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Wettability characterization of 3D printed polymer membranes with candle soot coating

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KEYWORDS

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Wettability
Oil-water separation

ABSTRACT

Wettability of a membrane plays an important role in enhancing separation efficiency and it can be assessed by determining the contact angle of liquid on a surface. In recent years, there is a lot of interest in developing hydrophobic surfaces for oil-water separation. However, implementation of candle soot on 3D printed polymer membranes has not fully explored. This study aimed to characterize the wettability of 3D printed polymer membranes with candle soot coating to improve the oil-water separation efficiency. Polyamide-based membranes were fabricated using Selective Laser Sintering (SLS) and modified with paraffin candle soot. Coated and non-coated membranes for both top and bottom surface morphology were monitored and the values of surface roughness and contact angle were recorded. Coated membranes recorded 16% and 29% higher surface roughness values for the top and bottom surfaces compared to the non-coated membranes; top and bottom surfaces of coated membranes also recorded increases in the contact angle values of 6.6% and 11.25%, respectively. Membranes' contact angle was affected by the roughness due to the coated carbon nanoparticle from the soot and printing technologies. It can be concluded, 3D printed polymer membrane with candle soot coating is an effective way in fabricating a hydrophobic membrane.

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1.0 INTRODUCTION

The membrane filtration technologies are currently being used in oil spill remediation, oily wastewater treatment and other oil-water separation processes (Ahmad et al., 2018; Rasouli et al., 2021; Ye et al., 2023). Oil-water mixtures comprise non-miscible and emulsified mixtures in the form of stable-state micro-oil-water droplets, making oil-water separation difficult. Therefore, breaking stable oil-in-water emulsions is essential to ensure high separation efficiency for emulsified oil-in-water mixtures (Yan et al., 2019). Since oil has a lower surface tension than water, it is possible to separate oil from water using porous interfacial materials with distinctive wettability (Wei et al., 2018). Currently, porous materials such as mesh-based materials (Gao et al., 2013), sponge-based materials (Kong et al., 2021), foam-based materials (Hailan et al., 2021; Li et al., 2016) and membranes (Rana and Matsuura, 2010; Yuan et al., 2017; Yuan et al., 2020) have been developed for oil-water separation. Polymer-based membranes play a key role in separating oil-water mixtures and have drawn great attention from previous studies (Pathreeker et al., 2021; Shirsat et al., 2019; Tüfekci et al., 2020) due to its advantages such as low cost, exceptional flexibility, superior processability and ease of operation (Cai et al., 2021). Polymer membranes possess smaller and more controllable pore diameters than metal meshes and textiles, making them more favourable as a medium for oil-water emulsion separation. However, the membrane manufacturing technique has been shown to cause detrimental effects on the membrane's crucial wetting properties such as low chemical stability and mechanical strength (Yuan et al., 2017; Yuan et al., 2020). Therefore, choosing the right method for fabricating the membrane specimens is important. Membranes can be fabricated through conventional and 3D printing methods. The most popular method to fabricate membranes through the conventional approach in which different kinds of polymers can be employed for various applications is phase inversion since it is straightforward and quick. Nevertheless, poor dispersion and agglomeration during the process caused by improper or excessive addition of nanofillers to the polymer matrix will deteriorate membrane formation (Jaafar and Nasir 2022).

3D printing technologies have been widely used, especially for the solventless 3D printing method, which offers flexibility to design and free form in three dimensions. To retain the best mechanical characteristics, 3D printing has been chosen as a way of creating structurally viable and porous membranes using polymer materials, especially polyamide powders. The sintering of polymeric particles can also be used to produce membranes (Remanan et al., 2018; Tan and Rodrigue, 2019). Selective Laser Sintering (SLS) offers the ability to fabricate completely functioning prototypes with similar mechanical properties as commonly manufactured parts by moulding (Martynková et al., 2021). SLS is a powder-based additive manufacturing method in which parts created from 3D Computer Aided Design (CAD) are lightweight, very robust and resistant to both heat and chemicals. During sintering, the powder will be heated to a partially melted state and then cool down to crystallise into semicrystalline solids (Xu et al., 2019). Compressing and heating particles slightly below their melting temperature induces bonding with spaces between the sintered particles becoming pores. The sintering process is very important in preparing microfiltration membranes.

