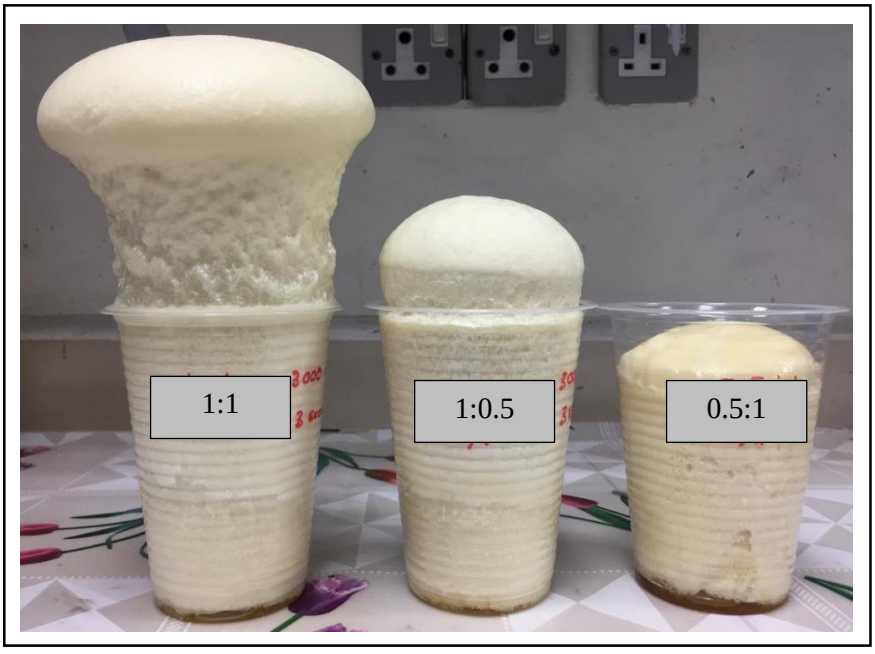


Figure 4.2 Polyurethane sample were mixed using speed of 3000 rpm with 7 seconds

This can be proven by Differential Scanning Calorimetry (DSC) result as shown in Figure 4.3. DSC is a method of thermal analysis used to examine whether materials

react to heat. DSC can offer valuable data on the heat stability and curing behaviour of thermoset polyurethane. It shows polyol to isocyanate, 1:1 ratio when using mixing speed and time of 3000 rpm and 5 seconds of the polyurethane were completely cure and no peak were observed. The mixing at 3000 rpm for 5 seconds was chosen because it ensures that the sample is completely cured and the value of cream time and rise time were according to the standard polyurethane grouting.

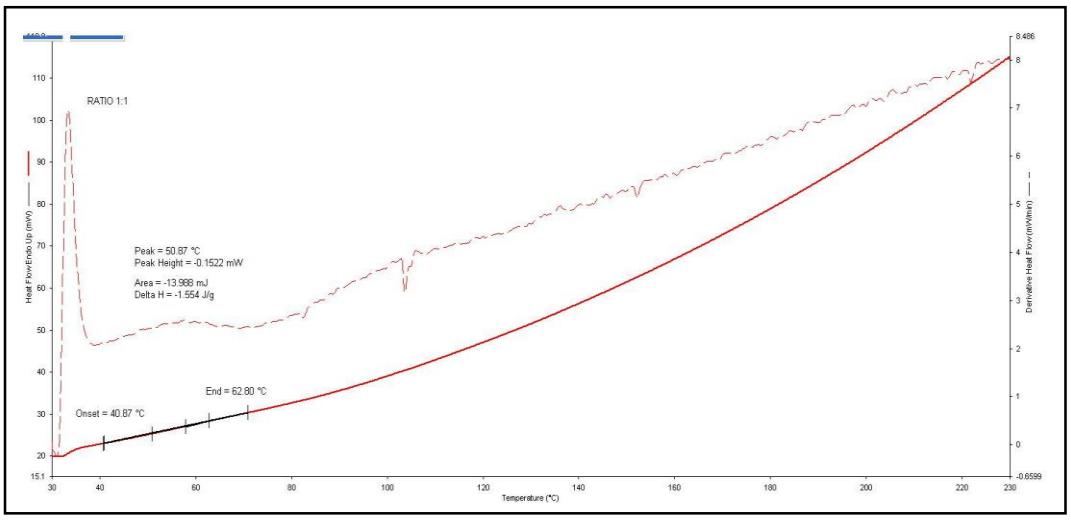


Figure 4.3 DSC analysis of Ratio 1:1 by using mixing speed of 3000 rpm for 3 seconds

4.3 Physical Observation Analysis

The hardener (isocyanate) introduce to resin polyol and mixed with the mechanical stirrer at a speed of 3000 rpm for 5 seconds. This steps were repeated for the other ratios. Produced sample were left for post cured for 24 hours before analysed.

Figure 4.4 shows the polyurethane samples with different ratios of polyol and isocyanate after curing. After 24 hours, the samples with ratios of 1:1 and 0.5:1 became hard and rigid, while the 1:0.5 ratio sample shrank and became soft when touch. Overall, the hard and rigid condition may be attributed to the 1:1 ratio, where all the isocyanate (-NCO) groups had reacted with all the hydroxyl (-OH) groups, resulting in the production of a fully reacted polyurethane foam (Triwulandari et al, 2021).

During the reaction, the polyol's hydroxyl groups react with the isocyanate groups, forming urethane linkages (NHCOO-) as shown in Figure 4.5 (Ivdre et al, 2020), This balanced stoichiometry ensures that no excess unreacted polyols or isocyanates are left in the final product, which can be essential for achieving the desired properties. Due to the low water content in the 1:1 ratio sample, the foam will not shrink

over time and is considered rigid (Sabri et al, 2021)

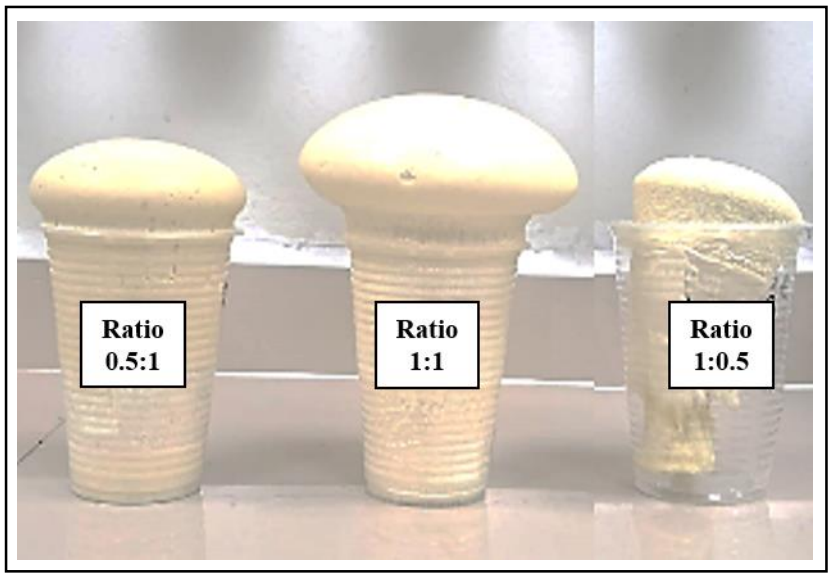


Figure 4.4 Polyurethane samples of 3 types ratios after curing

As the expansion height of the 1:1 ratio is high compare to ratio 0.5:1 as the ability for the polyurethane to expand is high. A decomposition reaction occurs and produces carbon dioxide gas, thus causing the solution to foam and expand in volume (Comte, 2021).

Polyols are a key component in the production of polyurethane, and they can affect the shrinkage behaviour of polyurethane products to some extent. Polyurethane shrinkage occur during the curing process, as the material transitions from a liquid state to a solid state. Moreover, the presence of reactive groups in the polyol, such as primary or secondary hydroxyl groups, might increase the degree of crosslinking in the final product, hence leading to shrinkage (Ma et al., 2022). This can be seen in Figure 4.4, that the 1:0.5 ratio polyurethane sample become shrink over time. The extent of shrinkage depends on a number of factors, including the type of polyol used, the processing conditions, and the formulation of the polyurethane (Członka et al., 2020).

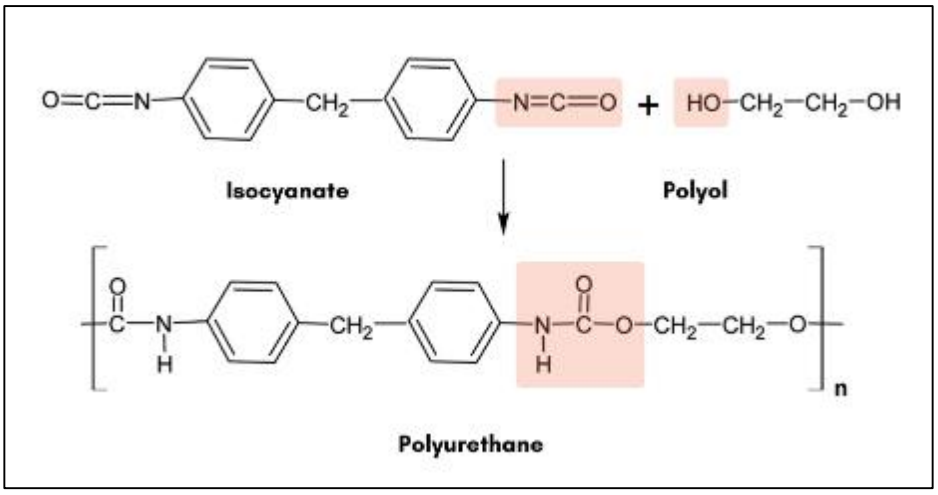


Figure 4.5 Chemical Structure of Polyurethane

4.4 Physical Properties

4.4.1 Foam Reaction Time

Time characterization entails measuring and recording the duration of the reaction between isocyanate and polyol during polyurethane synthesis, particularly as the mixture begins to foam.

Figure 4.6 illustrated the foam reaction time for the samples with ratios of 0.5:1, 1:1, and 1:0.5. The average cream time and rise time for all samples fell within the range of typical polyurethane grouting as shown in Table 4.2, with a cream time of 10–20 seconds and a rise time exceeding 40 seconds (Francés et al, 2018). The amount of time taken to form the gel was low. A low gel time in the production of rigid polyurethane implies a shorter period for the material to transition from liquid to gel, enabling faster curing and production cycles. This is crucial because it marks the point where the polymerization reaction has progressed enough that the mixture starts to lose its fluidity and become solid. Notably, the tack free times for the 0.5:1 and 1:0.5 ratio samples were considerably higher than for the 1:1 ratio sample, which averaged 2.99 seconds. This rapid loss of stickiness suggests an accelerated reaction rate ensuring maximum surface protection without disruption or damage upon contact (Bednarczyk et al, 2021; Kraitape et al, 2016). It also indicates that the surface has solidified enough to handle or move the product without marring it. This phenomenon also suggests that having limited tack free time increases the rate of reaction. In a reaction with a low rate, molecules combine

more slowly than in a reaction with a high rate. Additionally, it has a greater degree of crosslinking and a more rigid end product (Rao et al., 2017).

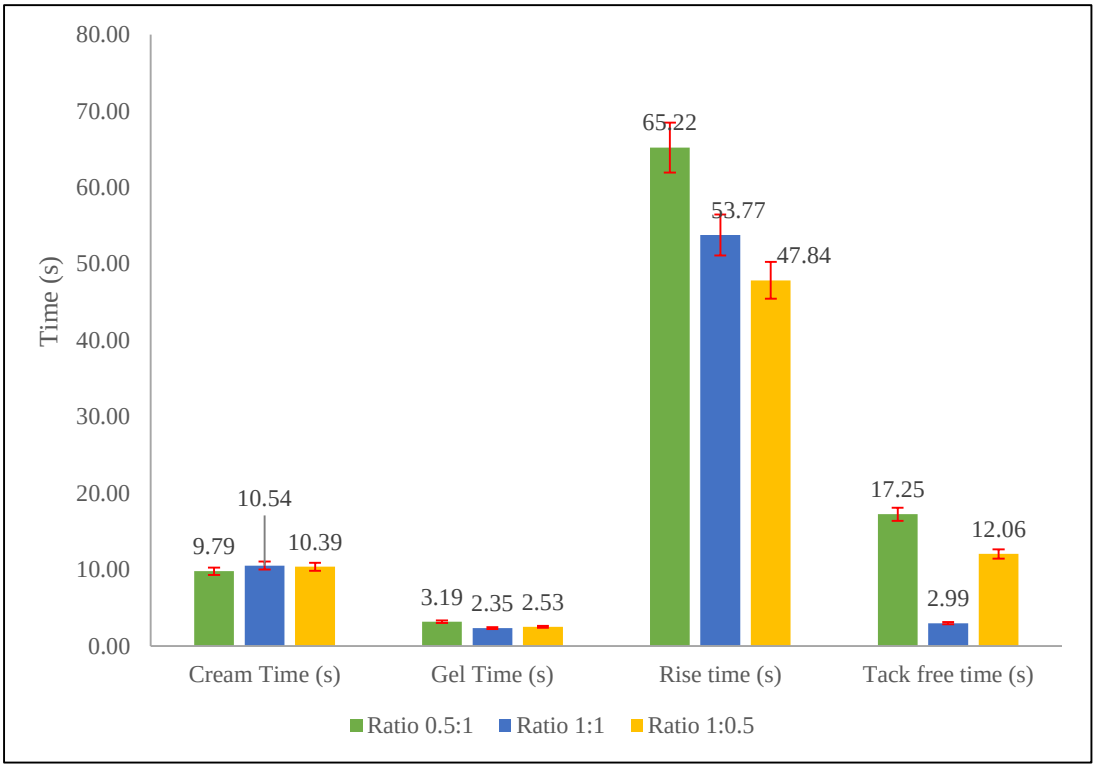


Figure 4.6 Characterization time of Ratio 0.5:1, 1:1 and 1:0.5

While the cream times and rise times of all samples were in line with polyurethane grouting standards as shown in Table 4.2, the prolonged tack free times for the 0.5:1 and 1:0.5 ratio samples indicate they may not be suitable for concrete raising applications due to their slower reaction rates, which may potentially hinder the maintenance process (Amran et al, 2021). From FTIR analysis in Figure 4.15(b) later reveal in section 4.4.1 reveals an excess of isocyanate in the 0.5:1 ratio and an excess of polyol in the 1:1 ratio, signifying incomplete reactions and contributing to the varying rates of reaction. A slow rate of reaction may indicate that not enough collisions are occurring with sufficient force to break the chemical bonds of the reactants, resulting in a slower production of the product. It can also cause the samples to not blend well with one another, leading to have voids between them (Libretexts, 2022)

Table 4.2

Standard Value of Polyurethane Grouting for Foam Reaction

Characterization of Time	Standard Time Value
Cream Time	>10 seconds
Gel Time	Low
Rise Time	>40 second
Tack-free Time	Low

Note: Aminuddin et al., (2022)

4.4.2 Apparent Density

The measurement of mass over volume determines the apparent density of polyurethane, which is an essential physical characteristic that may affect its strength and performance (Amran et al, 2021). Based on Equation (3.1) at section (3.2), the apparent density of each sample was determined. The behaviour of materials to applied forces contributes for the correlation between density and mechanical performance. There are more intermolecular bonds between molecules in materials with higher density as they contain more atoms or molecules per volume. This makes a material stronger and stiffer by increasing its resistance to deformation or failure under stress (Sun et al., 2021).

The Figure 4.7 shows the apparent density of all the samples, which averaged 56 kg/m^3 for the 1:1 ratio, 69 kg/m^3 for the 0.5:1 ratio, and 0 kg/m3 for the 1:0.5 ratio. These different values showcase the distinct properties and performance characteristics of each polyurethane sample (Lee et al, 2022). The 1:0.5 ratio apparent density cannot be measured due to shrinkage effect of the sample as shown in Figure 4.4.

The high density of polyurethane is directly related to crosslinking and foam rigidity, as a high isocyanate content in polyurethane leads to an increase in hard segments (Ivdre et al, 2021). Unreacted isocyanates can contribute to the material's reactivity, leading to potential issues with curing and crosslinking as shown in Figure 4.8. These unreacted isocyanates remain chemically active and can potentially react with other molecules in the material or the surrounding environment. Crosslinking refers to the formation of bonds between polymer chains that create a three dimensional network structure. The rise of hard segments (-NCO) leads to the production of extensive

urethane and allophanate crosslinking in the sample, which increases the density of the polyurethane (Morales-Cerrada et al ,2021).

Allophanate crosslinking, as shown in Figure 4.8, is a side reaction that happens when unbound isocyanate or unreacted NCO groups in a polyurethane sample react with the urethane form (Grzęda et al, 2023). The denser network results in a higher density material. High density polyurethane shows brittle characteristics compared to low density polyurethane, which exhibits elastoplastic characteristics (Lee et al, 2022). As illustrated in the SEM images in Figure 4.17, cell rupture occurs between the cells, making polyurethane easy to permanently deform under stress. Polyurethane with high number of soft segments in the polyol, make the sample become soft when touch (Jiang et al., 2018).

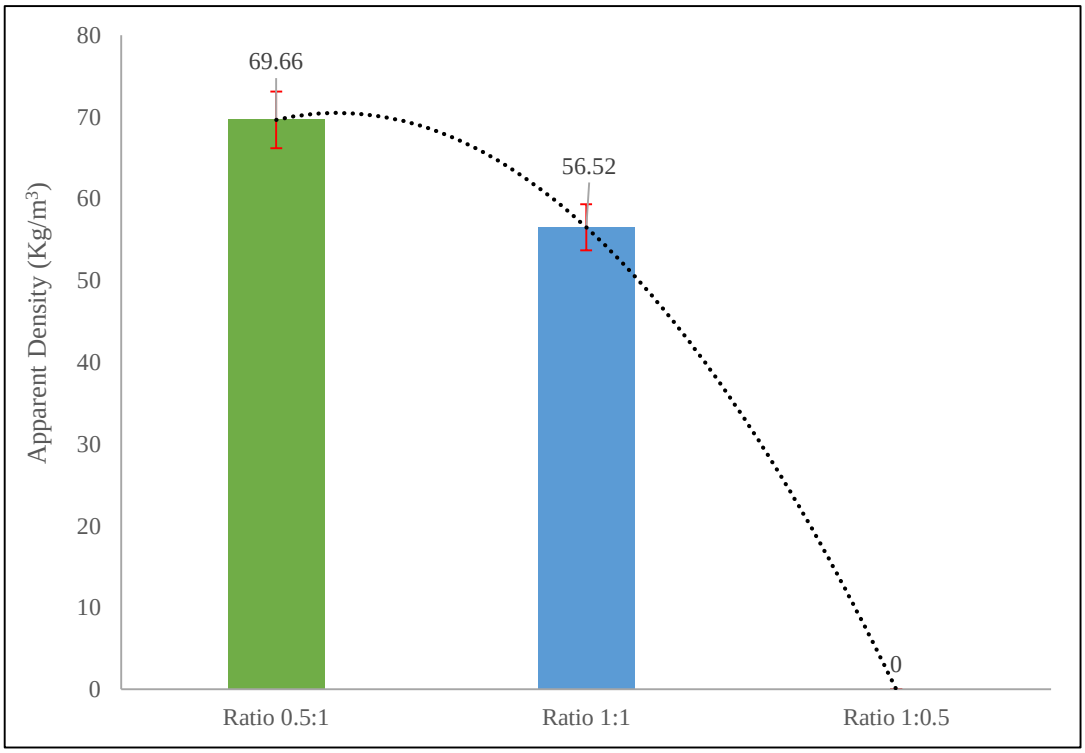


Figure 4.7 Apparent Density polyurethane of all ratios

The apparent density value for the 1:0.5 ratio sample was not calculated due to the shrinkage problem, and the physical properties of this polyurethane which was too soft. The presence of excess hydroxyl groups may increase the material's hydrophilicity or susceptibility to moisture absorption because it has voids in the cell structure, as shown SEM images in Figure 4.9 (Mohammed et al, 2023). These can lead to issues such as reduced mechanical strength, dimensional instability, and increased

vulnerability to degradation. With excess polyol, the density of crosslinking and the polymer network may be reduced, resulting in a less rigid or less durable material compared to a stoichiometric product (Lang et al, 2020).

The mechanical properties of high density polyurethane are more brittle than those of low density polyurethane, which has elastoplastic properties (Lee. et al, 2022). The density of polyurethane grouting used on road pavements varies between 56 and 76 kg/m^3 (Saleh et al, 2019). The values obtained are within the range of polyurethane grouting density.

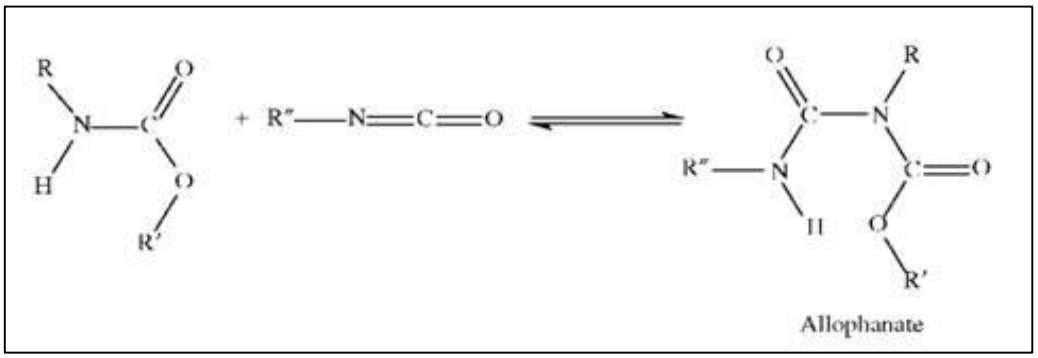


Figure 4.8 Allophanate crosslinking

4.4.3 Rheology Analysis

The purpose of the rheology test was to determine polyurethane's viscosity. By observing the polyurethane's ability to expand and flow, which stimulates the rheology into cracks in pavements or concrete bridges, the rheology index will indicate the viscosity of the polyurethane (Rig Worker, 2022). Understanding rheology and viscosity is essential for polymer processing. The calculation of rheology index was according to Equation (3.2) in section 3.3.3. The further the distance travelled, the higher the rheology index and the lower its viscosity, making the material suitable for injection into cracks or pothole (Jiang et al, 2019). Viscosity measures how internal friction affects the movement of polyurethane grouting through a medium. Low viscosity indicates low resistance, easy shearing, rapid flow, and a greater ability to expand and flow. This is advantageous in applications like injection moulding, where precise filling of moulds is essential for achieving desired part dimensions and properties. Polyurethane grout has a rheology index between 0.4cm/g and 5.0 cm/g (Münstedt, H., 2021).

According to Figure 4.9, all ratios fall within the polyurethane grout's range of the rheology index. The ratio 1:1 has the highest rheology index value, while the ratio 1:0.5 has the lowest. This indicates that the 1:1 ratio has less viscosity than other ratios due to its higher rheology index (Münstedt et al., 2021). Low viscosity is essential to the properties of polyurethane grout (Ren et al., 2021) and in this case, the 1:1 ratio sample was suitable for polyurethane grout. Polyols offer chemical advantages in terms of hydrolytic stability, cost, viscosity, and flexibility in polyurethane foam (Mohamed et al., 2021). Figure 4.10 shows the rheology testing in the hose of ratio1:1 after curing take places.

The 0.5:1 ratio sample that had a higher content of isocyanate produced polyurethane that was smaller in volume due to less expansion, and became brittle after curing (Münstedt, H., 2021). Isocyanates are more reactive, are accountable for the curing process, and contribute to the rigidity of polyurethanes. Additional isocyanates may increase the polymer chain’s mobility, potentially reducing its viscosity (De Souza, Kahol, et al., 2021). However, as shown in Figure 4.7 the higher density causes the polyurethane to become brittle after curing, which can compromise the injection process. This demonstrates that a ratio of 0.5:1 is impractical for polyurethane grouting applications.

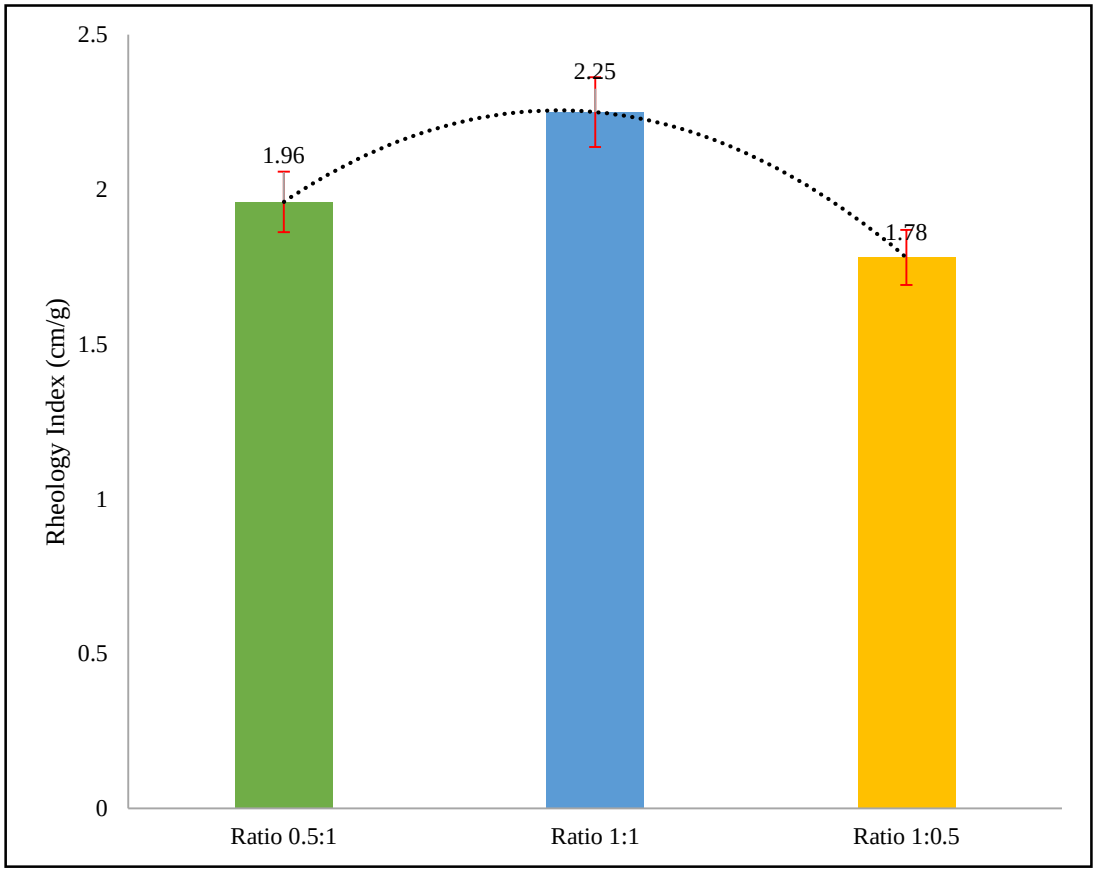


Figure 4.9 Rheology index value for polyurethane of ratios 0.5:1. 1:1 and 1:0.5

The 1:0.5 ratio sample had the highest viscosity of all. This may be due to the high content of polyol in the polyurethane product that shrunk in the hose after a day, same as shown in Figure 4.4. Introducing extra polyol into the reaction potentially enhances hydrogen bonding interactions within the polymer matrix. This increase in the interactions between molecules elevates its viscosity, making the material more resistant to flow. Despite the 1:0.5 ratio sample achieving a travel distance of 56 cm, its high viscosity rendered it unsuitable for use as polyurethane grouting (Jiang et al, 2019; De Souza, Kahol, et al., 2021)



Figure 4.10 Sample of 1:1 ratio after conducted rheology testing

4.4.4 Water Absorption & Thickness swelling

Water absorption analysis is essential for determining whether the polyurethane sample exhibits hydrophobic or hydrophilic properties. Figure 4.11 and Table 4.3 depicts the weight gain percentage when samples were immersed in distilled water without being subjected to pressure. When polyurethane foam encounters water, diffusion of water into the foam was observed as shown in Figure 3.5 in section (3.3.4).

