

**WETTABILITY OF RECYCLED 3D PRINTING POLYMER MEMBRANE  
WITH CANDLE SOOT COATING FOR OIL/WATER SEPARATION**

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**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

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WITH CANDLE SOOT COATING FOR OIL/WATER SEPARATION**

**MUHAMMAD RAIHAN**

**This report is submitted  
in fulfillment of the requirement for the degree of  
Bachelor of Mechanical Engineering**

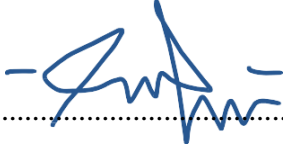
**Faculty of Mechanical Technology and Engineering**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**JUNE 2024**

## DECLARATION

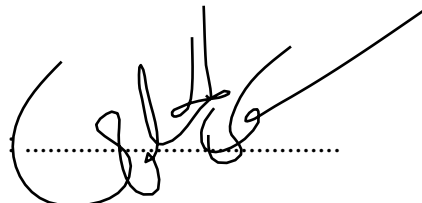
I declare that this project report entitled “Wettability of Recycled 3D Printed Polymer Membrane with Candle Soot Coating for Oil/Water Separation” is the result of my own work except as cited in the references

Signature :   
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Date : 10/06/2024

## APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature



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Date : 10/6/2024

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## WETTABILITY OF RECYCLED 3D PRINTING POLYMER MEMBRANE WITH CANDLE SOOT COATING FOR OIL/WATER SEPARATION

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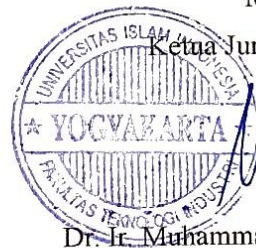
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## PERNYATAAN KEASLIAN

Demi Allah Yang Maha Segalanya, dengan ini saya menyatakan bahwa karya ini adalah hasil kerja saya sendiri kecuali kutipan dan ringkasan yang telah saya cantumkan sumbernya sebagai referensi. Apabila dikemudian hari terbukti bahwa pengakuan saya tidak benar serta melanggar peraturan yang sah dalam hak kekayaan intelektual, maka saya bersedia mengikuti hukuman maupun sanksi apapun sesuai hukum yang diberlakukan Universitas Islam Indonesia.

Yogyakarta, 21 Oktober 2024



Muhammad Raihan

## **DEDICATION**

To my beloved mother and father

## ABSTRACT

The evaluation of a membrane's wettability is crucial in improving the efficiency of oil/water separation; this can be achieved by calculating the liquid's contact angle with the surface. Creating hydrophobic surfaces for the purpose of separating oil from water has attracted a lot of attention recently. Nevertheless, the wettability behavior of three-dimensional (3D) printing membranes for oil and water separation is still under research. The objective of this study to determine the wettability characterization of 3D Printing polymer membrane with candle soot coating for oil/water separation. The research methodology included material characterization between scanning electron microscopy (SEM), surface roughness, porosity, and contact angle measurement on polyamide-12 (PA-12) membranes, coated with candle soot. Highest surface roughness value of 14.724  $\mu\text{m}$  is recorded for coated recycled bottom surface membrane with 80Watt of laser power and 0.12 mm layer thickness. Meanwhile, the lowest value of surface roughness (9.9357  $\mu\text{m}$ ) is measured for non-coated virgin top surface membrane with 80Watt of laser power and 0.12 mm layer thickness. The value for membrane contact angle of coated virgin 70Watt top, virgin 70Watt bottom, recycled 70Watt top, recycled 70Watt bottom, virgin 80Watt top, virgin 80Watt bottom, recycled 80Watt top, recycled 80Watt bottom are 143.5°, 148.2°, 141.7°, 147.3°, 138.2°, 145.4°, 146.1°, 151.8° respectively; for non-coated virgin 70Watt top, virgin 70Watt bottom, recycled 70Watt top, recycled 70Watt bottom, virgin 80Watt top, virgin 80Watt bottom, recycled 80Watt top, recycled 80Watt bottom, the measured contact angles are 137.8°, 142.3°, 137.8°, 138.9°, 136.9°, 139.2°, 144.1°, 145.9°, respectively. Based on SEM result, there are porous and melt characterization in every side of membrane depend the parameters that be used. The percentage for porosity result of coated membrane of virgin 70Watt, recycled 70Watt, virgin 80Watt, recycled 80Watt are 21.87%, 21.51%, 14.80%, 19.84%; as for non-coated virgin 70Watt, recycled 70Watt, virgin 80Watt, recycled 80Watt, the measured porosity are 20.18%, 19.95%, 13.78%, 16.51%. Results show that coated membrane performs the best which it can well separate water from oil. Recycled membrane has rough characterization naturally because it contains impurities from cooling and heating treatment before. Coating a membrane makes it rougher because the coating uses paraffin wax which it can hold coalescence of powder well. Moreover, paraffin wax has hydrophobic characterization itself and it can be higher contact angle measurement until superhydrophobic result with recycled membrane. Higher laser power also makes recycled membrane was more melting such as sapphire form which it can increase roughness. For conclusion, all of characterizations make air trap and behavior for creating a solid-water-air interface for super-hydrophobicity which it makes good separation of oil-water. Parameter production, coating, and material characteristic are important to consider to make good polymer membrane for oil/water separation.

## ABSTRAK

Penilaian keterbasahan membran adalah penting dalam meningkatkan kecekapan pemisahan minyak/air; ini dapat dicapai dengan mengira sudut sentuhan cecair dengan permukaan. Mewujudkan permukaan hidrofobik untuk tujuan memisahkan minyak dari air telah menarik banyak perhatian baru-baru ini. Walau bagaimanapun, tingkah laku kebolehbasahan membran cetakan tiga dimensi (3D) untuk pemisahan minyak dan air masih dalam penyelidikan. Objektif kajian ini adalah untuk menentukan pencirian kebolehbasahan membran polimer cetakan 3D dengan salutan jelaga lilin untuk pemisahan minyak/air secara graviti yang cekap. Metodologi penyelidikan termasuk pencirian bahan antara mikroskop elektron imbasan (SEM), kekasaran permukaan, keliangan, dan pengukuran sudut sentuhan pada membran poliamida-12 (PA-12), yang disaluti dengan jelaga lilin. Nilai kekasaran permukaan tertinggi sebanyak 14.724  $\mu\text{m}$  dicatatkan untuk membran permukaan bawah kitar semula yang disaluti dengan kuasa laser 80W dan ketebalan lapisan 0.12 mm. Sementara itu, nilai kekasaran permukaan terendah (9.9357  $\mu\text{m}$ ) diukur untuk membran permukaan atas dara yang tidak disaluti dengan kuasa laser 80W dan ketebalan lapisan 0.12 mm. Nilai sudut sentuhan membran bagi salutan dara 70Watt atas, dara 70Watt bawah, kitar semula 70Watt atas, kitar semula 70Watt bawah, dara 80Watt atas, dara 80Watt bawah, kitar semula 80Watt atas, kitar semula 80Watt bawah adalah 143.5°, 148.2°, 141.7°, 147.3°, 138.2°, 145.4°, 146.1°, 151.8° masing-masing; untuk dara yang tidak disaluti 70Watt atas, dara 70Watt bawah, kitar semula 70Watt atas, kitar semula 70Watt bawah, dara 80Watt atas, dara 80Watt bawah, kitar semula 80Watt atas, kitar semula 80Watt bawah, sudut sentuhan yang diukur adalah 137.8°, 142.3°, 137.8°, 138.9°, 136.9°, 139.2°, 144.1°, 145.9°, masing-masing. Berdasarkan hasil SEM, terdapat pencirian poros dan cair pada setiap sisi membran bergantung pada parameter yang digunakan. Peratusan hasil keliangan membran yang disaluti bagi dara 70Watt, kitar semula 70Watt, dara 80Watt, kitar semula 80Watt adalah 21.87%, 21.51%, 14.80%, 19.84%; manakala bagi dara yang tidak disaluti 70Watt, kitar semula 70Watt, dara 80Watt, kitar semula 80Watt, keliangan yang diukur adalah 20.18%, 19.95%, 13.78%, 16.51%. Hasil menunjukkan bahawa membran yang disaluti berfungsi dengan baik kerana ia dapat memisahkan air dari minyak dengan baik. Membran yang dikitar semula mempunyai pencirian kasar secara semula jadi kerana ia mengandungi kekotoran daripada rawatan penyejukan dan pemanasan sebelum ini. Menyalut membran menjadikannya lebih kasar kerana salutan menggunakan lilin parafin yang dapat menahan koalesens serbuk dengan baik. Selain itu, lilin parafin mempunyai pencirian hidrofobik sendiri dan ia boleh mencapai pengukuran sudut sentuhan yang lebih tinggi sehingga hasil superhidrofobik dengan membran yang dikitar semula. Kuasa laser yang lebih tinggi juga menjadikan membran yang dikitar semula lebih mencair seperti bentuk nilam yang boleh meningkatkan kekasaran. Untuk kesimpulan, semua ciri-ciri ini menyebabkan perangkap udara dan tingkah laku bagi mencipta antara muka pepejal-air-udara untuk super-hidrofobisiti yang menghasilkan pemisahan minyak-air yang baik. Parameter pengeluaran, salutan, dan pencirian bahan adalah penting untuk dipertimbangkan untuk membuat membran polimer yang baik untuk pemisahan minyak/air.



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I never stop to express my appreciation to all those who have contributed to be best result and successful of this project on analysis wettability of recycled 3D Printing polymer membrane with candle soot coating for efficient oil/water separation.

First of all, I am deeply grateful to my supervisor, Dr. Nurul Hilwa Binti Mohd Zini for their precious effort, knowledge, and her encouragement throughout the course of this project. Their insight was importance to guide the direction and breadth of this research.

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## LIST OF ABBEREVATIONS

SLS	Selective Laser Sintering
SEM	Scanning Electron Microscopy
ULCC	Ultra Large Cruide Carriers
VLSFO	Very Low Sulfur Fuel Oil
PAHs	Polycyclic Aromatic Hydrocarbons
PA-12	Polyamide-12
RF	Random Forest
SVM	Support Vector Machine
PTFE	Polytetrafluoroethylene
3D	Three Dimension
AAC	Autoclaved Aerated Concrete
CAD	Computer-Aided Design
UV	Ultra Violet

## LIST OF SYMBOLS

$P_r$	=	<i>Porosity</i>
$^{\circ}$	=	<i>Degree</i>
$\theta$	=	<i>Angle</i>
%	=	<i>Percent</i>
$\rho$	=	<i>Density of water</i>
$m_w$	=	<i>Mass of wet specimen</i>
$m_d$	=	<i>Mass of dry specimen</i>
$d$	=	<i>Thickness of membrane</i>
$\mu$	=	<i>Micro</i>
$A$	=	<i>Specimen sample area</i>
$\geq$	=	<i>More than or equal to</i>
$\leq$	=	<i>Less than or equal to</i>
$r$	=	<i>Half of diameter size</i>
$^{\circ}\text{C}$	=	<i>Celcius</i>

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background

Oil is one of the most important energy sources (Kushariyadi et al., 2024). From time to time, the demand for oil becomes higher. Almost everything needs oil energy right now. Example are vehicle, construction, beauty product, power plant, etc. Obviously, oil is produced from oil mine. However, not every country has oil resource for its necessary. Petroleum producers include Saudi Arabia, Russia, Canada, USA, etc. The transportation of oil from these countries to other places is normally via oil tankers

Oil tankers serve as oil shipments from place to place. It is transporting around 90% of all the oil around the world (J. Chen et al., 2020). Tankers are covered with various types of materials such as epoxy, stainless steel, and zinc silicate. It is meant to prevent oil spill.

Developments in this technology make human activities easier to transport oil to countries that do not produce oil. The transportation of oil to other locations creates risk such as oil spills. Oil spill should be prevented. It destructs the environment while it was happening. Additionally, the company gets loss to pay destruction and penalty.

In 2020, oil spill of MV Wakashio tanker happened when the ship grounded on a reef in Mauritius which released 1000 tons of very low sulfur fuel oil (VLSFO). It was reported 1000 tons of fuel oil spilled, around 0.5 tons of alkyl-phenanthrenes entered the environment. The most abundant Polycyclic Aromatic Hydrocarbons (PAHs) in the fuel oil were alkyl-naphthalenes (Scarlett et al., 2021). Oil spills are damaging not only economic aspects but also health. This health issue is damaging to both human and marine creatures. Moreover, the oil tanker's layer shall be environmentally friendly to the sea. Below, Figure 1.1 shows the MV Wakashio oil spill.



Figure 1.1: MV Wakashio oil spill

Oil spill is a very important environmental issue that needs to be considered. One of the latest possible solution to eliminate oil pollutants from both immiscible oil–water mixtures and bioinspired oil by using oil remediation procedures (Bhushan, 2019). In other hands, it can use mechanical and chemical membrane printed using Selective Laser Sintering (SLS) for separation of oil/water and immiscible organic mixtures (Yuan et al., 2020). SLS 3D printing is currently a popular method to fabricate separation membranes because it offers the ability to produce prototype

with similar mechanical properties as traditionally manufactured parts, making them lightweight, strong, and resistant to heat and chemical material (Martynková et al., 2021). Polyamide materials, like Polyamide-12 (PA-12) are chosen for membrane manufacturing as they offer excellent chemical stability and mechanical strength, essential for effective oil-water separation processes (Arbain et al., 2024). It is purposed for making high hydrophobic (superhydrophobic) membranes. Roughness material, surface morphology, porosity, contact angle, and candle soot coated membranes have been tested to determine hydrophobicity to ensure high separation of water and oil..

Wettability is a key aspect of creating good membrane. It can determined by measuring the water contact angle on the surface membrane. The contact angle defines the wetting behavior, whether it is hydrophilic, hydrophobic, or superhydrophobic (Arbain et al., 2024). In SLS application, usually membrane can be made from either virgin PA-12 powder or the recycled PA-12 powder. Naturally, recycled powder is more roughly than virgin PA-12 powder (Martynková et al., 2021). This thing probably can be more hydrophobic than the virgin powder. Furthermore, the recycled powder is more efficient in cost and eco-friendly.