In recent times, developing a superhydrophobic membrane has gained a lot of attention significantly in oil-water separation. Modifying the surface wettability of polymer-based membranes via chemical or physical is an effective method for increasing the oil-water separation efficiency (Wei et al., 2018). Coating is one of the methods that can be considered in modifying the surface properties due to its extremely flexible and adaptable materials and scale. A variety of coating processes can be used to deposit almost any material. Ye et al., (2023) developed superhydrophobic polylactic acid (PLA) nanofiber membranes using nanoscale fluorine-modified SiO2 (F-SiO2) clusters that are completely coated with low surface energy coatings, improving the superhydrophobicity of the entire nanofiber membrane resulting in above 99% separation efficiency. Yuan et al., (2017) also proposed a method to fabricate a 3D-printed polysulfone (PSU) membrane by implementing candle soot coating to enhance the hydrophobicity of the membrane which resulted in increases of the separation efficiency by 99%. Membranes comprising a rough surface structure and low surface energy coatings surpassed other porous materials in terms of separation efficiency and permeability for multiphase separation (Yuan et al., 2020). To produce hydrophobic particles, no other simpler and cheaper option is available than using candle soot. Candle soot is one of the alternatives that can be created directly via a bottom-up synthesis pathway from the incomplete combustion of a paraffin candle flame. Many superhydrophobic surfaces based on candle soot have been developed recently (Hussein et al., 2022; Li et al., 2017; Sun et al., 2022; Zhang et al., 2021). As a carbonaceous material with potential morphology, candle soot adapts itself well to the development of superhydrophobic surfaces (Song et al., 2022).

The term wettability refers to the interaction of a liquid with a solid surface (Prajitno et al., 2016). Wettability can be characterized by evaluating the surface's contact angle with a specified liquid (Frota et al., 2022; Jung and Bhushan, 2009; Juuso and Robin, 2018; Kung et al., 2019; Tanaka et al., 2013). Young's equation listed in Equation (1) below describes the contact angle of a liquid on a solid surface for three-phase interfaces.

$$\cos\theta = \frac{\gamma_{sv} - \gamma_{sl}}{\gamma_{lv}} \tag{1}$$

Where θ is the contact angle and $\gamma_{sv}, \gamma_{sl}, \gamma_{lv}$ represents surface tensions of solid-vapor, solid-liquid, and liquid-vapor interface respectively. The surfaces' wetting behaviour is defined by two categories either phobic (repelled) or philic (attracted) (Abbas, 2018). The wetting behaviour can be distinguished by determining the contact angle through the measurement of liquid on a solid interface. If θ is between 0° and 90°, it indicates the hydrophilic conditions where water is attracted to the surface. Meanwhile, if θ is between 90° and 150° it refers to hydrophobic conditions where water is repelled from the surface. For a contact angle more than 150°, a surface is considered superhydrophobic.

This paper aimed to characterize the wettability of printed polymer membranes coated with candle soot. Polymer membranes were fabricated using the SLS method and followed by surface modifications by physically coating the printed membrane with candle soot. The wettability characterization included surface roughness and contact angle measurement, and morphology analysis. This work highlighted the use of membranes for the oil-water separation process.

2.0 EXPERIMENTAL PROCEDURE

2.1 Materials Preparation

Virgin Polyamide-12 (PA12) powder from Farsoon Technologies (China) was used to fabricate the specimens. The paraffin candle soot particles were gathered by positioning a clean metal plate at a 2 cm on top of the mid-flame region for 10 minutes. The candle soot that had accumulated on the metal plate was then scraped off and kept for membrane modification.

2.2 Fabrication Process

SLS Farsoon FS402P printer was used to fabricate 3D printed membranes. Figure 1 illustrates the mechanism of the SLS printing. The laser scanned the specimens of 5 cm circular diameter and 2.5 cm length square shapes with 1 mm thickness. Before the CO_2 laser exposure, the powder bed was preheated to 169.5 °C. The layer-by-layer printing followed the layer thickness that had been set in the printer based on 3D CAD drawing data of the specimens. The parameters configuration shown in Table 1 was used in specimen fabrication.

Table 1: SLS printing parameters configuration.

Parameters	Configuration values
Laser Power	70 Watt
Slicing Thickness	0.12 mm
Laser Beam Velocity	7.6 m/s
Chamber Temperature	169.5 °C