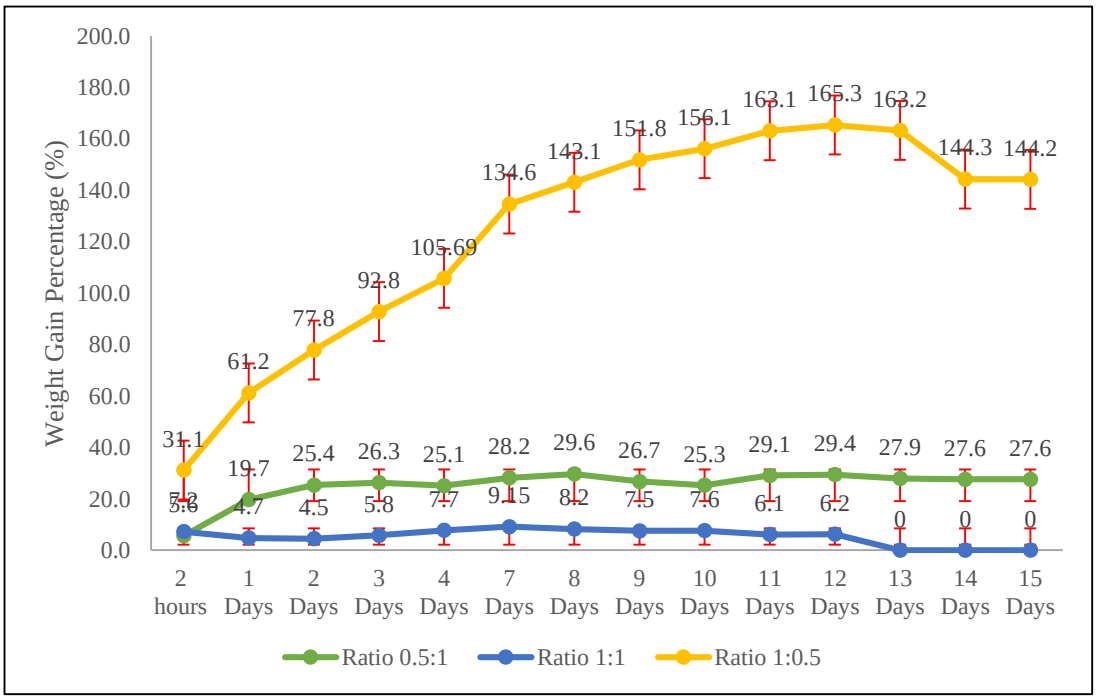


Figure 4.11 Weight gain percentage for Polyurethane of ratios).5:1, 1:1 and 1:0.5

Table 4.3
Data table of Weight Gain Percentage of Ratio 0.5:1, 1:1 and 1:0.5

Sample	Ratio 0.5:1	Ratio 1:1	Ratio 1:0.5
2 hours	5.6	7.2	31.1
1 Days	19.7	4.7	61.2
2 Days	25.4	4.5	77.8
3 Days	26.3	5.8	92.8
4 Days	25.1	7.7	105.69
7 Days	28.2	9.15	134.6
8 Days	29.6	8.2	143.1
9 Days	26.7	7.5	151.8
10 Days	25.3	7.6	156.1
11 Days	29.1	6.1	163.1
12 Days	29.4	6.2	165.3
13 Days	27.9	0	163.2
14 Days	27.6	0	144.3
15 Days	27.6	0	144.2

Overall, the 1:1 ratio sample showed an increased weight percentage but it remained within the acceptable range for grouting PU, thus water absorption did not exceed 12%. This ratio had the lowest average percentage of weight gain compared to the others. This was proven by the surface morphology images depicted in Figure 4.20 later in section 4.6 which showed no intercellular spaces in the structure, making water diffusion difficult (Panda et al, 2017). The closely packed arrangement of the cells renders the sample hydrophobic, which almost repels water. This occurs because there is a greater surface area available for the diffusion of the substance, but a comparatively smaller surface area through which the substance can enter the cell.

For the 0.5:1 ratio sample, the water absorption percentage increased and continued to rise until day 15. The highest percentage of weight gain was 29 %, and the lowest was 7.2 %. This indicates that water diffused into the sample, making equilibrium difficult to achieve (Faure et al, 2019). The tendency of high density substances that was proven in Figure 4.6, sink instead of float in water allows them to absorb water easily.

Figure 4.10 illustrates how a higher crosslinking density in polyurethane can diminish water absorption by restricting the movement of polymer chains and impeding the diffusion of water molecules into the material (Possart et al, 2022).

Moreover, the size dimension of the sample remains unchanged when submerged in water, indicating that it possessed sponge like properties, including a soft texture and the ability to absorb any liquid (Raja et al., 2016). This was demonstrated by the microscopic image later in Figure 4.22 which indicating that when there are holes in each cell wall, water can diffuse in and spread throughout the sample, causing the sample to swell with water and prevent it from shrinking (Saberon, 2021). Density is a characteristic property of a substance that is independent of its quantity (Yeoh et al., 2020). Cell rupture in the cell arrangement of Figure 4.21's micrograph, which allows water to diffuse. It was anticipated that the water absorption would increase with increasing density due to its brittleness, which corresponds to the sample breaking/cracking during compression testing due to the presence of voids and cell ruptures between the cells (Wang et al, 2022).

The morphology cell surface was larger for the 1:0.5 ratio. When we increase the surface area of the adsorbent, the adsorption of gases increases. This is because the number of adsorbing sites increases as the surface area increases (Sahu et al., 2020). Consequently, finely divided solids and certain porous substances are effective adsorbents. This prevents water from escaping when the object was lifted out of the water. Therefore, a 1:05 ratio polyurethane has the characteristics of a porous material. A porous material is any solid that contains void spaces, mean the space not occupied by the main framework of atoms that comprise the solid's structure which not in character with grouting properties (Gao et al., 2021).

Lastly, in the 1:0.5 ratio sample, the water absorption percentage rose for 12 days before decreasing slightly on day 13 although continuing to absorb water. The highest it achieved was 165 %. This indicates that the material has more capillaries or is more porous, resulting in greater water absorption and increased 15 Days weight (Wang et al,2022). The microscopic image in Figure 4.22 demonstrates this, showing holes in each cell wall, which allows water to diffuse in, spread throughout the sample and cause it to swell. Therefore, a 1:05 ratio of polyurethane did not meet the requirements for a grouting material due to its porous characteristics.

Lastly, the 1:1 ratio marginally increased and decrease until 6.2% of the weight percentage, but remained within the range of grouting properties where water absorption

should not exceed 12% (Possart et al., 2017). This ratio has the lowest average percentage of weight gain compared to the others ratios. According to the surface morphology depicted in Figure 4.20, there are no intercellular spaces, making water diffusion difficult. The closely packed arrangement of the cells renders the sample hydrophobic, which can almost repel water. A hydrophobic surface is one that repels water and has a low surface energy that resists wetting (Rasouli et al., 2021). The surface morphology of the 1:1 ratio also indicates that the cell has a small surface area. When there is less cell surface area, diffusion is slower and less efficient. This is because there is a larger surface area that must receive the substance being diffused, but a smaller surface area through which the substance can enter the cell (Sahu et al, 2020)

Water resistance or hydrophobic is a very important property of rigid polyurethane foam that can be advantageous in certain applications. Due to its closed cell structure and hydrophobic nature, rigid polyurethane foam has a low propensity for water absorption. Polyurethane is a common chemical used to waterproof exposed surfaces, such as roofs (Balasubramanian, 2022). Apart from that, polyurethane is composed of polyol and isocyanate, where polyol serves as the base and isocyanate as the reactor component. The combination of these two substances in a predetermined proportion yields polyurethane for waterproofing applications. Additionally, polyurethane is a popular material due to its simplicity of installation (Facilitator, 2021). Additionally, it indicates that the sample has reached equilibrium on day 13 which conclude that 1:1 ratio has a good grouting applications. Figure 4.12 shows the mechanism of water diffusion on polyurethane.

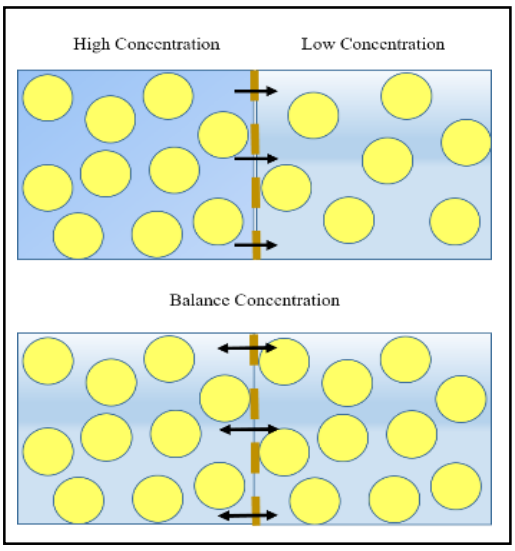


Figure 4.12 Mechanism diffusion of water into polyurethane

4.4.5 Moisture Content

Polyol and isocyanate are the main material on producing polyurethane. Each constituent is hygroscopic. A hygroscopic material is one that readily absorbs moisture from the surrounding environment, such as air. This absorption can be due to either a physical attraction between the material and water molecules (adsorption) or the water actually entering the material (absorption). Hygroscopic materials can be solids, liquids, or even gases (Wang et al., 2024). Condensation, humidity, etc. are typically polyurethane's enemies in terms of formula and product preservation (Ansell et al., 2017). Carbon dioxide is produced when moisture reacts with isocyanate. Polyol is largely inactive. This material will absorb moisture. The magnitude of outgassing is proportional to the amount of moisture and the sensitivity of the isocyanate (Ann, 2022).

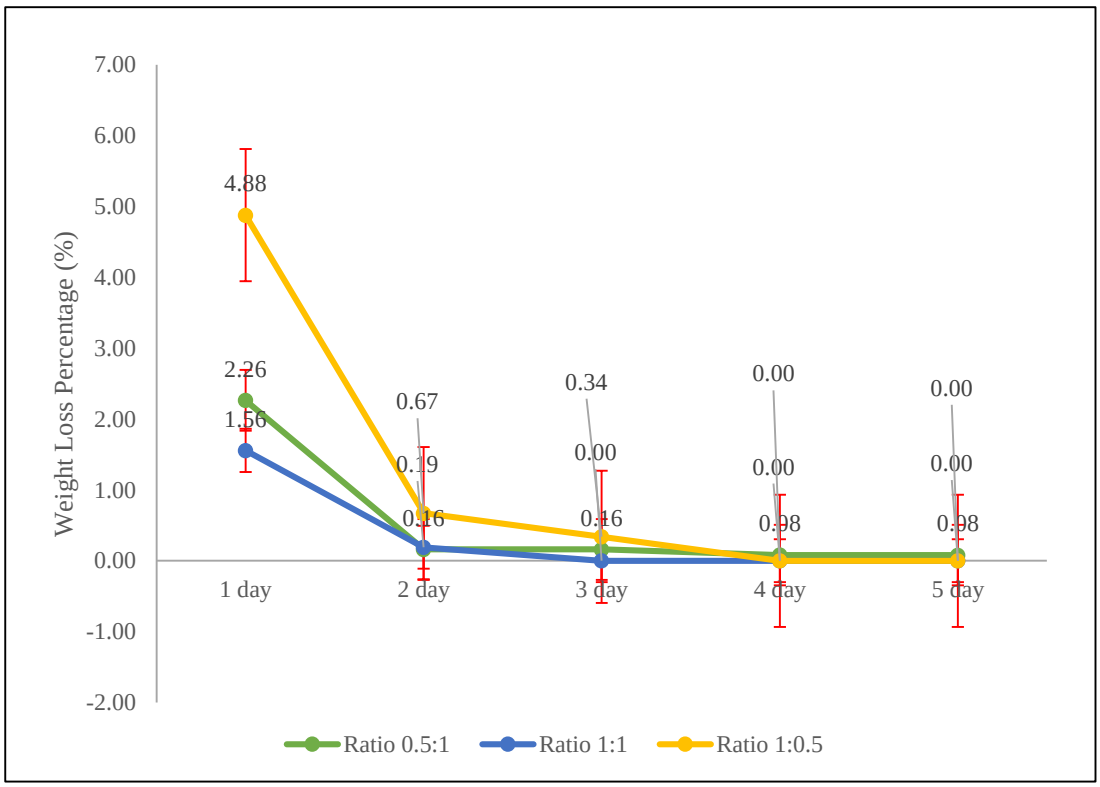


Figure 4.13 Moisture content percentage (%) for all ratios of Polyurethane

Table 4.4
Moisture Content Value of Ratio 0.5:1, 1:1 and 1:0.5

Sample	1 day	2 day	3 day	4 day	5 day
Ratio 0.5:1	2.26	0.16	0.16	0.08	0.08
Ratio 1:1	1.56	0.19	0.00	0.00	0.00
Ratio 1:0.5	4.88	0.67	0.34	0.00	0.00

It was determined that the moisture content percentage of each ratio does not exceed 10%, and that the moisture content of each ratio conforms to polymer specifications (Chamas et al., 2020). Based on Figure 4.13 and Table 4.4, shows that the 1:0.5 ratio has the highest moisture content, at 4%, before dropping rapidly to 0.6% overnight. Due to its high polyol (OH) content, which can evaporate and cause sample weight loss, the 1:0.5 ratio has the highest percentage of weight loss in comparison to other ratios (Amran et al., 2021). This was proven by FTIR analysis on Figure 4.14 shows the OH band broad indicating unreacted polyol was high. Also, the heat relaxes the sample's molecules, causing the polyurethane to return to its original in not oriented size and shrink due to a weight reduction (Ronca, 2017).

For the 0. 5:1 ratio, there was high concentration of flammable isocyanate, which can contribute to a slight increase in the sample's percentage of weight loss. Isocyanates are a group of low molecular weight, highly reactive compounds with a functional isocyanate group. Also, it is highly flammable liquid that evaporates quickly when exposed to the air specially to heat air (Acuña et al., 2019). Polyurethanes and other polymers may have decomposed and produced airborne particulates, gases, and/or vapours same as smoke when heated above normal processing temperatures, such as during hot work activities. This was the results in a loss of mass (Technology, 2022). This deterioration is commonly known as thermal degradation.

The sample has a lower percentage of weight loss for the ratio 1:1, which stabilises the chemical in the production of polyurethane, when compared to other ratios as there is no weight loss starting at day 3. The highest moisture content of 1:1 ratio can have achieved was 1.5 % and decrease to 1% after a day in the oven. Less moisture content indicating that mould cannot form and that bacteria and viruses cannot propagate easily (Wang. et al, 2022). Polyurethanes, unlike other potential synthetic compounds, are resistant to heat and do not melt when heated (Bengtström et al., 2016). Without fear

of melting or overheating, these materials are the ideal solution for elements that emit large amounts of energy, such as automobiles (Guo et al, 2018).

4.5 Fourier Transmittance Infrared Spectroscopy (FTIR) Analysis

FTIR is used to analysed the chemical structure and interactions between the polyols and isocyanates in polyurethane. The FTIR peaks correspond to the functional groups present in the samples. The spectra of raw polyol and isocyanate are presented in Figure 4.14 and Table 4.5. The spectrum of polyol exhibits absorption bands within the range of 3500 to 3200 cm^{-1}, which correspond to the stretching of O-H functional groups. The isocyanate groups were identified by the bands at 2276 cm^{-1}, which correspond to the stretching of -N-C-O- bonds.

A comparison of the spectra of the three samples is presented in Figure 4.15 and Table 4.5. According to the FTIR spectrum, demonstrate that different ratio gives different size peak cause by the different concentration of polyol (OH) and isocyanate (NCO) of polyurethane. The spectrum of the 1:1 ratio sample shows the presence of polyol and isocyanate functional groups that correspond to the C=O, C-N and C-O-C at 1700 cm^{-1}, 1224 cm^{-1} and 1074 cm^{-1}, respectively, which are almost identical to the peaks in the spectra of raw polyol and isocyanate. For ration 0.5:1 shown in Figure 4.15 depict the narrow wavelength on N=C=O stretching peak proven there were excess of isocyanate left untreated thus occurs the allophanate crosslinking as discuss in section 4.3. It is also shown on ratio 1:0.5 have the excess hydroxyl group indication there were untreated polyol reacted with isocyanate which make the foam become soft. This proves the material has a highly cross linked structure containing a much higher proportion of urethane and substituted urea groups, which induces strong hydrogen bonding exchange forces (non-covalent interactions) (Lee et al, 2018). The ratio of 1:1 also shows the best in terms of the formability of the foam giving the uniform cells size and the best mechanical characteristics as compared to other ratios (Mohamed et al., 2021).

In addition, the infrared spectra of the polyurethane samples with ratios of 1:1 and 0.5:1 displayed smaller and broader bands than the sample with a ratio of 1:0.5, indicating that there was no excess polyol in the samples and all of the isocyanates in the pre polymer had reacted with each other (Lee et al, 2018). Furthermore, the C-O-C stretching peaks exhibited a greater intensity compared to the other samples. This can

be attributed to the presence of excess polyol in the polyurethane formulation with a ratio of 1:0.5.

The isocyanate (N=C=O) content of the 1:0.5 ratio sample was lower than other samples, indicating that this ratio contained a higher composition of polyol, causing the sample to shrink throughout the curing process, as seen in Figure 4.4. Thus, polyurethane with a 1:1 ratio comprised all functional groups that constituted a well-made polyurethane. Excess polyol in the 1:0.5 ratio sample reduced its tendency to form urethane linkages (Lang et al, 2020). Fewer urethane bonds were formed with a lower isocyanate concentration in the polyurethane system, resulting in a weaker interaction between polyurethane chain molecules.

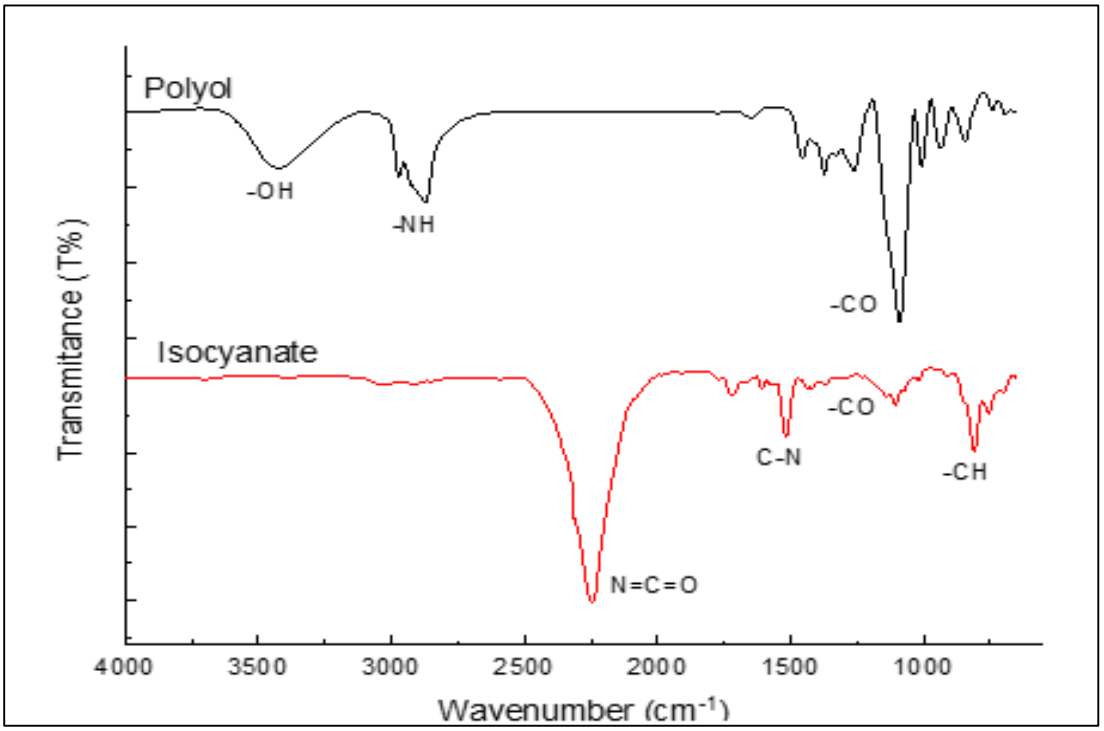


Figure 4.14 FTIR Analysis of polyol and isocyanate

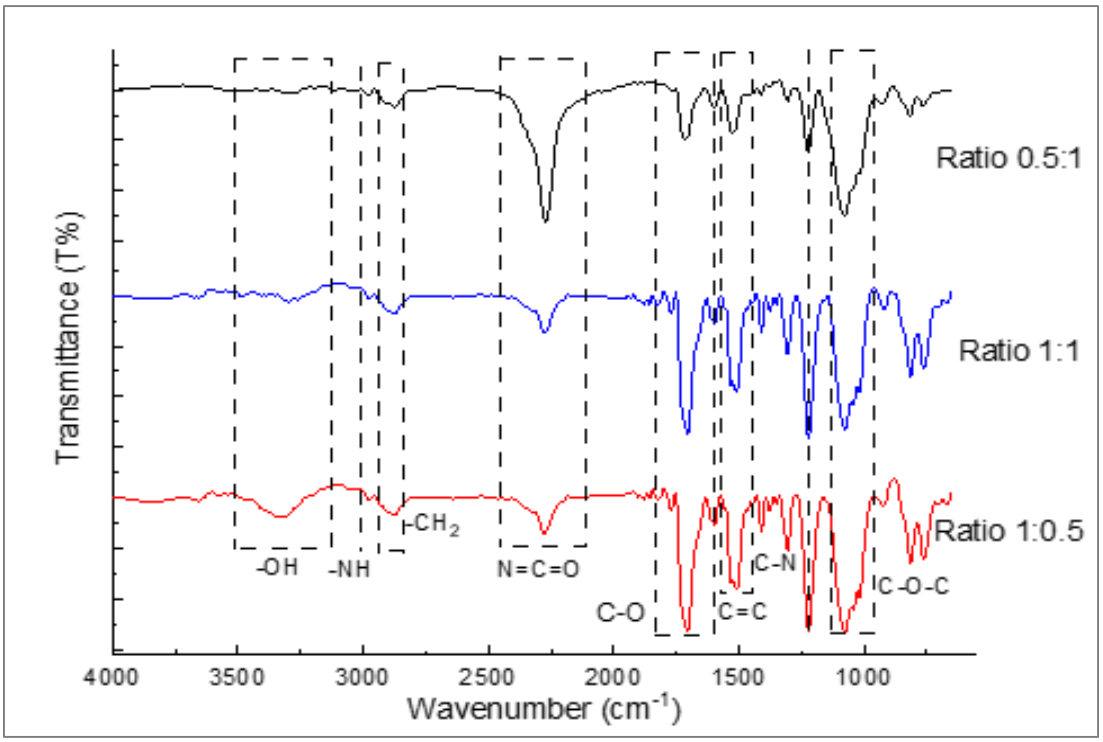


Figure 4.15 FTIR Analysis of all type ratio

Table 4.5
FTIR Spectrum Value of the Different Ratio Polyol and Isocyanate

Reference Wavenumber (cm^{-1})	**Ratio 0.5:1 Wavenumber (cm^{-1})**	**Ratio 1:1 Wavenumber (cm^{-1})**	**Ratio 1:0.5 Wavenumber (cm^{-1})**	**Functional Group Wavenumber (cm^{-1})**
3200-3600	3330	3324	3336	O-H
2270-2300	2274	2276	2278	N=C=O
1700-1735	1706	1700	1704	C=O
1220-1320	1226	1224	1224	C-N
1100-1200	1184	1174	1178	C-O-C

4.6 Mechanical Properties: Compression Strength and Modulus

The analysis of compression is depicted in Figure 4.16. This experiment was conducted to determine whether each sample ratio of polyurethane has the ability to withstand pressure for an extended period of time because it is not easily broken, cracked, or deformed. From methodology state in section 3.5 with a compressive force of 50 kN and a cross speed of 5 mm/min, the sample takes a considerable amount of time to deform. Hence the compression strength and modulus were calculated. These characteristics are necessary to ensure the safety of overly extensive pavements, structural stress and deformation caused by temperature variations, and regular pressure application (Dang et al., 2013).

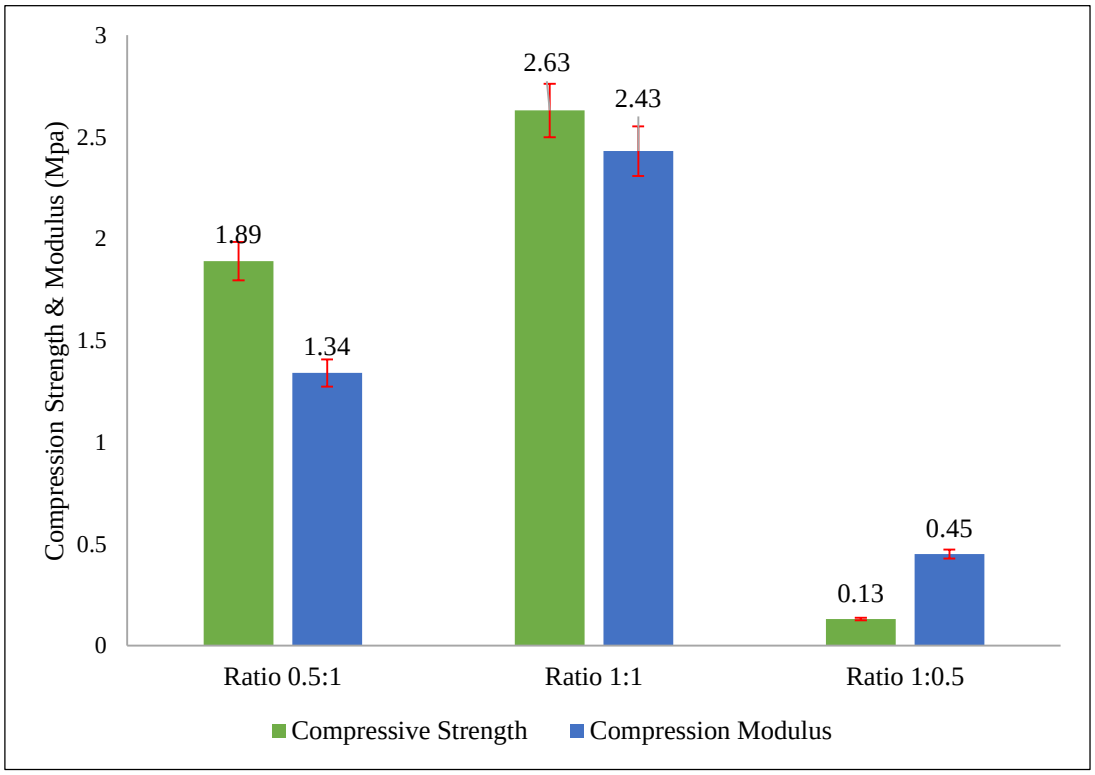


Figure 4.16 Compression Analysis of polyurethane with all types of ratio

Compressive strength is the maximum compressive stress on a plastic specimen can endure before rupturing or deforming by a specified percentage (Prajapati et al., 2022). The Figure 4.16 demonstrates that the compressive strength and modulus of ratio 1:1 are 2.63 MPa higher than ratio 0.5:1 and also the 1:0.5 showed 0.13 MPa value due soft like properties as shown in Figure 4.4. The diminished compression strength may be attributed to the increase in moisture content, as illustrated in Figure 4.13. The porosity of a material is dependent upon its moisture content, with a high water

absorption exceeding 50% weight increase, as illustrated in Figure 4.11. This condition leads to a reduction in compressive strength and does not comply with polyurethane grouting standards.