## **1.2 Problem Statement**

However, polymer membrane has wettability and hydrophobic characterization itself. For best separation of water from oil, polymer membrane should be more hydrophobic (repel water). Moreover, it is a challenge itself for the researchers to find superhydrophobic surfaces which is described by  $150^\circ$  or over of contact angle result. Candle soot is one of the options for researchers to use as coating for the virgin-based polymer membrane as a way to increase hydrophobicity of the membrane surface (El Assimi et al., 2020). To minimize waste of printing

powder, more research has utilized recycled powder for specimen fabrication (Rafi Omar et al., 2022). It is considered a new material to fabricate polymer membrane for oil/water separation, therefore more studies on its wettability characterization need to be conducted to understand how it works. This characterization depends with parameters and testing result. Thus, this work should answer these questions:

- What is the effect of 3D printing parameters on the wettability characteristics of polymer membrane?
- What is the difference in terms of wettability performance between recycled- and virgin-based polymer membrane?

### **1.3 Objective**

The objectives of this project are as follows:

- To investigate the effect of 3D Printing process parameters on the wettability of recycled polymer membranes.
- To compare the wettability performance between 3D printed membranes is made of virgin powder and recycled powder.

### **1.4 Scope of Project**

The scopes of this project are:

1. The specimen was made from virgin and recycled Polyamide-12 (PA-12) powder. Every specimen was set with 0.12 mm of layer thickness.

2. To make specimens, virgin and recycled membranes were produced by Selective Laser Sintering (SLS) 3D Printing. Its parameters used 70 and 80 watts of laser power.
3. Virgin and recycled specimens were treated with candle soot coating. A mixture of candle soot and hexane solution was made by mixing 80 mg of candle soot particles into 80 mL of hexane using sonication for 30 minutes
4. Surface morphology (SEM), surface roughness, porosity, and contact angle testing checked the characterization of each specimen. Moreover, all testing was used for all sides among the top and bottom sides of the specimen membrane.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Oil Spill Pollution**

Oil spill pollution is a major environmental issue that has long-term effects on human health and marine ecosystems. Although, more than 5000 tons of oil leftovers were removed from the Brazilian Northeastern coast between August 2019 and January 2020, little tar balls can still be observed on a number of beaches (Lourenço et al., 2020). Figure 2.1 shows chromatogram of oil found in Brazil. Regretfully, if some of the oil is still stuck in reefs, marine sediments, and shallow coastal areas, this condition might continue. Furthermore, marine oil spill contamination is caused by a variety of oil products, such as crude oil and its emulsions (J. Yang et al., 2023). Oil spills can harm fish, seabirds, and people also such as financial losses.

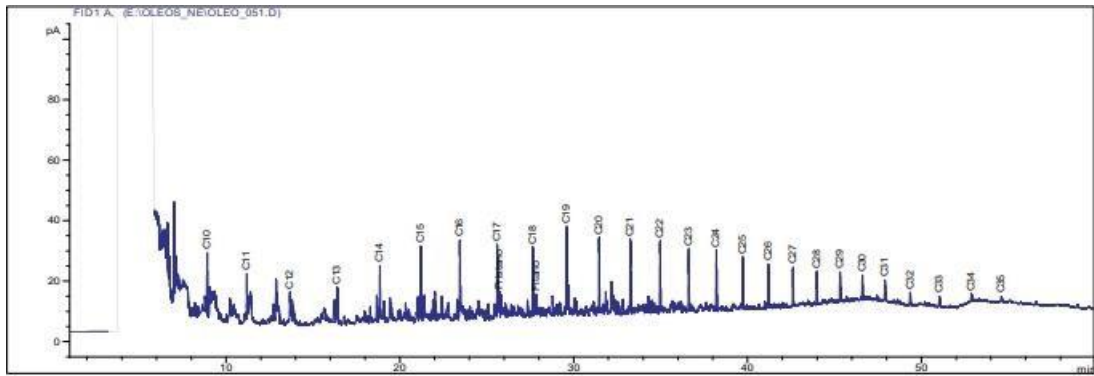


Figure 2.1: Chromatogram of the oil found at beaches of northeastern Brazil in 2019  
(Source: <https://doi.org/10.1016/j.marpolbul.2020.111219>)

One of the most significant types of pollution in the waters is oil. The severity of oil spill cause damage and losses (Nelson & Grubestic, 2021). There are four categories of severity in the degree of damage and severity of oil spills. The categories of impact are coastal and marine resources, total amount of beached oil, the spill extent, and the rate of plume expansion. Studies on the oil disaster at Dalian New Port demonstrate how persistent oil contamination is in coastal sediments, which serve as long-term storage for leftover oil (Guo et al., 2022). Whether it happens on land or in the sea, oil spills endanger wildlife, humans, and aquatic species, thus there is a need for solution from scientist how to clean up and care sea water. Figure 2.2 shows the effect of oil spill on wildlife.



Figure 2.2: Brown pelican coated on sea by heavy oil wallows (Source: <https://edition.cnn.com>)

## 2.2 Method to Separate Oil Spill

Oil spills are dangerous because they disrupt the marine ecology and unnecessarily risk the survival of marine life forms. It is now essential to explore for oil in oceanic resources. Therefore, since accidents happen unintentionally, it is crucial to use a variety of cleanup techniques for oil spills. Variations in the type of oil, the spill's location, and the current weather all contribute to this. Furthermore, a

number of chemical, physical, and biological mechanisms affect how spilled oil behaves and spreads across the ocean.

As oil is less dense than water, therefore oil spill will form a layer along the surface; one can easily picture the difficulties in cleaning up an oil spill. However, we should quickly detect wherever oil spills spread. An oil spill detection method can be based on machine learning approaches including Random Forest (RF) and Support Vector Machine (SVM) and optical images was introduced to detect oil (Nadia Abbaszadeh Tehrani et al., 2023). Four steps included performing, preprocessing, feature extraction and selection, classification, and sensitivity analysis and validation. Based on outputs, Sentinel-2 outperforms Landsat-8 in detecting oil spills. Since most oil spills happen unintentionally, it's critical to use a variety of cleanup techniques to minimize the threat they may represent to the marine ecology. Afterwards, various methods have been developed to separate oil spills effectively. One solution to solve oil spills is filtering by separation membranes that have mechanical and chemical capability to separate effectively oil and water emulsions.

### **2.2.1 Chemical Aspect**

To achieve chemical stability that can be hydrophobicity on a polyamide-12 (PA-12) membrane, various strategies have been explored. One approach involves physically modifying the PA-12 membrane through SLS 3D printing with a polymer containing recycled powder, resulting in a hydrophobic membrane with improved roughness and contact angle testing. Recycled PA-12 powder is used because it has rougher characterization that can enhance wettability. Additionally, SLS 3D printing produces parts that can be lightweight, high strength and resistant to chemical and heat (Yuan et al., 2020). Another method includes designing a functional PA-12 membrane with mixture of candle soot/hexane solution to enhance the roughness and

porosity, leading to highly stable superhydrophobicity (Arbain et al., 2024). So, these chemical aspects demonstrate the variety of methods used to enhance hydrophobic qualities on membranes for a range of purposes.

### **2.2.2 Mechanical Aspect**

Mechanical stability is used also for superhydrophobicity on a polyamide-12 membrane which various approaches can be considered. One method involves utilizing SLS 3D printing (Yuan et al., 2020). Fabrication of superhydrophobic PA-12 membrane can provide the desired mechanical stability. Laser power SLS 3D printing makes membrane more porous and rough, thus better hydrophobicity. These approaches collectively contribute to achieving mechanical stability for superhydrophobicity on a PA-12 membrane.

### **2.3 Selective Laser Sintering (SLS) 3D Printing**

In an industrial setting, selective laser sintering, or SLS, is a common 3D printing technique. SLS 3D printing offers significant benefits for manufacturing complex geometries (Hagemann et al., 2022.). Also, printing process that using SLS 3D Printing method can get high performance of membrane specimens included lightweight, strongfull, heat and chemicals resistance (Yuan et al., 2020). This additive manufacturing technique utilizes polymer powders to create 3D components, with the quality of the final part being influenced by the distribution and orientation of printing samples in the build chamber (Sulaymon et al., 2022). SLS 3D Printing enables the production of intricate structures that may not be feasible using traditional manufacturing methods due to constraints like tooling limitations (Matuš et al., 2023). Additionally, SLS 3D printing allows for the fabrication of complex geometries with high precision and detail, overcoming

challenges associated with conventional manufacturing processes (Sajadi et al., 2020.). The ability to print complex geometries with SLS opens up new possibilities for creating intricate designs and functional parts in various industries, showcasing the versatility and potential of this advanced manufacturing technology.

Furthermore, SLS 3D printing can enhance hydrophobic properties in various materials. There are key feature SLS 3D printing, between:

- Material versatility: SLS can print with a wide range of materials, including nylon, thermoplastic elastomers, metals, ceramics, and glass-filled materials.
- No need for supports: Unlike other 3D printing methods, SLS does not require support structures because the unsintered powder supports the part during printing.
- High detail and resolution: SLS can achieve high levels of detail and resolution, suitable for producing complex geometric shapes and functional prototypes.
- Batch production: SLS is capable of printing multiple parts simultaneously within the same build volume, making it ideal for batch production of small to medium-sized parts.
- Material recycling: Unused powder from previous prints can be recycled and reused for future prints, reducing material waste and costs.

However, SLS 3D printing technology gives hydrophobicity benefits by improving surface treatments on PA-12 parts, reducing water absorption and enhancing contact angle which leads to increased hydrophobicity (Yuan et al., 2020). Additionally, the fabrication of 3D structures with dual roughness scales through 3D printing can achieve superhydrophobic surfaces, enhancing water repellency and reducing solid-liquid contact areas (Elizabeth C. A. et al., 2023). Overall, SLS 3D

printing proves to be a valuable tool for enhancing hydrophobic characteristics in various materials, offering improved performance and durability.

## **2.4 Polyamide-12 Powder**

Polyamide 12 can be made more hydrophobic by incorporating specific additives or modifying its structure. One approach involves preparing optically transparent hydrophobic coatings using polyamide 12-SiO<sub>2</sub> nanocomposites, which exhibit superhydrophobicity with increased roughness due to higher SiO<sub>2</sub> content (Prasad et al., 2017). Another method is the synthesis of semi-crystalline hydrophobic polyamidoamines (H-PAAAs) by combining different bis-sec-amines with bis-acrylamides, resulting in water-resistant and thermally stable materials (Marcioni et al., 2021).

Moreover, PA-12 has characterization that be useful for oil and water separation, between:

- **Chemical Structure:** PA-12 is a thermoplastic polymer characterized by the presence of amide (CONH) groups in its chemical structure. It is a semi-crystalline material.
- **Mechanical Properties:**
  - a. **Strength:** PA-12 exhibits good tensile strength and impact resistance.
  - b. **Flexibility:** It remains flexible at low temperatures, which is beneficial for cold weather applications.
  - c. **Fatigue Resistance:** PA-12 has good resistance to repetitive stress and is often used in dynamic applications.

3. Thermal Properties:

a. Melting Point: The melting point of PA-12 is typically around 178-190°C

b. Heat Resistance: It can withstand moderately high temperatures without significant deformation.

4. Chemical Resistance:

PA-12 has good resistance to chemicals, oils, greases, and many solvents. It is less susceptible to hydrolysis compared to some other polyamides, making it suitable for applications where exposure to moisture is a concern.

5. Barrier Properties:

PA-12 provides a good barrier against gases and moisture, making it useful in daily applications.

Thus, PA-12 is better powder for SLS 3D printing. This powder can be used with SLS 3D printing and has high effectiveness of oil/water separation (Yuan et al., 2020). Naturally, virgin PA-12 powder has  $101 \pm 3^\circ$  of contact angle, which mean the powder is hydrophobic basically. This hydrophobicity can be enhanced by using coatings such as candle soot.

## **2.5 Candle Soot Coating**

Coating with candle soot refers to a methods known as carbon black deposition or candle blackening, where a surface is covered or coated with a thin layer of fine carbon particles derived from burning a candle. This method has been used historically for various purposes, including in scientific experiments and artistic applications. Candle soot coating uses for many functions and benefit, such as:

- Blackening Surfaces

Candle soot coating is used to darken or blacken surfaces for aesthetic or functional purposes. Historically, this technique has been employed in art, particularly in creating sketches and drawings (such as with the use of a "smokestick" or "fusain" for charcoal-like effects).

- Scientific and Research Applications

Candle soot coatings have been used in scientific experiments and research, particularly in materials science. The carbon coating can be utilized to create less wettability because of rough and hydrophobic characteristics.

- Surface Modification

Candle soot coatings can change surface properties, such as enhancing surface roughness or providing a protective layer against corrosion or oxidation.

Candle soot coating exhibits excellent hydrophobic properties. It has shown that candle soot can be utilized to create superhydrophobic surfaces with outstanding water-repellent capabilities. Moreover, candle soot coatings have been proven effective in reducing hydrate adhesion on surfaces, promoting slipperiness, and inhibiting water bridges between hydrates and substrates. So, the superhydrophobicity of candle soot-based coatings can be enhanced by adjusting the loading amount of candle soot, resulting in coatings with varying roughness levels and improved superhydrophobic properties (Zhang et al., 2022). Moreover, candle soot composite coatings have been developed to enhance the durability of superhydrophobic surfaces, demonstrating high resistance to mechanical abrasion, ultrasonication, and corrosive solutions while maintaining their hydrophobic characteristics (Sun et al., 2022).

## 2.6 Wettability

Wettability refers to the ability of a liquid to spread over to a solid surface. It is a fundamental property that influences how liquids interact with solids, and it is important in various scientific, engineering, and everyday contexts. The degree of wettability is typically quantified by the contact angle formed between the liquid droplet and the solid surface. The measurement of contact angle is shown in Figure 2.3.

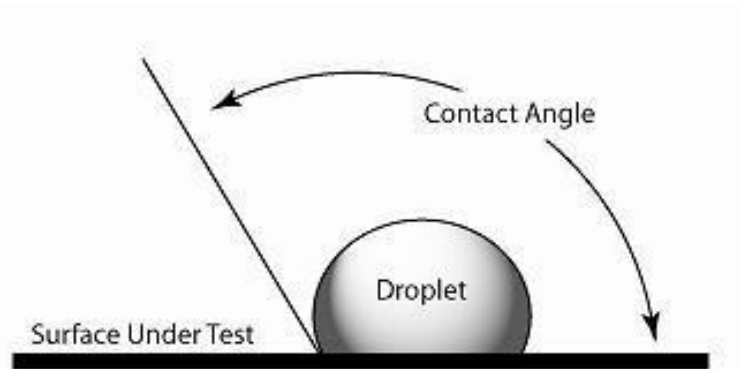


Figure 2.3: Contact angle measurement (Source: <https://inf-wiki.eecs.umich.edu/wiki>)

There are various type of classifications for wettability: hydrophobic, hydrophilic, oleophobic, oleophilic, and superhydrophobic characterization.