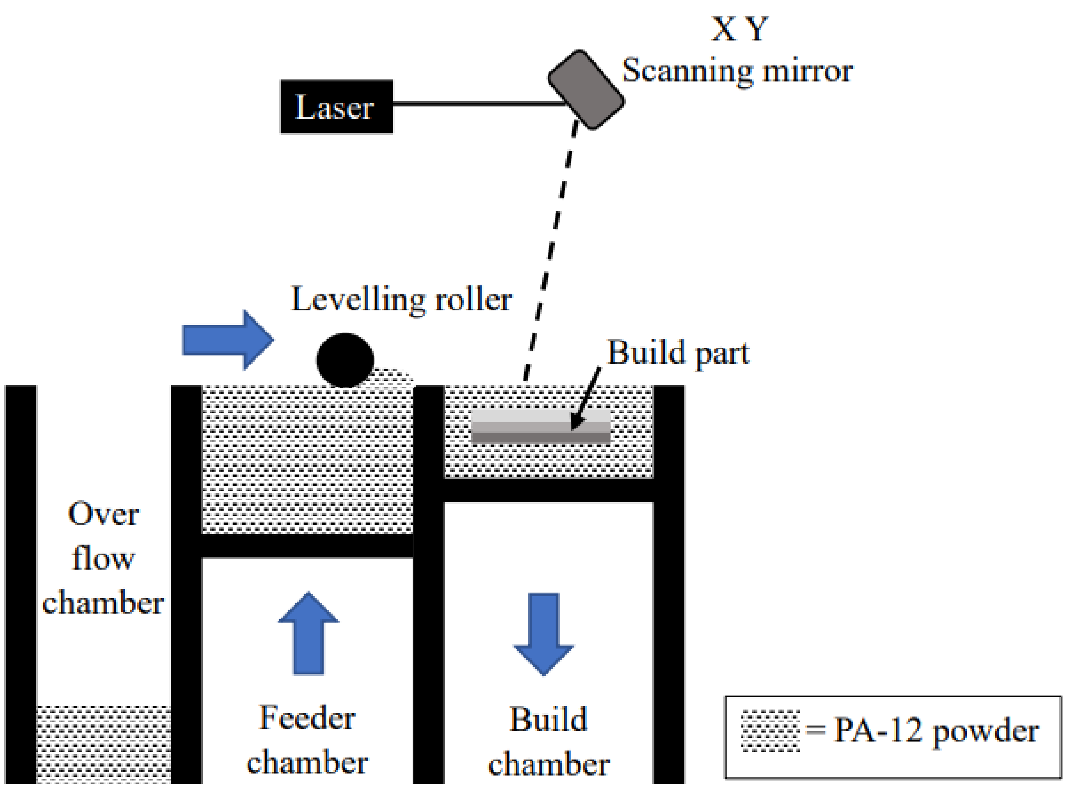

Figure 1: The SLS schematic diagram for Farsoon FS402P 3D printer.

2.3 Membrane Modification

The process of candle soot carbon coating on the printed specimens was done after the specimens were printed. As shown in Figure 2 (a), 0.04 g of candle soot was dispersed in 40 ml hexane under sonication using the ultrasonic bath cleaner for 30 minutes in a sealed bottle to form a candle soot/hexane solution (0.1 wt%). Then, the sonication process continued with a PA-12 membrane specimen immersed in the candle soot/hexane solution for 40 minutes. The white printed PA-12 membrane turned black which indicated the carbon candle soot particles have been deposited and coated the membrane as shown in Figure 3. The coated PA-12 membrane was then placed in an oven at 60°C for 10 minutes for drying.

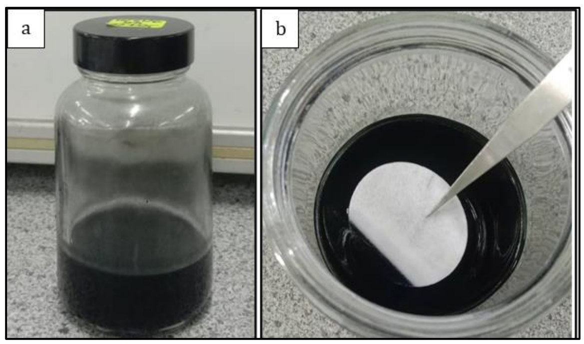

Figure 2: (a) The mixture of candle soot/hexane solution; (b) printed specimen immersed in the solution.

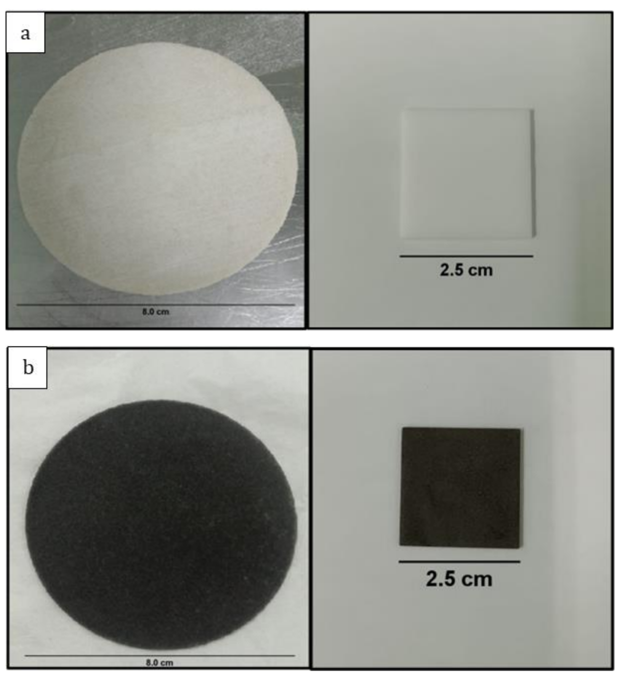

Figure 3: 3D printed polymer membrane specimens (a) before coating and (b) after coating.