The compression modulus characterises the material's resistance to changes in length when subjected to compressive loads. The greater a material's modulus of compression, the more rigid it is (Corrosionpedia, 2019). Figure 4.16 showed the compressive modulus of 1:1 ratio was 2.43 MPa which was higher than ratio 0.5:1 and ratio 1:0.5. Sample for the ratio 1:0.5 also have the lowest value compared to the other ratios, 0.45 MPa of compression modulus.

The density of polyurethane could influence the mechanical properties of polyurethane foam (Manjun et al., 2020). Figure 4.7 showed that the ratio 1:1 has a lower amount density than the ratio 0.5:1. Research indicates that the lower the density of the polyurethane, the less impact properties have in comparison to the higher density (Wang, et al., 2022). Higher isocyanate content in ratio 0.5:1, leads to higher crosslink density, resulting denser foam structures but exhibits brittle like properties. This is because the sample become break after compression testing conducted upon it as shown in Figure 4.17.



Figure 4.17 Sample of ratio 0.5:1 after compression

In addition, the size of the material's cells plays an important role in its mechanical properties (Gama et al., 2018). It is possible that larger sample cell sizes are weaker than smaller ones, but they may also be stronger (Andrade, 2020). Figure 4.20 demonstrates that the 1:1 ratio is rigid polyurethane, which has closed cell foam confirm by the SEM analysis. Closed cell foam is defined as a cell totally enclosed by its walls and hence not interconnecting with other cells (Mike et al., 2017). The ratio 1:1 is sufficient to prevent the cell from rupturing, as evidenced by the cell's inability to

rupture. Figure 4.18 illustrates the sample ratio of 1:1 following the applied compression. The sample exhibits no obvious break instead, only deformation is observed as shown in figure 4.19. As depicted in Figure 4.16 for a 1:1 ratio, the uniform cell arrangement also indicates the sample has an elastic property to the material which give bouncing effect on the material. Cell structure distinguishes polyurethane rigid foams from their flexible or semi flexible counterparts.



Figure 4.19 Sample of ratio 1:1 after compression

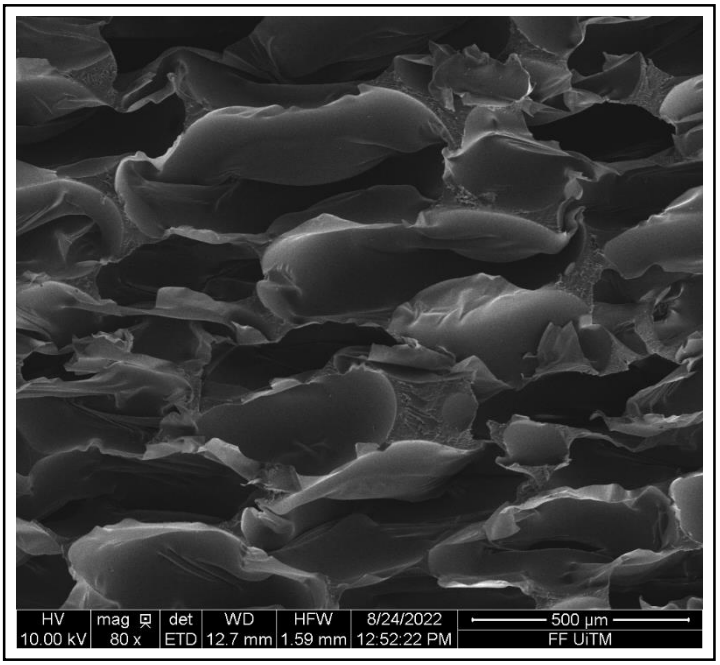


Figure 4.18 SEM images of ratio 1:1 after compression testing

4.7 Morphology Analysis: Scanning Electron Microscopy (SEM)

Figures 4.20, 4.22 and 4.24 shows the microscopic photographs of a cross sectional each ratio polyurethane sample at with magnifications of 80X before and after conducting compression testing. Based on compression result on Figure 4.16, the ratio 1:1 give the highest compressive strength and modulus. Whereas, ratio 0.5:1 which have high density resulting the sample rupture during compression testing as shown in Figure 4.7. For ratio 1:0.5, explicit the lowest value due to sample's shrinkage condition.

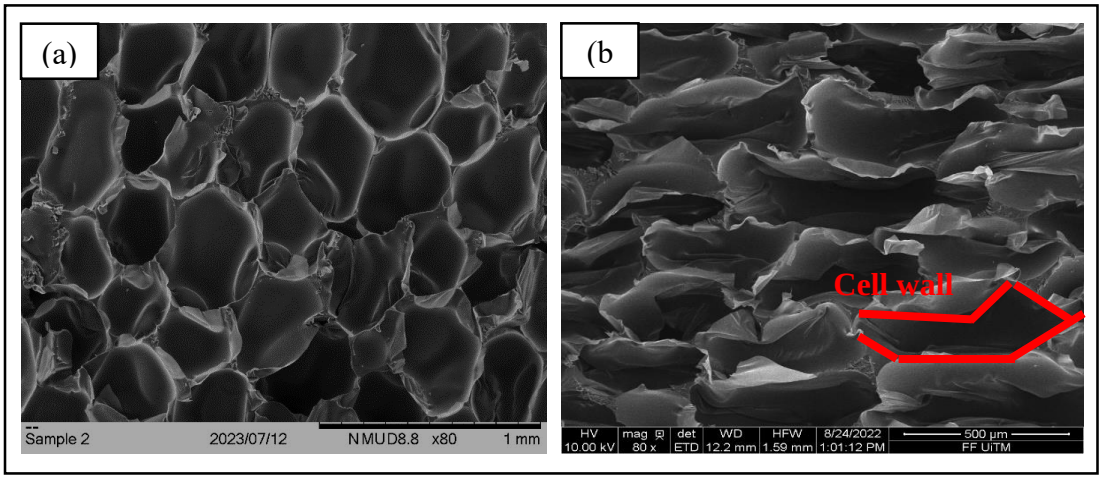


Figure 4.20 SEM images of ratio 1:1 uniform cell and good arragement (a) before compression testing;(b) after compression testing

The morphology of polyurethane foam was characterised using SEM since the mechanical properties are mostly determined by the apparent density and size of the cell. This approach was used to observe the standard rigid and flexible polyurethane foam cell structure (Tudor, 2018). Based on the SEM analysis portrayed in Figure 4.20 (a) and (b), the similar cell size and regular cell arrangement before and even after compress, demonstrate the excellent polyurethane mechanical and morphological properties. The same cell size and linear cell arrangement represent the rigidity of the sample which composed of <90% of closed cell (Skleničková et al., 2020). The morphology of pure polyurethane foam shows stable cell structure and is smaller in size (uniform cell), it will give better strength and modulus of the polyurethane rather than foam with irregular and bigger cell size (Soberi et al, 2017). It can be proven by Figure 4.21 as shown the cell size of for the ratio 1:1 has the average of 40 to 190 µm. A smaller cell size implies a higher number of cells per unit volume. This means the foam has

more closed cell foam and struts to support heavy load, leading to the high compression strength as shown in Figure 4.16 (Blazejczyk, 2018).

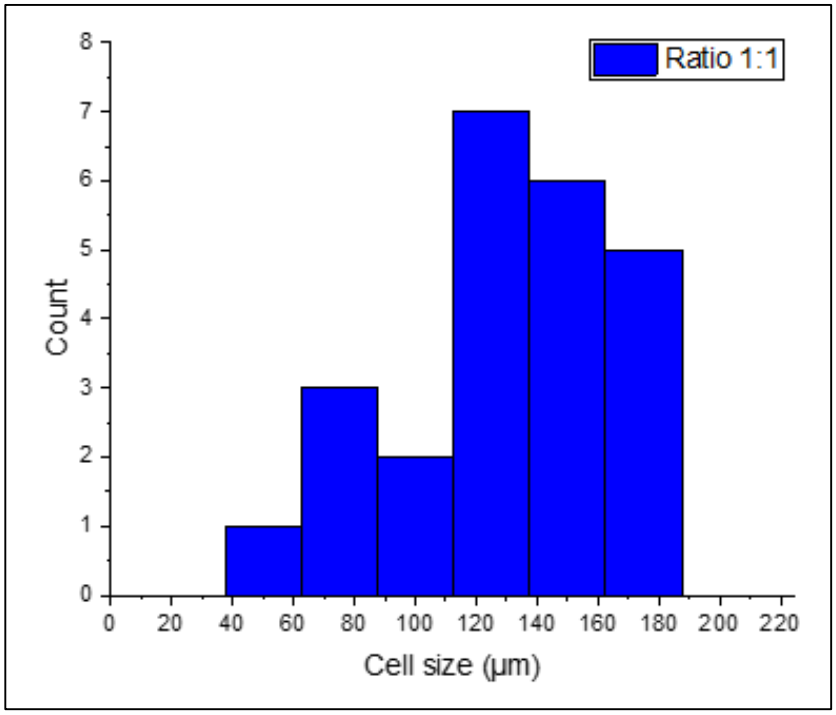


Figure 4.21 Cell count of the cell foam size for Ratio 1:1

Based on the FTIR analysis as illustrate in Figure 4.15, ratio 1:1 indicates that the material has a highly cross linked structure containing a much higher proportion of urethane and substituted urea groups, which induces strong hydrogen bonding exchange forces (non-covalent interactions) made up the polyurethane as lower density (Tudor, 2018). The low density characteristic of the 1:1 ratio sample, as illustrated in Figure 4.7, indicated that the material was both lightweight and highly efficient regarding its performance-to-weight ratio (Mouchati et al., 2023). The low density features of a 1:1 ratio imply that the cell wall is unable to endure compression as stress levels increase, resulting in the compression of cell structures and a significant decrease in volume. There were no visible cracks in the cell structure during the compress stage, but their deformation is irreversible as shown in the Figure 4.20. In other words, it conforms to the gradual increase of stress with increase compression strength observed in the microscopic experiment (Panda et al., 2017).

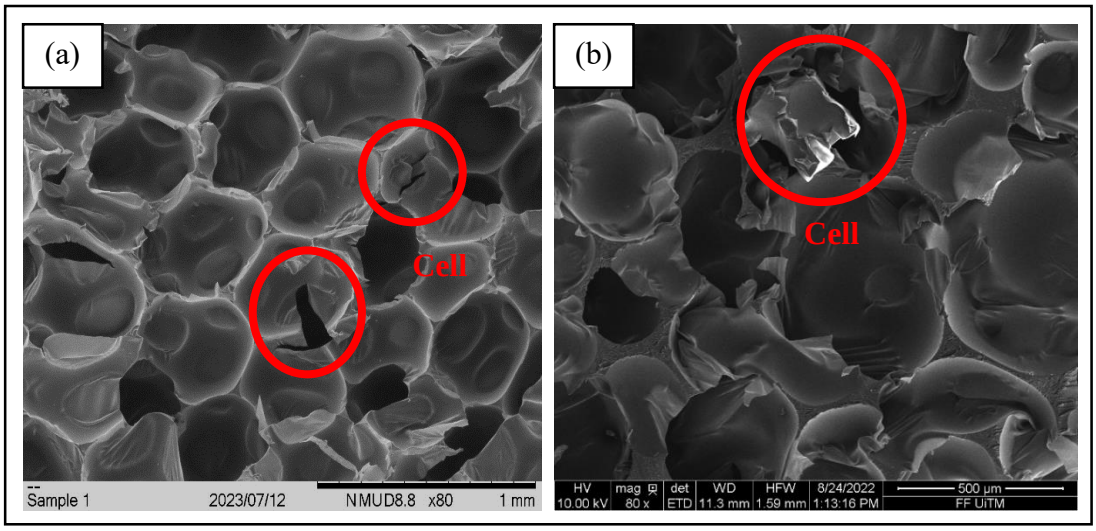


Figure 4.22 SEM Images of ratio 0.5:1 uniform cell with cell rupture; (a) before compression testing;(b) after compression testing

From Figure 4.22 (a) and (b) illustrate that the cell foam has nearly uniform cell size and arrangement with a few numbers of broken cell walls even before compression testing was conducted. The different cell arrangement can increase the likelihood of defects. The damaged foam within the cell structure demonstrates their brittle and dense characteristics. The increased cell size also indicated that fewer cells formed in a given volume, resulting in a decrease in foam density, which corresponded with the following apparent density as showed in Figure 4.7 (Wu et al., 2022).

Moreover, microscopic evidence suggests that the cell wall cannot support the load. The structure of the sample cell not only resulted in compression, but also in the formation of a few cracks, which can be considered brittle fractures or failures (Liu et al, 2019). It is also can be prove using Figure 4.22 (b) as sample was broken after compression testing was conducted. The foam conformation is important for the determination of the mechanical characteristics of the polymer, which will influence its application (Tudor, 2018). Therefore, SEM images have demonstrated that brittle and plastic failure can be resulted from a high density polyurethane and not suitable to be used in grouting applications.

Figure 4.23 shows the cell size of the SEM images for ratio 0.5:1 in between 75 to 375 µm. These cell size contributes to high density as shown in Figure 4.7 which depicts ratio 0.5:1 have the high amount of apparent density compared to ratio 1:1. Based on FTIR analysis in Figure 4.15, shows the presence of high content of isocyanate

which the primary reason for the difference in cell size leading to relatively uniform cell. The unreacted isocyanate promotes the formation of allophanate linkages which can increased its density and also the harden the polyurethane foam which also contribute to its brittleness (Chae et al., 2024).

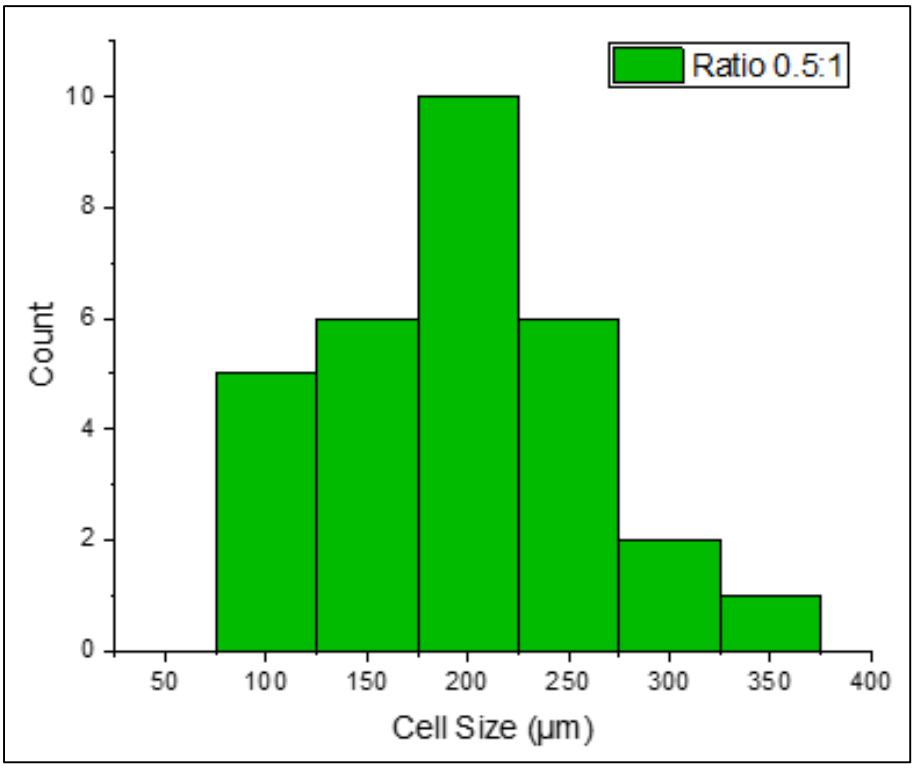


Figure 4.23 Cell count of the cell foam size for Ratio 0.5:1

High polyol content structures have very few shrinkage foam cells, larger cell sizes, and holes or tears in every single cell of the foam. This demonstrates that a 1:0.5 ratio has a high viscosity in the system, inhibiting the expansion of the cells. These microscopic images reveal that an increase in polyol causes a greater number of cells to open with a smaller size, resulting in a denser foam, but poor interfacial adhesion between the polyol and isocyanate due to agglomeration in the polyurethane foam matrix (Liu et al, 2019). As illustrated in Figure 4.24, a decrease in the isocyanate index produced more open cell structures and void in between the cell with somewhat larger cell sizes at the expense of increased irregularities. This outcome was consistent with the well-established behaviour of flexible polyurethane foams (Kaikade et al, 2022).

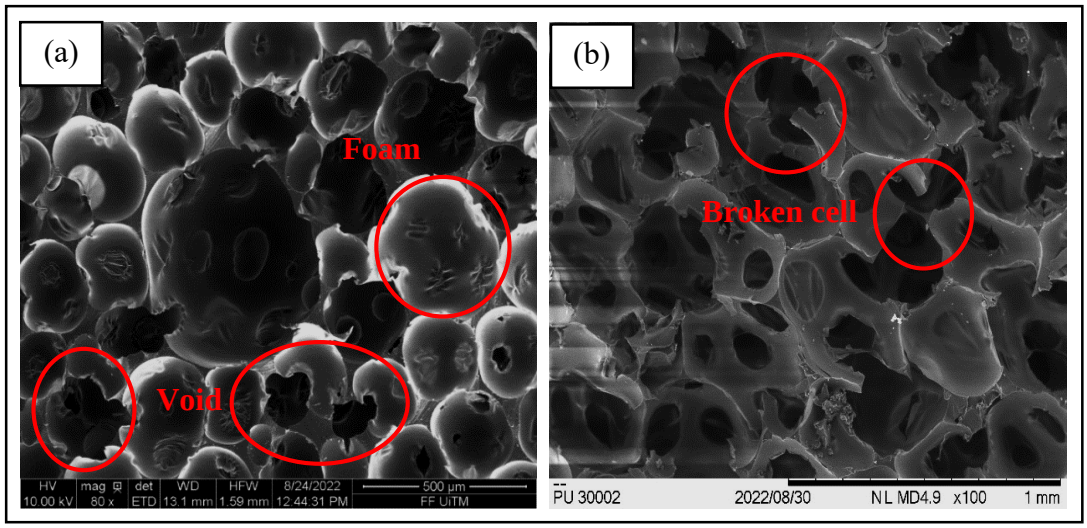


Figure 4.24 SEM images of ratio 1:0.5 non uniform size cell ; (a) before compression testing; (b) after compression testing

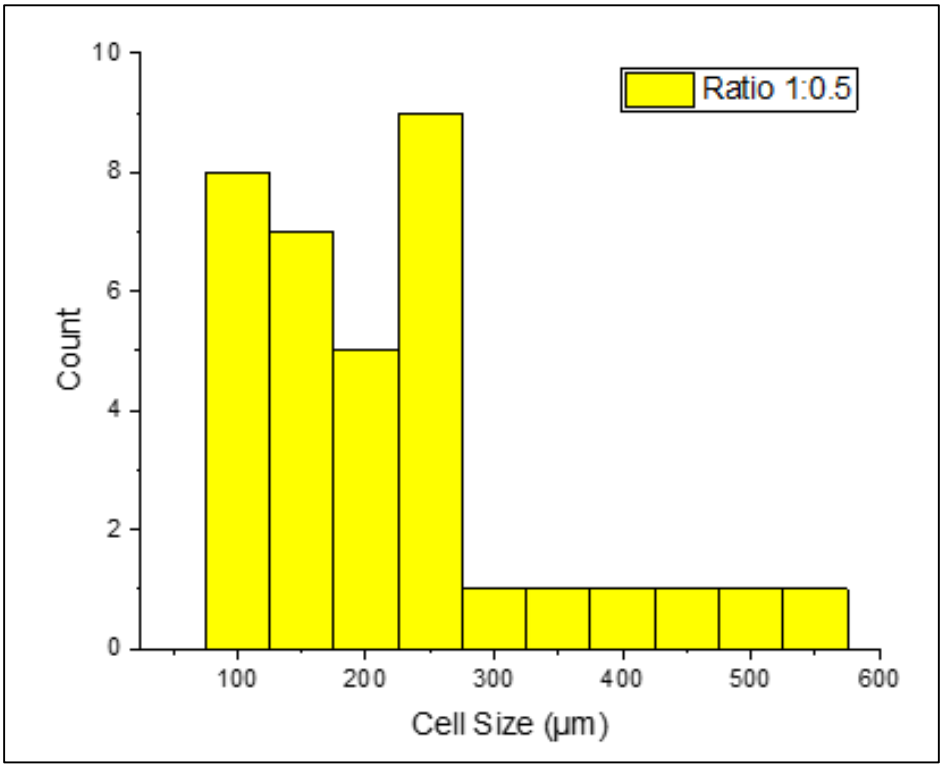


Figure 4.25 Cell count of the cell foam size for Ratio 1:0.5

Based on Figure 4.25, the cells size is in between 100 to 600 µm which represent the average diameter of micro cells with pores. This range is considered characteristics of standard flexible polyurethane foam which the cell large enough to be seen with the naked eye but small enough to provide good thermal can strength (Ju et al., 2025). By using high reactivity polyol, it can lead to a faster foaming process, which can trap more

gas in smaller bubbles initially. This can be proven by the time taken for the rise time in Figure 4.6 has the fastest time compare to the other ratios. Also, with high content of polyol in polyurethane foam, it can cause the cell walls become weak, leading to cell coalescence (smaller cells merging into larger ones) (Chae et al., 2024).

4.8 Summary of Ratio Study

Three different polyurethane ratios were successfully synthesized; the 1:1 ratio exhibits favourable characteristics in terms of characterization time, density, rheology, and water uptake compared to the 0.5:1 and 1:0.5 ratios. From these findings, the 1:1 ratio a well suited polyurethane grouting material requirement, imparting hardness and rigidity to the product. FTIR analysis indicates that the 1:1 ratio possesses a complete urethane linkage than the other ratios. Ratio 1:1 yields good results in apparent density, rheology analysis and water absorption. Water absorption of the 1:1 ratio also does not exceed 20%, demonstrating its suitability as a grouting material, which was proven by microscopic images revealing uniform cell size and arrangements of ratio 1:1 and balance stoichiometry between the polyol and isocyanate. Hence, ratio 1:1 was chosen as the formulation of polyol and isocyanate in order to produce good properties polyurethane for grouting applications.

CHAPTER 5
THE COMPARISON BETWEEN INDUSTRY AND GREEN POLURETHANE USING CASTOR OIL BASED POLYOL WITH DIFFERENT BLOWING AGENTS

5.1 Introduction

This chapter compared the best blowing agent for castor oil polyol as a replacement for industrial polyurethane. Additionally, various blowing agents, including 1,1-dichloro-1-fluoroethane (HCFC 141b) and water (H_2O), were used, and their properties were compared to determine which is suitable for polyurethane grouting. Improving Malaysia's building industry's framework and infrastructure requires comparing the two types of green polyurethane and making industrial polyurethane (PI) the benchmark for polyurethane grouting applications.

5.2 Synthesis of Castor Oil polyurethane

The blowing agents were introduced into the castor oil polyol for a specific time, then isocyanate was added. Both samples were left to rest for 24 hours for the post-cure process. Based on the ratio analysis of the polyurethane industry (PI) in section 4.0, the formulation of polyol to isocyanate was 1:1. Formulation for both types of blowing agents has been described in Table 3.3 at Section 3.7. Figure 5.1 shows the synthesis of green polyurethane using castor oil as the polyol and HCFC 141b (PCH) and water (PCW) as blowing agents.

In the PI, a 1:1 ratio indicates a balance between polyol and isocyanate, with no blowing agent required. The ratios are optimized to attain specific foam properties, including a balance of mechanical strength and lightweight characteristics with closed cells. The 1:1:0.25 PCH ratio indicates a small HCFC 141b requirement, attributed to the high fatty acid content of castor oil, which can affect cell structure and foam stability. The hydroxyl value of castor oil directly influences the formulation of HCFC 141b-blown polyurethane by limiting the number of available reaction sites for isocyanate. A reduced amount of HCFC 141 b is required to achieve the desired closed-cell structure and adequate mechanical strength. An imbalance could result in poor foam quality due

to under cross-linked matrices that cannot retain the blowing agent vapour effectively. The PCW exhibits a 1:1:0.015 ratio, indicating a notably reduced water content to prevent isocyanate depletion. Water reacts with isocyanate, potentially consuming it. Castor oil has a relatively low hydroxyl value (161.83 mg KOH/g) compared to that of petroleum-based polyols. This affects the reactivity with isocyanates. In PCW, the available isocyanates must cover both urethane formation and gas generation via the water reaction, hence the very low water ratio (0.0015) to avoid depleting isocyanate excessively.

Figure 5.1 shows a sample of both types of castor oil polyurethane. The PCH sample has a darker white colour compared to PCW. The difference in colour was because of the types of blowing agents used. As PCH was using HCFC 141b, a physical blowing agent, HCFC 141 b is non-reactive with isocyanate, so the stabilizer interaction changed the colour due to residual catalyst. It also has undergone oxidation of aromatic isocyanates. As for PCW, the side reaction of reaction water to isocyanates may lead to more urea groups, which are typically less coloured and may bleach the polymer matrix over time as the foam cures and oxidizes slowly.



(a) Castor oil- HCFC 141b (PCH)



(b) Castor oil-water (PCW)

Figure 5.1 Sample of (a) PCH and (b) PCW

5.3 Physical Properties

5.3.1 Foam Reaction Time

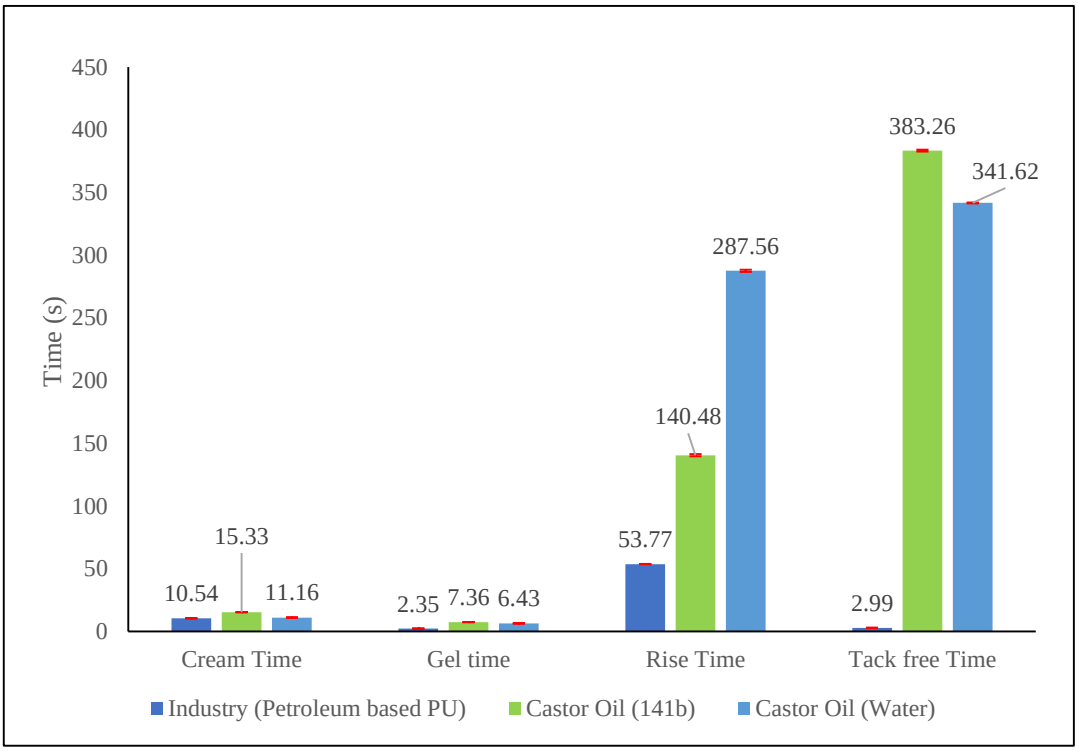


Figure 5.2 Foam reaction time of industrial and castor oil based polyol with HCFC 141b and water as blowing agent

The graph in Figure 5.2 shows how long the foaming process took to produce polyurethane with different blowing agents: water and HCFC 141b. It is essential to record the cream time, gel time, rise time, and tack-free time to ensure the material's consistency, quality, and optimal performance in world applications.