- **Hydrophilic**

These surfaces have low contact angles ( $\leq 90^\circ$ ) as shown in Figure 2.4. It promotes the spreading of water and other polar liquids. Examples include glass and certain metals treated with hydrophilic coatings.

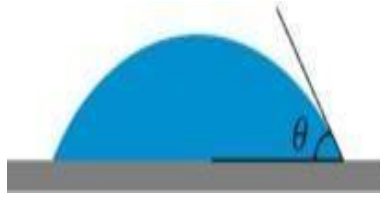


Figure 2.4: Hydrophilic contact angle

Hydrophilic compounds are often defined by their favorable interactions with water, which is why many chemical and biological processes depend on them. Hydrophilic part in hydrogel invention can improve swelling and dissolution behavior.

- **Hydrophobic**

These surfaces have high contact angles ( $\geq 90^\circ$ ) visually shown in Figure 2.5.



Figure 2.5: Hydrophobic contact angle

Hydrophobic means the phobic surface rejects water from the surface. Water rolls off on hydrophobic surfaces. Examples include Teflon (PTFE) and some silicones. Different methods, such as using surfactants, nanoparticles, and alkalis, have been explored to alter wettability in porous media, especially in carbonate rocks (Hachem et al., 2022).

- **Oleophilic**

These surfaces have high contact angles ( $\leq 90^\circ$ ) which is similar with hydrophilic. However, oleophilic is defined for oil and non polar liquid. Oleophilic surface is shown in Figure 2.6.



Figure 2.6: Oleophilic contact angle

"Oleophilic" refers to the property of having a strong affinity or attraction for oils and other non-polar organic compounds. According to the given papers, surfaces of materials like MoS<sub>2</sub> and graphite are composed of "oleophilic" regions that have a strong affinity for long-chain normal hydrocarbons (Zuo et al., 2020). Micro-/nanostructured interfaces, which are modeled after natural survival strategies, are essential to liquid manipulation. The way surfaces interact with liquids is determined by their morphology at the micro- and nanoscale, which results in characteristics like (super)wettability. Applications in drag reduction, oil-water separation, water collection, antifogging, antiicing, anticorrosion, and liquid transport are made possible by fabrication techniques and surface alterations. Achieving controlled liquid manipulation on par with biological systems still presents challenges. Comprehending the connection between liquid manipulation and micro-/nanostructured surfaces is crucial for the progress of several fields in the future.

- **Oleophobic**

Oleophobic happens when oil can make  $\geq 90^\circ$  of contact angle measurement, as shown in Figure 2.7 below.



Figure 2.7: Oleophobic contact angle

Oleophilic is often defined to show high hydrophobicity and superoleophilicity, which made them effective for oil absorption and environmental cleanup. Oleophobic surfaces repel oils, making them valuable for various applications like self-cleaning and oil-water separation. Different methods have been explored to achieve oleophobicity, such as coating plastics with perfluoropolyether (PFPE) and using hydrophobic agents on porous grains (Matsuno & Kawamoto, 2022). The research evaluated the hydrophobicity/oleophilicity of porous particles produced from autoclaved aerated concrete waste covered with oleic and stearic acids for the purpose of oil/water separation. The oil/water separation efficiency using the coated AAC grains through oil adsorption and fixed-bed column experiments

There are many factors that can influence wettability such as:

1. Surface Chemistry

The chemical composition of the solid surface determines its interaction with different liquids. Polar surfaces mean to be more hydrophilic, while non-polar surfaces are typically hydrophobic.

- Surface Roughness

Microscopic roughness on a surface can enhance wettability. For instance, nanostructured surfaces can create superhydrophobic behavior by minimizing solid-liquid contact area. It means rougher surface is better for hydrophobicity.

- Surface Energy

The surface energy of a material influences its wettability. Materials with higher surface energy are more likely to be wetted by liquids.

- Liquid Properties

The surface tension and polarity of the liquid also affect wettability. Polar liquids like water interact differently with surfaces compared to non-polar liquids like oils.

Thus, understanding wettability alterations through various methods is essential for optimizing processes like oil recovery and fluid flow in porous media. In summary, wettability is a key property controlling how liquids behave on solid surfaces. It is influenced by surface chemistry, roughness, and the properties of the liquid itself. Understanding and controlling wettability have broad implications across diverse fields, from materials science to everyday product development.

## **2.7 Summary of Past Research**

The research of Yuan et al. (2020) discussed on the fabrication of a robust membrane using SLS 3D printing for separating oil/water and immiscible organic mixtures. The membrane is hydrophobic, superoleophilic, and underwater

superoleophobic, showing excellent stability during various tests. It demonstrated high separation efficiency for oil/water mixtures and immiscible organic mixtures, expanding the use of selective laser sintering for functional membranes. On the other hand, candle soot coated then used to fabricate hydrophobic 3D printed membranes via SLS, demonstrating a rough and porous surface structure composed of partly sintered PA-12 particles. The porosity and morphology of the membranes are influenced by the laser parameters during fabrication, with higher energy input leading to denser structures and less porous surfaces. The successful coating and membrane fabrication process highlight the potential of using candle soot for functionalizing membranes and improving their stability and separation efficiency. Moreover, this journal discusses the limitations of traditional polymeric membranes and the benefits of using additive manufacturing like SLS 3D printing. Therefore, this journal highlights combination of candle soot and SLS 3D printing to functionalize PA-12 powder for membrane fabrication which showcasing improved stability and separation efficiency.

Another research by Yuan et al. (2017) describes a method to fabricate superhydrophobic surfaces on SLS 3D printed polysulfone (PSU) membranes. The paper also discusses the PSU membranes using candle soot to enhance oil/water separation efficiency. By immersing the PSU membranes in a candle soot/hexane solution, a rough membrane surface with a loose network structure is created, leading to superhydrophobic properties. The amount of candle soot deposited on the membrane surface and the water contact angle increase with longer immersion times, reaching over  $155^\circ$  after 28 minutes. It is caused the loose network structure traps air, creating a solid-water-air interface for superhydrophobicity. The membranes also exhibit superoleophilicity and stability due to the candle soot and network structure.

Varying processing parameters affect membrane structure and performance, influencing water permeability and porosity. Mechanical strength increases with higher parameters, while wettability changes with surface roughness. The surface shows excellent mechanical stability under sonication and chemical stability in harsh environments (e.g., 1 M HCl, 1 M NaOH, 1 M NaCl, and hot water). Furthermore, the bottom surface shows improved hydrophobicity due to its roughness. Significantly, the optimal membrane is chosen for candle soot coating to achieve super-hydrophobicity for efficient oil/water separation. For our research, it will be good opinion that compare with our research proven.

The paper of Dadbakhsh et al. (2017) details the morphological changes in PA-12 powders used for SLS post-ageing, including cracking, thermal behavior, viscosity increase, and molecular weight alterations. It discusses how ageing influences the powder's morphology and properties, potentially impacting the quality and performance of SLS-produced parts. On the other hands, the paper explores microstructural variations in SLS parts from virgin, mixed, and aged powders which affect mechanical properties. It highlights how the ageing status of the powders can lead to variations in the microstructure, ultimately influencing the mechanical behaviour of the SLS parts. Variations in mechanical properties of SLS parts are influenced by powder ageing. SLS parts from virgin powders show higher tensile strength due to higher crystallinity and fibrillar-like lamellae, resulting in increased stiffness and strength. Otherwise, aged-SLS parts show lower tensile strength attributed to lower crystallinity and a coarse spherulitic microstructure, leading to decreased stiffness and strength. Additionally, the aged-SLS parts demonstrate lower shear strength due to lower viscosity, affecting bonding strength, while mixed-SLS parts exhibit the highest ductility under tension, balancing strength and mobility of

interlamellar amorphous regions. These findings underscore the significant impact of powder ageing on the mechanical behaviour and performance of SLS parts. Although, this result will combine toward porosity result which connecting with tensile strength also. This research also concludes the consequences of powder ageing on material coalescence, crystallinity, and thermal traits of SLS parts, emphasizing viscosity, crystallinity, and melting modifications. It discusses how ageing affects the coalescence behaviour, crystallinity, and thermal characteristics of SLS parts, shedding light on the changes in viscosity, crystallinity, and melting behaviour. Therefore, this paper provides insights into the impact of powder ageing on the properties and performance of SLS-produced parts, emphasizing the importance of understanding these changes for quality control and process optimization.

The study of Rafi Omar et al. (2022) focused on investigating the impact of different PA-12 material compositions on the mechanical properties and surface morphology of SLS 3D printed parts. Various types of materials, including virgin, reheat, and recycled materials, were utilized to create test specimens under different process parameters. Tensile strength, surface roughness, and surface morphology were evaluated following ASTM standards. Results indicated that samples made of 100% virgin material exhibited higher stiffness, lower plastic deformation at maximum tensile stress, and smoother surface roughness. Scanning Electron Microscopy (SEM) analysis revealed partial melting of specimens with a notable amount of PA-12 powder before sintering and coalescing. The research findings serve as a valuable guide for SLS 3D printer users interested in printing composite material products. The study demonstrated that the mechanical properties and surface quality of SLS-printed products are influenced by the composition of PA-12 materials.

Specifically, samples with 100% virgin material displayed superior tensile strength and surface smoothness, highlighting the importance of material composition in achieving desired mechanical properties and surface characteristics in SLS 3D printing. But, recycled powder has higher roughness testing. This testing result will add point of view to prove higher contact angle that because higher roughness also.

Arbain et al. (2024) investigated the modification of polymer membranes using candle soot carbon coating. By dispersing candle soot in hexane and applying it to PA-12 membranes, changes in color and surface properties were observed. The coated membranes displayed increased roughness due to the deposition of carbon particles, affecting surface texture and crystallinity. SEM analysis revealed differences in surface morphology between coated and non-coated membranes, with the former showing more pores and roughness. The study also examined surface roughness, morphology, and contact angle measurements to assess the effects of membrane modification. The research methodology involved fabricating polymer membranes through the SLS 3D Printing method and physically coating them with candle soot for modification. The characterization process included evaluating surface roughness, contact angle measurement, and morphology analysis. This work emphasized the application of membranes in oil-water separation processes, underscoring the importance of surface modifications in enhancing membrane properties for specific separation tasks.

However, result of recycled polyamide 12 can change depends to its treatment before producing. So, the primary goal is to understand how repeated exposure to high temperatures during the SLS process affects the material properties of PA-12 powder. This understanding is crucial for improving part quality, reducing costs, and managing powder recycling more efficiently.

Repeated recycling and high-temperature exposure can degrade the powder, impacting its flowability and thermal characteristics, which in turn affect the quality of sintered parts (Pham et al., 2008). The study revealed several critical findings regarding the deterioration of PA-12 powder properties in the laser sintering (LS) process. Firstly, it was observed that powder heated above 140°C showed significant deterioration in properties, with an increase in melting temperature ( $T_m$ ) and a decrease in glass transition temperature ( $T_g$ ) over time, highlighting the temperature sensitivity of the material. Additionally, the Melt Flow Rate (MFR) of new PA-12 powder decreased significantly within the first 15-20 hours of exposure, indicating rapid initial deterioration, although the rate of deterioration slowed down afterward. This decline in MFR had a direct impact on part quality, as powder with lower MFR resulted in poor surface quality, exhibiting defects such as an 'orange peel' texture. A blend with an MFR of 25-27 g/10 min was identified as a reference for maintaining acceptable part quality. Furthermore, the study suggested that to restore the quality of heavily used (2-3 times recycled) powder to acceptable levels for SLS processes, about 50% of new powder is needed. These results underscore the importance of temperature control and efficient powder management in maintaining the quality of PA-12 powders and the parts produced using SLS processes.

There is study to analyze recycled polyamide 12 condition because of the printing cycle time, especially in the neighborhood of the printed part contour (Gomes et al., 2022). Analysis through Differential Scanning Calorimetry (DSC) revealed a consistent decrease in melting enthalpy and a slight reduction in melting temperature with successive reuse of PA-12 powder. These findings suggest a decline in crystallization and potential chain scission within the polymer structure

after more recycling process. Scanning Electron Microscopy (SEM) examination of the powder surface after 12 printing cycles unveiled a notable increase in roughness and porosity compared to virgin powder. Furthermore, Computed Tomography (CT) analysis further corroborated these observations, revealing a progressive increase in porosity within printed parts, particularly evident after the 6th cycle. This escalation in porosity adversely affects functional part reliability and dimensional accuracy, highlighting the cumulative impact of powder degradation on final product quality. These comprehensive analyses underscore the significance of monitoring and mitigating thermal and structural degradation in PA12 powder to uphold the integrity and performance of selective laser sintering (SLS) manufactured components.

Moreover, another study shows effect of aging powder influences crystallinity and melting behavior due to an increase in molecular weight and viscosity caused by post-condensation phenomena (Dadbakhsh et al., 2017). This increase in molecular weight holds the ordered chain folding, leading to reduced crystallinity in SLS parts made from aged powders. Aged or recycled powder is leading to change in melting behavior, mechanical properties, and coalescence behavior of the SLS parts. In this study highlights the importance of powder quality in the SLS process and provided insights for developing protocols for powder reuse in SLS processes to maintain its quality. The protocol maintains aged powder quality involves removing a portion of the aged powder after a certain number of cycles, especially from regions in excessive contact with heating, as wasted powder for increase its mechanical quality. Therefore, number of cycles influence wettability result.

Furthermore, maintenance while storing time is needed to maintain powder quality. The study meticulously examines the influence of storage time on polyamide

12 (PA-12) powder, used in selective laser sintering (SLS). It was observed that the prolonged storage of these powders led to notable changes in several key properties (Sanders et al., 2024). PA-12 storage exhibited a sharp change in color, often manifesting as yellowing. This color shift is typically indicative of chemical changes, such as oxidation, occurring within the powder. Additionally, the particle size distribution was changed, potentially due to the grouping of particles over time. These changes in particle size can negatively impact the flowability of the powder, a critical parameter for ensuring uniform layer deposition during the SLS process. A significant finding was the reduced oxidation resistance in stored powders. Oxidation can lead to the degradation of the polymer chains, resulting in shorter molecular chain lengths. This degradation was directly linked to the observed yellowing and reduced mechanical strength of the powder.