2.4 Characterization

There are four different types of specimens were prepared for this study as shown in Table 2.

Table 2: Membrane specimens ID.

Specimens ID	Description
CMT	Coated Membrane Top
CMB	Coated Membrane Bottom
NCMT	Non-Coated Membrane Top
NCMB	Non-Coated Membrane Bottom

2.4.1 Surface Roughness Testing

Average surface roughness (*Ra*) was measured on every printed specimen using the surface roughness tester Mitutoyo SJ-410 with a travelling length of 5 mm; ISO 4287: 1997 was followed for the surface roughness measurement. The reading was taken at three different positions on a 5 cm diameter specimen. Both the top and bottom surfaces of each specimen were measured, applicable for coated and non-coated membranes.

2.4.2 Surface Morphology Examination

Surface morphology of the printed polymer membrane was examined using Scanning Electron Microscope (SEM) JEOL 6010 PLUS with a magnification setup of 500µm (50x) and 100µm (200x). The image was visualized with Electron High Tension (EHT) of 5kV in Secondary Electron Images (SEI) mode.

2.4.3 Contact Angle Measurement

Contact angle measurement was conducted using self-fabricated contact angle measurement equipment by implementing the sessile drop method. Water droplets of about 5 µL in volume with a spherical droplet radius of about 1 mm in the atmospheric environment were gently deposited on the specimen using a micropipette. Before measuring the contact angle, the stabilization time of 15 seconds was given before measurements can be started. Five data points were collected at five different positions on the specimen surface and the average values were calculated. The droplet image was taken using the Digital Viewer software and Image J software evaluated the contact angle for the specimens.

Figure 4 : The self-fabricated contact angle measurement set-up.

3.0 RESULTS AND DISCUSSION

3.1 Surface Roughness and Morphology

Figure 5 shows the measured mean values of surface roughness, *Ra* were in the range of 9-16 μm for three position measurements on both coated and non-coated surfaces. The measurement was repeated in three positions to obtain average values for each type of specimen. From Figure 5, NCMT specimen has the lowest value of surface roughness obtained in this study with a total roughness of 9.97±1.0592 μm while the CMB specimen has the highest values of surface roughness of 14.364±1.1227 μm. This indicated that the top part of the non-coated membrane has the finest surface of all specimen types while the bottom part of the coated membrane has the roughest surface.

Referring to Figure 5, the top surface of the coated membrane, CMT has approximately 16% higher mean average roughness compared to the non-coated membrane, NCMT; similar trend can be seen for the bottom surface where CMB has 29% higher roughness compared to the NCMB. The roughness of coated membranes increased as the carbon particles from the candle soot deposited on the membrane and covered the membrane. Candle soot particle sizes are approximately 30 nm (Shooto and Dikio, 2011) which consisted of about 91% of carbon and contained some amounts of hydrogen, nitrogen, oxygen, and other insoluble hydrophobic compounds (Wei et al, 2017). As previously mentioned by Sahoo and Balasubramanian (2014), utilizing the candle soot particles as a coating can increase surface texture and roughness of the polymer as well as its crystallinity. Carbon particles formed by incompletely burning paraffin, which gradually accumulated on the substrate and resulted in a rough surface have been composed of candle soot (Chen et al. 2022; Thamaraiselvan et al. 2021). Iqbal et al., (2017) discovered that candle soot particles embedded in a mixture of PDMS+n-hexane exhibited nanoscale surface asperities with an average roughness of 187nm which increased in surface roughness and resulted in very high water repellency. In addition, the SEM results shown in Figures 6 and 7 found that the non-coated membrane exhibited more melting and fewer pores on the surface compared to the coated membrane for both top and bottom. Thus, more melting and fewer pores on the surface resulted in lower average surface roughness (*Ra*)(Omar et al. 2022; Yuan et al. 2017).