The cream time for PCW was 11.16 seconds, and PCH took about 15.33 seconds, resulting in a 4-second difference. A higher concentration of hydroxyl groups in water accelerated the foaming process. Water initiated the polyurethane foam reaction faster than HCFC 141b (1,1-dichloro-1-fluoroethane), producing carbon dioxide rapidly through its reaction with isocyanates. Gel time, which shows the transition from liquid to solid, was slightly longer for HCFC 141b (7.36 seconds) than for water (6.43 seconds), indicating that water forms gel a little faster. Rise time, referring to how long it took for the foam to reach its full size, was longer for water (287.56 seconds) compared to HCFC 141b (140.48 seconds), which means that HCFC 141b makes the

foam expand more quickly due to its vaporizing rapidly upon mixing and creating gas bubbles that expand the foam efficiently.

Finally, the tack-free time, which is the amount of time it takes for the foam surface to stop sticking, was longer for water (341.62 seconds) than for HCFC 141b (383.26 seconds). Water cured more slowly because the reaction between water and isocyanates produced carbon dioxide gas more gradually, resulting in a slower expansion and curing process (Chen et al., 2018). Overall, HCFC 141b reacts faster most of the time, except during the cream time. On the other hand, water reacts more slowly and steadily, but becomes fully cured after a day. Results show that water can be used as a blowing agent for polyurethane production because the foam reaction time is similar to and consistent with that of HCFC 141b.

5.3.2 Apparent Density

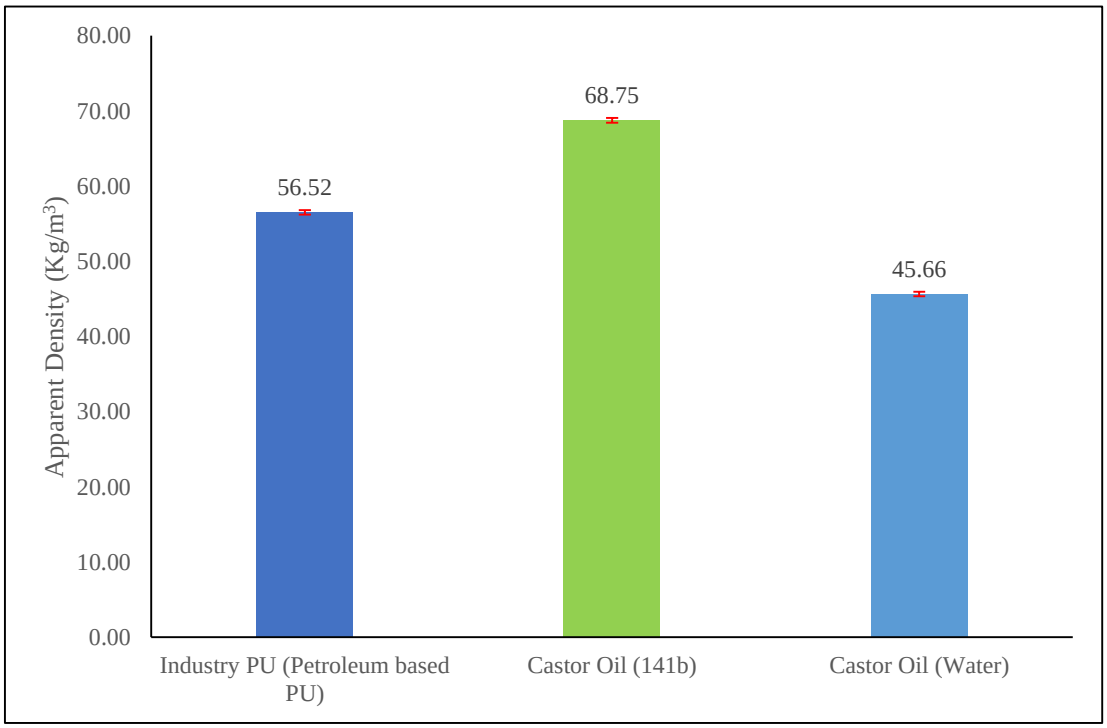


Figure 5.3 Comparison value of apparent density of PCH, PCW and PI

Figure 5.3 shows the apparent density of polyurethane using two distinct blowing agents, HCFC 141b and water. An important factor in figuring out the properties and usefulness of polyurethane foam for various applications is its apparent density. This shows that the foam made with HCFC 141b has a much higher apparent density (68.75 kg/m³) than water (45.66 kg/m³). Water is lighter than HCFC 141b

because it has a lower molecular weight (approximately 18 g/mol) compared to HCFC 141b (approximately 117.5 g/mol).

HCFC 141b makes the foam structure much denser and maybe even stiffer than water polyurethane. Higher-density foams are usually stronger, with better load-bearing ability and structural integrity. Because of this, they work well in standard polyurethane grouting, where strength and durability are very important, for applications such as foundation support, soil stabilization, and hole filling. The dense foam ensures that the grouted area can withstand high stress and offers a strong, durable option (Chen et al., 2021). The apparent density of standard polyurethane foam typically ranges from 30 to 50 kg/m³ for flexible foams and from about 100 to 200 kg/m³ for rigid foams, though specific values can vary depending on the formulation and application.

The water-based polyurethane foam is lighter, more flexible, and may be better at insulating because it conducts heat less well. It can be useful in situations where weight reduction is important or where heat insulation is more important than structural support. For example, lower-density foams can be used to fill gaps in building insulation without adding significant weight to the structure. This makes it more energy-efficient by slowing heat transfer through the structure (Chen et al., 2021).

HCFC 141b has been used extensively because it is effective for producing dense foam. It is known, though, that it harms the climate and contributes to ozone depletion and global warming. Water as a blowing agent is better for the environment because it poses fewer risks. Water-based foams could be a desirable property of polyurethane grouting by modifying and varying the composition of isocyanate and polyol. Modification of the composition of the water blowing agent might improve the properties of water-based PU.

5.3.3 Rheology Analysis

Rheology index for polyurethane foam produced with two separate blowing agents, water and HCFC 141b, is shown in Figure 5.4. The flow behaviour and viscosity of the foam mixture were represented by the Rheology Index, which measures in cm/g. These are two essential properties for conventional polyurethane grouting projects. The foam created with HCFC 141b has a higher Rheology Index (2.4 cm/g) than the foam made with water.

A higher rheology index is beneficial in standard polyurethane grouting because it ensures the material can penetrate small areas and fill voids more effectively, thereby producing a more uniform and comprehensive coverage (Izarra et al., 2021). In structural repairs and soil stabilization projects where complete infiltration is required to guarantee the integrity and stability of the grouted region, this is especially crucial. On the other hand, the lower Rheology Index of the foam produced with water results in a thicker, less fluid consistency, which would be advantageous in situations where slower flow is needed to control placement and prevent excessive spreading. The rheology index of HCFC 141b offers better flow characteristics for applications requiring deep penetration and coverage than water. However, both polyurethanes exhibited rheological properties consistent with industrial grouting standards. Polyurethane grout has a rheology index between 0.4cm/g and 5.0 cm/g (Münstedt, H., 2021).

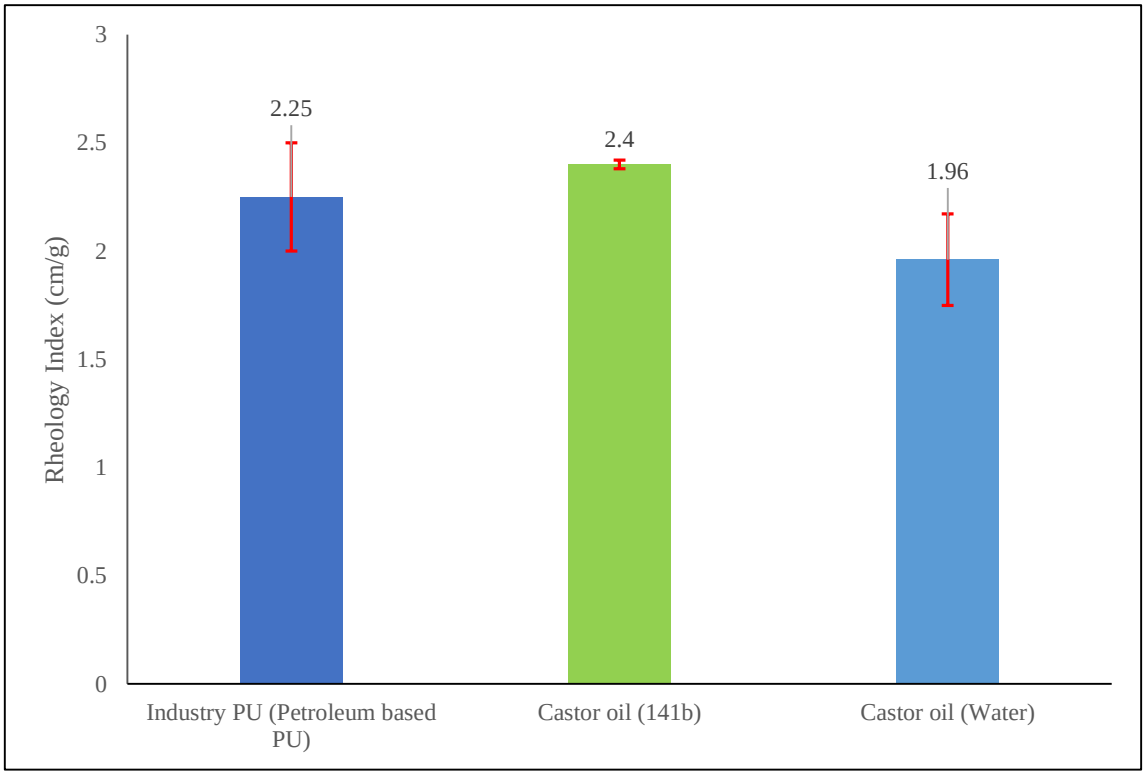


Figure 5.4 Rheology index analysis of all types of polyurethane

5.4 FTIR Analysis

The Fourier Transform Infrared Spectroscopy (FTIR) revealed the transmittance patterns for industrial polyurethane (PI) and green polyurethane made with two different blowing agents: water and HCFC 141b. Producing an infrared absorption spectrum is a powerful analytical technique for identifying organic, polymeric, and, in some cases, inorganic materials. Polyurethane is formed by the reaction between an isocyanate and a polyol. In castor oil-based polyurethane, castor oil (a triglyceride with hydroxyl groups) is used as the polyol. HCFC 141b and water are commonly used blowing agents in polyurethane foam production. These agents help to create the cellular structure of the foam by generating gases during the polymerization process.

Typically, industrial polyurethane (PI) is derived from petroleum-based polyols. The FTIR spectrum in Figure 5.5 shows strong, well-defined peaks corresponding to the urethane linkages, with little interference from other natural substances. Castor oil-based polyurethane has additional peaks related to the ester groups in the triglyceride structure of castor oil. These appear around 1730 cm^{-1} (C=O stretching in esters). Castor oil's hydroxyl groups (–OH) also contribute to the broad peak around 3300–3500 cm^{-1}. 141b may introduce slight variations in the FTIR spectrum, particularly in the C–H stretching region (2800–3000 cm^{-1}) due to the presence of halogenated alkanes.

Water reacts chemically with isocyanates to form urea linkages (–NHCO–NH), generating carbon dioxide gas that expands the foam. This reaction produces changes in the FTIR spectra. The presence of water as a blowing agent leads to new urea linkages, detectable through shifts or new peaks in the N–H and C=O regions. 141b, being chemically inert, does not contribute significantly to the FTIR spectrum but may introduce subtle changes in the aliphatic C–H stretching region.

Characteristic peaks are seen in the green polyurethane; both spectra match several functional groups in the polyurethane foam. Usually associated with the polyol component in the polyurethane formulation, the wide band between 3200-3600 cm-1 typically indicates the presence of hydroxyl (OH) groups. This peak in the spectrum for polyurethane with water as the blowing agent was more pronounced, suggesting a higher concentration of residual hydroxyl groups, possibly due to a less complete reaction or higher moisture content (Yang et al., 2023).

By comparison, the spectra for polyurethane with HCFC 141b indicate less intensity in this range, suggesting fewer OH groups. This variation can be attributed to

the more thorough interaction between the isocyanate (NCO) groups and the polyol upon HCFC 141b, which is consistent with the faster foam reaction times observed in Figure 5.1. The NCO groups correspond with the peaks seen at approximately 2270 cm^{-1}. Here, the water-blown foam exhibits a higher transmittance, suggesting more unreacted NCO groups and hence a slower reaction or less effective crosslinking than the HCFC 141b-blown foam.

Other important peaks indicate the carbonyl (C=O) stretching vibration at about 1700 cm-1 and the C-O-C stretching vibration of the urethane linkage at roughly 1100 cm^{-1}. These peaks facilitate the growth of the polyurethane network. The variations in the chemical environment and degree of crosslinking in foams generated with different blowing agents are reflected in the intensity and sharpness of these peaks.

The broader peak spectrum observed in the water-blown foam suggests a more homogeneous structure, likely due to the higher moisture content and side reactions that form urea bonds (Yang et al., 2023). The faster rise and tack-free times for the HCFC 141b-blown foam correspond to a more complete reaction and effective formation of the polyurethane network, as revealed by the FTIR spectra, compared with these chemical insights into the foam reaction times.

The polyol component in industrial polyurethane is synthetic, resulting in cleaner spectra with fewer overlapping peaks. The spectrum typically shows well-defined absorption bands for urethane and urea. FTIR analysis of both industrial and castor oil-based polyurethane foams with HCFC 141b and water as blowing agents provides crucial information about the material's chemical structure. The technique identifies the key functional groups involved, highlights the differences introduced by the type of polyol used, and shows the effects of blowing agents, especially the water-induced formation of urea linkages. These spectra confirm the success of the polymerization process and ensure the desired material properties have achieved.

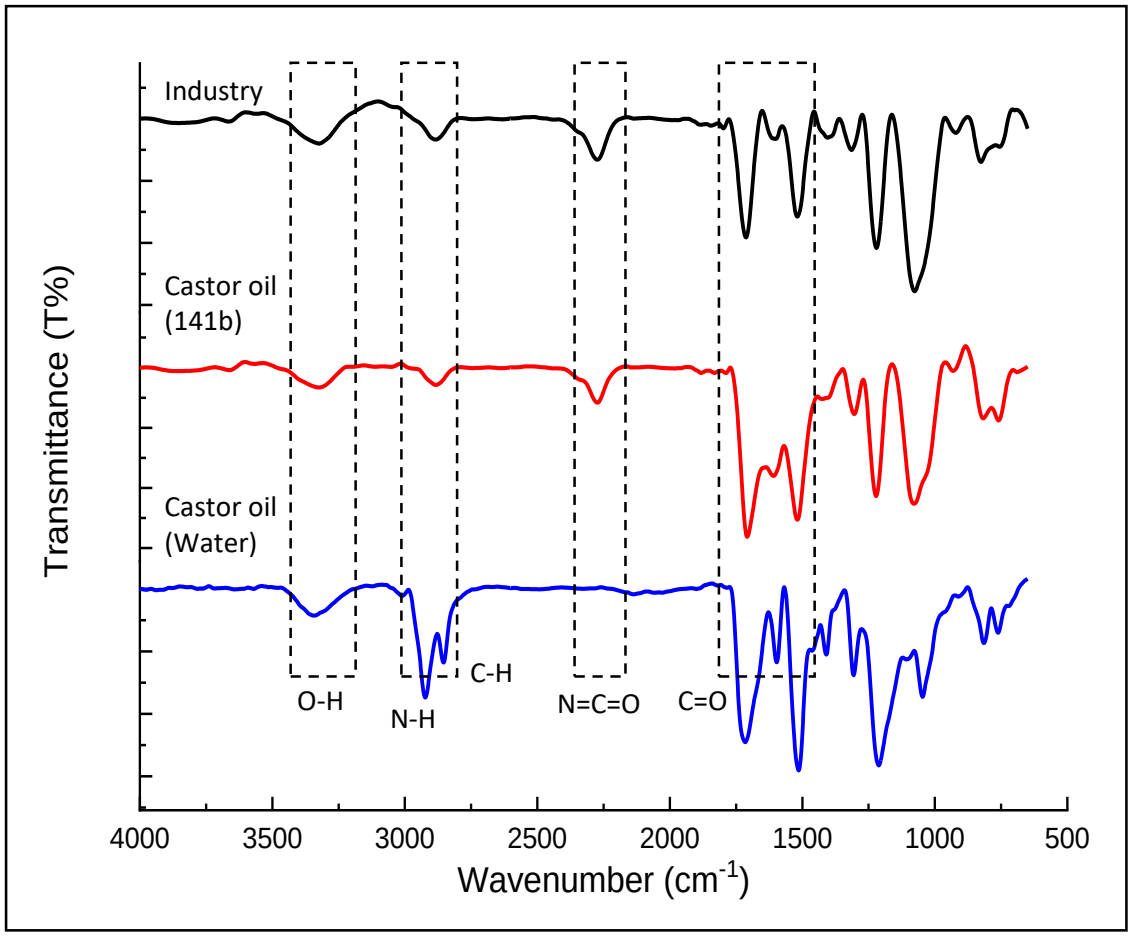


Figure 5.5 Comparison of FTIR analysis between polyurethane industry, castor oil-HCFC 141b and castor oil-water

Table 5.1
FTIR Spectra obtained for the 3 type of Polyurethane (Industry, Castor oil HCFC 141b and Castor oil Water) and possible Functional Group

Reference Wavenumber (cm^{-1})	**Industry (PI) (Wavenumber cm^{-1})**	**Castor oil HCFC 141b (PCH) (Wavenumber cm^{-1})**	**Castor Oil Water (PCW) (Wavenumber cm^{-1})**	**Functional Group (Wavenumber cm^{-1})**
3200–3600	3322	3326	3344	O-H
3100-3500	3124	3166	3145	N-H
2800–3000	2874	2888	2854	C-H
2270–2300	2272	2278	2288	N=C=O
1725	1710	1702	1716	C=O

5.5 Compression Analysis

Figure 5.6 provides a comparative analysis of the compression strength of polyurethane foam when produced using two different blowing agents: water and HCFC 141b. The data reveal a substantial disparity in the resulting foam's mechanical properties across blowing agents.

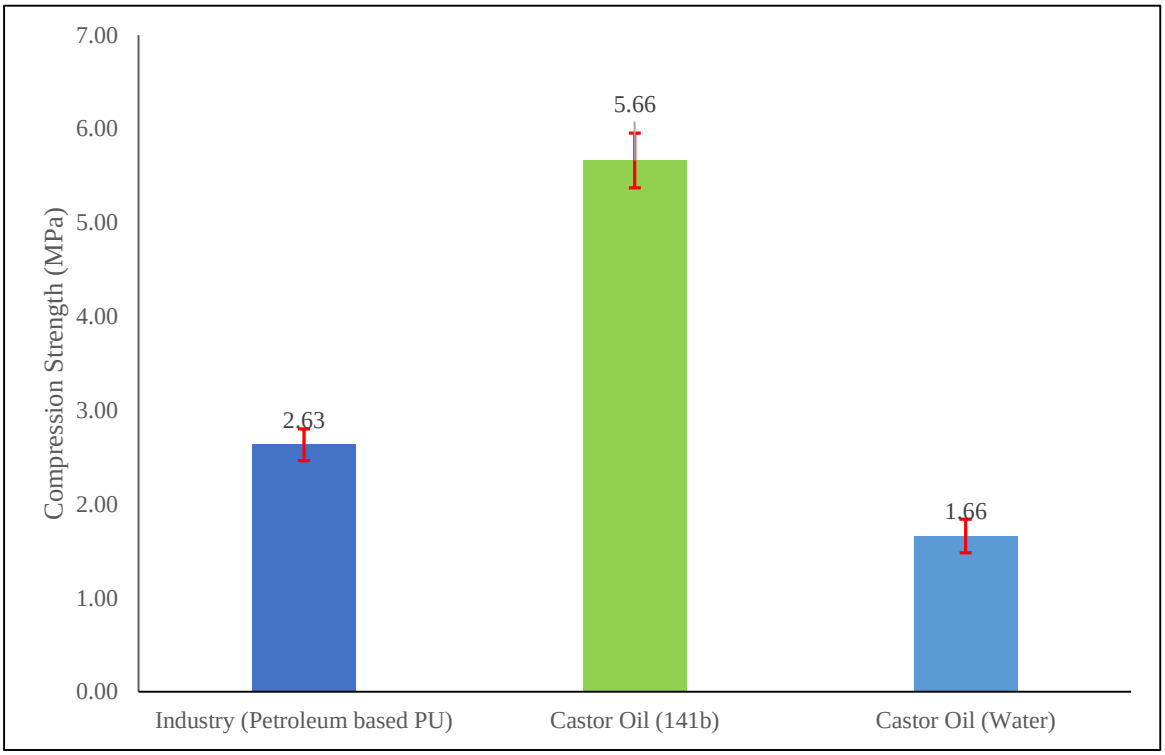


Figure 5.6 Compression strength of all types of polyurethane

The polyurethane foam produced using HCFC 141b as the blowing agent demonstrates a significantly higher compression strength of 5.62 MPa, within the range of industrial standards. This marked difference indicates that HCFC 141b promotes the formation of a denser, more uniform cellular matrix within the foam. The chemical characteristics of HCFC 141b likely led to a greater number of closed cells and fewer defects, thereby enhancing the foam's mechanical robustness. The threefold increase in compression strength with HCFC 141b suggests that it is a superior blowing agent for applications requiring high-strength polyurethane foams (Lin et al., 2023).

Overall, the graphs underscore the critical impact of the blowing agent choice on the mechanical properties of polyurethane foam. Water provides more environmentally friendly and cost-effective solutions, while HCFC 141b offers

significantly better compression performance, making it more suitable for applications requiring structural integrity and durability. Modification of the composition of the water blowing agent might improve the properties of water-based polyurethane.

Figure 5.7 compares the compression modulus of polyurethane foam using two different blowing agents: water and HCFC 141b. The compression modulus is a measure of the foam's stiffness and its ability to resist deformation under compression (Lee et al., 2023).

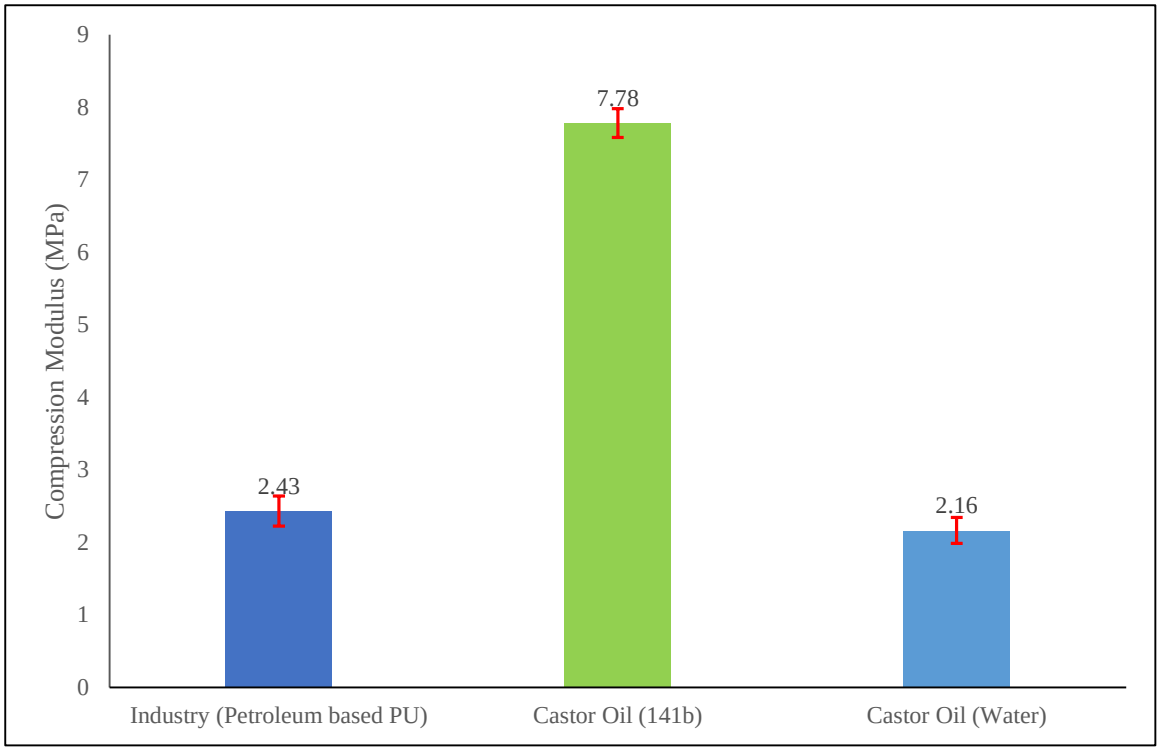


Figure 5.7 Compression modulus of all types polyurethane

The polyurethane foam produced with water exhibits a compression modulus of 2.2 MPa. This value indicates that the foam is relatively flexible and less rigid, which can be attributed to the type of cellular structure formed when water is used as the blowing agent. This type of foam may be more suitable for applications where some degree of compressibility and cushioning is desirable.

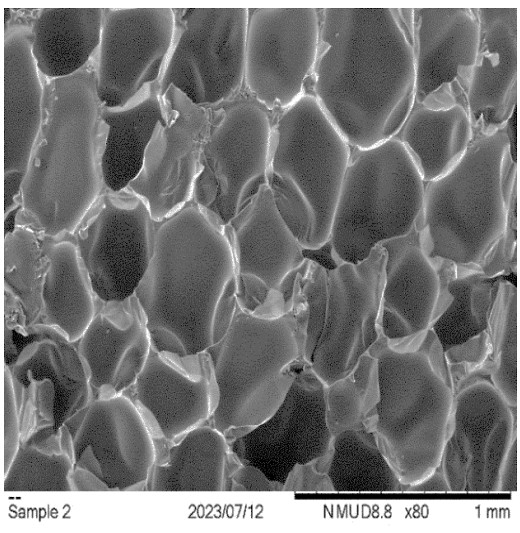
In contrast, the polyurethane foam produced with HCFC 141b as the blowing agent shows a significantly higher compression modulus of 7.8 MPa. This indicates that the foam is much stiffer and more resistant to compression. The higher modulus suggests that HCFC 141b promotes the formation of a denser, more uniform cellular structure,

with smaller, more closed cells. This results in a more rigid foam that can bear higher loads without significant deformation.

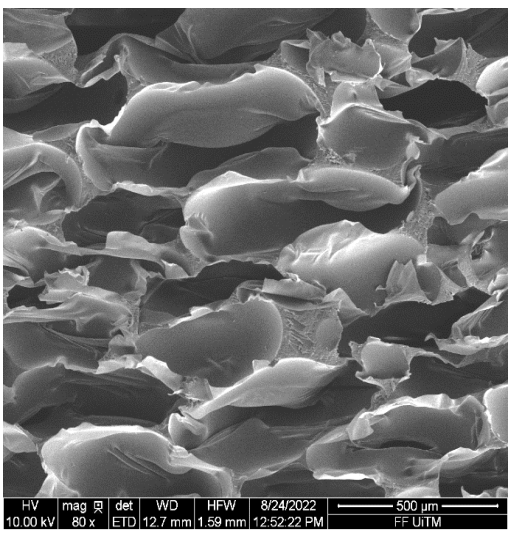
Overall, Figures 5.6 and 5.7 highlight the substantial impact of the blowing agent on the mechanical properties of polyurethane foam. While water produces a softer, more compressible foam, HCFC 141b yields a much stiffer, more robust foam. PCW is a good replacement for PI as a new green grouting material.

5.6 Morphological Analysis

The morphological properties of polyurethane, including the size and distribution of polymeric chains and cross- inking density, significantly affect their performance. Scanning electron microscopy (SEM) and other imaging techniques reveal that industrial polyurethane (PI) often exhibits a more homogeneous, consistent morphology, contributing to its high mechanical performance. Green polyurethanes, while historically more heterogeneous due to variations in bio-based feedstock, have seen improvements in recent years. Through advanced processing techniques, green polyurethanes can now achieve a morphology that rivals or surpasses that of PI, particularly in terms of cross-linking density and uniformity. The shape and size of the polyurethane foam cell are important to the mechanical properties.