Moreover, decision controls are factor to determine quality of polyamide 12 powder. PA-12 storage in a wet atmosphere, such as nitrogen, can significantly reduce oxidation and moisture absorption. This approach helps maintain the powder's original properties over a longer period. Additionally, young recycled powders should be prioritized for such applications to ensure the highest quality and reliability of the parts. Aged of powder in storing time influence quality of mechanical and chemical.

Overall, the study aimed to enhance the wettability of 3D printed polymer membranes by characterizing the impact of candle soot coating. The investigation focused on improving oil-water separation efficiency through surface modifications. By analyzing surface roughness, contact angle values, and membrane morphology, the study provided insights into the effectiveness of candle soot coating in creating hydrophobic membranes. The results indicated that the modification process influenced the roughness and wettability of the membranes, showcasing the potential

of candle soot coating as an effective method for fabricating functional polymer membranes.

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Introduction**

In this chapter, we discussed methodology used to achieve the objectives of the project. Material preparation, material production, and experiment testing were included as steps in the methodology. Moreover, the procedure towards materials is also provided. The methodology was chosen to ensure a comprehensive and reliable experiment of the research questions, focusing on qualitative data to characterize a well recycled and virgin membrane of this topic.

Methodology helps researchers and practitioners to organize their work, make informed decisions, and replicate experiments or studies. Different disciplines have their own methodologies that are adapted to their own requirements and goals. In this research, the overall flow of the work is provided in Figure 3.1



Figure 3.1: Flow Chart Methodology

### 3.2 Material Preparation

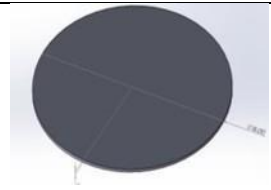

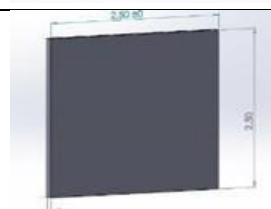
The preparation of materials for this study ensured the reliability and validity of the research findings. This involved the development of recycled and virgin membrane specimens, between accuration size, well ingredient, correct processing of experiment, and well design of specimen. Moreover, candle soot should be prepared as well as membrane specimen. Candle soot had its process to prepare coating membrane specimens. Well testing depend how good materials are prepared for

coating, included recycled and virgin membranes. Therefore, the research was done to prove characterization of membrane specimens.

### 3.2.1 Specimen Design

Specimen had critical result for this research. Specimen described qualitative of separation water from oil. For enhancing its separation, specimens should be designed as long as well procedure. The design and fabrication of specimens for this research were accomplished using advanced Selective Laser Sintering (SLS) 3D printing technology, ensuring precision and consistency across all specimens. The process started with the creation of detailed digital models using CAD (Computer-Aided Design) software (SolidWorks 2022). These models were carefully designed to meet the specific requirements of the research, including precise dimensions, geometries, and material properties. The models of the specimen are shown in Table 3.1.

Table 3.1: Membrane geometries that be tested

No	Drawing	Geometries	Testing
1		Geometry: Circular Diameter: 8 cm Thickness: 1 mm	1) Surface morphology 2) Surface roughness
2		Geometry: Circular Diameter: 5 cm Thickness: 1 mm	1) Porosity
3		Geometry: Square Diameter: 2.5 cm x 2.5 cm Thickness: 1 mm	1) Dry contact angle measurement

### **3.2.2 Collection of Candle Soot**

Candle soot was important for this research as it was used to coat membrane specimen to provide superhydrophobicity that is useful for separation of water from oil. Proper handling techniques and a controlled environment are crucial to prevent contamination and ensure the integrity of the collected candle soot coating. Nevertheless, for scientific research or creative projects, the collection of candle soot coating requires a methodical approach to get optimal results. So, candle soot is very important with wettability qualitative.

In order to collect candle soot, firstly, a clean metal plate was set above a paraffin candle in the center of the flame and kept there for 10 minutes, by 2 centimetres of distance. The accumulated candle soot on the metal plate was then scraped off and stored for further experiment of the PA-12 powder. The, the candle soot soot was kept in a storage to ensure it was free from contamination.

### **3.3 Material Production**

After material was prepared, the membranes were ready to be produced. Material production included the 3D Printing process and coating membrane by candle soot.

#### **3.3.1 3D Printing Process**

Membrane specimens were being produced with its geometries, based on Table 3.1, using SLS 3D printing method to get high performance of membrane specimens (lightweight, high strength and heat and chemicals resistance). Raw material for the printing process involved recycled and virgin PA-12 powder. For its production, the setting of the printing process was set in 70 and 80 watts of laser

power and 0.12 mm of layer thickness, listed in Table 3.2 below. SLS 3D printing process is described in Figure 3.2.

Table 3.2: Printing Parameters

Specimens		Parameters			
Material powder	Laser Power (Watt)	Layer Thickness/Slicing (mm)	Laser Beam Velocity (m/s)	Hatch Distance (mm)	Chamber Temperature (°C)
Virgin	70				
Recycled		0.12	7.6	0.30	169.5
Virgin	80				
Recycled					

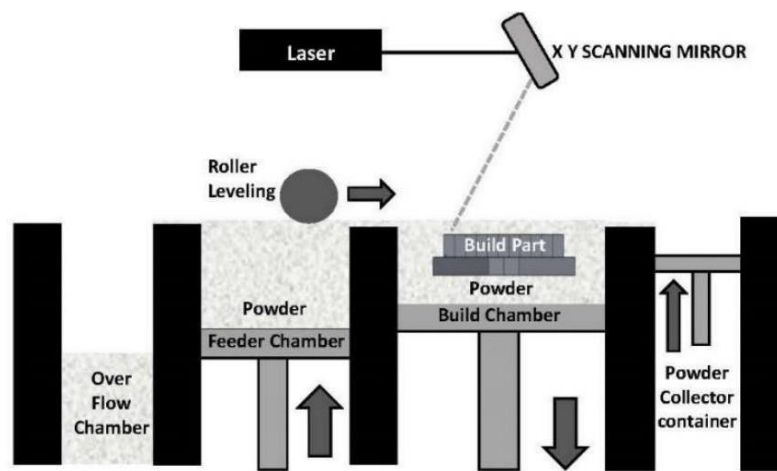


Figure 3.2: SLS process for Farsoon FS402P (Source: (Rafi Omar et al., 2022))

### 3.3.2 Coating of Polyamide-12 powder with candle soot.

All specimens of various printing parameters were coated with candle soot. Firstly, candle soot was calculated with 80 mg of weight. Afterwards, hexane was accounted also with 80 mL of volume. So, weight and volume ration was 0,1 wt%

and both mixture was then sonicated for 30 minutes by ultrasonic cleaner. The powder particles were coated and distributed onto the membrane surface using sonication methods. A uniform coating was produced by properly distributing and attaching the powder, which is made possible via sonication. Next, membranes that were produced was put inside of mixture for 40 minutes by ultrasonic cleaner. The bottle of mixture was opened a bit for good air circulation in sonication processing. And then, the membranes were put in oven that was set at 60 °C for 10 minutes to remove the hexane from the surface of the PA-12 membranes. Afterwards, membranes had been coated and moved to dry in ambient temperature. Coating process was described in Figure 3.3 and Figure 3.4.



Figure 3.3: Coating for specimen membrane

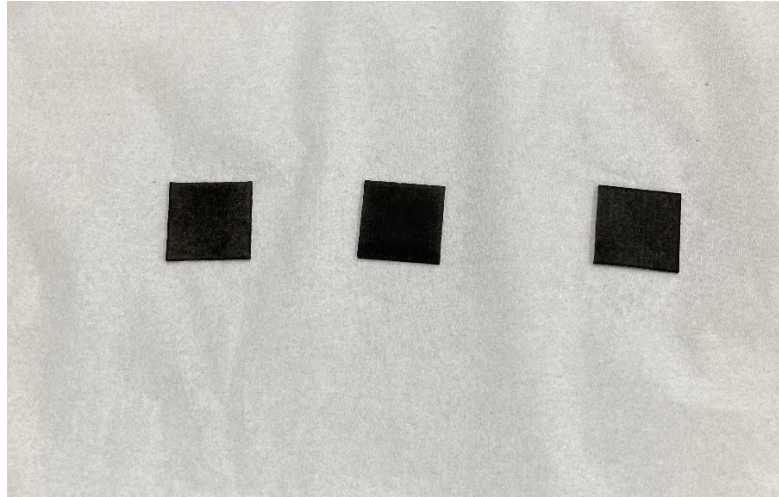


Figure 3.4: Specimen after coating

### **3.4 Characterization of Membrane**

After producing process, membranes underwent characterization. There were four tests conducted to determine membrane characterization.

#### **3.4.1 Surface Roughness**

The ISO 4287: 1997 standard specifies parameters for quantifying surface texture through roughness measurements. This standard outlines the parameters for evaluating surface roughness, including amplitude parameters such as Ra (average roughness). For surface roughness testing, Mitutoyo SJ-410 is equipped with a wide range of probing and analysis capabilities, enabling detailed assessments of surface texture. Its robust design ensured durability and consistent performance, making it a valuable tool for quality control and ensuring compliance with industry standards. Mitutoyo SJ-410 is represented in a Figure 3.5 below.

To measure surface roughness, first Mitutoyo SJ-410 roughness tester was calibrated. After that, both side, top and bottom were measured for each kind of

membranes. This measurement per surface was done by three times with different position. The result was recorded afterwards.



Figure 3.5: Mitutoyo SJ-410 tool

### 3.4.2 Contact Angle Measurement

Contact angle measurement is a crucial test in surface science used to evaluate the wettability of a material. This method involves placing a droplet of liquid on a solid surface and measuring the angle formed between the liquid's edge and the surface. The contact angle provides insight into the surface's hydrophobicity or hydrophilicity, indicating how well the surface interacts with liquids.

First step, researcher put membrane in front of a micro camera. 5mL of water volume was dropped with micropipette on surface membrane. Next, the micro camera was set to focus to membrane and capture the results. Contact angle setup is shown in Figure 3.6.

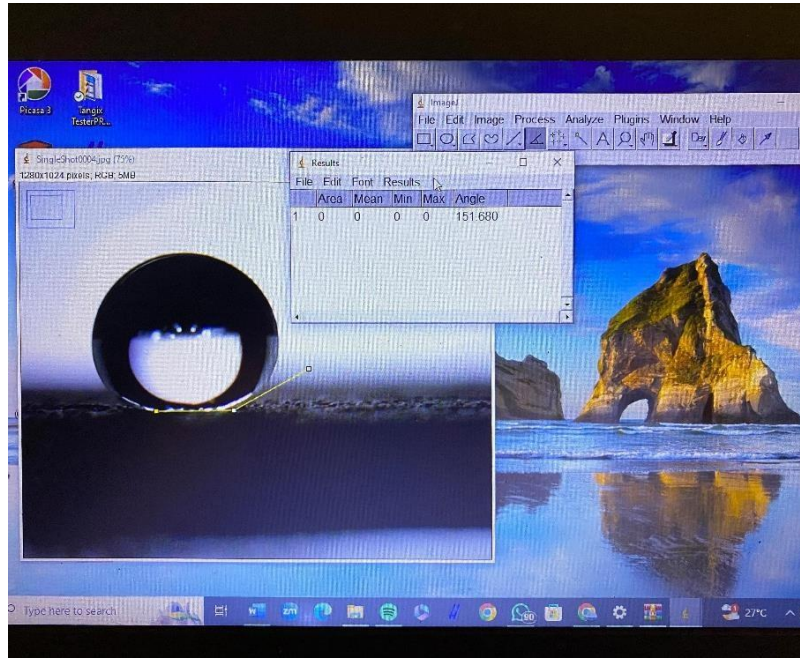


Figure 3.6: Contact angle measurement process

### 3.4.3 Porosity

Porosity testing is a fundamental procedure used to determine the volume and distribution of pores within a material. This process is essential for evaluating the structural integrity and performance characteristics of various materials, recycled and virgin PA-12 powder. Formula for porosity is described in equation 3.1 below.

$$P_r = \left( \frac{m_w - m_d}{Ad\rho} \right) \times 100\% \quad (3.1)$$

In this testing, measurement was repeated three times to ensure reliable result. Membranes were weighed when it was dry. Then, membranes were rinsed into ethanol liquid for 5 minutes. It was meant to clean membranes from powder and contaminations. Membranes should be removed from ethanol by deionized water.

After that, membranes were rinsed in deionized water for 10 minutes long and lifted for weighing next. Porosity testing setup is shown in Figure 3.7 and Figure 3.8.



Figure 3.7: Porosity testing setup

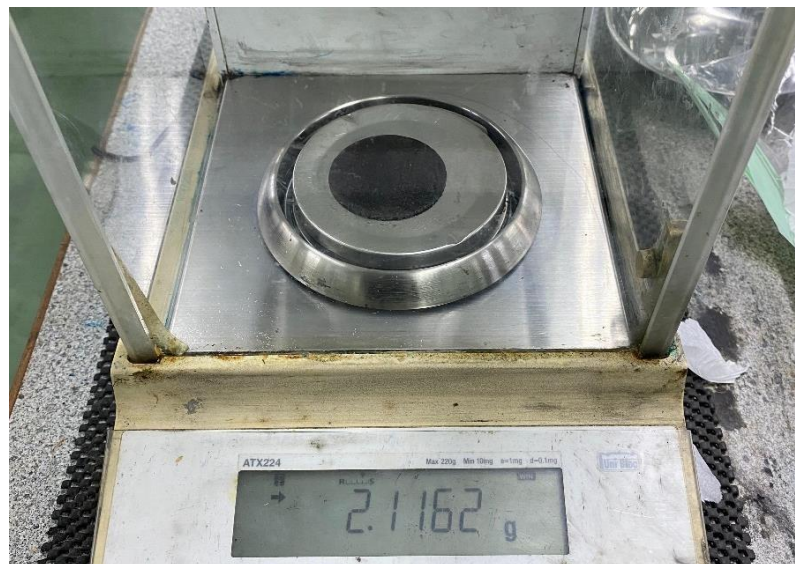


Figure 3.8: Weighing of wet recycled specimen after porosity testing

### 3.4.4 Surface Electron Microscopy (SEM)

Surface electron microscopy (SEM) is a powerful analytical method used to investigate the topography, composition, and properties of material surfaces at very high resolutions. This method provides a focused beam of electrons to scan the surface of a specimen, producing detailed images that reveal surface structures at the micro to nanometer scale. B. If membrane that is captured by SEM has a lot of pores, it means that the membrane is considered porous, which is represented as having high roughness value. All set up testing was meant to define well wettability and performance separation water from oil.