The top and bottom surfaces have distinct results for both coated and non-coated membranes. The roughness values of the bottom surface specimens for both coated (CMB) and non-coated membranes (NCMB) membranes were higher compared to the top surface specimens, CMT and NCMT; this is due to the variation of energy absorbed by both surfaces during the sintering process. Sintering requires a particular amount of laser power because of the laser-powder interaction, which varies depending on the layer thickness (Golhin et al. 2023). It is noteworthy that the energy distribution from the laser power during the printing process influences the surface quality of the printed parts. The surface of the powder bed absorbs the greater energy from the laser during selective laser sintering, generating the top layer of the membrane and is substantially reduced as it goes down to the powder bed. The remaining laser power leads to the partial melting of polymer particles at a specific depth in the powder bed, which results in deficient coalescences of the particles at the bottom layer of the membrane; this is because the full amount of laser energy is incapable to go through into the deeper depth of the bed. The minimal energy received by the bottom surface of the membrane results in a rough surface compared to the top surface of the membrane. The polymer type, particle size, morphology and density of the powder bed may influence the value of the minimal energy (Yuan et al., 2020). Yuan et al., (2017), mentioned that the roughness of both top and bottom surfaces for the PSU membrane was different as the water contact angle for the bottom surface was larger than the top. In addition, Kingman and Dymond (2022)discovered the surface of the top and bottom showed a significant difference in voids sizes left between the extruded filaments developed during the fabrication process using fused filament fabrication methods utilized PLA filament. The bottom part exhibited likely larger voids compared to the top part which affected the wetting properties. The printing process could affect the roughness of both sides of the printed parts. Figures 6 and 7 displayed the differences in surface morphology for both the top and bottom surfaces respectively in which NCMB has a rough surface due to the coalescence behaviour of the PA-12 powder during the printing process.

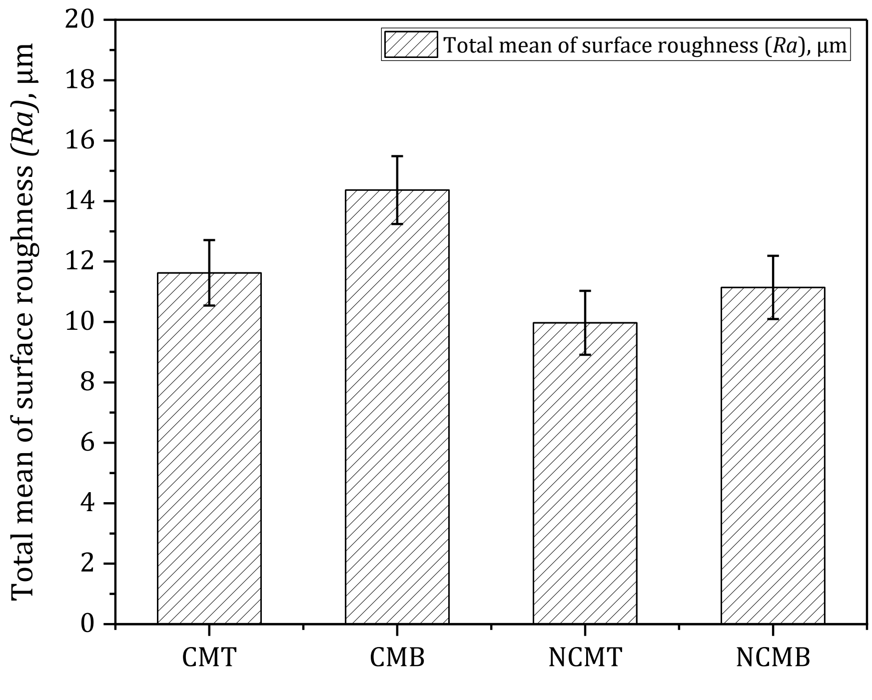

Figure 5: The total mean of surface roughness value for all specimens.

Figure 6 (b) and Figure 7 (b) displayed the initial condition of the PA-12 membrane surface that originally exhibited the formation of pores due to the printing method. Pores can be generated by the insufficient coalescence between polymer particles in a membrane created by SLS (Yuan et al. 2017). However, Figures 6 (a) and Figure 7 (a), show that the implementation of candle soot coating amplifies the pore sizes on the membrane surfaces. Yuan et al., (2020) discovered that more porous structure generated on the coated membrane due to the candle soot coating layer, which inhibited the coalescence of molten polyamide particles. Overall SEM analyses in Figure 6 illustrates the sizes and number of pores on top surface are smaller and fewer to be compared the bottom surface as shown in Figure 7 for both coated and non-coated membranes, resulting in a smoother surface.