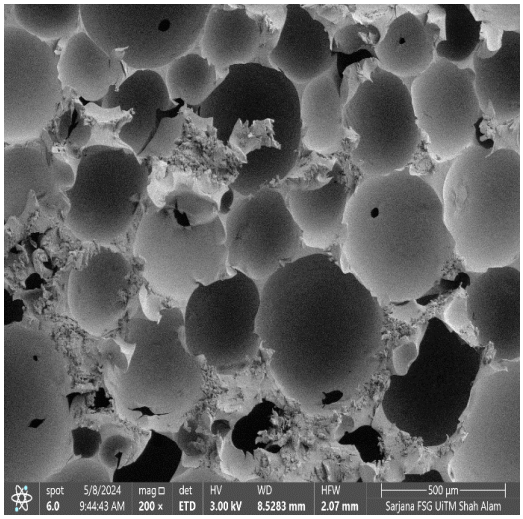
(a) PI



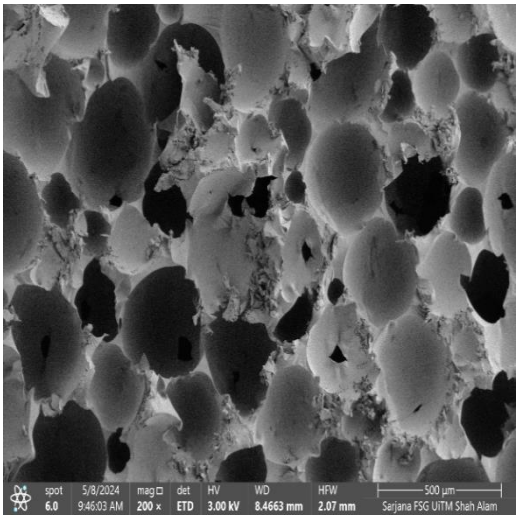
(b) PI after compress

Figure 5.8 SEM images of before and after compression of polyurethane industry

Based on the SEM analysis shown in Figure 5.8 for the polyurethane industry (PI), the similar cell size and regular cell arrangement even after compression demonstrate excellent polyurethane mechanical and morphological properties. There is no broken cell wall in the cell arrangement. The same cell size and linear cell arrangement represent the rigidity of the sample, which is composed of more than 90% of closed cells. The morphology of the industry-based polyurethane foam, if the foam shows a stable cell structure and is smaller in size (uniform cell), will give better strength and modulus than foam with irregular, larger cell size.



(a) PCH



(b) PCH after compress

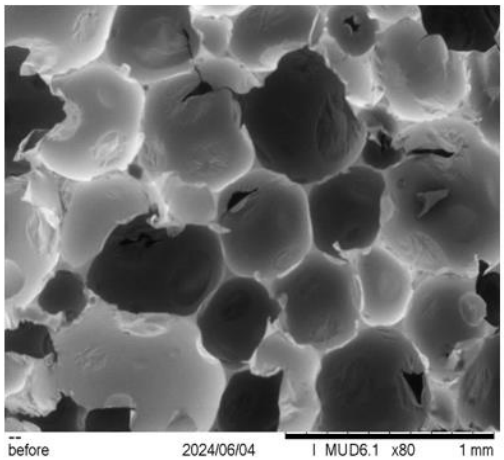
When a polyol and an isocyanate react, the mixture's temperature increases due to an exothermic reaction. A physical blowing agent, such as HCFC 141b, vaporizes, utilizing the reaction heat. Figure 5.9 shows that castor oil 141b has an irregular arrangement and cell size due to the vaporization of HCFH 141b using the reaction heat. This is happening during the rise time, when a bubble has already formed: a new bubble nucleates, which makes it take a longer time to foam, as shown in Figure 5.2. But after conducted the compression testing, the cell wall did rupture but not enough changing the shape of the cell. This may be due to the denser properties as shown in Figure 5.3.

Figure 5.9 SEM images of castor oil based polyurethane with HCFC 141b (PCH) (a) before and (b) after compression

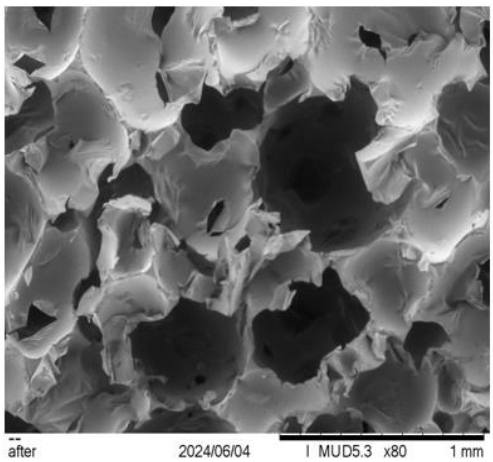
The images provided depict the microstructure of polyurethane foam produced using water as the blowing agent, observed both before and after compression. Figure 5.10 reveals the foam's initial microstructure. In this state, before compression testing was conducted, the cell was well-formed and uniformly distributed, with relatively smooth walls. The interconnected cells suggest that the foam can effectively absorb energy and compress under load, making it particularly suitable for applications that require shock absorption and insulation (Izarra et al., 2021).

Figure 5.10 displays the foam's microstructure after compression. There are noticeable changes in cell structure, with many cells appearing deformed. The walls of these cells show signs of crumpling and breakage, indicating that the foam has undergone significant plastic deformation. This transformation highlights the foam's ability to absorb energy through structural deformation, a characteristic essential for materials designed to protect against impact. The crumpled cell walls suggest that the foam has effectively distributed the compressive forces, preventing localized failures and enhancing overall durability (Li et al., 2019).

Comparing the images, it is evident that compression has a profound impact on the foam's microstructure. The initially well-defined and uniform cell structure becomes more irregular and damaged after compression. The deformation process involves the compression and collapse of cells, which dissipates energy and reduces the force transmitted through the material. However, the extent of cell wall breakage also implies that while the foam can absorb impact efficiently, repeated compressive stresses could lead to a gradual loss of structural integrity.



(a) PCW



(b) PCW after compress

Figure 5.10 SEM images of castor oil based polyurethane with water (PCW) (a) before and (b) after compression

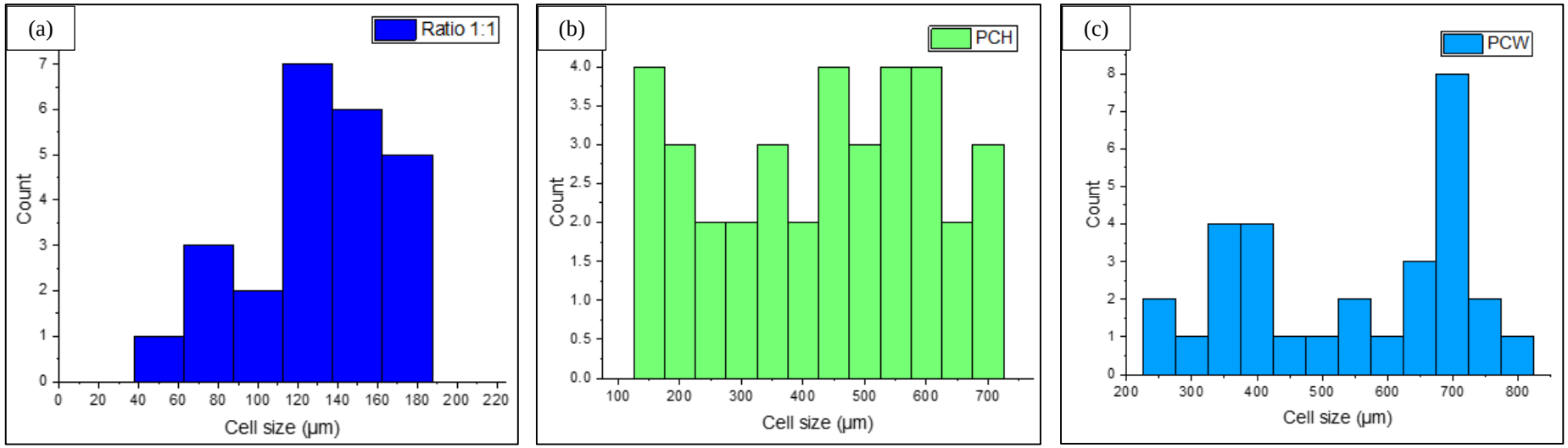


Figure 5.11 The cell count of cell foam size of (a) Polyurethane Industry (PI), (b) Castor oil - HCFC 141b (PCH) and (c) Castor oil - Water (PCW)

Figure 5.11 shows the difference of the cell count of the foam size between PI, PCH and PCW. It shows the cell size for the ratio 1:1 ranges from 40 to 190 µm. A lower cell size indicates a greater number of cells per unit volume. This means the foam contains more closed cell foam and struts to withstand huge loads, resulting in a high compression strength (Blazejczyk, 2018). Pure polyurethane foam with a stable cell structure and smaller size (uniform cell) has a higher strength and modulus than foam with irregular and larger cell sizes (Soberi et al, 2017). But on PCH, shows the average cell size of the foam is between 125 to725 µm. It can be defined as the size within the rigid foam structure with typical micro cellular structure and uniformly distributed cells that can provide superior compressive strength (Soloveva et al., 2022). This can prove by the compression analysis on Figure 5.6 shows the compression modulus of PCH have higher value compare to PI and PCW. Also the range of the cell size represent

a well closed cell structure which is critical as the HCFC 141b is one of powerful blowing agent that can remains trapped inside the closed cell (Kim et al., 2024). Next, on PCW shows the average of cell size foam is between 250 to 800 μm. It can be defined as the cell foam has gas bubbles trapped within the solid polymer matrix. The range is typically large, indication semi-rigid structure which gives out lower compression strength but have good elasticity properties (Kurańska et al., 2020). With the use of castor oil as polyol and water as blowing agent, often results in an increase in cell size. The high viscosity of the castor oil can make the cell structure heterogeneous (non-uniform). By decreasing the water content can helps reduced the cell size, but too little water can make the polyurethane foam become brittle (Lee et al., 2021).

However, based on the overall analysis the compression strength and modulus of PI and PCW exhibits approximately same value. But based on the microscopic image of PI and PCW, PI cannot withstand the buckling due to have thinner cell wall. PCW has larger foam cell size resulting thicker and stronger cell wall that can withstand greater stress before buckling (Liu et al., 2020). Thus, PCW can become an ideal material for grouting applications needling to support heavy load without deforming (Li et al., 2025)

5.7 Environmental Stress Cracking Resistance (ESCR)

5.7.1 Soil Burial Testing

Environmental Stress Cracking Resistance (ESCR) is an essential characteristic for evaluating the durability and efficacy of polyurethane materials, especially under soil burial settings. This investigation contrasts conventional polyurethane (PI) with green polyurethane, namely castor oil polyol utilizing water as a blowing agent (PCW). Both polyurethane varieties were exposed to soil for 10, 20, 30, 60, and 90 days, as shown in Figure 5.12. The results demonstrate distinct variations in stress cracking behaviour, offering insights into the materials' long-term performance.

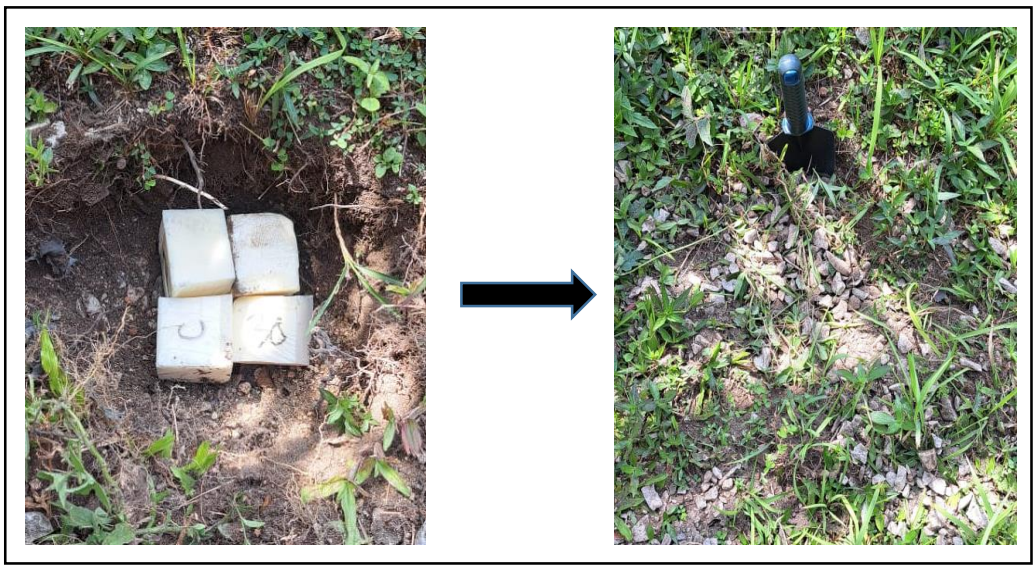


Figure 5.12 Soil burial testing of polyurethane

Based at Figure 5.13, at the 10 day interval, Castor oil-water (PCW) exhibits markedly more value of the weight gain percentage (31.82%) than PI (11.20%). The heightened values indicate that PCW's chemical composition, originating from castor oil polyol and water, demonstrates increased vulnerability to premature environmental stress cracking. The early fragility may be ascribed to the molecular configuration of castor oil based polyurethanes, which tend to exhibit lower stability than petrochemical based formulations commonly employed in PI (Smith et al., 2021). After 20 days, both materials exhibit a decline in stress cracking values, with PCW reducing to 18.17% and PI to 15.36%. Although both materials stabilize, the findings demonstrate that PI has a greater resistance to weight gain of the soil burial. This trend illustrates the intrinsic

structural benefits of PIs that guarantee superior performance in adverse environmental circumstances (Zhang et al., 2020).

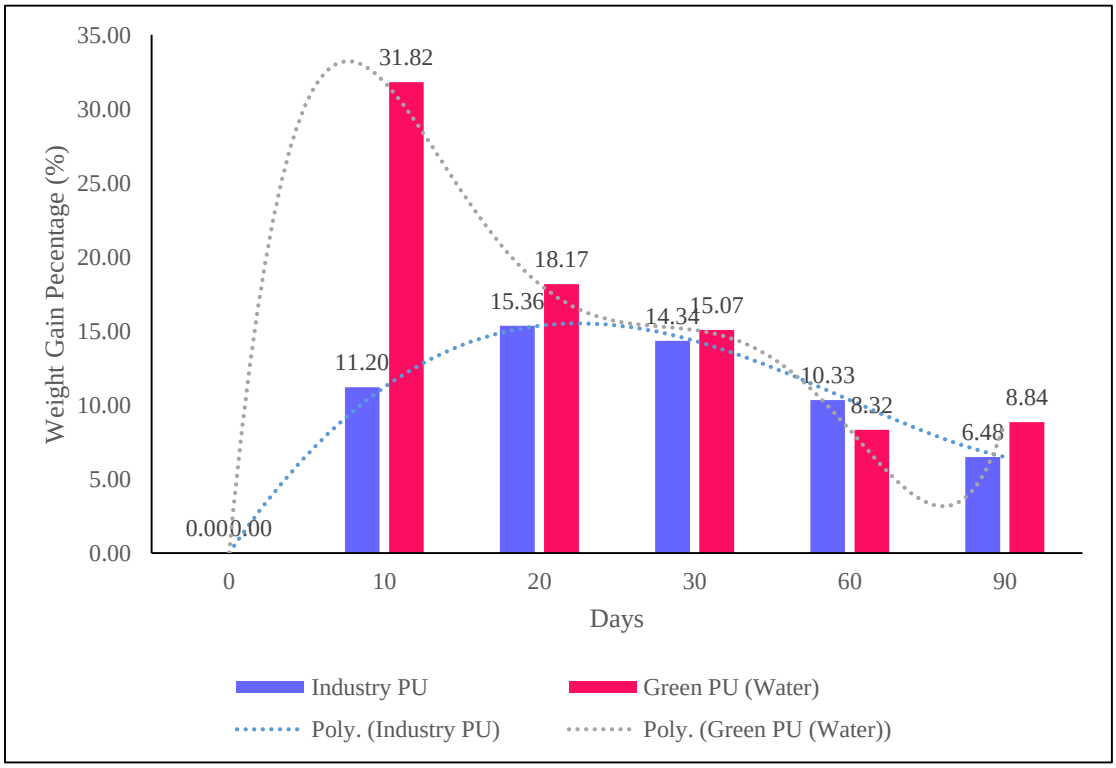


Figure 5.13 Weight gain percentage when PI and PCW buried in the soil

After 30 days, the disparity between the two materials diminishes significantly, with values of 15.07% for PCW and 14.45% for PI. This convergence indicates that both materials undergo stabilization, possibly due to chemical hardening or reduced external stresses over time. Nonetheless, PCW continues to demonstrate somewhat elevated weight gain, underscoring its continued susceptibility relative to the conventional alternative. At 60 days, the trend of diminishing stress cracking persists, with PI retaining a 10.33% superiority over PCW's 8.32%. This phase emphasizes the resilience of PI, which maintains superior resistance to degradation throughout time. Ultimately, at 90 days, PI surpasses PCW, with values of 6.48% and 8.84% of the weight gain, respectively. This outcome highlights the enduring advantage of PI in resistance to environmental stress cracking, whereas PCW stabilizes at an elevated cracking threshold.

The comparative investigation indicates that castor oil–water (PCW), although environmentally sustainable, exhibits greater vulnerability to weight gain, especially during the early phases of soil exposure. Over time, both materials show a decreasing percentage of weight gain; nonetheless, PI consistently exhibits higher long-term durability. The findings indicate that although castor oil-based polyurethanes show promise as environmentally friendly alternatives, further formulation enhancements, such as improved molecular cross-linking or the integration of reinforcing agents, are necessary to optimize their performance (Rahman et al., 2019).

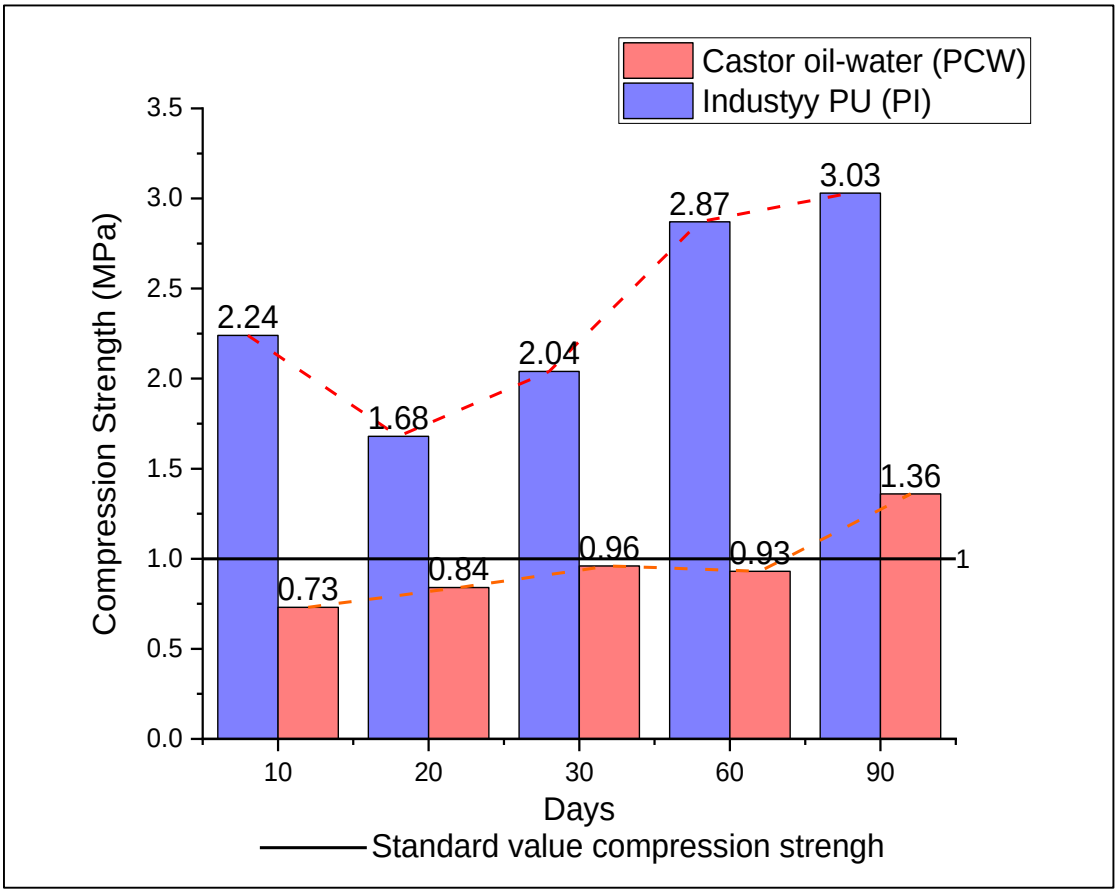


Figure 5.14 Compression Strength of PI and PCW after soil burial testing

Figure 5.14 illustrates the compression strength of two polyurethane samples: polyurethane industry (PI) and castor oil–water (PCW), assessed at 10, 20, 30, 60, and 90 days. Analysis of weight gain and environmental stress cracking resistance (ESCR) during soil burial reveals notable performance differences between the two materials, indicating their durability and structural stability under these conditions.

At the 10-day mark, PI exhibits a significantly greater compression strength of 2.24 MPa, in contrast to PCW, which measures 0.73 MPa. This initial difference

underscores the structural advantages of PI, which exhibits enhanced mechanical integrity under external forces. The reduced strength of PCW is attributed to its castor oil-based composition and water-blown cellular structure, which may lead to defects or weaker bonds within its polymer matrix (Rahman et al., 2019).

At 20 and 30 days, both materials show progressive increases in compressive strength. At 30 days, the PI increases to 2.04 MPa, whereas the PCW shows a slight improvement to 0.96 MPa. The observed increase in strength may be attributed to matrix densification processes occurring as the buried material interacts with environmental factors, notably moisture absorption. The disparity between the two materials is significant, suggesting that PI maintains a more resilient structure under environmental conditions.

At 60 days, the compressive strength of both samples continues to increase, with PI reaching 2.87 MPa and PCW rising to 0.93 MPa. At this stage, the weight gain observed in earlier tests, attributed to moisture absorption, likely contributes to the slight increase in PCW's strength. Moisture absorption may serve as a temporary filler in the cellular structure, improving load distribution while potentially compromising long term structural stability (Zhang et al., 2020).

At 90 days, the trend continues, with PI reaching a peak of 3.03 MPa, whereas PCW continues to increase to 1.36 MPa. This notable distinction highlights the enhanced mechanical performance of PI, which is particularly resilient to prolonged exposure to environmental stressors. In contrast, the limited performance of PCW indicates that moisture absorption and potential degradation compromise its mechanical properties over time. Figure 5.15 shows the condition of the soil burial sample after 90 days. The soil only covers the outer surface of the foam and the soil did not visible in the inside layer of the foam.

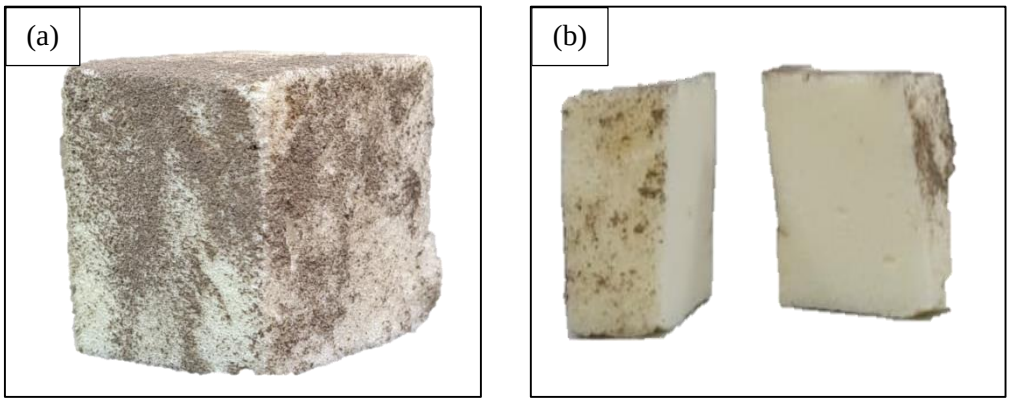


Figure 5.15 Polyurethane sample after 90 days of soil burial(a) condition of outer foam (b) cross section condition of polyurethane foam

The PI exhibits increased resistance to moisture absorption and environmental degradation, as indicated by its enhanced compression strength and reduced stress cracking behaviour. The dense, well-cross-linked structure enables the maintenance of mechanical integrity and resistance to moisture-induced weight gain. The analysis of compression strength indicates that PI consistently surpasses PCW across all time intervals, demonstrating superior structural stability and resilience to environmental stressors. PCW exhibits marginal improvements over time due to moisture absorption; however, its overall efficacy is limited by its susceptibility to environmental stress cracking and weight gain. This connection underscores the necessity for additional formulation enhancements in castor oil-based polyurethanes to improve their mechanical properties and long-term durability under environmentally challenging conditions.

Figure 5.16 presents the compression modulus of polyurethane industry (PI) and castor oil-water (PCW) across intervals of 10, 20, 30, 60, and 90 days. The compression modulus quantifies a material's stiffness, reflecting its capacity to withstand compressive deformation. The analysis, when combined with weight gain data from Figure 5.13, provides insights into the structural stability and environmental performance of these materials.

PCW demonstrates a greater compression modulus of 0.94 MPa in contrast to PI, which has a modulus of 0.73 MPa, after 10 days of soil burial. This difference highlights the superior stiffness and structural integrity of PI, resulting from its dense and cross-linked polymer matrix derived from petrochemicals. PCW exhibits greater stiffness, likely attributable to the inclusion of castor oil-based polyols and water as blowing agents, resulting in a more flexible, less dense cellular structure.

Over a period of 20 days, both materials exhibit a minor increase in modulus, with PI measuring 0.84 MPa and PCW showing a slight increase to 1.1 MPa. The slight increase in PCW’s modulus is due to moisture absorption during soil burial, which temporarily enhances the material's stiffness by filling voids within its structure (Smith et al., 2021). The modulus of PI increases to 0.96 MPa after 30 days of immersion, whereas the modulus of PCW rises to 1.50 MPa. This trend indicates that PI exhibits structural stabilization, preserving its stiffness under environmental exposure. The ongoing increase in modulus for PCW may be associated with moisture absorption, as indicated by previous weight-gain analyses. This improvement does not completely close the performance gap between the two materials. The PI exhibits a downward trend,

reaching 20.93 MPa, while PCW improves to 1.32 MPa over a 60 day immersion period. At 90 days, both PI and PCW reached their maximum peak, 1.36 MPa and 1.58 MPa, respectively. The continuous rise in PI and PCW modulus indicates enhanced resistance to environmental degradation, preserving stiffness even under extended exposure.