SEM setup is shown in Figure 3.9. SEM machine can focus on surface until 5 nm of size. This research used Scanning Electron Microscope (SEM) JEOL 6010 PLUS. It can reach magnification settings of 500 $\mu$ m (50x) and 100 $\mu$ m (200x), but for this research only used 50x and 200x magnification. The power for imaging the material under research was 5 kV, which is how the microscope was set up to operate.



Figure 3.9: SEM Setup

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 Surface Morphology

SEM images can be used to prove the visual characterization which show the morphology of the surface. Results of SEM is categorized based on the coating.

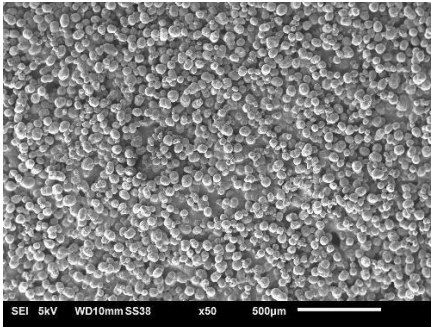
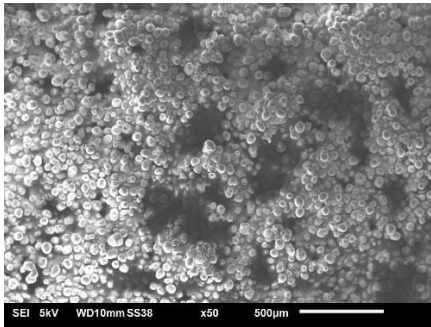
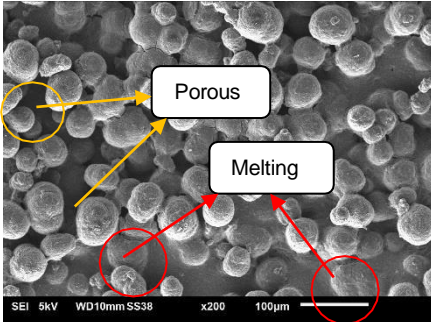
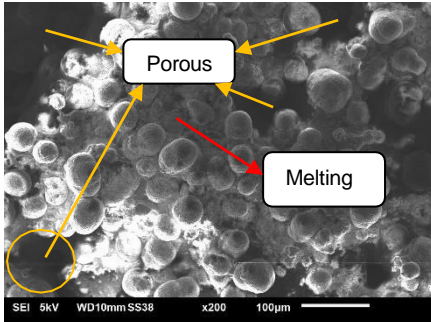
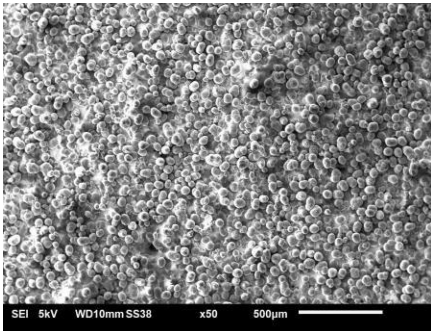
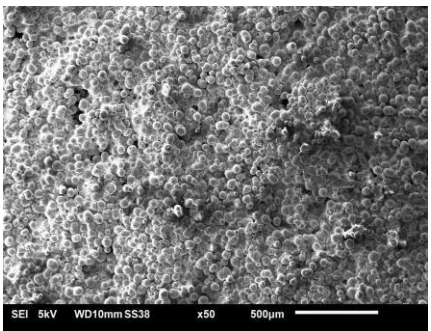
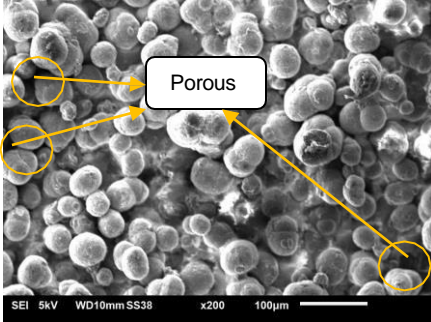
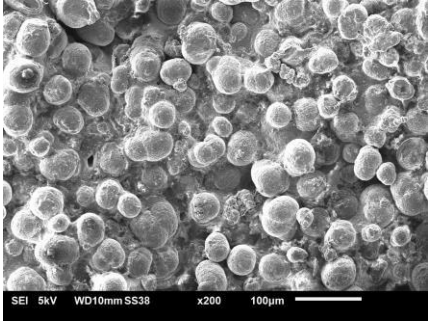
##### 4.1.1 Coated Membrane

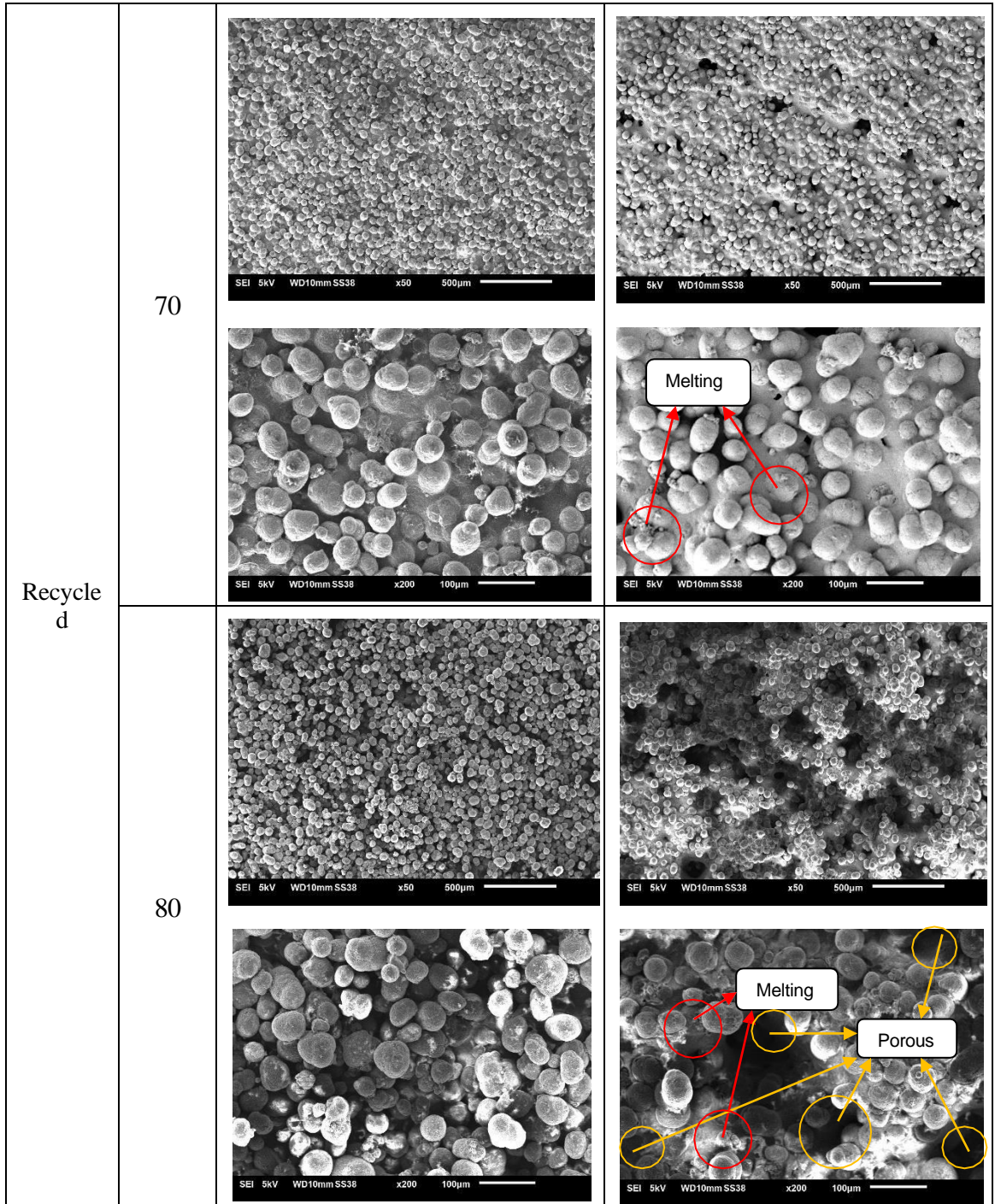
The specification of the specimen for SEM is provided in Table 4.1 while the result of SEM image testing is listed in Table 4.2.

Table 4.1: Coated membranes magnification

Powder	Laser Power (watt)	Magnification	
		Top	Bottom
Virgin	70	50	50
		200	200
	80	50	50
		200	200
Recycled	70	50	50
		200	200
	80	50	50
		200	200

Table 4.2: SEM surface morphology of 3D printed PA-12 coated membranes with magnification x50 and x200

Powder	Laser Power (Watt)	Top	Bottom
Virgin	70		
			
	80		
			



As we can see in Table 4.2, recycled powder specimen with 80 watt of laser power has more pores than others. This thing is good news for increasing hydrophobic value. Irregular or unflat surface results in higher roughness values, therefore is more hydrophobic (Yuan et al., 2017).

Additionally, the bottom surface has more pores than the top surface. Candle soot coating makes more pore for each membrane. The loose network structure that these nanoparticles candle soot create on the membrane's surface raises the porous of the surface (Ntaote Shooto & Dixon Dikio, 2011). Therefore, more pores is caused by the growth of porous candle soot nanonetwork (Hussein et al., 2022). Moreover, this nanoparticle contribute to superhydrophobic properties to the surface. The rough structure of the candle soot plays a crucial role in achieving water-repellent properties and stretchability (Wurong Ren et al., 2023).

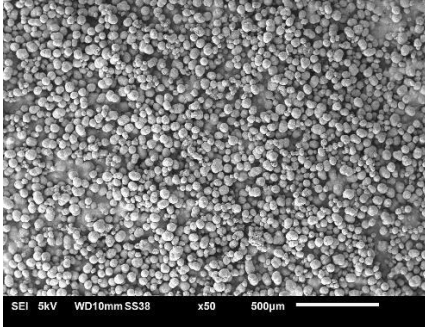
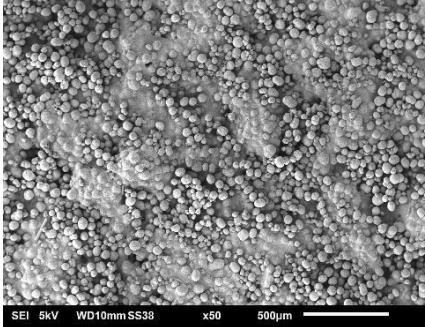
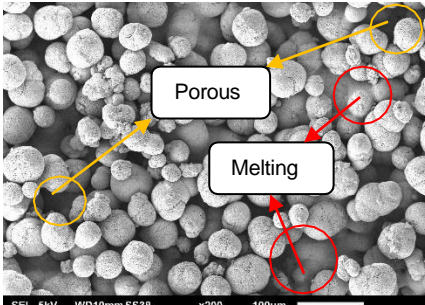
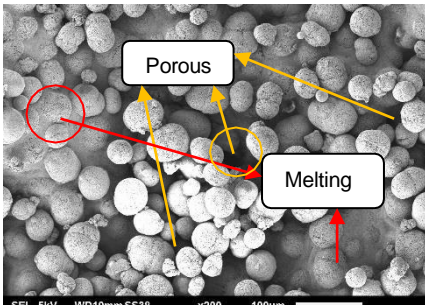
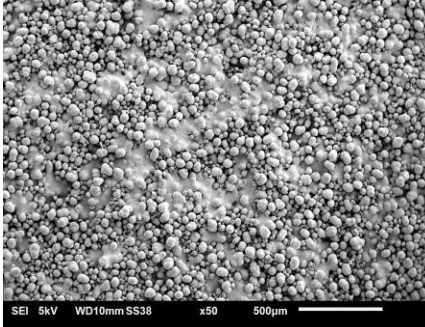
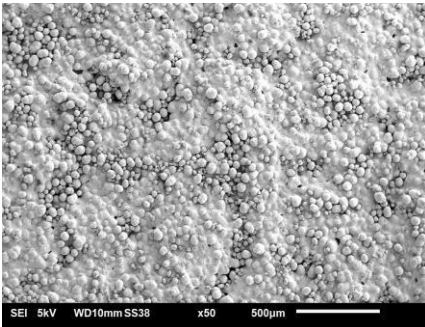
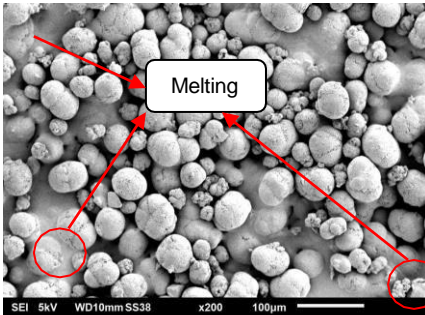
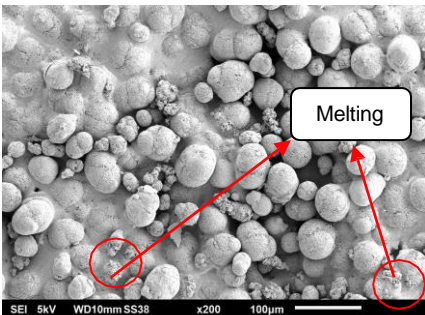
#### 4.1.2 Non coated Membrane

The specimen specification of non-coated membranesis provided in Table 4.3 while the result of SEM images is listed in Table 4.4 below.

