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# A Review on Surface Characterization Techniques of Polymeric Membrane in Forward Osmosis

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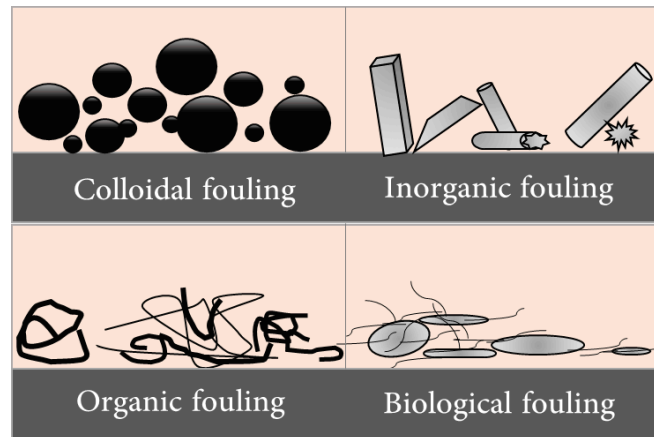
**Abstract.** Forward osmosis (FO) is an emerging osmotic process that has been extensively investigated in the past decade. There are many literatures, discussing on the available methods in producing FO membranes as well as researches that emphasis on the type of FO membranes. The quality of the FO membrane often valued based on its intrinsic parameters and its morphological characteristics. Unlike the intrinsic parameters, the surface characteristics regularly described in adjectives thus it is hardly to be consistent. This paper presents a comprehensive review on surface characterization specifically for FO membranes. Regardless the types of FO membranes, the surface characterization comprises of FTIR, XPS, FESEM/SEM, AFM and contact angle were discussed in the way that how the findings should be reported. Thus, through this work, the relevant discussion that should be measured and included when deliberating the analysis result for FO membranes is presented. Insufficient and misinterpreted characterization data might have not changed the novelty fact of the research, yet it may lead to lower its impact. A strong connection between the intrinsic values and surface characteristics would have been compromised as well.

## 1. Introduction

Forward osmosis (FO) is an emerging technology that promises to provide a sustainability of the global water supplies and is currently drawn considerable attention in the membrane process researches. This is probably because FO process does not require high pressure during operation as it works based on the naturally occurring process where water permeate across the membrane driven by the osmotic gradient between two solutions with significantly different concentrations. Therefore, the process consumed very low energy for water production [1]. As no hydraulic pressure is applied in FO, the potential of the membrane fouling can be reduced [2]. Nevertheless, fouling is considered significant issue in FO as it is also contributed to the lower water flux. Generally, in a pressure-driven process, fouling was categorized to four different classes namely colloidal fouling, inorganic fouling, organic fouling and biological/microbial fouling as depicted in Figure 1 [3]. Colloidal fouling is typically described when there are particles or colloidal deposition on the membrane surface while organic fouling is the result of the adsorption of macromolecular organic compounds that is usually found in natural organic matters (NOM). Whereas, inorganic fouling is caused by the accumulation of inorganic precipitates that initially forms scales on the membrane surfaces and then followed by the



crystallization and crystal growth [4]. The adhesion and accumulation of the microorganism at the surface of the membrane generates a biofilm that known as biological fouling.



**Figure 1** Schematic illustration of four different types of fouling. Redrawn from [4, 5]

Similarly, all the fouling mechanisms in pressure-driven process also occurring in FO except that it might be occurring at both surfaces; the active layer and support layer [6]. This is because in FO process, the direction of the flow involved two ways which is the water flux from feed solution (FS) towards draw solution (DS) and the solute flux from DS towards FS. The flow between both sides of the membrane in FO process shows great non-linearity behavior compared to pressure-driven process in regard to the permeability of the membrane and the osmotic pressure difference between the two solutions (FS and DS). There seems to be some evidence to indicate that internal concentration polarization (ICP) is the reason of the hindered linearity in the mass transport of the FO process [7-9]. Tiraferri et al, have previously examined the water flux and the solute flux in FO process obtained from various types of membranes including hand-cast thin film composite (TFC) membrane, commercial (cellulose triacetate (CTA) from Hydration Technology Innovation) FO membrane, TFC (SW30, Dow Chemical Company, Midland, MI) reverse osmosis membrane and TFC FO membrane (from Oasys Water Inc., Boston, MA) [10]. In their analysis, they found that the water flux for the membrane (that is specifically produced for FO process) which is hand-cast TFC, CTA FO and Oasys TFC FO are greatly high as compared to the TFC SW30 (reverse osmosis) membrane.