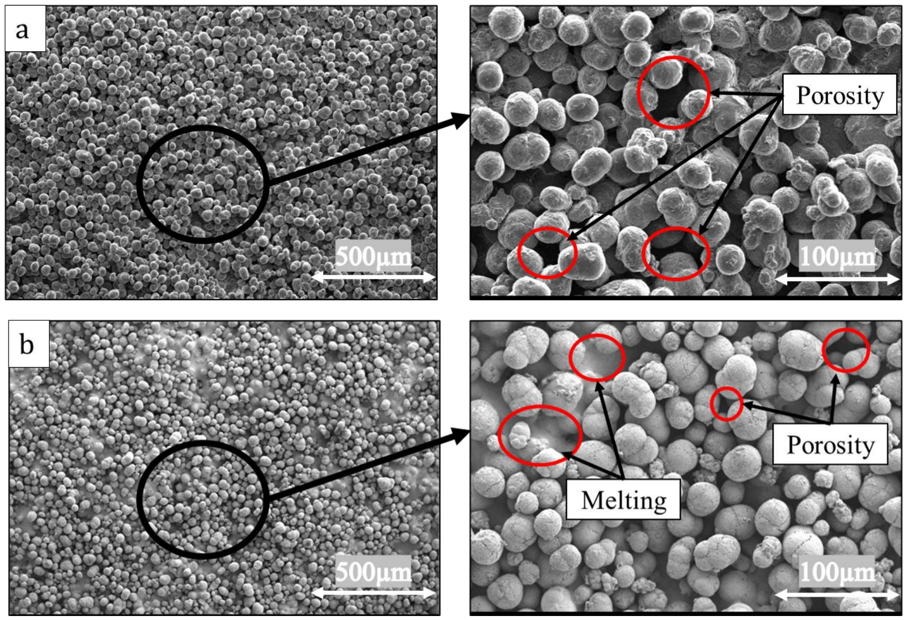

Figure 6: The morphology of membrane surfaces under SEM for top surfaces (a) coated membrane (CMT); (b) non-coated membrane (NCMT).

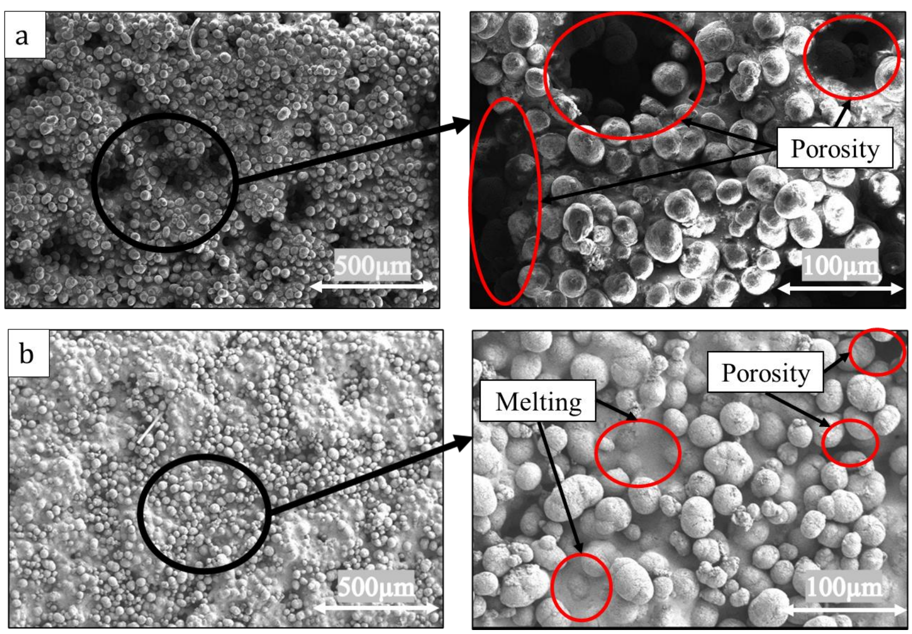

Figure 7: The morphology of membrane surfaces under SEM for bottom surfaces (a) coated membrane (CMB); (b) non-coated membrane (NCMB).

3.2 Contact Angle Measurement

In determining the wettability of the membrane, water contact angle measurement was evaluated for top and bottom surfaces of both coated and non-coated membranes. In the oil-water separation process, the hydrophobicity of a membrane is a crucial factor. As presented in Figure 8, the water contact angle for all of the specimens was above 90° which has resulted in a hydrophobic behaviour. The printed polymer membrane has the capability to produce a hydrophobic nature which can be utilised in the oil-water separation process. However, the modification of the membrane is important to improve the feasibility of the oil-water separation by enhancing the hydrophobicity.