Table 4.3: Non coated membrane magnification

Powder	Laser Power (watt)	Magnification	
		Top	Bottom
Virgin (Non Coated)	70	50	50
		200	200
	80	50	50
		200	200
Recycled (Non Coated)	70	50	50
		200	200
	80	50	50
		200	200

Table 4.4: SEM surface morphology of 3D printed PA-12 non-coated membranes with magnification x50 and x200

Powder	Laser Power (Watt)	Top	Bottom
Virgin	70		
			
	80		
			

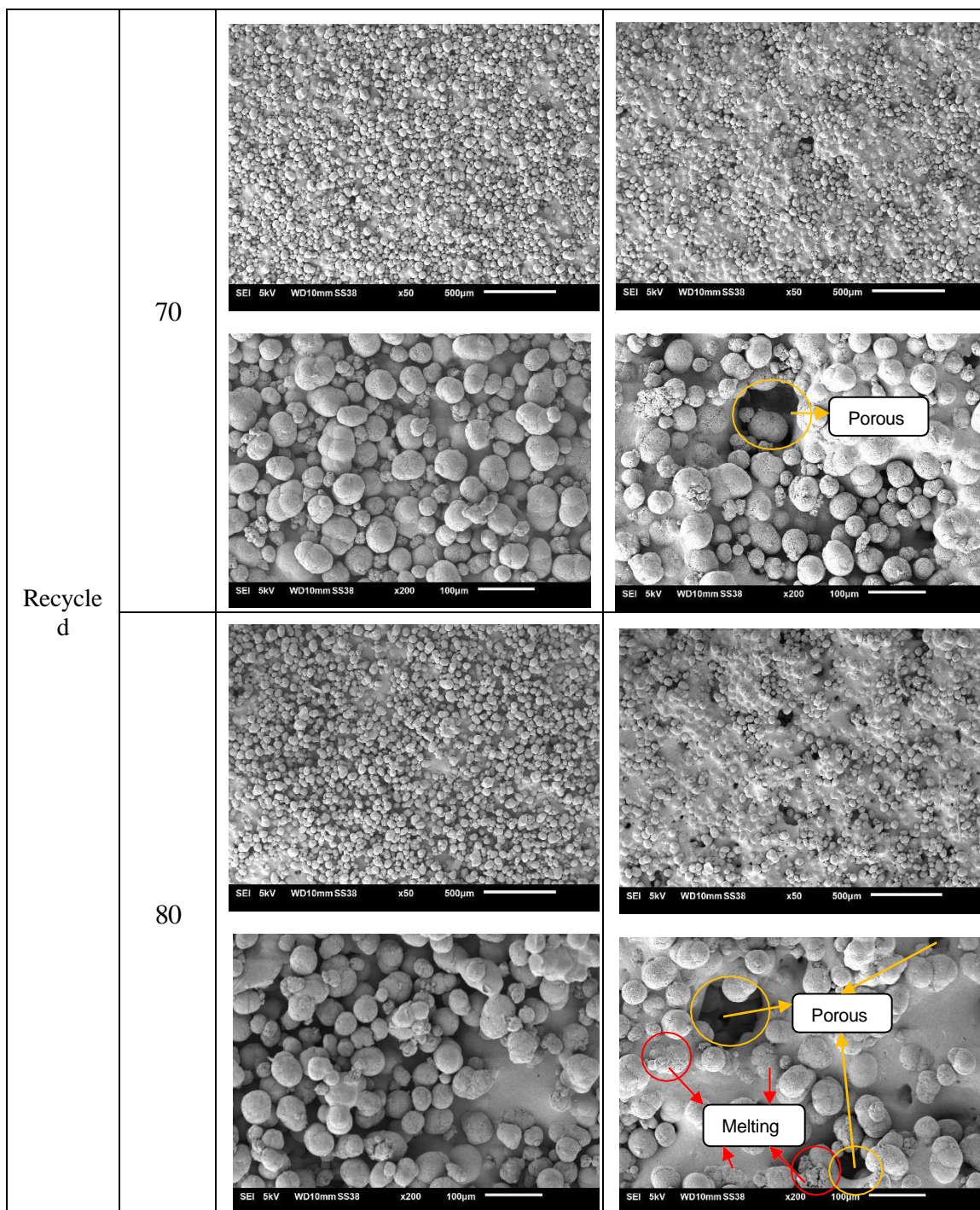


Table 4.4 above show the surface morphology of non-coated membranes for various specimens. Based on the data above, the top surface membrane images are denser than the bottom surface. This suggests that pores are smaller in size. Moreover, the melting powder is lesser. This pores and melting means the surface membrane had more part that be covered. Therefore, the roughness value decreases.

Furthermore, based on Table 4.4, the bottom surfaces are shown to be rougher than the top surfaces which occasion happens in every specimens This result is obtained because the laser power spreads unequal into bottom surface (Yuan et al., 2020). Because of that, the bottom surfaces is observed to have more pores than the top surfaces. Additionally, bottom surface without coated can be shown to have more melting. It is due to recycled membrane has lower melting point than virgin membrane (P. Chen et al., 2018). Therefore, it is appeared as SEM image that bottom surface membrane has more melting state than top surface membrane despite no coating for this membrane.

## 4.2 Surface Roughness Testing

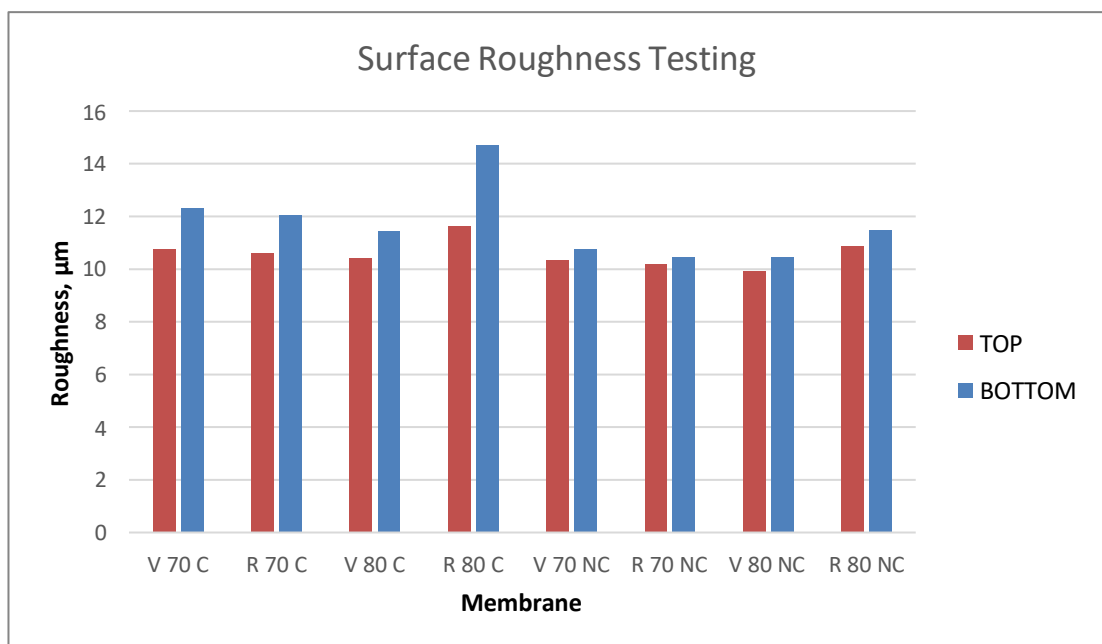
Table 4.5 and Table 4.6. list the surface roughness result. which was graphically plotted in Figure 4.1.

Table 4.5: Result for surface roughness testing of coated membrane

Material	Laser Power (watt)	Layer Thickness (mm)	Surface	Roughness Testing ( $\mu\text{m}$ )	Standard Deviation
Virgin	70	0, 12	TOP	10.7597	0.4754
			BOTTOM	12.3153	0.4387
Recycled	70		TOP	10.6063	0.8554
			BOTTOM	12.068	1,2934
Virgin	80		TOP	10.4053	0.3859
			BOTTOM	11.4291	0.4822
Recycled	80		TOP	11.6427	0.7813
			BOTTOM	14.724	0.9129

Table 4.6: Result for surface roughness testing of non-coated membrane

Material	Laser Power (watt)	Layer Thickness (mm)	Surface	Roughness Testing ( $\mu m$ )	Standard Deviation
Virgin	70	0, 12	TOP	10.362	0.5170
			BOTTOM	10.7510	0.4487
Recycled	70		TOP	10.1980	0.7038
			BOTTOM	10.4547	0.4927
Virgin	80		TOP	9.9357	0.2415
			BOTTOM	10.4603	0.5558
Recycled	80		TOP	10.8687	0.3268
			BOTTOM	11.4947	1.2356



- |  |   |
|--|---|
| 1. V 70 C = Virgin, LP: 70 w, Coated   | 5. V 70 NC = Virgin, LP: 70 w, Non Coated   |
| 2. R 70 C = Recycled, LP: 70 w, Coated | 6. R 70 NC = Recycled, LP: 70 w, Non Coated |
| 3. V 80 C = Virgin, LP: 80 w, Coated   | 7. V 80 NC = Virgin, LP: 80 w, Non Coated   |
| 4. R 80 C = Recycled, LP: 80 w, Coated | 8. R 80 NC = Recycled, LP: 80 w, Non Coated |

Figure 4.1: The result of coated and non-coated surface roughness graph

Surface roughness is important because it relates with wettability result (Yuan et al., 2020). Rougher membranes will result in more hydrophobic surface. The relationship between surface roughness and low wettability is a fundamental concept in surface science and material engineering. Surface wettability, which refers to the ability of a liquid to maintain contact with a solid surface, is typically quantified by the contact angle of a liquid droplet on the surface. A low contact angle (less than  $90^\circ$ ) signifies good wettability, indicating that the liquid spreads well on the surface. Conversely, a high contact angle (greater than  $90^\circ$ ) indicates poor wettability, where the liquid forms droplets and does not spread easily. In this case, we want to improve contact angle until more than  $150^\circ$  which mean superhydrophobic.

Surface roughness plays a crucial role in influencing wettability. It can amplify the inherent wettability characteristics of a surface, a phenomenon described by models such as the Wenzel and Cassie-Baxter models (Rauter et al., 2021). These models explain how the micro- and nano-scale features of a rough surface interact with a liquid droplet, affecting its contact angle and thus its wettability. Understanding this relationship is vital for designing materials with specific wetting properties tailored to various applications in industries ranging from biomedical to

aerospace engineering. Thus, high surface roughness can result in larger contact angle which leads to superhydrophobic surface.

Membrane which be produced by recycled powder has higher roughness than virgin powder. It is reasonable because when recycled polyamide 12 powder is used in SLS 3D Printing, it causes poor component surface finish with uneven textures and a lot of unsintered particles (F. Yang et al., 2021). Moreover, many reasearcher make combination powder to improve these surface and mechanically part qualiatative. Fully recycled powder part has increased roughness, porosity, unflat and unmolten particles.

Moreover, variations in surface characteristics or the presence of contaminants may also have an impact on the recycled powder particles' surface roughness (Rafi Omar et al., 2022). The modification of recycled powder such as heat treatment, cooling, and melting when recycling process and also dirty contaminants make the powder particle more hydrophobic. However, this virgin powder contrasts with recycled powder that recycling process can unintentionally produce.

Furthermore, recycled powder needs higher melting point (P. Chen et al., 2018). The powder's peak melt temperature slightly increases dependent with recycled powder's age. It indicates that crystalline reorganization needs a higher melting point to be crystal structure. Crystal structure makes recycled powder more-rough. However, high laser is good to promote higher temperature for making crystal nucleation in recycled powder, which in this case 80 watt is better than 70 watt.

On the other hands, additional variables were introduced by the candle soot coating. The coating is needed for increasing contact angle which can be described

by surface roughness. The coating makes higher roughness because it is made from nanoparticle that this characterization is loose structure (Hussein et al., 2022). Therefore, coating soot makes surface rougher. Carbon nanoparticles that be putted as coated membranes are rougher than non-coated membranes. Additionally, paraffin wax nanoparticles are hydrophobic (Atta et al., 2019) . These nanoparticles can also increase hydrophobicity.

### 4.3 Porosity Testing

Table 4.7 and Table 4.8 list the result of porosity, plotted graphically in Figure 4.2.

Table 4.7: The result of coated membrane porosity testing

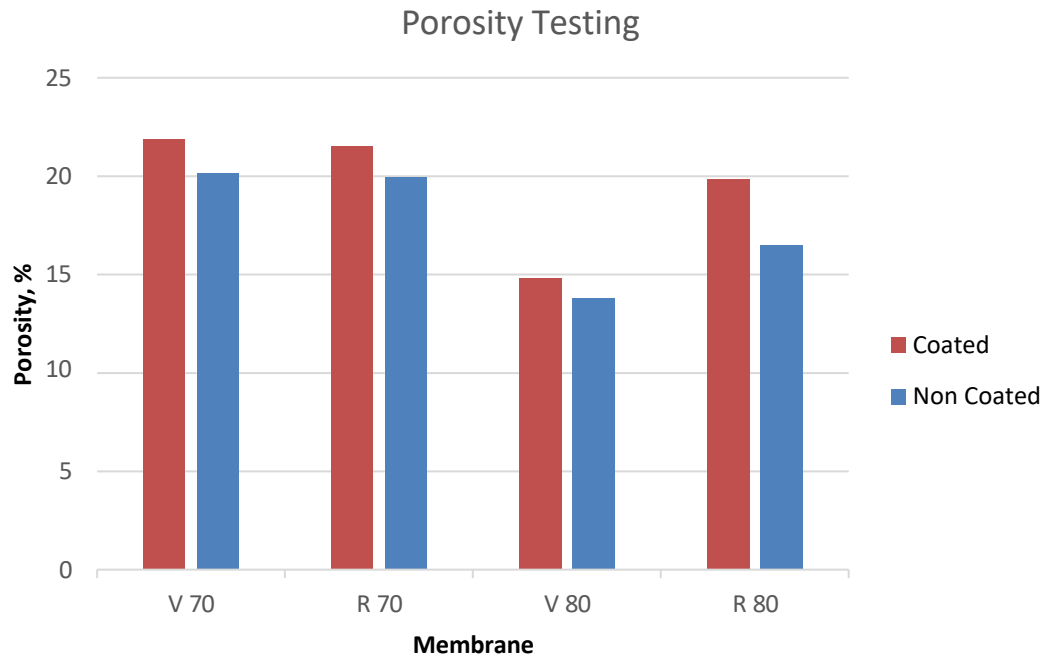
Powder	Laser Power (watt)	Layer Thickness (mm)	Readings	Sample		
				Sample 1	Sample 2	Sample 3
Virgin	70	0, 12	m <sub>d</sub>	1.9627	1.9466	1.7752
			m <sub>w</sub>	2.431	2.3479	2.1941
			Porosity(%)	23.85	20.438	21.334
			Average	21.874		
Recycled	70	0, 12	m <sub>d</sub>	2.1247	1.8112	1.8257
			m <sub>w</sub>	2.603	2.237	2.1892
			Porosity(%)	24.359	2.237	2.1892