The concentration polarization (CP) commonly found in pressure-driven membrane where it exists at the interface of the membrane and the solution. This CP is referred to external concentration polarization (ECP) as it mainly accumulates at the surface of the active layer (denser layer) [11]. On the other hand, ICP basically the results of accumulation or buildup of solutes that creates a concentration barrier between the two solutions inside the porous layer of the membrane in which it highly affecting the osmotic pressure difference [12]. Considering the osmotic pressure difference is the driving force of the FO process, it is fair to say that the existence of ICP is significantly distressing the water flux, hence the efficiency of the membrane process. Thus, developing a membrane with less porosity in support layer for FO process is necessary. Therefore, many researches were reported in innovating more practical membranes for FO via varieties of methods including all available surface modification methods, layer-by-layer method, and not so long ago introduced biomimetic methods [13-17]. Prior to understand the ideal characteristics of produced membrane, it is necessary to recognize the fouling issues and mass transport inside FO process. The ideal membrane for FO ought to have minimal porosity for the support layer, highly hydrophilic, mechanically strong enough for reasonable pressure tolerance, anti-fouling, very thin and stable in contact with FS and DS [18]. The

membrane characteristics is typically observed through various surface characterization methods and instruments.

Although the surface characterization is widely adopted almost in every research, but the complete discussion on the characteristic values often not provided. This paper focuses on the comprehensive review of the surface characterization method during fabrication and/or modification of the FO membrane. Various methods used to fabricate and modify membranes for FO process will be revised followed by the discussion on discrete membrane characteristics and surface characterization methods in FO. Specifically, the appropriate way to deliberate the surface characterization method is addressed. By highlighting these proper discussions, a vital information to guide researcher to extant complete explanation of the data in their works is presented.

## **2. Fabrication and modification methods of FO membrane**

Due to various advantages offered by FO technology, many researchers are interested innovatively to prevail and vanquish its limitation and improve its efficiency [19-22]. Practically, an FO membrane must consist of a very thin and dense active layer together with more porous layer also known as a support layer. Since 1960s, the usage of cellulose acetate (CA) and CTA based FO membrane have been extensively studied and commercially available for FO application regardless of the modules [23, 24]. These membranes were fabricated through phase inversion, a technique that can produced very thin and fine membrane layer thus providing very low structural parameter (S) value for the membrane which make them perfectly suit for FO membrane. Currently, the commercially available FO membrane is developed by the Hydration Technologies, Inc. (HTI) made of the CTA [25]. However, it is poor in water permeability and have a low percentage of salt rejection Li, Yan [26], [27]. It is also can easily degrade to certain draw solution compositions [28].

The cellulose-based membranes were dominant till the initiation of the TFC membranes in 1970s [29-32]. TFC is another type of membrane that is fabricated for FO applications. Commonly, it involves interfacial polymerization techniques on the RO and NF based membranes specifically on polysulfone (PS) or polyethersulfone (PES) [33-36]. It is either coated on hand cast membrane or commercial membrane. The crosslinked process responsible to increase the selectivity level of the TFC membrane specifically for the active layer while making sure the support layer remains intact. Nevertheless, the existence of support layer has caused the ICP issue in TFC FO membranes [37]. Therefore, a low structural parameter (S) value in TFC FO membrane will reduce the ICP and thus increase the efficiency of the membrane [38]. Moreover, TFC is still the current growing membranes fabricated for FO applications [39]. Table 1 presented the summary of the most recent TFC-FO membrane with its intrinsic parameters. An alternative technique using nanofibers by means of an electrospinning technique has also been previously discussed [31]. This technique provides higher permeability and low tortuosity membranes, thus a better version of TFC FO membrane [40].