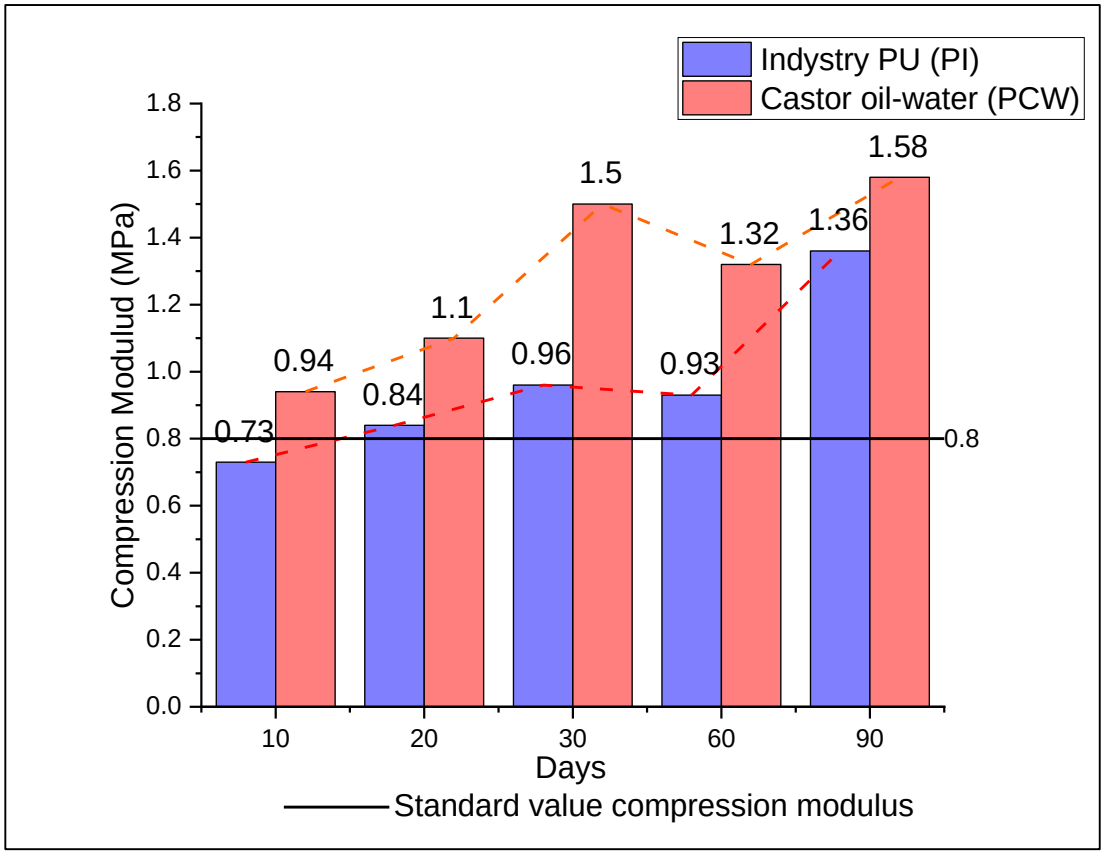


Figure 5.16 Compression Modulus of PI and PCW after soil burial testing

The weight gain analysis shown in Figure 5.13 indicated that PCW exhibited increased moisture absorption over time due to its hydrophilic properties. The absorption of moisture led to a temporary stiffening of the material by occupying voids in its cellular structure, as indicated by the gradual rise in its compression modulus. Excessive moisture absorption can result in structural degradation and diminished long term stability, thereby constraining its modulus enhancement relative to PI. The interplay of compression modulus, weight gain, and stress cracking indicates that PI is more appropriate for prolonged environmental exposure, attributed to its durable molecular structure and reduced vulnerability to moisture-related degradation.

The analysis of compression modulus demonstrates that PI exhibits superior performance, consistently surpassing PCW regarding stiffness and structural stability

over time. The correlation between weight gain and modulus trends indicates that although PCW experiences short-term advantages from moisture absorption, its long-term efficacy is hindered by structural degradation. The findings indicate that enhancements to the PCW formulation, including increased crosslink density and the addition of moisture-resistant additives, are necessary for it to serve as a viable alternative to traditional PI under environmentally challenging conditions.

The SEM imaging in Figure 5.17 reveals morphological changes that provide significant insight into the degradation behaviour of industrial polyurethane (PI) and castor oil–water (PCW) foams subjected to soil burial over time. On day 30, the foam exhibited a uniformly preserved cellular structure, with few signs of tearing or wall disruption on PI, suggesting limited environmental interaction. Nonetheless, the presence of cell ruptures in the foam matrix and early indications of surface roughening suggest initial moisture absorption infiltration. This is associated with a minor rise in weight gain percentage, suggesting the initiation of fluid absorption within the polymer network (Panaitescu et al., 2017). The compression strength and modulus remained high at this stage, exhibiting only slight reductions compared to the control sample.

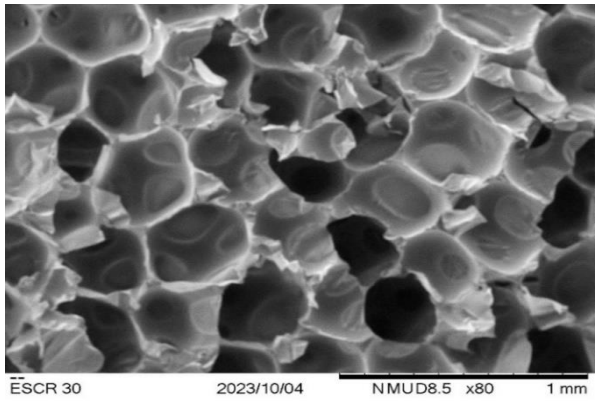
On Day 60, the SEM image demonstrates notable morphological distortion, characterized by a few wrinkled cell walls, localized collapse, and pore deformation on PI. The observed changes indicate progressive hydrolysis or microbial activity, correlating with increased weight gain attributed to enhanced water permeability and soil diffusion into the foam. The partial collapse of cellular structures disrupts the load distribution mechanism in the foam, resulting in a slight reduction in compressive strength and modulus. This degradation trend is consistent with earlier research indicating that soil moisture and microbial enzymes can enhance polyurethane degradation through chain scission and soft-segment disruption (Guo et al., 2021; Poussard et al., 2020).

Based on Figure 5.17, 90 days of foam exposure shows considerable structural degradation, characterized by observable fragmentation of pore walls, pore collapse, and roughened or eroded surfaces on PI and PCW. The observed features suggest significant degradation, likely due to prolonged microbial colonization and enzymatic hydrolysis, undermining the foam's mechanical integrity. This condition is associated with the highest percentage of weight gain, due to significant fluid absorption, and exhibits the lowest values for compression strength and modulus. The pronounced pore deformation indicates the foam's lack of recovery under load, corroborating earlier

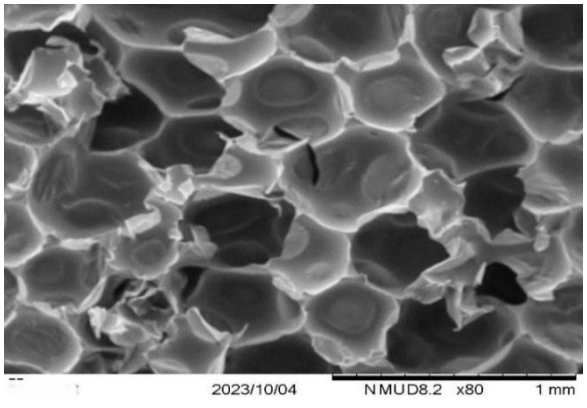
findings that prolonged soil burial markedly diminishes the structural resilience of polyurethane (PU) based materials (Yusuf et al., 2022). The observed degradation behaviour closely corresponds to recognized mechanisms of environmental aging in biodegradable and partially bio-based polyurethanes.

Based on Figure 5.18, shows the average cell size foam of (a) polyurethane industry (a) and (b) castor oil-water (PCW) after soil burial testing. The cell size of PI has the average between 225 to 525 μm. This proves PI has relatively uniform cell size. Also, the foam undergoes a complex process of biodegradation which can contribute to the physical structure failure. It is noticed in SEM image in Figure 5.17, the cell of the foam is thin and it breakdown into a few smaller fragment (Bhavsar et al., 2023). This can me soil particle act as fillers within the foam which significantly make the foam become stiffer thus has the increasing value of compression strength and the modulus based on Figure 5.15 (Chamas et al., 2020).

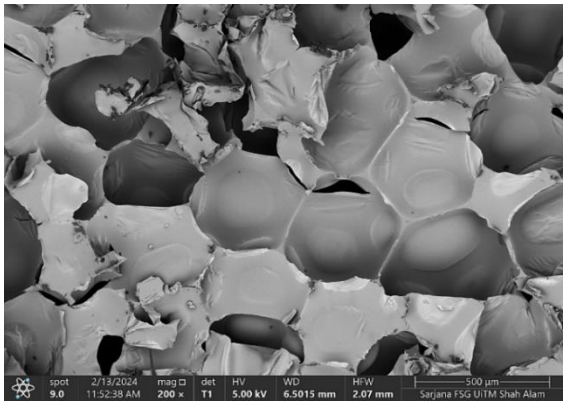
Furthermore, the cell size of PCW has average of 125 to 625 μm. This indicates the small cells remain intact but the others have coalesced (merged) as the thin walls between them become degrades as shown on Figure 5.17. As the compression strength of PCW also become increase over time, this is due to the polymer chain wall breakdown and the fine soil particle and minerals migrates into the open voids. Thus, a higher relatively percentage of hard segments which naturally make the foam become stiffer and hard leading to the increase of strength of the PCW foam (Bher et al., 2022).



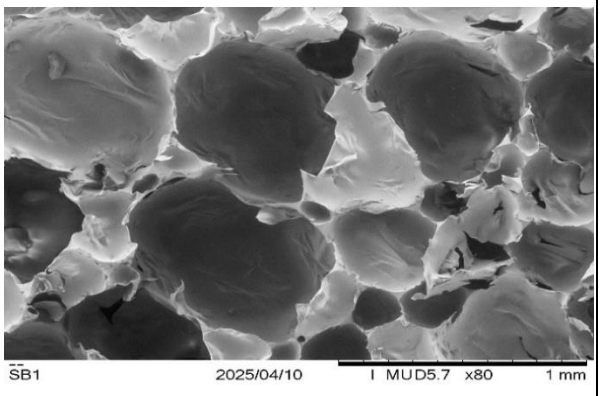
PI 30 Days



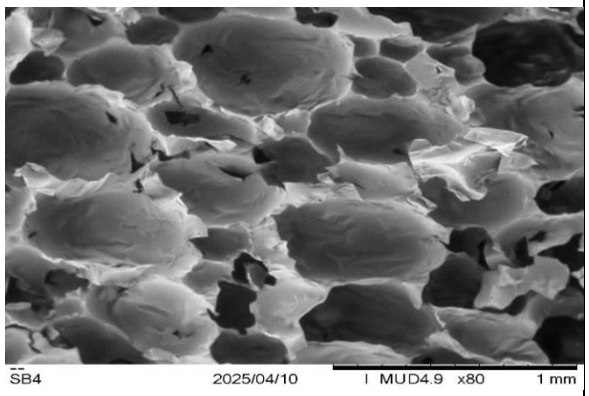
PI 60 Days



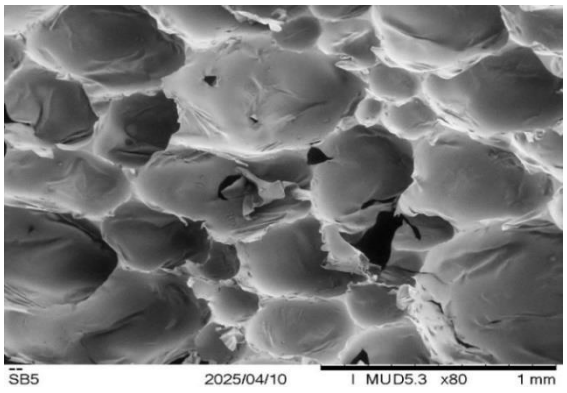
PI 90 Days



PCW 30 Days



PCW 60 Days



PCW 90 Days

Figure 5.17 SEM images on 30, 60 days and 90 days of industry polyurethane (PI) and castor oil-water (PCW) after soil burial testing

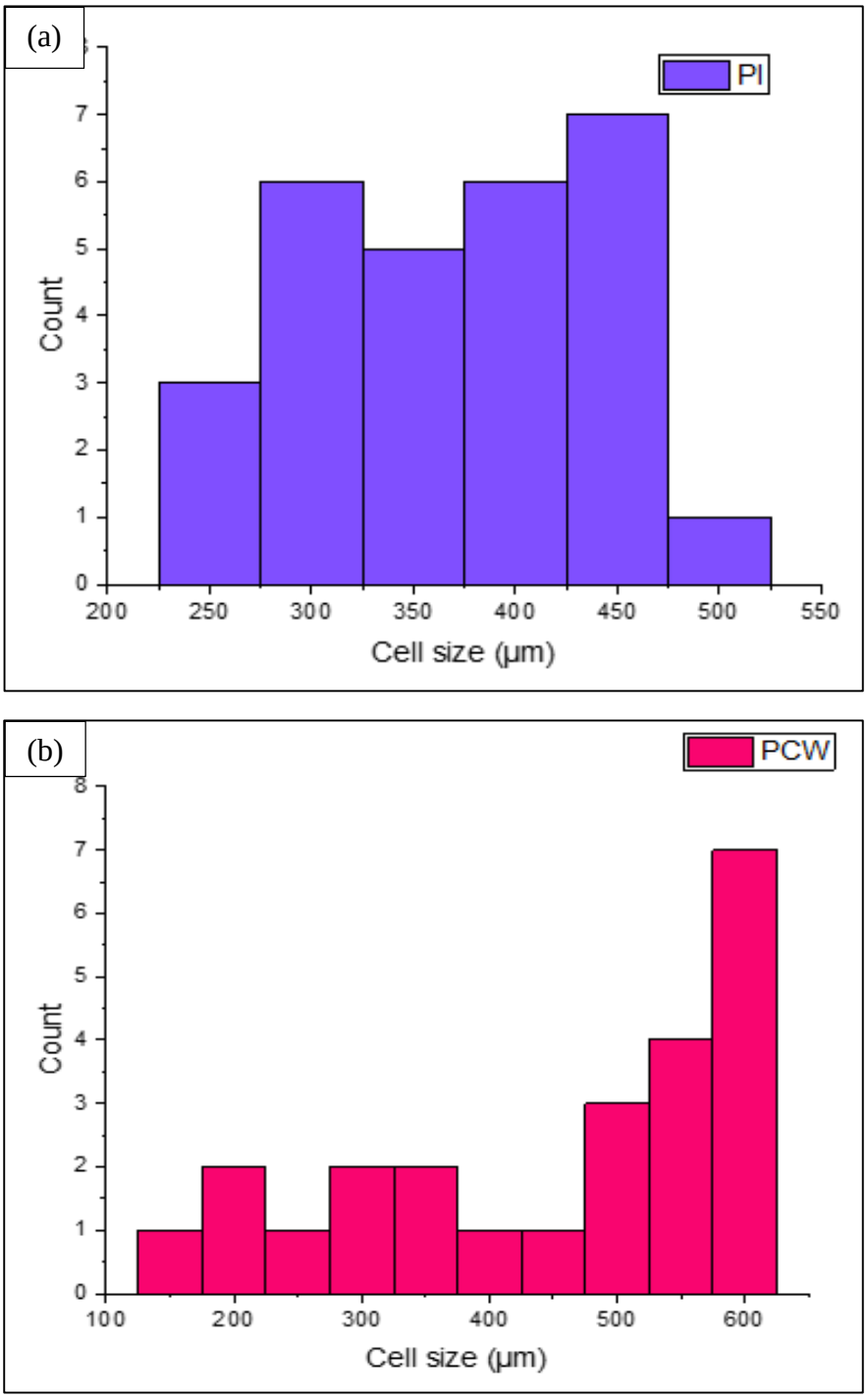


Figure 5.18 Average of the Cell size foam of (a)polyurethane industry (PI) and Castor oil–water (PCW) after soil burial

5.7.2 Solubility Test

5.7.2.1 Immersed in Hydrochloride (HCl) solution

The significant weight gain observed in both industrial polyurethane (PI) and castor oil-based polyurethane with water as a blowing agent (PCW), Figure 5.19, shows that when polyurethane samples are immersed in hydrochloric acid (HCl) solution, this can be attributed to a combination of chemical and physical interactions between the polyurethanes and the acidic environment. These interactions involve processes such as acid diffusion, polymer swelling, hydrolysis of urethane linkages, and hydrogen bonding, which collectively affect the materials' integrity and performance (Zhang et al., 2023). Hydrochloric acid molecules are small and highly mobile, enabling them to penetrate the microstructure of polyurethane materials. This diffusion leads to the absorption of acid and water molecules into the polymer matrix. As the acid penetrates, it causes polymer swelling, which increases the material's weight (Snetkov et al., 2020). This effect is more pronounced in PCW due to its higher porosity and less dense cellular structure, which allows for deeper acid penetration. The porous morphology of PCW, resulting from its castor oil-based composition and water as the blowing agent, facilitates higher absorption than that of the denser PI.

Polyurethanes are composed of urethane groups (-NH-CO-O-) that are susceptible to hydrolysis under acidic conditions. When exposed to HCl, these linkages undergo hydrolysis, yielding primary amines and alcohols. This reaction not only contributes to weight gain by increasing the material's hydrophilicity but also compromises the structural integrity of the polyurethane. The hydrolysis process is more significant in PCW due to its less cross-linked and more hydrophilic nature, which makes it more vulnerable to chemical degradation in acidic environments (Horváth et al., 2024).

While both PI and PCW exhibit weight gain in HCl in the early days and gradually decrease their weight gain percentage, the magnitude of this effect differs significantly due to their intrinsic structural properties. Industry polyurethane (PI) is typically more cross-linked and hydrophobic, which provides resistance to acid penetration (Patel et al., 2018). However, any unreacted components or structural imperfections can still allow acid and water molecules to be absorbed, leading to a moderate weight gain. Castor oil-based polyurethanes are less cross-linked and

inherently more hydrophilic, making them more susceptible to acid diffusion, swelling, and hydrolysis.

Consequently, PCW exhibits a higher degree of weight gain (25.53%) than PI (15.93%). The weight gain in both polyurethanes correlates directly with their mechanical and structural integrity. The absorbed acid and water can temporarily improve stiffness due to swelling, but also result in permanent structural degradation. For PCW, the combination of higher weight gains and reduced environmental resistance highlights its vulnerability in acidic environments. On the other hand, PI demonstrates better durability, retaining its mechanical properties over time.

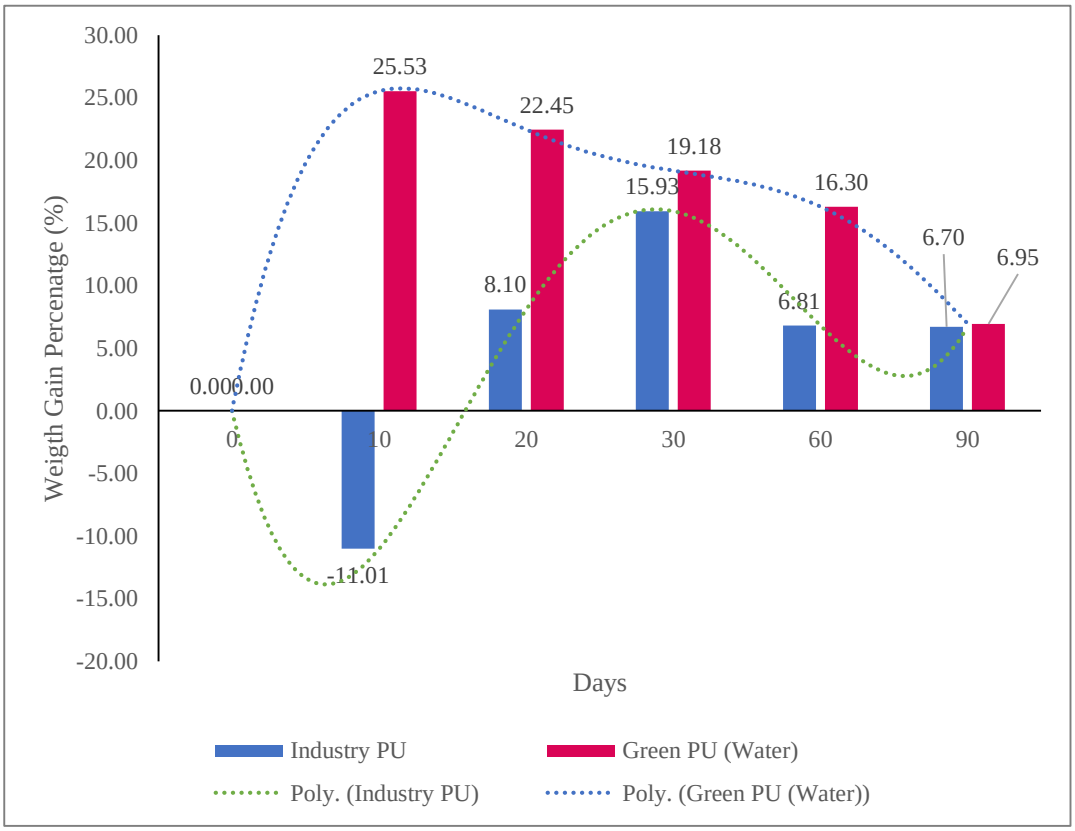


Figure 5.19 Weight gain analysis when immersed in HCl solution

Figure 5.20 illustrates the compression strength and modulus of the PI and PCW. It shows that the compression strength gradually increases until 90 days, despite the weight gain over time, as shown in Figure 5.19. It shows the compression strength of PI, slightly increasing from 2.12 MPa to 2.78 MPa, and for PCW from 2.39 MPa to 3.02 MPa. This may be due to swelling and penetration of the HCl solution, which can be attributed to several physical and chemical changes within the material. The HCl

solution can act as a catalyst, enhancing chemical interactions or secondary crosslinking between polymer chains. Increased crosslinking density can improve the rigidity of the polymer network, contributing to higher compression strength (Chen et al., 2020).

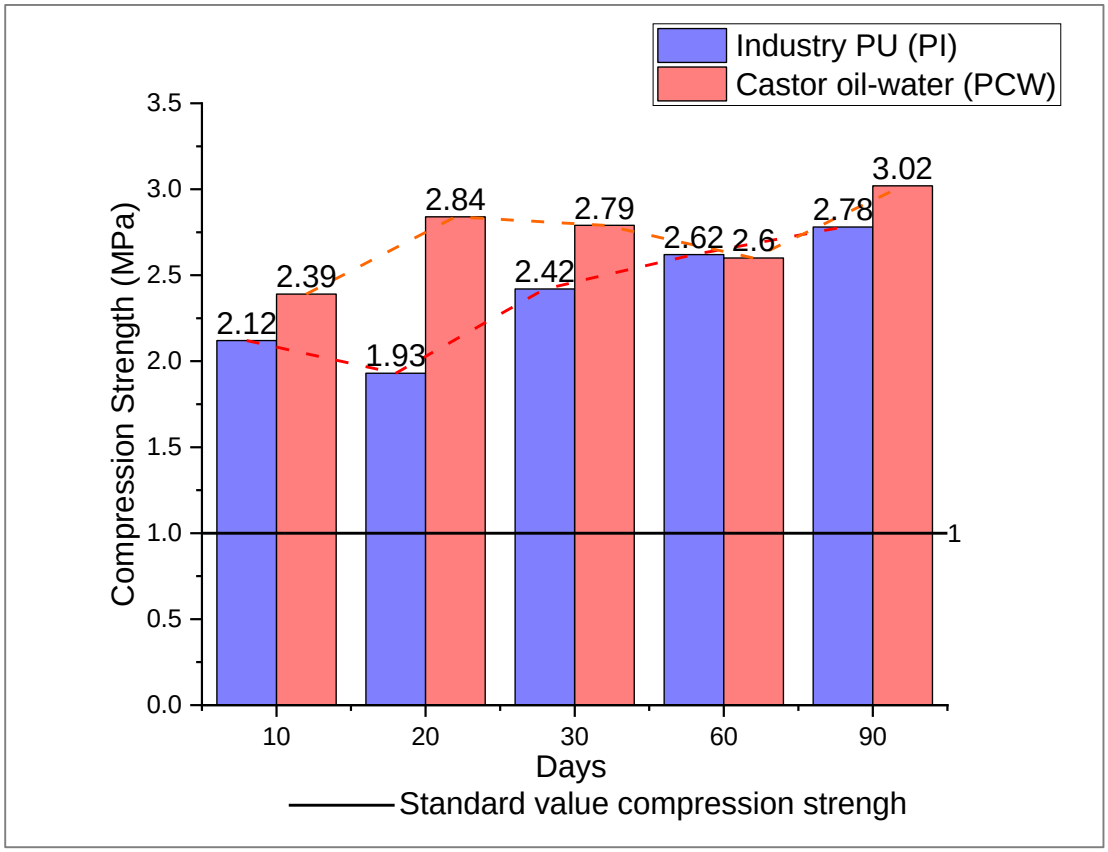


Figure 5.20 Compression strength of PI and PCW after immersed in HCl solution

The swelling caused by the solution penetration may induce uniform internal stresses within the polymer matrix. These stresses can reduce localized weak points and redistribute mechanical loads more evenly, improving the overall compression strength. (González et al., 2012). The ingress of HCl solution may initially act as a plasticizer, softening the material. However, subsequent water evaporation and ionic interactions between HCl and polymer components (e.g., urethane groups) can reinforce the material by forming ionic crosslinks or crystallites (Wu et al., 2019). The presence of HCl can promote crystallization of the hard segments in polyurethane. This crystallization improves mechanical properties, particularly under compressive loads (Zhao et al., 2016). The gradual increase in compression strength due to swelling and penetration of the HCl solution is likely the result of increased crosslinking density, stress redistribution, ionic interactions, microstructural changes, and hard segment

crystallization. These combined effects improve the mechanical stability of the polymer network.

The immersion of polyurethane foam of polyurethane industry (PI) and castor oil-water (PCW) in hydrochloric acid (HCl) resulted in a gradual increase in the compression modulus for both materials over the 90-day period, as illustrated in Figure 5.21. Specifically, PI exhibited an increase from 0.76 MPa at day 10 to 1.23 MPa at day 90, while PCW increased from 0.86 MPa to 1.27 MPa across the same duration. Despite this common trend, the weight change profiles of PI and PCW demonstrated distinct behaviours, suggesting different degradation and structural responses to the acidic medium.

For PI, a reduction in weight was observed at day 10 as shown in Figure 5.19, indicating an initial phase of degradation. This weight loss may be attributed to the leaching of low-molecular-weight oligomers or to the dissolution of unreacted components, which are particularly vulnerable to acid hydrolysis (Singh et al., 2021). Following this early stage, the polymer gradually regained stability, with its modulus increasing from 20 to 90 days. The stiffening behaviour is likely associated with acid-induced chain scission that reduces the mobility of the amorphous fraction, thereby promoting localized ordering and partial crystallinity within the polymer network. Such structural densification has been reported to enhance stiffness while simultaneously reducing ductility, a phenomenon commonly referred to as embrittlement (Lyu et al., 2022).

PCW exhibited a different trend: weight initially increased up to 30 days, then declined between 60 and 90 days. The initial increase in weight suggests acid diffusion and polymer matrix swelling, leading to water or acid uptake. This phenomenon is consistent with the behaviour of bio-based polyurethanes, which possess more polar functional groups that can absorb acidic media (Zhao et al., 2021). After prolonged exposure, the swelling phase was replaced by chain scission and polymer backbone erosion, resulting in a gradual reduction in weight. Nonetheless, as observed with PI, the compression modulus continued to rise throughout immersion, reflecting structural stiffening. This may be due to increased packing density of the residual network, enhanced hydrogen bonding, or the formation of micro domains with higher crystallinity, which reinforce the foam structure despite overall mass loss (Chen et al., 2023).

Both PI and PCW demonstrated a progressive increase in compression modulus during immersion in HCl, though weight change behaviours differed. PI experienced an initial degradation followed by stabilization, while PCW underwent swelling before degradation dominated. These findings highlight that acid exposure induces a two-stage process, which includes early swelling or dissolution, followed by chain scission and reorganization of the polymer microstructure. The net result is a stiffer but potentially more brittle material, consistent with acid-induced embrittlement mechanisms reported in recent literature.