			Average	21.5193		
Virgin	80		m <sub>d</sub>	1.9043	1.8363	1.8714
			m <sub>w</sub>	2.1743	2.1289	2.1808
			Porosity(%)	13.751	14.912	15.7576
			Average	14.8069		
Recycled	80		m <sub>d</sub>	1.7642	1.6338	1.7346
			m <sub>w</sub>	2.1113	2.0739	2.1162
			Porosity(%)	17.68	22.41	19.43
			Average	19.84		

Table 4.8: The result of non-coated membrane porosity testing

Powder	Laser Power (watt)	Layer Thickness (mm)	Readings	Sample		
				Sample 1	Sample 2	Sample 3
Virgin	70	0, 12	m <sub>d</sub>	1.8601	1.711	1.7293
			m <sub>w</sub>	2.306	2.0794	2.104
			Porosity(%)	22.706	18.757	19.0833
			Average	20.1821		
Recycled	70		m <sub>d</sub>	1.8391	2.0065	1.8737
			m <sub>w</sub>	2.2005	2.4033	2.2547

			Porosity(%)	18.4050	20.056	21.38
			Average	19.947		
Virgin	80		m <sub>d</sub>	1.8118	1.8763	1.9058
			m <sub>w</sub>	2.116	2.156	2.1335
			Porosity(%)	15.4928	14.245	11.5967
			Average	13.7782		
Recycled	80		m <sub>d</sub>	1.7559	1.7413	1.6553
			m <sub>w</sub>	2.1497	1.9937	1.9786
			Porosity(%)	20.056	12.855	16.645
			Average	16.5187		



Number of membrane	
1. V 70 = Virgin, LP:70 w,	3. V 80 = Virgin, LP:80 w
2. R 70 = Recycled, LP: 70 w	4. R 80 = Recycled, LP: 80 w

Figure 4.2: The result of coated and non-coated membrane porosity testing graph

Porosity refers to the presence of voids or pores within a material. These pores can arise due to incomplete melting, insufficient energy input, or other factors. During the manufacturing process, incomplete melting can result in trapped gas bubbles or voids within the material. This often happens when the material does not fully liquefy, leading to inconsistencies in its structure. Similarly, insufficient energy input during processes like SLS 3D printing can cause incomplete fusion, which also contributes to porosity. Additionally, some materials release gases, such as hydrogen, during solidification, leading to the formation of pores. Solidification shrinkage is another common issue; as the material cools and solidifies, it contracts, which can

create voids. These factors collectively impact the integrity and qualitative of the manufactured product and wettability.

Porosity testing is conducted because it describes result that show a strong correlation between porosity and mechanical properties, linked to energy input. A strong correlation was observed between energy input, porosity/bulk density, and mechanical properties. Energy power relates to porosity results in the context of the degradation of the powder and its impact on the printed parts characterization. The porosity level in printed parts, as observed in the study, is influenced by factors such as the increased porosity of reused powders due to thermal degradation (Sanders et al., 2024). Reused powder indicated a decrease in melting enthalpy and a slight decrease in melting temperature, suggesting a decrease in the degree of crystallization throughout printing cycles. This degradation process, characterized by chain scission mechanisms, results in changes in the material properties, such as molecular weight variations and effects on chain mobility. These variations can lead to an increase in porosity in the printed parts, which is crucial for their mechanical performance and dimensional accuracy.

However, there are cases where excessive energy inputs may have a negative impact on porosity (Zhu & Majewski, 2020). Excessive energy input during SLS can indeed lead to increased porosity, negatively affecting mechanical properties. Higher porosity reduces material strength, stiffness, and fatigue resistance. Therefore, balancing energy input to minimize porosity while maintaining desired mechanical properties is crucial.

The aging behavior of PA12, which can result in increased porosity that compromises the mechanical integrity of printed parts (Wudy & Drummer, 2019). It

has investigated the impact of porosity on recycled or aged PA-12 in powder bed fusion processes. Additionally, discussing about the systematic mechanism of PA12 aging, emphasizing the importance of understanding how powder aging can affect melt viscosity and overall part qualitative.

There are both internal and external factors that might accelerate the aging or degradation of polymers, especially recycled powder (Sanders et al., 2024). The material's physical and chemical interactions with its environment, such as weathering, UV radiation, humidity, and temperature. This interaction is being the most common degrading reason of this powder as the external causes. The material's thermodynamically unstable internal causes are states that, when triggered, usually by heat stress, result in measurably different properties. Incomplete polycondensation, residual stress, and unstable crystallization times are some examples of internal causes also.

In conclusion, based on Figure 4.2, the porosity of virgin with 70 watt of laser power is highest value. However, porosity does not directly indicate roughness because it primarily deals with the internal structure and volume of voids within the membrane. Meanwhile, roughness pertains to the external surface characteristics. Porosity value is influenced by many factors including membrane condition and producing method. Porosity is tested to prove how big influence to assist hydrophobic happening.

#### **4.4 Contact Angle Testing**

Contact angle testing is important for determining wettability result. Furthermore, contact angle determines whether the surface is hydrophobic,

superhydrophobic, and sequence of hydrophobic values. Table 4.9 until Table 4.10 list all of contact angle result for coated membranes and plotted in Figure 4.3 below.

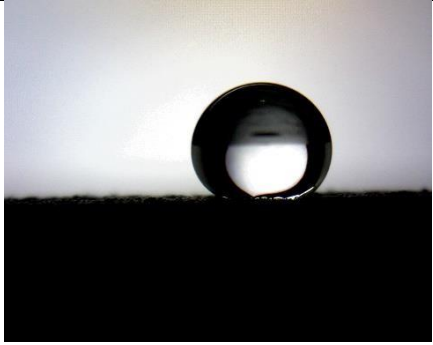
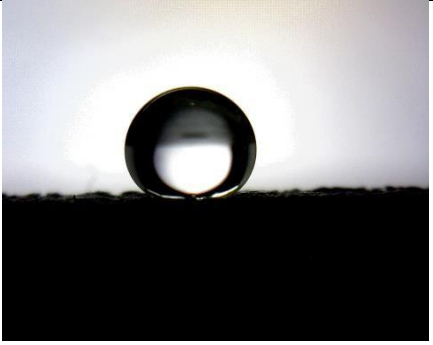

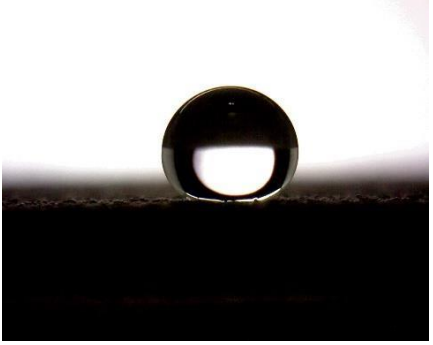


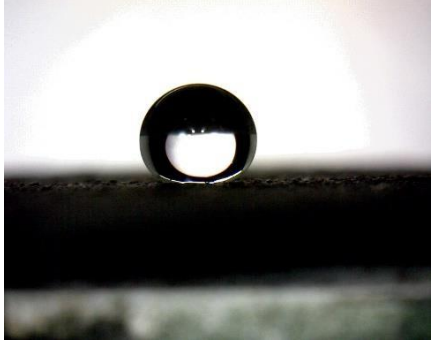

### 1) Coated Membrane

Table 4.9: Contact angle result for coated membranes

Material	Laser Power (watt)	Thickness (mm)	Surfaces	Average Contact Angle (°)	Standard Deviation
Virgin	70	12	Top	143.5784	4.7075
			Bottom	148.171	2.6578
Recycled			Top	141.6644	2.9314
			Bottom	147.3376	5.5393
Virgin	80		Top	138.1748	2.9380
			Bottom	145.4308	3.4506
Recycled			Top	146.0916	2.9675
			Bottom	151.8148	2.3444

Table 4.10: Droplet picture for coated membranes

Material	Laser Power (watt)	Top	Bottom

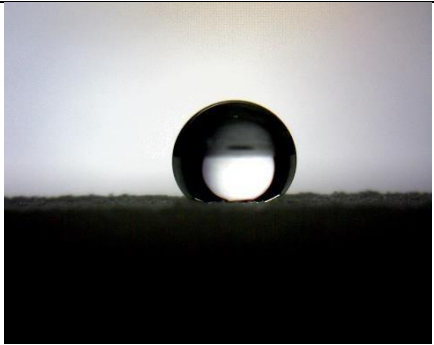
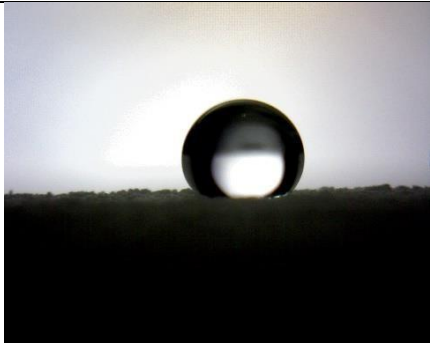
Virgin	70		
		143.5°	148.2°
Recycled	70		
		141.7°	147.3°
Virgin	80		
		138.2°	145.4°
Recycled	80		
		146.1°	151.8°

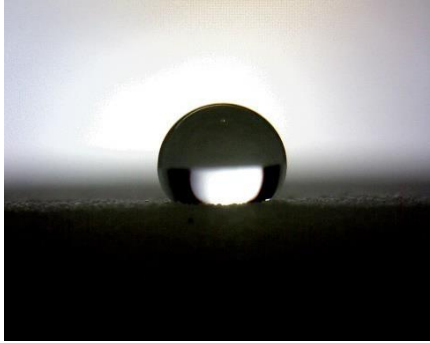
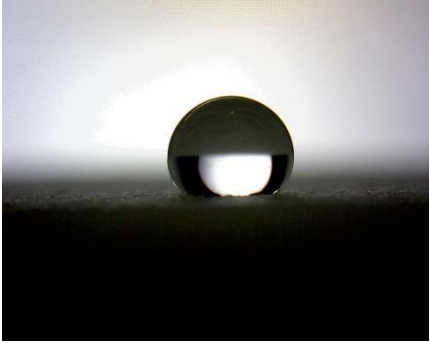
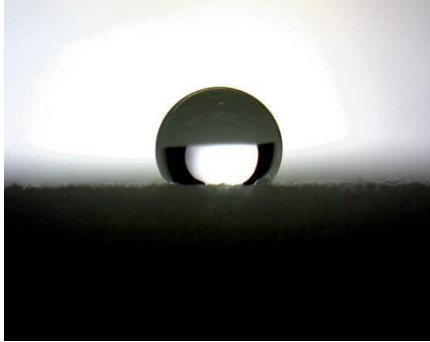
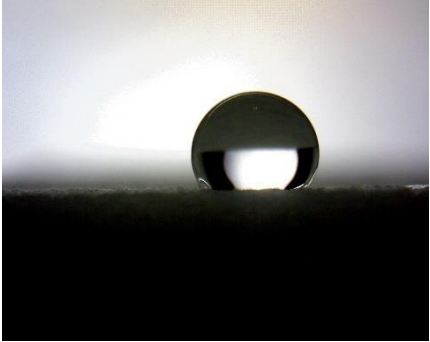
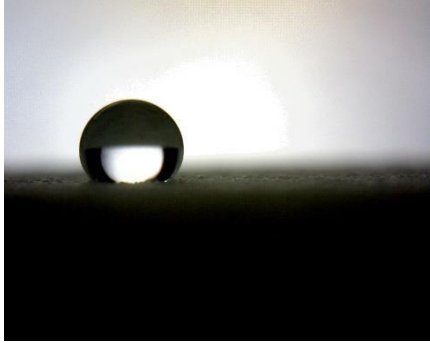
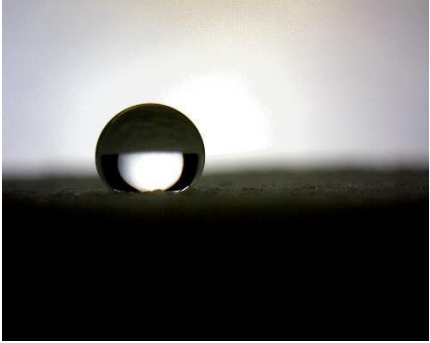
## 2) Non Coated Membrane

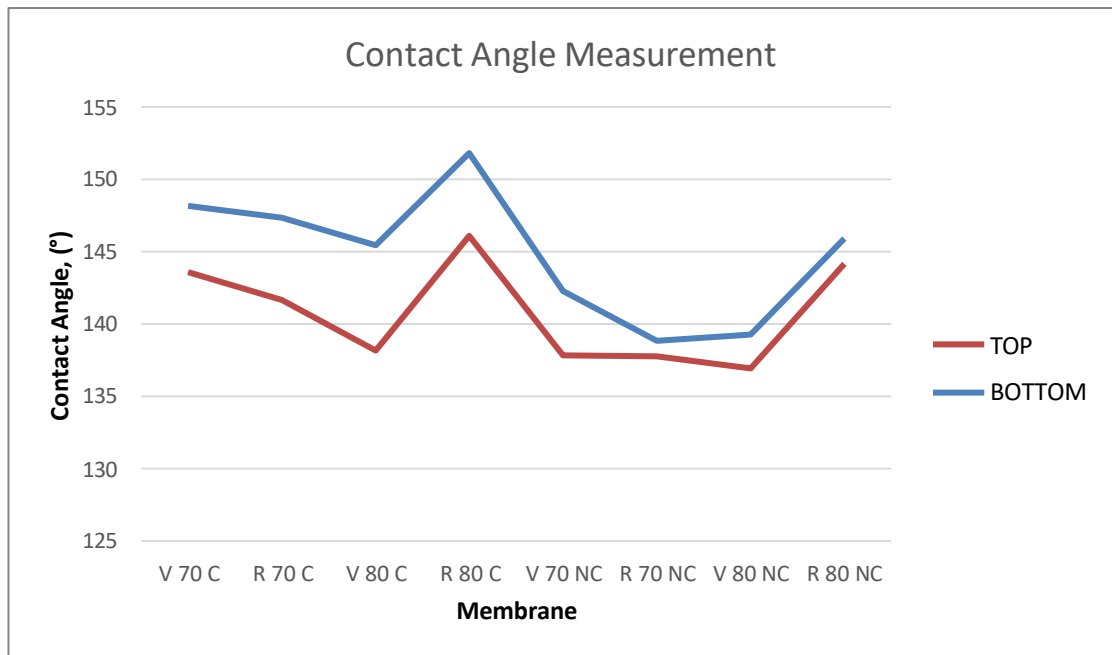
Table 4.11: Contact angle result for non-coated membranes

Material	Laser Power (Watt)	Thickness (mm)	Surfaces	Average Contact Angle (°)	Standard Deviation
Virgin	70	12	Top	137.823	5.5800
			Bottom	142.279	2.5977
Recycled			Top	137.764	5.0006
			Bottom	138.834	5.2493
Virgin	80		Top	136.927	4.2919
			Bottom	139.260	2.4733
Recycled			Top	144.147	2.3279
			Bottom	145.899	2.8689

Table 4.12: Droplet picture for non coated membranes

Material	Laser Power (watt)	Top	Bottom
Virgin	70		

		137.8°	142.3°
Recycled			
		137.8°	138.9°
Virgin	80		
		136.9°	139.2°
Recycled			
		144.1°	145.9°



- |  |   |
|--|---|
| 1. V 70 C = Virgin, LP: 70 w, Coated   | 5. V 70 NC = Virgin, LP: 70 w, Non Coated   |
| 2. R 70 C = Recycled, LP: 70 w, Coated | 6. R 70 NC = Recycled, LP: 70 w, Non Coated |
| 3. V 80 C = Virgin, LP: 80 w, Coated   | 7. V 80 NC = Virgin, LP: 80 w, Non Coated   |
| 4. R 80 C = Recycled, LP: 80 w, Coated | 8. R 80 NC = Recycled, LP: 80 w, Non Coated |

Figure 4.3: Graph of contact angle measurement

Based on this result, we can determine that bottom recycled membrane coated with 80 watt of laser power is the most hydrophobic. This result is 151.8° of contact angle measurement. Another hand, this result is included into superhydrophobic which over than 150° of result measurement. However, the worst result in this observation is top virgin membrane non coated with 80 watt of laser power.