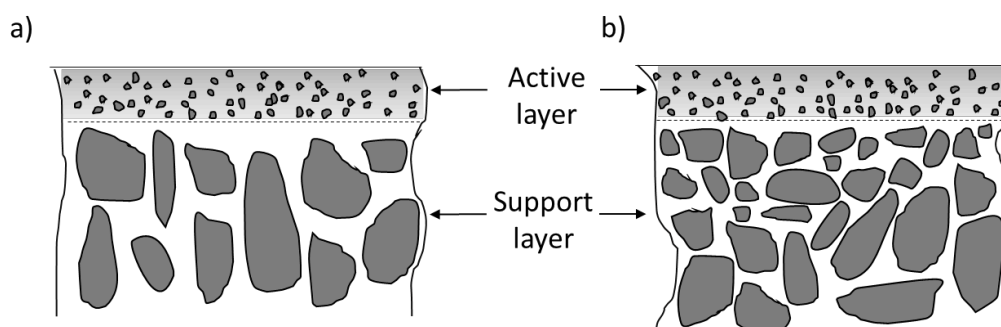
As far as the ICP issues is highly concerned, no solutions to this issue has been found so far. A potential way to auxiliary this membrane process is by using polyelectrolytes-based membranes [14]. Over the last decades, the study on polyelectrolytes membranes have been broadly investigated [41-43]. This type of membrane is usually consisting of a layer-by-layer (LbL) deposition of cationic and anionic electrolyte-films onto a membrane support. Common polyelectrolytes employed for LbL membrane fabrications are chitosan (CS), poly (sodium styrene sulfonate) (PSS), poly(ethyleneimine) (PEI) and poly (acrylic acid) (PAA) [44-46]. The LbL polyelectrolyte membranes are high in selectivity and flux [14]. Moreover, they have great solvent resistance and thermal stability [14, 42]. However, it is found that it is difficult to assemble the membranes through LbL method in large scale hence limit the option for industrial applications [32].

**Table 1** Summary of current fabricated TFC-FO membranes

Membrane	Water Permeability (LMH bar <sup>-1</sup> )	Solute Permeability (LMH)	Structural Parameter (μm)	Ref.
Sulfonated poly (ether ether ketone) casted- PSF TFC	2.16	0.16	191	[47]
Nanofiber Polyamide TFC	3.68	0.32	340	[48]
Carbon Nanotube TFC-FO	2.45	0.119	126	[49]
PES TFC-FO	1.93	0.573	324	[49]
Polyamide TFC	1.94	0.52	289	[50]
Polyimide microporous nanofiber	15.9	28.5	350	[39]

Another potential way to circumvent the problem of ICP is surface modification, whereby grafting a hydrophilic monomer onto the hydrophobic or less hydrophilic monomer [51, 52]. In general, there are three ways of modification processes in membrane namely bulk modification, surface modification by physical methods and chemical methods [53]. Over the past century, there has been a dramatic increase in the surface modification of the membrane compare to bulk modification. This probably because the modification of bulk membrane will involve the whole membrane layers which in the case of enhancing the hydrophilicity of the active layer, the support layer will also increase in hydrophilicity and thus become swelling and plasticize or it could swell differently in different substrates hence provide less mechanical stability [54]. Despite varieties of membranes surface modifications, grafting seems to be the simplest modification techniques. Several methods for membrane surface modifications are including chemical, photochemical, plasma, enzymatic and UV grafting [55].

To date, the most available membranes in industries are fabricated for pressurize membrane processes and typically comes with asymmetric structure [56]. The asymmetric membranes are usually consist of a thin active layer, backed by thicker layers of porous polymer and fabric support [57, 58]. Figure 2 presents the comparison between the membrane structure for FO membrane and pressurized RO/NF membrane. Apparently, the structure of pressurize membrane (RO/NF membrane) is thicker and more tortuous than the FO membrane which has been the main reason why the ICP issues in pressurize membrane is very significant [9]. It has been reported that it performs poorly when used for FO process undoubtedly because of the boundary layers that build up within the supporting layers [59].