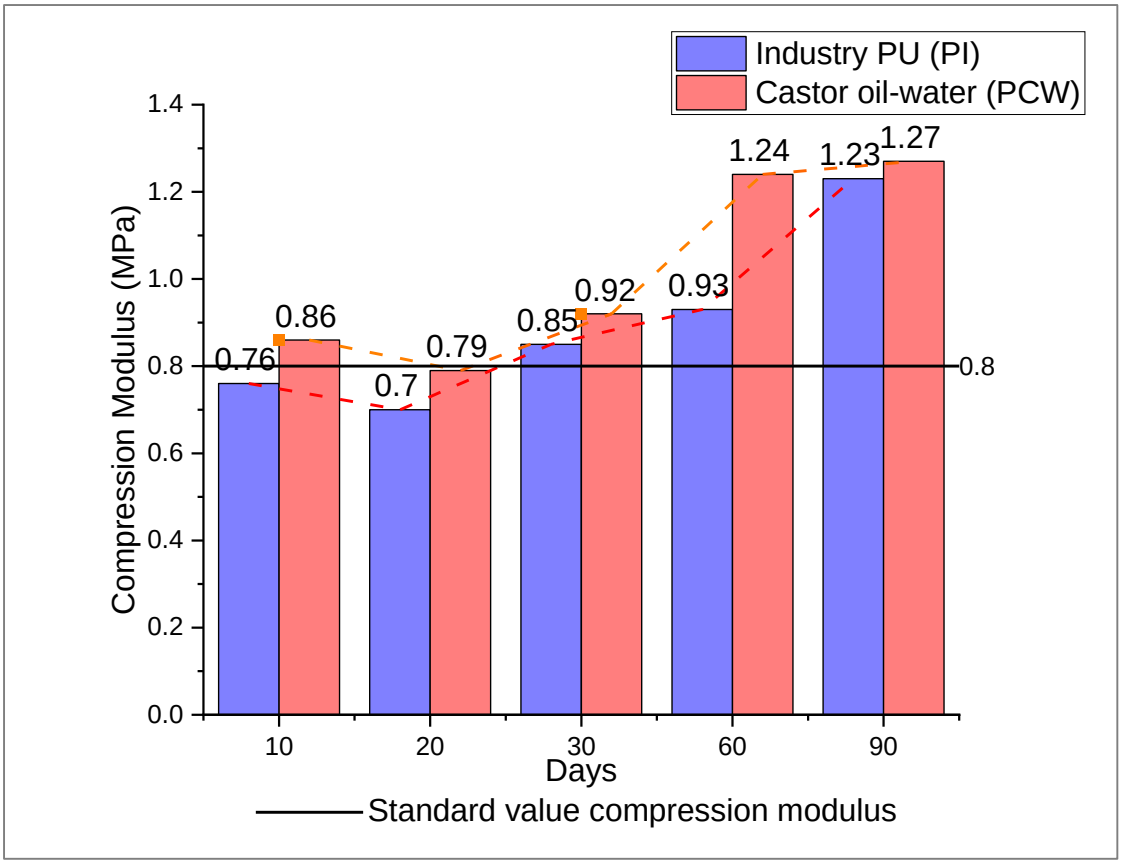


Figure 5.21 Compression modulus of PI and PCW after immersed in HCl solution

The SEM micrographs in Figure 5.23 show significant morphological evolution of the PI microstructure under prolonged HCl exposure. The initial 30-day period is characterized by the emergence of enhanced porosity and irregular voids, attributed to the acid hydrolysis of the more susceptible polyurethane linkages. These structural defects create interconnected diffusion pathways, facilitating the rapid increase in fluid absorption observed during this interval. Conversely, between 60 and 90 days, the microstructure undergoes a transition toward stabilization and localized densification.

This phenomenon suggests that degraded chains of molecular units resulting from the breakdown (degradation) of a larger molecule or polymer fragments may undergo secondary interactions or precipitation within the pore network. This structural consolidation effectively restricts further ingress of the penetrant, providing a mechanistic explanation for the plateau and subsequent decline in absorption percentage (Xu et al., 2021).

The SEM images for PCW in Figure 5.21 indicate a gradual reduction in surface roughness and porosity over time. After 30 days, the material exhibits notable surface roughness and microvoids, indicating early degradation and swelling attributed to water absorption. At 60 and 90 days, the structure exhibits increased compactness and reduced porosity, suggesting that the absorbed water has promoted hydrolytic degradation. However, the degradation products may have precipitated or occupied the voids, thereby limiting further penetration of the solution. This phenomenon corresponds with the documented reduction in absorption percentage over the period from 10 to 90 days. The polyol derived from castor oil in PCW likely enhances this process due to its hydrophilic characteristics, which increase its susceptibility to initial absorption and subsequent degradation, in contrast to the hydrophobic structure of PI (Rana et al., 2019).

The differing behaviours of PI and PCW in HCl immersion highlight their fundamental material distinctions. The rapid absorption of PI within the first 10 to 30 days is due to structural breakdown and enhanced porosity, whereas the subsequent decline indicates structural stabilization. The persistent decrease in absorption percentage in PCW is attributed to hydrolytic degradation, which fills voids with degradation products, resulting in an increasingly dense structure over time. The chemical composition and structural integrity of polyurethane materials influence their interactions with acidic environments.

Figure 5.23 shows the average of cell size foam of (a) polyurethane industry (PI) and (b) castor oi-water (PCW) after immersed in hydrochloride (HCl) solution. For (a)PI, the average cell size is between 125 to 375 μm. This indicates the polyurethane foam signifies high chemical resistance and structural ability (Mpusa et al., 2022). The significant increase in cell siew oud indicate swelling of the polymer chain as illustrated on Figure 5.22. Polyurethane foam that can maintain the original cell dimension after endure chemical resistance considered highly durable for harsh environment (Hodná et al., 2022). As for PCW as illustrated in Figure5.23(b) a cell size range of 75 to 550 μm after immersed in HCl solution for 90 days indicates the morphology stability and

chemical resistance of the foam's cellular structure under harsh acidic conditions. Castor oil based polyuretnae are often characterized by the high degree of cross linking due to multifunctional nature of castor oil (Akram et al., 2022).With the average cell starting from 75 μm represent smaller size, often more rigid that contributes to higher compression strength as shown in Figure 5.20 (Lee et al., 2021) Also, the long fatty acid chains in castor oil provide hydrophobic barrier that limits the penetration of the aqueous HCl solution into the polymer matrix as the weight gain analysis become decrease over time as shown in Figure 5.19 (Kim et al., 2025).

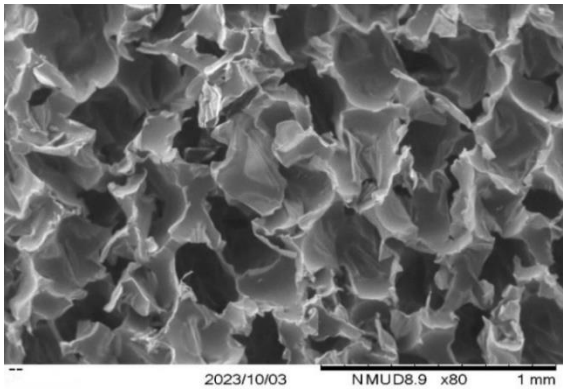
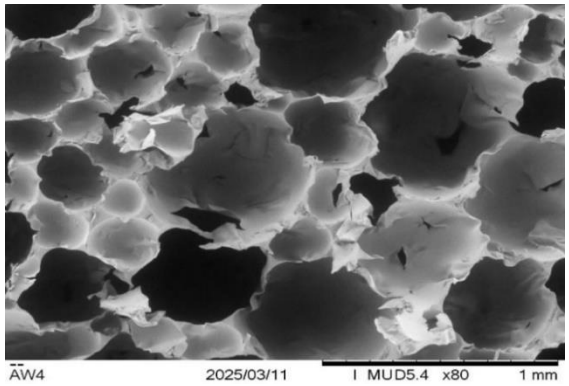
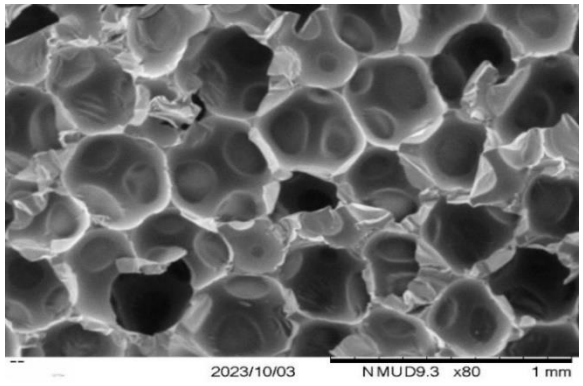
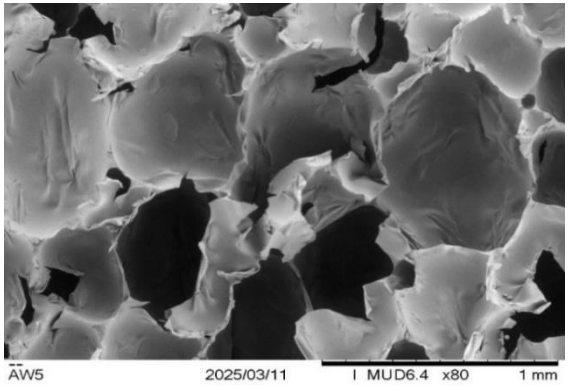
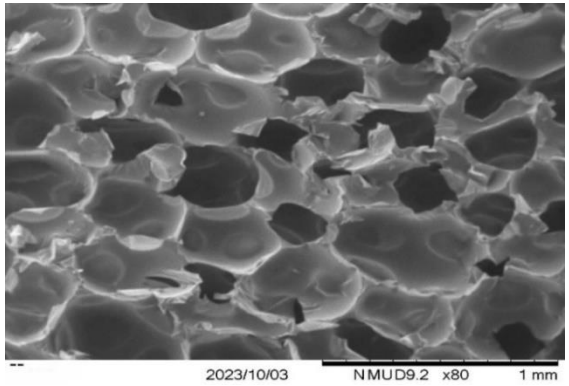
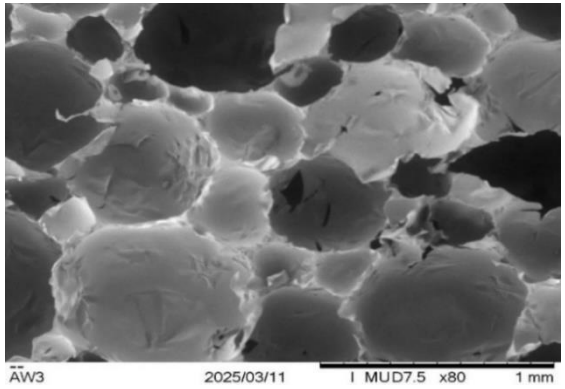
		
PI 30 Days	PI 60 Days	PI 90 Days
		
PCW 30 Days	PCW 60 Days	PCW 90 Days

Figure 5.22 SEM images of polyurethane industry (PI) and castor oil-water (PCW) after immersed in HCl solution for 30, 60 and 90 days

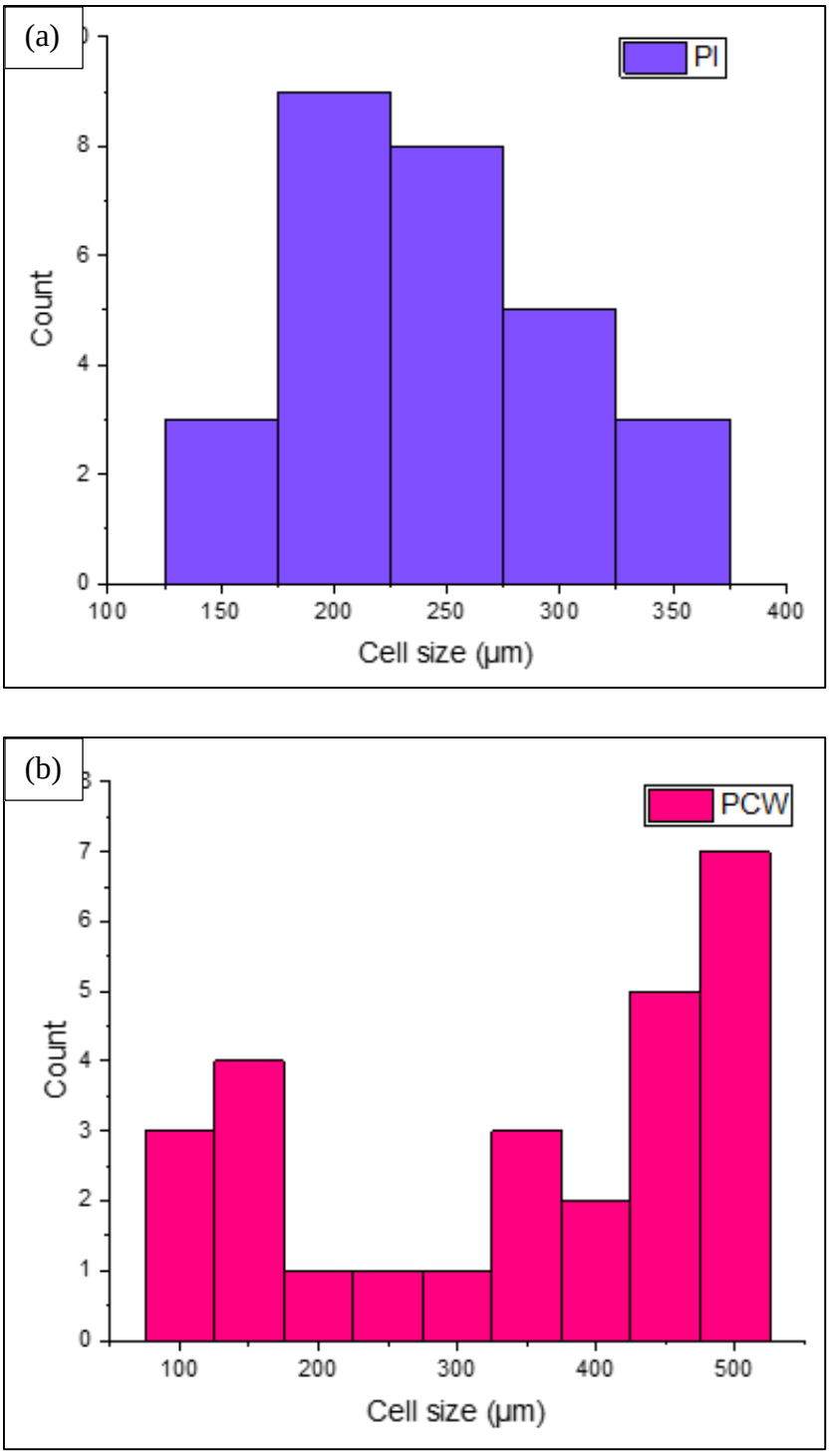


Figure 5.23 Average of Cell size foam of (a) polyurethane industry (PI) and (b) castor oil-water (PCW) after immersed in hydrochloride (HCl) solution

5.7.2.2 Immersion in Sodium Hydroxide (NaOH) solution

Figure 5.24 shows the weight gain percentage analysis, indicating that both the polyurethane industry (PI) and castor oil–water (PCW) absorb water upon immersion in a NaOH solution. The PCW exhibits a significantly higher weight gain percentage than Industry PU at all immersion times, peaking at 20 days (22.53%) and decreasing thereafter. This pattern can be attributed to the hydrophilic nature of the castor oil polyol, which enhances water absorption and, in turn, degrades the polymer network in the alkaline environment. The swelling observed in PCW likely results from water molecules infiltrating the polymer and disrupting its chains, leading to expansion and increased weight (Wang et al., 2015). Conversely, PI demonstrates lower weight gain, indicative of its relatively hydrophobic nature and greater resistance to NaOH-induced hydrolysis.

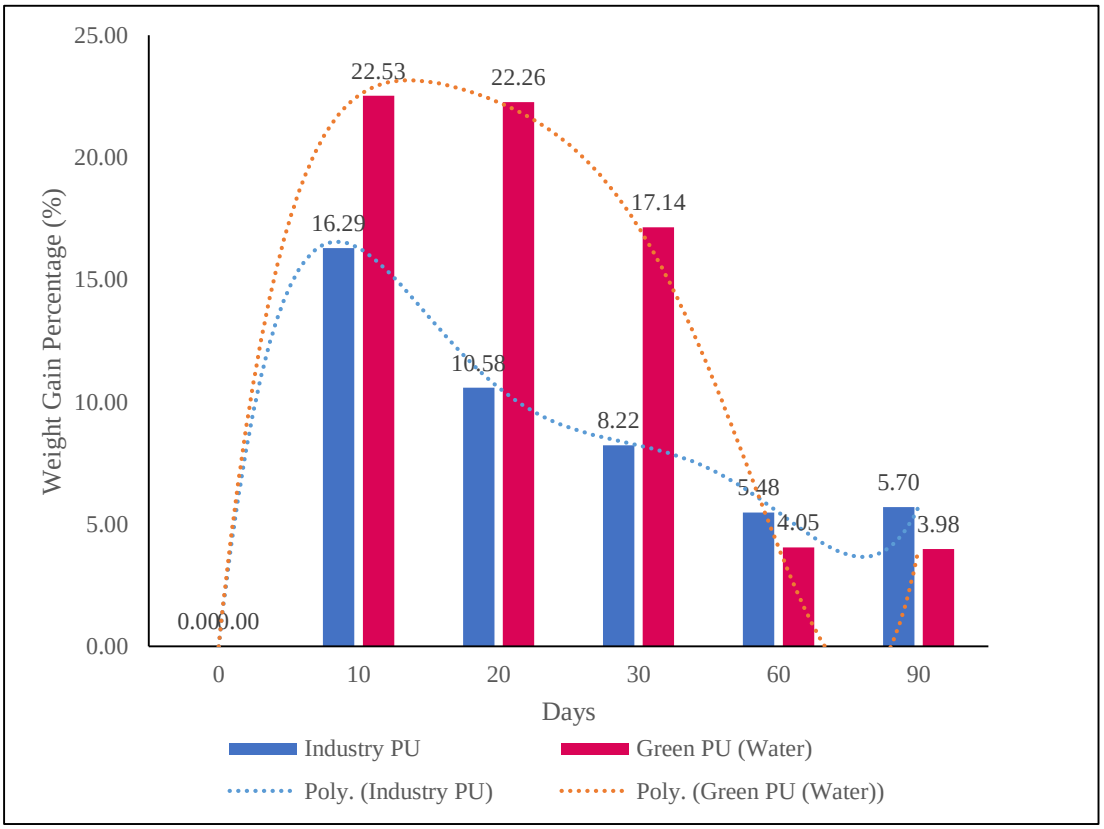


Figure 5.24 Weight gain percentage of PI and PCW immersed in NaOH solution

The comparison of compression strength between PI and PCW in Figure 5.25 shows a consistent increase over time, from 1.77 MPa at 10 days to 2.48 MPa at 90 days for PI and 1.99 MPa to 2.65 MPa for 10 to 90 days for PCW. This trend suggests a hardening effect, possibly due to secondary reactions between the polyurethane matrix and NaOH that enhance cross-linking or reduce internal defects. On the other hand, PI exhibits much lower compression strength, which correlates with its water absorption and subsequent degradation, as shown in Figure 5.24. The infiltration of water molecules disrupts the polymer structure, thereby diminishing load-bearing capacity (Rana et al., 2019).

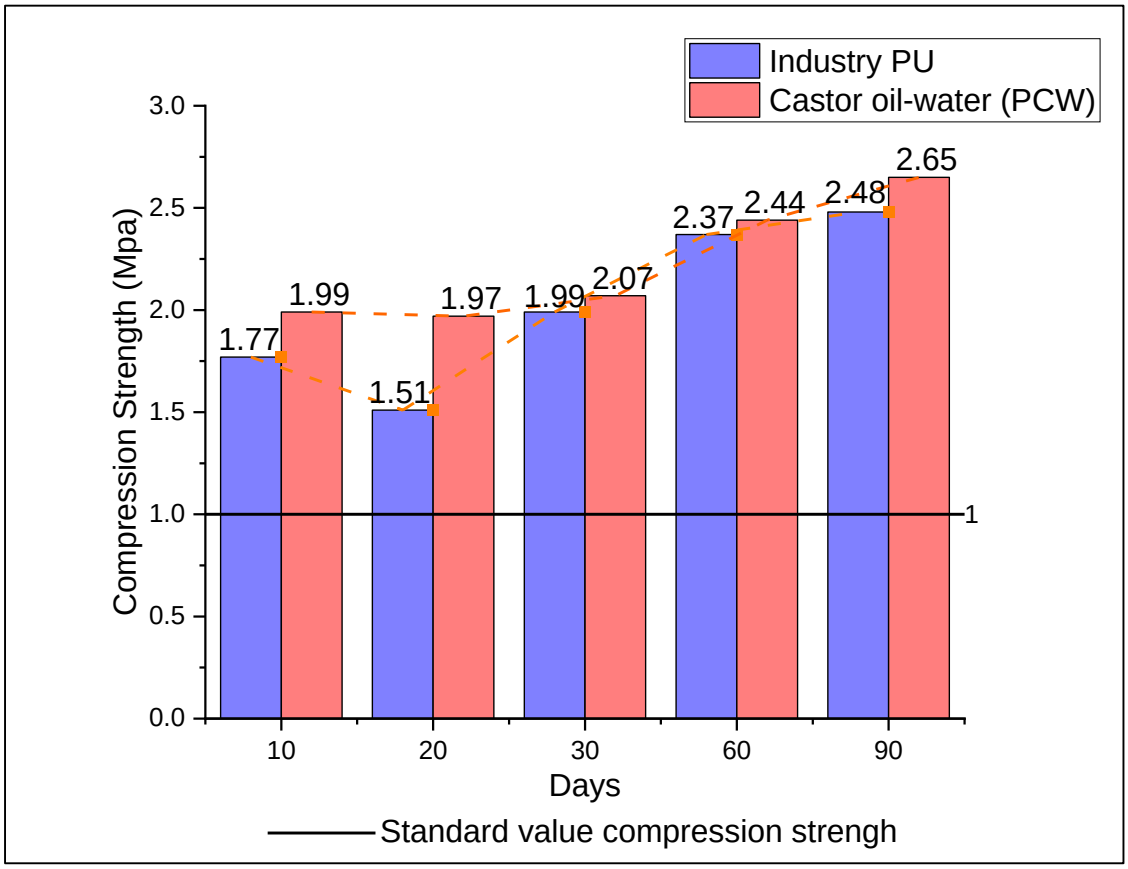


Figure 5.25 Comparison of compression strength between PI and PCW after immersed in NaOH solution

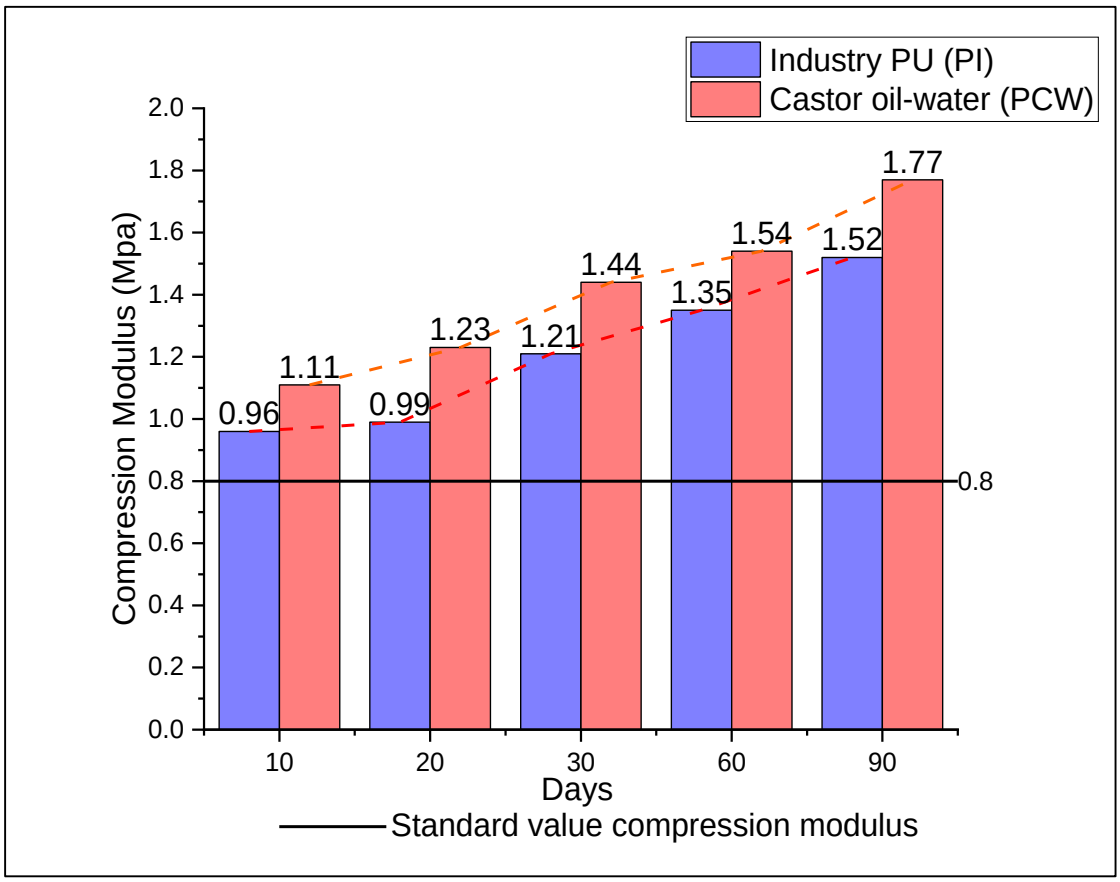


Figure 5.26 Comparison of compression modulus between PI and PCW after immersed in NaOH solution

Figure 5.26 shows the compression modulus of PI and PCW as a function of immersion time in a sodium hydroxide (NaOH) solution over a 10 to 90-day period. It also shows that the compression modulus follows a similar trend to compression strength. Based on the results, both PI and PCW exhibited a progressive increase in compression modulus as PI rose from approximately 0.96 MPa to 1.52 MPa, and PCW from about 1.11 MPa to 1.77 MPa. Concurrently, both materials experienced substantial weight loss: PI decreased from around 16.25% to 5.70%, and PCW from about 22.53% to 3.98% relative to their initial mass at day 10. These contrasting trends indicate mechanical stiffening despite mass reduction, suggesting complex underlying degradation mechanisms.

The weight loss is primarily due to alkaline hydrolytic degradation of ester or urethane linkages in the polymer networks. Hydroxide ions act as strong nucleophiles, attacking vulnerable bonds, leading to chain scission and leaching of small molecular fragments—thus causing mass loss over time. The observed increase in compression

modulus despite mass reduction can be attributed to several concurrent structural changes. As the amorphous regions of the polymer degrade preferentially, the remaining material becomes enriched in more crystalline or rigid domains, effectively increasing stiffness through densification or reorganization of polymer chains (Haryńska et al., 2023).

The immersion of PI and PCW in NaOH induces a two-phase degradation process. In the first phase, alkaline hydrolysis causes progressive material loss, especially from amorphous, weaker regions. As these degrade, the residual polymer network becomes more densely packed, as shown by the SEM analysis in Figure 5.25, with relative increases in crystallinity or inter-chain interactions that bolster mechanical stiffness. Consequently, even as total mass declines, the residual material exhibits a higher compression modulus. This behaviour highlights the susceptibility of green polyurethane (castor oil polyol) to environmental stress, as water-induced swelling and chain degradation reduce its rigidity (Xu et al., 2021).

The trends in compression strength and modulus corroborate this observation. The observed increase in compression strength and modulus even after 30 days indicates structural stabilization. The stabilization may arise from the interaction of NaOH with degraded polymer fragments, resulting in densification or the reformation of cross-links within the material (Rana et al., 2019). The correlation among weight gain, compression strength, and modulus highlights the essential distinctions in material properties between PI and PCW. The increased weight gain noted in PCW correlates with its diminished mechanical properties, as water absorption promotes hydrolytic degradation and undermines the integrity of the polymer network.