First of all, recycled powder has higher contact angle than virgin powder. It is due to recycled powder is naturally rougher than virgin powder PA-12 due to its processing history and material properties (Martynková et al., 2021). Recycled or reused PA-12 powder tends to exhibit surface irregularities and a coarser texture

compared to virgin powder. These differences arise from the degradation and contamination that happen during the recycling process, which affects the powder's flowability and melting behavior (Pham et al., 2008). Consequently, the final product made from recycled PA-12 powder may have a less smooth finish and slightly inferior mechanical properties compared to those made from virgin powder.

Additionally, the presence of coating soot hinders the coalescence of powder during the SLS 3D printing process (Yuan et al., 2020). This interference results in a rougher membrane surface, which in turn promotes a higher contact angle. The soot particles act as barriers, preventing the powder particles from fully merging and forming a smooth, cohesive surface. Consequently, the increased roughness affects the membrane's surface area and wettability characteristics, leading to a higher contact angle. This change in surface properties can impact the performance and application of the printed material. Another hands, paraffin wax which used for coating soot, has hydrophobic characterization itself (Atta et al., 2019). Moreover, paraffin wax's nanoparticle helps increasing contact angle with loose structure in its membrane (Hussein et al., 2022).

Furthermore, the bottom surface of SLS 3D printed membranes is rougher than the top surface primarily due to the minimal energy input during selective laser sintering (Yuan et al., 2020). This reduced energy at the bottom layer leads not enough coalescence of PA-12 particles, resulting in a rougher texture compared to the top layer. The lower energy input during the printing process not only affects the coalescence of particles but also impacts the embedment of candle soot, leading to a less smooth surface.

The distribution of energy during the SLS 3D printing process is crucial in determining the surface quality of the printed membrane (Yuan et al., 2020). At the bottom layer, where energy input is minimized, the particles do not fuse as effectively, causing a rougher texture. In contrast, the top layer receives sufficient energy to facilitate better particle coalescence, resulting in a smoother surface. This differential energy distribution is a key factor in the varying surface roughness observed between the top and bottom layers of the printed membrane.

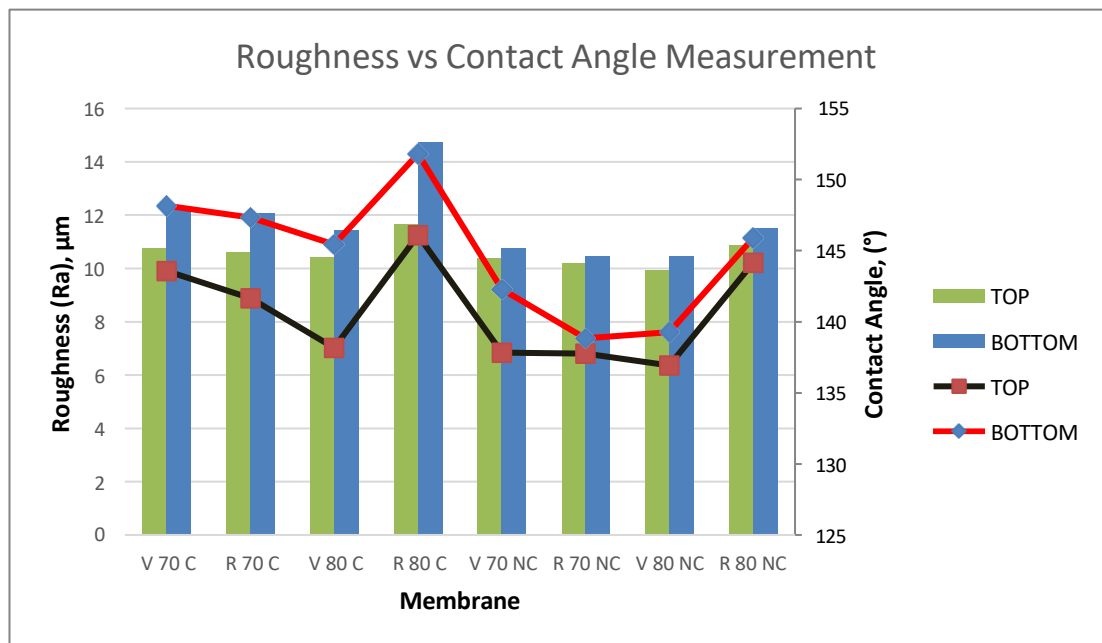
Moreover, the presence of candle soot makes worse texture and rougher of the bottom surface (Yuan et al., 2020). The minimal energy input at the bottom layer means that the soot particles are not fully embedded within the polyamide matrix, leaving them more exposed. This increased exposure of soot particles contributes to the heightened roughness of the bottom surface. On the other hand, the top layer, with higher energy input, allows for better embedment of soot particles, leading to a comparatively smoother surface. The overall roughness of the bottom surface has implications for the membrane's performance, particularly in applications where surface texture and contact angle are critical factors.

Laser power is important or crucial parameter in this case. Recycled powder needs higher temperature to be melting point (Rafi Omar et al., 2022). The recycled material will require a higher melting point depending on the previous recycling times and the increase in the amount of adhered powder. Each recycling cycle tends to degrade the material's structure slightly, causing the polymers to break down and become more susceptible to thermal stress. As a result, achieving a homogeneous melt during subsequent recycling processes often necessitates higher temperatures. Additionally, adhered powder, which may include contaminants or residual particles from previous cycles, complicates the melting process further. These substances can

create nucleation sites, leading to uneven melting and potential defects in the final product.

#### 4.5 Connection between Surface Roughness and Contact Angle

Surface roughness and contact angle should be parallel in result. Roughness on surface promotes higher contact angle measurement also (Yuan et al., 2020). Below, Figure 4.4 shows the connection between roughness and contact angle result, for both coated and non-coated membranes.



- |  |   |
|--|---|
| 1. V 70 C = Virgin, LP: 70 w, Coated   | 5. V 70 NC = Virgin, LP: 70 w, Non Coated   |
| 2. R 70 C = Recycled, LP: 70 w, Coated | 6. R 70 NC = Recycled, LP: 70 w, Non Coated |
| 3. V 80 C = Virgin, LP: 80 w, Coated   | 7. V 80 NC = Virgin, LP: 80 w, Non Coated   |
| 4. R 80 C = Recycled, LP: 80 w, Coated | 8. R 80 NC = Recycled, LP: 80 w, Non Coated |

Figure 4.3: Graph of roughness vs contact angle

As shown above, surface properties such as contact angle and surface roughness play crucial roles in determining the hydrophobicity of the membrane. Both characterizations are related to each other. Supposedly, high roughness also results in high contact angle (Yuan et al., 2017). The bottom recycled coated membrane, with an 80-watt coating, exhibits the highest contact angle and surface roughness, indicating superior hydrophobic or even superhydrophobic characteristics. This enhancement in hydrophobicity is beneficial for applications requiring effective separation of water from oil, as the increased contact angle suggests a reduced affinity for water, thus promoting better oil-water separation.

Therefore, the connection between surface roughness and contact angle is complex and influenced by several factors. The Wenzel equation and roughness corrected contact angles are key methods used to understand and characterize the relationship between surface roughness and contact angles. The wenzel equation provides a quantitative framework to understand how surface roughness influences wettability. According to this equation, the apparent contact angle on a rough surface ( $\theta'$ ) is related to the basic of contact angle on a smooth surface ( $\theta$ ) and the roughness factor ( $r$ ) by the equations

$$\text{Cos} (\theta') = r \text{Cos} (\theta) \quad (4.1)$$

Here, ( $r$ ) is the ratio of the actual surface area to the projected area. When the surface is rougher,  $r$ , becomes greater than 1, which amplifies the inherent wettability dictated by the surface chemistry.

In the case of the bottom recycled coated membrane, the high roughness factor combined with the hydrophobic surface chemistry leads to a significant increase in the contact angle, resulting in superhydrophobic behavior. This amplified

hydrophobicity is advantageous for applications like oil-water separation, where the membrane needs to repel water effectively while allowing oil to pass through.

The Wenzel equation also helps explain why the top virgin non-coated membrane exhibits lower roughness and a blunt contact angle. The absence of additional surface roughness means that the intrinsic wettability of the material is less pronounced. As a result, the contact angle remains relatively low, reflecting the smoother surface's limited hydrophobic enhancement.

Understanding the interplay between surface roughness and contact angle through the wenzel equation is crucial for designing membranes with desired wettability characteristics. By manipulating surface roughness, engineers can set membrane surfaces to achieve optimal performance for specific applications, such as creating superhydrophobic surfaces for efficient water separation. This ability to engineer surface properties through controlled roughness opens up new possibilities in various fields, including environmental engineering, biotechnology, and materials science. Each parameters has influenced itself, such as coated, laser power, bottom or top surface, and recycled or virgin powder as main topic in this case.

Furthermore, the top virgin non-coated membrane subjected to an 80-watt treatment demonstrates the lowest surface roughness and a relatively blunt contact angle. This indicates a smoother surface and less pronounced hydrophobic properties. Such membranes may not perform as efficiently in hydrophobic applications but could be advantageous in scenarios where lower roughness is required for different functionalities, such as reduced dirt in filtration processes.

The contrast between these two membranes highlights the impact of surface treatment on the performance characteristics of the membranes. The coating process,

particularly at the specified power settings, changes the surface morphology and wettability, thus setting the membrane for specific applications. Understanding these relationships is crucial for designing and optimizing membranes for targeted industrial uses, especially in sectors where liquid separation is important.

Thus, the study of these surface modifications provides insights into the mechanisms by which surface roughness and contact angle can be controlled and set, offering pathways for the development of advanced materials with requested surface properties. This knowledge not only enhances the successful of membrane technologies but also contributes to the broader field of material science, where surface engineering is key to developing innovative solutions for a range of technical challenges.

## CHAPTER 5

### CONCLUSION AND RECOMENDATIONS

#### 5.1 Conclusions

In conclusion, result of the wettability of recycled 3D printing polymer membrane with candle soot coating for oil/water separation had been conducted. This project analyzes all of characterization between surface morphology, surface roughness, porosity, and contact angle. Furthermore, all of parameter printing were also tested between top and bottom surface, laser power, coated and non coated membranes. All of the tests is aimed to give insight and elaborate material characteristics and human behaviour as operator.

Based on analysis, the application of paraffin candle soot coating influences result to be poor wettability. This poor wettability is caused superhydrophobic behavior of this membrane, appropriate with our aimed and function. On the other hands, this project uses recycled membrane that make better separation water from oil result and eco-friendly environment goal. Furthermore, printing parameters were importance to make well-recycled membrane performance. This case connects to be higher surface roughness which increase contact angle water to be superhydrophobic.

Thus, recycled membrane with coated is good choice which had good result to separate water from oil. Otherwise, operator should concern about printing parameter to make well polymer membrane.

## 5.2 Recommendations for Future Study

For future studies, several sector can be investigated more to be better result and application for separation water from oil.

- Underwater contact angle testing can be considered by using an inverted sessile drop experiment setup to understand underwater wettability behavior. Underwater contact angle testing captures the interactions between the membrane surface and water, including any changes in wettability due to submersion.
- Storage period of recycled powder should also be tested to know how much it affects wettability of membrane. Prolonged storage might lead to change in particle size distribution, impacting how the powder interacts with the membrane material. Understanding how storage affects wettability makes sure that membranes produced at different times maintain consistent quality and performance.
- Previous heat treatment of recycled powder can also influence wettability of polymer membrane. Heat treatment can cause oxidation or reduction reactions on the surface of the powder. The particle size and shape of the recycled powder change which can modify its wettability characteristics.
- Amount of cycled number reused powder is importance and should be considered in future research. The number of recycling cycles can affect the wettability of the membrane. Repeated recycling might lead to changes in surface characteristics that impact hydrophilicity or hydrophobicity, influencing membrane efficiency in applications such as filtration or separation.

- To get the best result of strength and wettability, user should be tested on the proper composition of virgin and recycled powder. Virgin powder typically provides better mechanical strength due to its unaltered structure, while recycled powder can contribute to cost savings and sustainability. Finding the optimal blend ensures that the final product maintains necessary mechanical strength while achieving the desired wettability.

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## APPENDIX A

Appendix 5.1: Gantt Chart of timeline research for PSM 2

	Activity/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	CAD Modelling															
2	Material Producing															
3	Candle soot coating															
4	Experimental Setup															
5	Surface Roughness															
6	SEM Testing															
7	Porosity Testing															
8	Contact Angle															
9	Progress Report Submission															
10	Preparation Slide Presentation															
11	Seminar PSM II															
12	Submission Hardbound Final Report															



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