**Figure 2** Schematic illustration of skinned asymmetric a) FO membrane and b) RO/NF membrane structures [2]

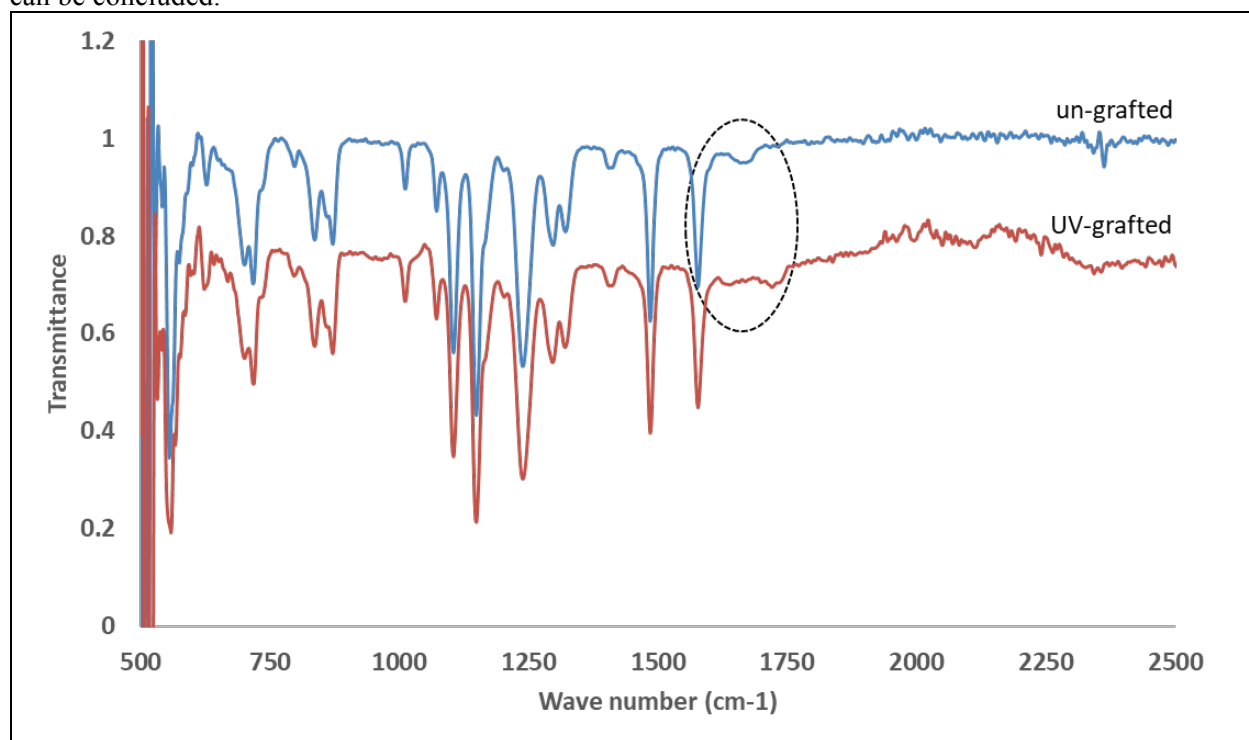
Apart from that, several attempts applying the commercial RO membrane in FO system shows depressing results as a very low flux reported [28, 60]. The same performance of FO was also obtained earlier through RO asymmetric membrane and main issue of the ICP occurrence in which is related to the thickness of the membrane is identified [7, 61]. Similarly, the performance of the FO were too low when the modified commercial NF membrane is employed in the system [62, 63]. Considering this issue, significant flux was obtained when the fabric support layer of polyester (PET) were removed from the commercial RO membrane [10, 64]. Moreover, the effective structural parameters observed on the removed-PET membrane were found to be very small compare to the membrane with PET support. Since the fact that abundance of available commercial NF/RO membrane in industries, there seems to be some suggestion to indicate that enhancing the surface hydrophilicity of the pressurized-based membrane leading to a great opportunity in solving the issue of FO membrane design [65, 66]