The reduced weight gain in PI is associated with its enhanced mechanical performance, attributed to its hydrophobic structure that effectively resists water infiltration and degradation. The findings highlight PI's enhanced resistance to environmental stress cracking, indicating its suitability for harsh alkaline environments. Conversely, although PCW exhibits significant environmental benefits, its mechanical shortcomings in alkaline environments underscore the necessity for additional modifications or reinforcements to enhance its durability.

The SEM images in Figure 5.27 of PI illustrate changes in surface morphology over time in the NaOH solution. After 30 days, the microstructure shows enhanced porosity and surface irregularities, likely due to hydrolysis and degradation of weaker urethane bonds induced by NaOH. The observed degradation corresponds with a weight

gain of 16.29% at 10 days, decreasing to 10.58% by 20 days, attributed to the porous structure facilitating greater solution penetration (Xu et al., 2021). At 60 and 90 days, the structure exhibits stabilization, accompanied by decreased porosity, corresponding to weight gain percentages of 5.48% and 5.70%, respectively. This indicates a partial restoration of the material's integrity or saturation of the polymer matrix.

Based on Figure 5.28 (a) shows the average cell size of the PI is between 73 to 335 μm which proven the cell has chemical degradation and cell wall breakdown. The average range of the cell size also suggest a transition from a fine, uniform cell structure to heterogeneous as the cell wall dissolved from the adjacent smaller cells merged into larger, irregular voids, shifting the average cell size upward (Du et al., 2022).

The SEM images for PCW in Figure 5.27 indicate considerable structural degradation over time. After 30 days, the surface exhibits roughness with significant voids, reflecting initial swelling and hydrolytic degradation attributed to the NaOH solution. This is associated with a rapid weight gain of 22.53% at 10 days, followed by a decrease to 17.14% by 30 days, as the material starts to disintegrate. At 60 and 90 days, the structure exhibits increased compactness, with smaller voids presumably occupied by degradation products, which accounts for the ongoing decrease in weight gain, 3.98% at 90 days.

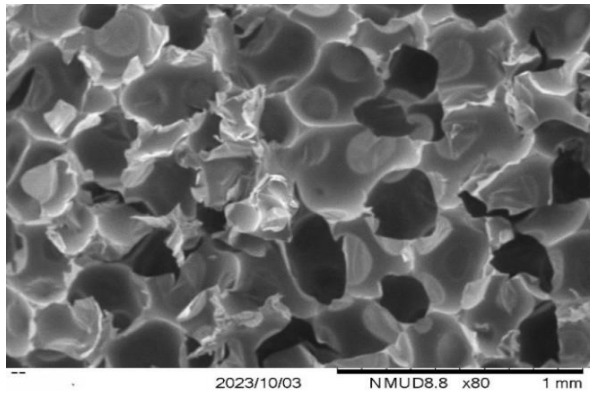
Bases on Figure 5.28(b) shows the average cell size of PCW is between 325 to 725 μm after immersed in NaOH solution. This average range indicates significant morphological degradations and structural expansion due to alkaline hydrolysis as shown in Figure 5.24. The increase of the cell size reflects the rupture of cell wall which NaOH facilitates the breaking of urethane and ester bonds within the polymer matrix (Gotkiewicz et al., 2024).

The PCW trends in compression strength and modulus further corroborate this behaviour. The lower compression strength and modulus values at 1.99 MPa and 1.11 MPa, respectively, after 90 days, compared to PI, indicate reduced structural integrity of PCW, attributed to the hydrophilic nature of castor oil-based polyurethane, which is more susceptible to degradation under alkaline conditions. The compact yet weak structure observed in later stages indicates the accumulation of degradation by products rather than structural recovery (Liu et al., 2018).

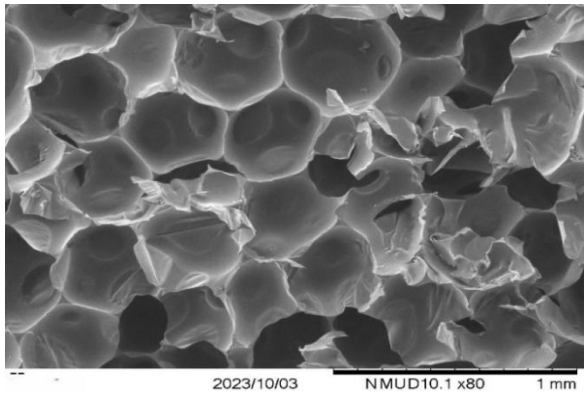
The differing performances of PI and PCW in NaOH solution highlight the impact of their unique chemical properties. PI exhibits an initial degradation phase, followed by structural stabilization, whereas PCW undergoes continuous hydrolytic

degradation with minimal stabilization. The disparities are apparent in weight gain, compression strength, modulus trends, and SEM observations.

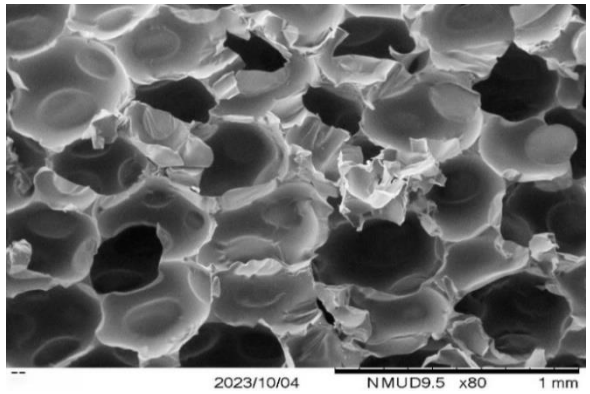
The higher resistance of PI to NaOH degradation is due to its denser, cross-linked structure, which restricts hydrolysis and enables partial recovery in subsequent stages. The hydrophilic characteristics of PCW, arising from castor oil-based polyols, facilitate water absorption and hydrolytic degradation, thereby diminishing mechanical properties and increasing vulnerability to alkaline conditions (Lee et al., 2023).



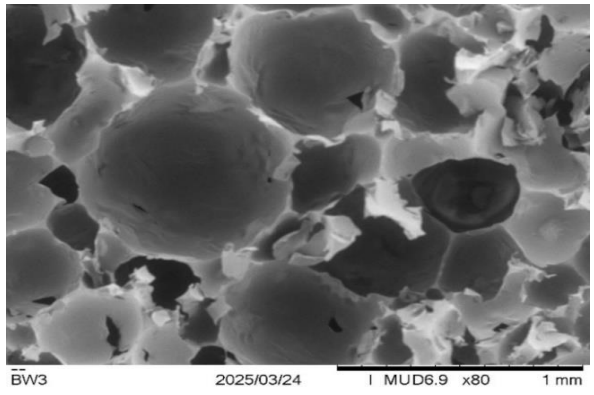
PI 30 Days



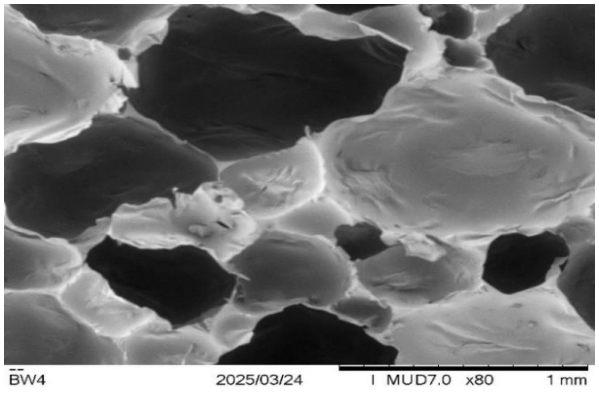
PI 60 Days



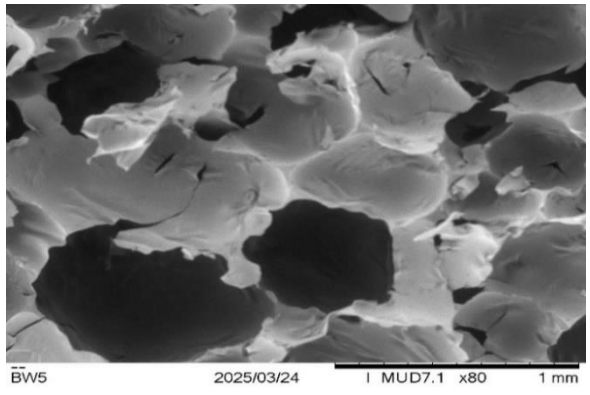
PI 90 Days



PCW 30 Days



PCW 60 Days



PCW 90 Days

Figure 5.27 SEM images of polyurethane industry (PI)and castor oil-water (PCW) after immersed in NaOH solution for 30, 60 and 90 days

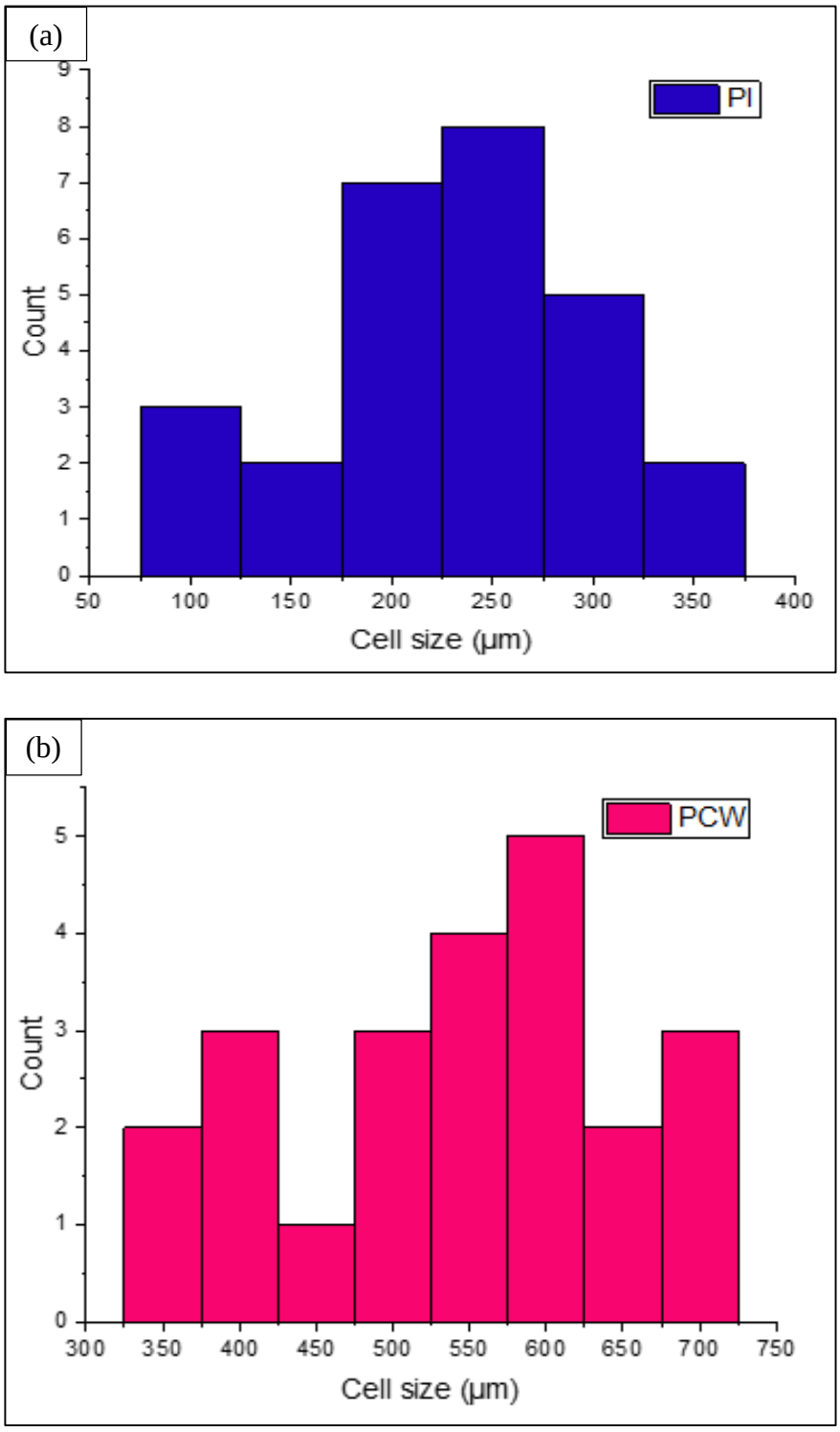
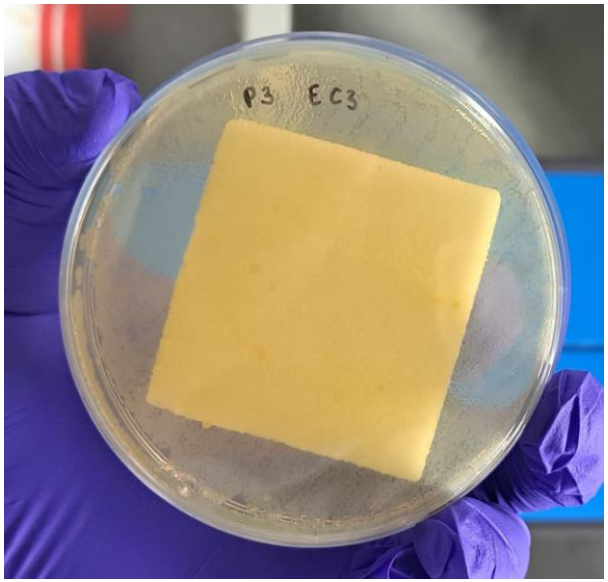


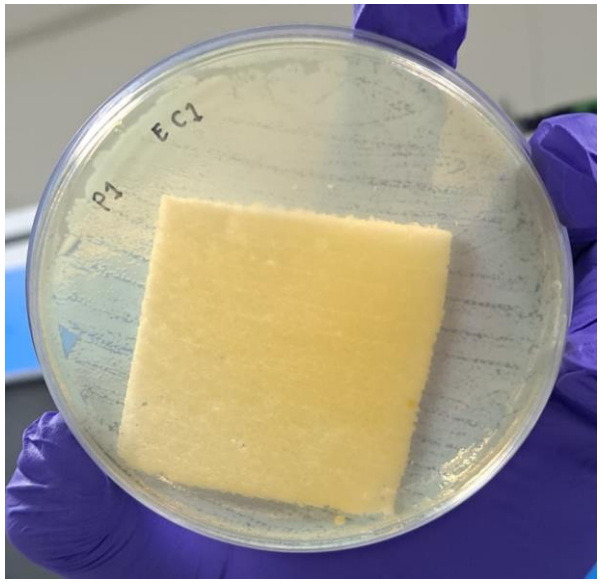
Figure 5.28 Average of Cell size of (a) polyurethane industry (PI) and (b) castor oil-water (PCW) after immersed in NaOH solution

5.8 Antibacterial Testing

The Figures 5.29 and 5.30 show the antimicrobial testing for the industry (PI) and castor polyol polyurethane water (PCW) as blowing agents. It is conducted by placing the foam on top of agar that has already been streaked with bacteria and observing the conditions after 10 days. The bacteria used were *E. coli and B. subtilis*, both of which can degrade polyurethane (Maheswaran et al., 2024).



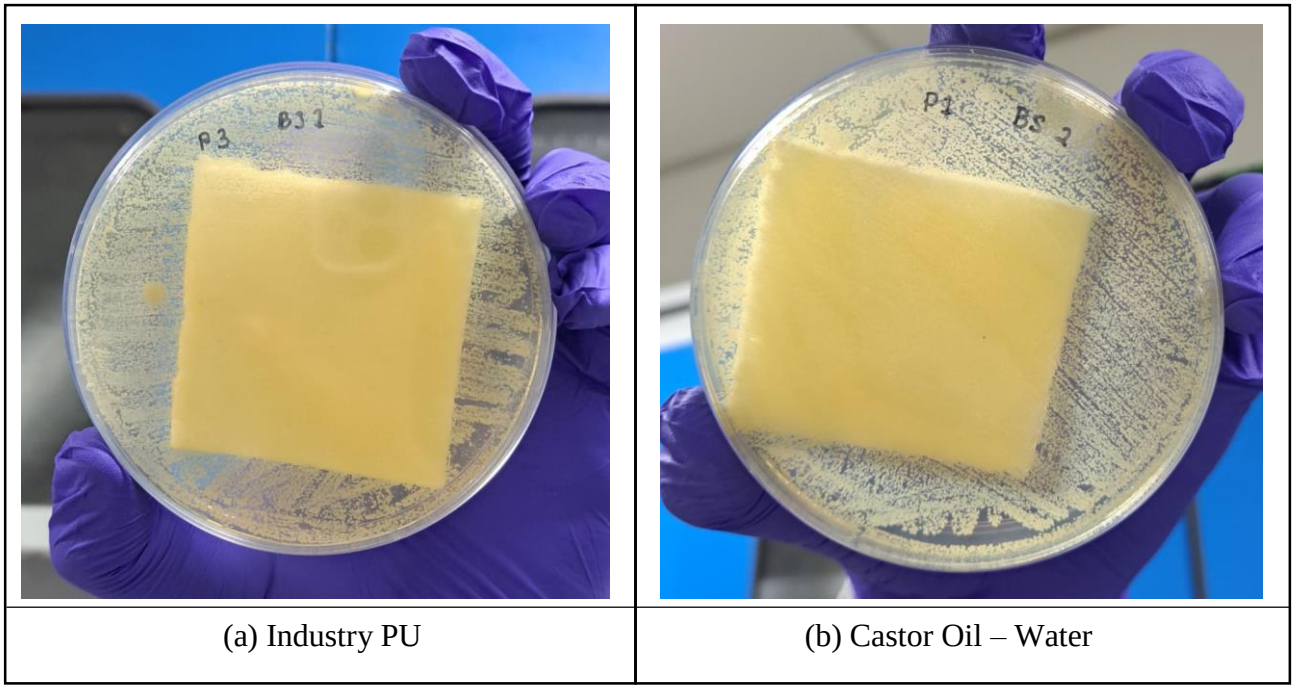
(a) Industry PU (PI)



(b) Castor Oil – Water (PCW)

Figure 5.29 Antimicrobial Testing on (a) PI and (b) PCW using *E.coli*

Based on Figures 5.29 and 5.30, no inhibition zone is observed. The lack of an inhibition zone is expected, as no antimicrobial agents (e.g., silver nanoparticles, triclosan, or quaternary ammonium compounds) were incorporated into the polyurethane matrix. This indicates that the foams do not release any diffusible antimicrobial agents into the surrounding agar medium to actively kill or inhibit the growth of *E. coli* or *B. subtilis*. Hence, they show no biocidal activity**.** Despite the absence of active inhibition, both the PI and PCW foams remained visually unaffected by bacterial colonization or fungal/mold growth on top of the polyurethane foam over the observation period (1-14 days).



(a) Industry PU | (b) Castor Oil – Water

Figure 5.30 Antimicrobial Testing on (a) PI and (b) PCW uisng *B.subtilis*

Several mechanisms could be contributing to this passive antimicrobial**.** Polyurethane foams, particularly those made with hydrophobic polyols and blowing agents such as HCFC 141b, often exhibit low surface energy and hydrophobicity. This can inhibit microbial adhesion, especially that of Gram-negative bacteria such as E. coli, which rely on surface wetting and moisture retention to establish colonies. The semi-open cell structure of the foam may not retain sufficient moisture or nutrients on its surface to support microbial or fungal growth. If the foams were clean and uncontaminated with organic matter (e.g., oils, sweat, proteins), microbial attachment would be minimal. This aligns with studies showing that microbial growth on PU foam surfaces often requires surface contamination.

The hydrophobic characteristics of polyurethane surfaces are essential in preventing microbial attachment and colonization, although they do not inherently possess bactericidal properties. Polyurethane foams synthesized with hydrophobic polyols or blowing agents, such as, demonstrate low surface energy and restricted water absorption. The specified conditions impede the ability of bacteria, particularly E. coli, to form biofilms or adhere to the foam surface, as moisture is generally necessary for colonization to initiate. Nevertheless, the antimicrobial test depends on the diffusion of antibacterial agents into the agar to create a clear inhibition zone. The lack of active agents results in no observable zone of inhibition, even in the presence of surface-level antimicrobial resistance (Liu et al., 2020; Ghosh et al.,2019).

The polyurethane foams utilized in this study did not contain any antimicrobial additives, including silver nanoparticles, zinc oxide, or quaternary ammonium compounds. The material does not release biocidal substances into the surrounding agar medium. The lack of such zones indicates that the foam's resistance arises from contact-based or passive surface characteristics, rather than from active chemical inhibition. This is consistent with the literature highlighting the necessity of additives for the formation of clear antimicrobial zones in polyurethane systems (Tischer et al., 2012).

The differential microbial adhesion behaviour observed between Gram-negative *E. coli* and Gram-positive *B. subtilis* may elucidate variations in their interactions with foam surfaces. Gram-negative bacteria have an outer lipopolysaccharide membrane that increases their susceptibility to surface hydrophobicity, which may restrict their capacity to adhere and form colonies on non-polar polyurethane surfaces. In contrast, Gram-positive bacteria, characterized by the absence of an outer membrane and the presence of thicker peptidoglycan layers, may exhibit greater tolerance to such conditions;

however, no inhibition zones were observed due to the non-leaching nature of the polyurethane matrix. This supports the conclusion that the absence of inhibition zones does not indicate microbial susceptibility but rather reflects the absence of diffusible antimicrobial agents, thereby confirming the material's passive resistance to microbial growth (Yazdani et al., 2021). The results clearly show that bacteria did not attack the samples; they may possess anti-biofouling or biostatic properties due to their chemical structure, surface hydrophobicity, and nutrient deprivation, leading to resistance to colonization by bacteria and fungi. This passive resistance is valuable in grouting applications where surface contamination without the use of active antimicrobial agents remains a concern.

CHAPTER 6
CONCLUSION AND RECOMMENDATION

6.1 Conclusion

In conclusion, all three objectives have been accomplished. First objective in focus on finding the optimum ratios of polyol and isocyanate for the industry polyurethane. Three distinct ratios of polyurethane were successfully synthesized (0.5:1, 1:1 and 1:0.5). The findings for the initial objectives indicate that a 1:1 ratio is suitable for polyurethane grout, yielding a hard and stiff material with superior physical, mechanical, and morphological qualities. Ratio 1:1 yields a rapid reaction rate relative to other ratios, indicating that the reaction is time efficient, hence minimizing the duration needed for pavement maintenance and repair. The FTIR data indicate that all ratios exhibit complete urethane linkage, since all functional groups of polyurethane are present. The compressive strength and modulus of the 1:1 ratio were significantly high, which are superior than the other ratios. The SEM study indicates a 1:1 ratio, demonstrating that the array cells are uniform in size and exhibit no rupture, hence significantly enhancing waterproofing capability and resistance to fracture. This signifies that a 1:1 ratio has been chosen for usage in grouting applications as it follows the standard value of polyurethane grouting.

On the second objective, it was to evaluate the efficiency of water as blowing agents (PCW) properties compare with other blowing agents HCFC 141b (PCH) by using castor based polyol. The results show all types of polyurethane (PI, PCH and PCW) depicts the properties of polyurethane grouting. PCH shows good results as compared to PCW. Results shows the apparent density of 68.75 Kg/m^3 with compressing strength 5.62 MP with high viscosity compare to PI and PCW. The compression modulus of PCH also contribute the highest value of 7.78 MPa. However, PCW poses the potential to function as an alternative material for grouting application as the performance metrics of PCW were observed to be within the accepted specifications or established standards for polyurethane grouting materials. The rate reaction time for the production of polyurethane take place was high and the mechanical properties of PCW still within the standard of polyurethane grouting.

Third objectives of this study were the comparison of the environmental stress

cracking resistance (ESCR) and the antibacterial properties between PCW and PI. The ESCR analysis demonstrates that PCW is appropriate for use as an alternative in the polyurethane grouting sector. The mechanical properties of PCW and PI shows no significant towards the value of compression strength and modulus. It is shows the durability of the PCW can be adequate with PI. It also shows no bacteria detect on the surface of the sample after 2 weeks of exposure. This is proven the castor oil did not contribute to the bacterial attack even derived from vegetable oil based polyol polyurethane.

6.2 Recommendations

While castor oil based polyurethane foams present clear environmental advantages, their relatively lower compressive strength and higher water uptake compared to industrial polyurethane (PI) indicate a need for formulation optimization. Future research should focus on chemical modification of bio based polyols or blending with higher functionality components to improve mechanical and durability performance without compromising sustainability.

Furthermore, by extended aging experiments under real world environmental conditions (cycling humidity, temperature fluctuations, and soil chemistry) are needed to assess the long term durability of bio based PU foams. Combined studies of water absorption, Environment Stress racking (ESCR) and mechanical properties of retention over time would provide more accurate predictions for field applications.

The antimicrobial testing be augmented through the incorporation of fungus and mold. The testing indicates the duration required for the degradation of polyurethane, and the identification of fungi that may enhance the degradation time of polyurethane warrants investigation. To prolong or improve the longevity of polyurethane, antibacterial or antifungal agents, such as silver nanoparticles, can be used into the mixture. It can also augment the durability and strength of the polyurethane.

It is also, recommended to study to conduct a Life Cycle Assessment (LCA) to comprehensively evaluate the environmental impacts associated with the production and application of both industrial and green polyurethane foams. LCA is a systematic methodology that accounts for the entire life cycle of a material from raw material extraction and processing, to manufacturing, usage, and end of life disposal. By adopting this approach, a holistic understanding can be obtained of how different polyol

sources and blowing agents contribute to environmental burdens or sustainability benefits. Moreover, LCA can also highlight trade-offs in sustainability. For instance, while bio based polyols such as castor oil and water as blowing agent reduce dependence on fossil resources, their cultivation may involve land use changes, water consumption, and agricultural inputs that carry their own environmental impacts. Therefore, applying LCA will help to balance the advantages of renewable resources with potential ecological footprints.

These future research can enhance industry collaboration, offer incentives for bio based alternatives for a long term advantages of green polyurethane, as limited awareness, higher costs, and perceived poor performance hinder the adoption of green polyurethane in sectors dominated by traditional materials.

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AUTHOR'S PROFILE



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LIST OF PUBLICATION

Fauzi, F. N. a. M., Anuar, A. U., Bonnia, N. N., Hussin, N. I. a. M., & Redzuan, A. A. (2025). Potential of green binders as matrices in composite materials. In Engineering materials (pp. 141–163).

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CONFERENCE PRESENTATIONS

The physical properties of polyurethane to act as concrete raising material. 11-12 October 2022. 5th International Conference on Nano-engineering Technology Devices and Materials 2022 (IC-NET 2022) & IOS-FSG Postgraduate Seminar 2022, Glenmarie Resort, Shah Alam, Malaysia.

Superb performance, economic and durable Polyurethane-RCB as grouting materials. 21-25 November 2022 Invention, Innovation & Design Exposition 2022, Shah Alam, Selangor, Malaysia.

Different types of polyol and ratio of isocyanate for polyurethane for polyurethane grouting: physical, mechanical, and morphological properties. 30 November 2022. 30TH Scientific Conference of Microscopic Society Malaysia 2022, MSU University, Shah Alam, Malaysia.

Superb performance, economic and durable polyurethane as grouting materials. 22-23 May 2023. Penang International Invention, Innovation & Design (PIID) 2023, UiTM Pulau Pinang, Permatang Badak, Pulau Pinang, Malaysia.

Enhancing infrastructure sustainability with polyurethane as grouting material. 11-12 Novemner 2023. 2nd International Conference on Biodiversity and Sustainable Development 2023 & SDG Innovation@IBSD: Building a Resilient Nation, UiTM Shah Alam, Shah Alam, Malaysia.

Waste tire: a potential source of graphene oxide for future antibacterial fabrics. 20 June 2024. Innovative Research Exhibition (IReX) 2024, Universiti Kebangsaan Malaysia, Bangi, Malaysia.

Evaluating the efficiency of petroleum based polyurethane versus green polyurethane: A comparative study on the physical, mechanical and morphological properties for enhancing grouting application. 6-7 Febuary 2025. International Conference on Discoveries in Applied Science and Technology 2024 (DASAT), Genting Highland, Pahang, Malaysia.