The average water contact angle values in Figure 8 represent the wettability of the top and bottom surfaces of the PA-12 membrane, both coated and non-coated. The coated membrane's water contact angle was higher than the non-coated membrane's. The candle soot coating is composed of carbon-containing carboxylic species, branching saturated hydrocarbons, methylene, and methyl groups (Mulay et al., 2019). Hydrocarbons are naturally nonpolar and the development of the hydrophobic effect was created by the ability of hydrocarbons to resist water molecules; a hydrocarbon chains promote low surface energies (Alexander et al. 2016) and form a barrier that prevents water from passing through.

For the water contact angle comparison, for the top surface of the coated membrane (CMT) had 6.6 % higher contact angle than the non-coated membrane (NCMT); the trend is similar for the bottom part which showed coated membrane has 11.25 % higher contact angle. It is proven that the use of the carbon nanoparticle from the candle soot coating on the membrane can enhance the hydrophobicity which make it possible to reach superhydrophobic. In addition, there were distinct values for the top and bottom surfaces, of both coated and non-coated membranes. For the non-coated membrane, the water contact angle for the bottom surface (NCMB) was larger than the top surface (NCMT), same as for the coated membrane. It can be seen the contact angle also can be affected by the texture and roughness of the surfaces. Jothi Prakash and Prasanth, (2021)reported that the surface characteristics such as surface roughness, surface energy, and porosity, all have an impact on liquid wettability on the surface in terms of the liquid contact angle values.

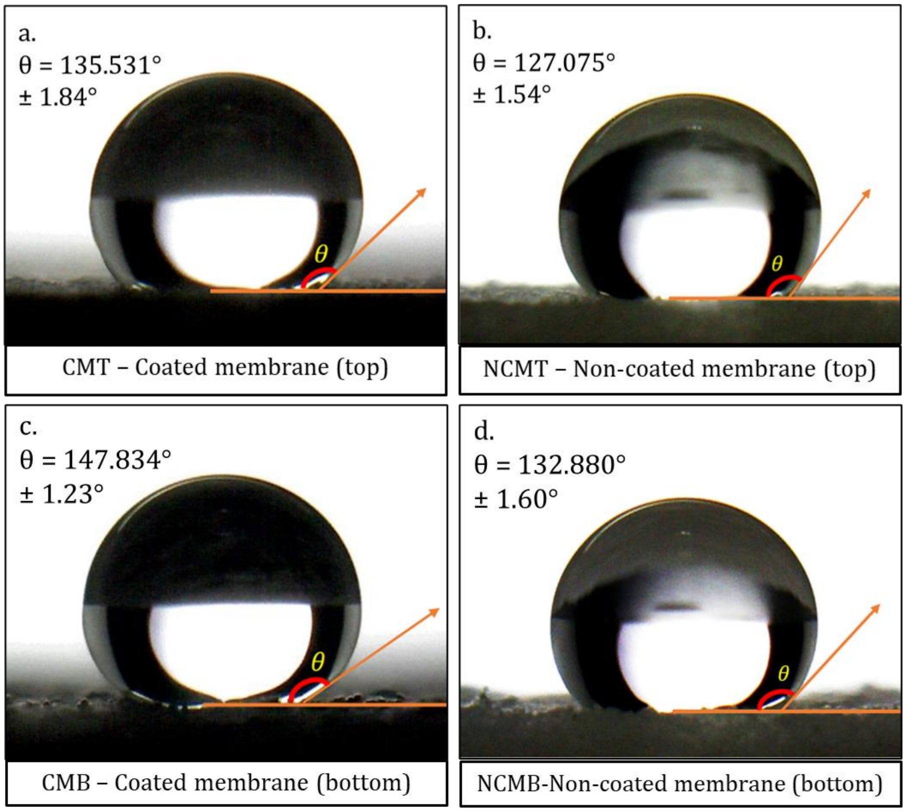

Figure 8: The mean values of water contact angle for top surface (a) coated membrane (CMT) and (b) non-coated membrane (NCMT) ; For bottom surface (c) coated membrane (CMB) and (d) non-coated membrane (NCMB).

3.3 The Effect of Surface Roughness and Contact Angle on Wettability

A correlation study was performed to determine contact angles and surface roughness of non-coated and coated surfaces as well as for both top and bottom surfaces, with the result shown in Figure 9. According to the results, the contact angle of the membrane was influenced by the surface roughness. The higher the roughness, the higher the contact angle which results in low wettability. The roughness of the surface was significantly affected by the printing process which different energy received by top and bottom surfaces. Besides, the roughness of the membrane was also affected by the modifications that had been made by the deposition of the candle soot coating. Since the candle soot layer comprises hydrocarbons with low surface energy, molecules in water droplets are more attracted to each other than to the surface, resulting in poorer wettability.

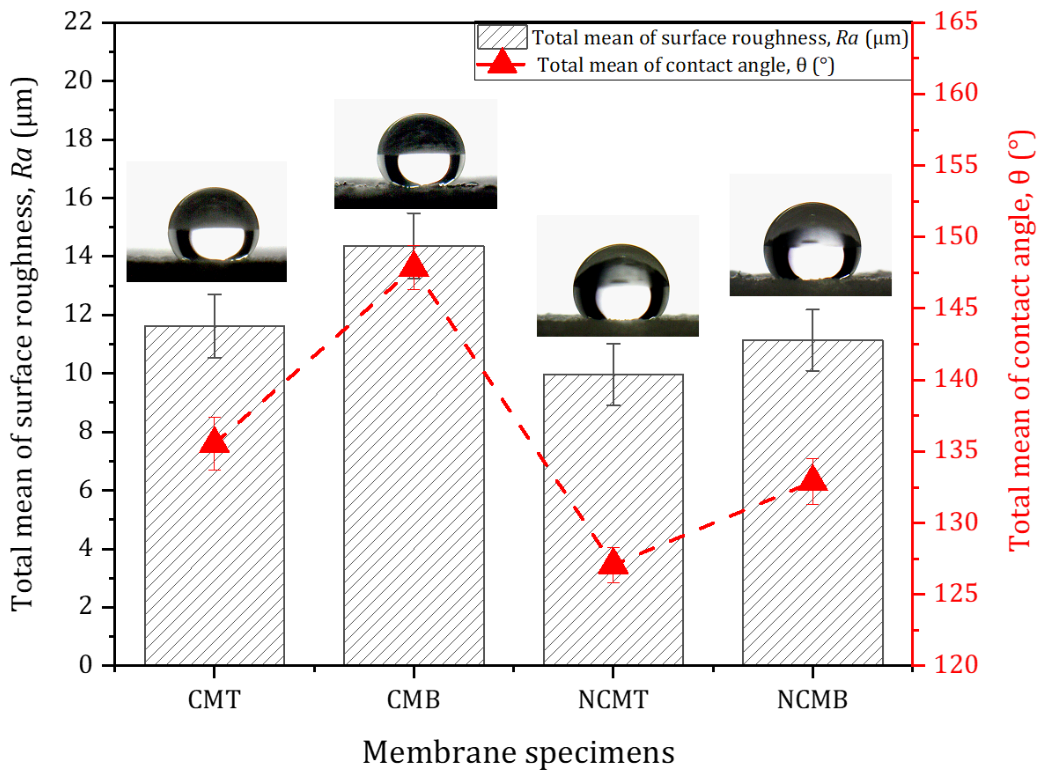

Figure 9: The correlation between surface roughness and contact angle.

Surfaces with low wetting properties will allow oil/water mixture to separate the substances which oil will go through the surface while the water will be repelled. The surface's hydrophobicity increases, resulting in a larger contact angle. As mentioned by Saji (2021), higher surface roughness, nano/micro-hierarchical surface structures, surface reduction procedures (removal of hydrophilic surface groups) and additional low surface energy treatments can enhance hydrophobicity which results in higher contact angle values.

CONCLUSIONS

The effective and economical application of candle soot coating in this study is shown to improve the hydrophobicity which enhances the separation efficiency of oil/water mixtures. The deposition of candle soot coating on the membrane increases the water contact angle and the hydrophobicity of the membrane until almost approaching the superhydrophobic behaviour. As a result, combining the functional candle soot particles with the 3D printed polymer membrane is an attractive approach to produce a membrane with high separation performance. However, this study also discovers that the top and bottom surfaces for the 3D printed polymer membrane had different roughness which results in different wettability behaviour. The higher the surface roughness, the higher the water contact angle, contributing to low wettability which could potentially exhibit a superhydrophobic surface. From the results obtained in the study, the coated membrane with the bottom surface had the highest mean values (147.834°±1.23°) for the contact angle indicating the best performances for oil-water separation compared to the other types of specimens. Thus, it can be concluded that both roughness and surface modification are important factors that can affect the wettability of 3D printed polymer membranes.

Previous findings by Barrios and Romero (2019) the variation of printing setting also can affect surface roughness. In future works, the effect of printing parameters can be investigated in combination with the application of candle soot coating to further improve the wettability of 3D printed membranes. This discovery shall open the way for a greater opportunity for the involvement of candle soot particles in the fabrication of functional 3D printed polymer membranes, especially for oil-water separations.

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