### 3. Characterization of the functional groups (FTIR)

The common techniques employed for surface characterization are Fourier transform infrared spectroscopy (FTIR). This method is probably the utmost prevailing analytical techniques for the clarification of the structure, composition and functional groups of polymer surfaces including membranes [67]. The results of the FTIR analysis are usually presented in the graph figures with either absorbance or transmittance as a function of the wavelength. Frequently, if the membrane is fabricated for FO application, the spectrums of the chemical functional group must first be known and thus it can be directly spotted on the peaks of FTIR results [68]. Whereas in the case of membrane modifications, the functional groups are typically interpreted based on the structure and number of the peaks exist in the modified sample [67]. The infrared characteristics of the unmodified sample are first to be figured, thus, new peaks on the modified sample can be easily noticed. For instance, in the case of UV-grafting of the commercial asymmetric PES NF membrane with N-vinyl pyrrolidone (NVP), the amide group of C=O is one crucial criterion in defining the successful grafting of NVP monomer on a PES substrate. The amide group is presented in the range between 1640-1690  $\text{cm}^{-1}$  [69, 70]. As the remarkable functional group of the new monomer is determined, it can be easily marked on the modified spectrums as shown in Figure 3 while compare it to the spectrum of unmodified PES. Else, the common spectrum of the PES membrane can be referred from Table 2 though the new peaks that outstandingly exist after the modification can be spotted.

In an investigation on modified polyvinylidene difluoride (PVDF) nanofibers for FO process, [71] presented both the peaks of FTIR spectrum for unmodified PVDF and the nylon 6,6 deposited PVDF together with the nylon 6,6 itself. The new peaks were observed at 1661 $\text{cm}^{-1}$  and 1544 $\text{cm}^{-1}$  exhibit the peaks for C=O stretching of amide and C-N stretching of amide II respectively. The observed peaks are very cleared, and it represent the new functional group of the modified fibers as the evidence of successful integration between nylon 6,6 and the PVDF support [71]. Another way of presenting convincing FTIR results analysis were shown by Emadzadeh et al. [72] where they incorporated titanium dioxide ( $\text{TiO}_2$ ) on the polysulfone matrix. They have included the spectra for pristine PSf and PSf substrate together with the PSf- $\text{TiO}_2$  nanocomposite membrane. These provide substantial evidences for the existence of Polyamide (PA) on the membrane where both pristine PSf and PSf substrate shows no indication of the characteristic peaks. Nevertheless, in some cases the obtained peaks could appear very indistinct or almost not appear, thus does not signify the exact peaks that were expected. For instance, the FTIR spectrum that were presented by [73] where they compare the spectrum for unmodified polyacrylonitrile (PAN) membrane with the mixed matrix membrane of silica gel deposition on PAN membrane. Although, they claimed that a new peak was observed at 1050 $\text{cm}^{-1}$  which represents the Si-OH stretching, the peak was rather the increase in intensity of the existed peak as it is also observe at very low intensity on the unmodified spectrum. Thus, if any changes are to be expected on the spectrum of the membrane after the modification has been

implemented, one must know what are the expecting spectrum that might appear, hence proper idea can be concluded.



**Figure 3** FTIR spectra of the pristine NF PES membrane and the NVP UV-grafted NF PES membrane. Adapted with permission of the authors [69]

**Table 2** Infrared characteristic for the unmodified PES membrane [74]

Functional group	Type of vibration	Characteristic absorptions (cm <sup>-1</sup> )	Intensity
C=C	stretching	1400-1600	strong
C-O-C	stretching	1000-1300	strong
O=S=O	asymmetric stretching	1321 & 1296	two bands
O=S=O	symmetric stretching	1147	sharp peak
C-H	aromatic stretching	3000-3100	medium
C-H	aliphatic stretching	2850-3000	strong

The low intensity peaks were too observed in an investigation of LbL polyelectrolyte membranes for FO applications, where both poly (allylamine hydrochloride) (PAH) and poly (sodium 4-styrenesulfonate) (PSS) were crosslinked with glutaraldehyde and UV light exposure respectively on the PAN membrane [75]. Although, it was found at low intensity, they are clearly new peaks as it did not appear in the sample before the UV exposure was applied. Additionally, the authors confirmed the existence of the chemistry elements through X-ray photoelectron spectroscopy which will be discussed in the later section. Similarly, the spectrum of the PAN membrane is presented by [76]. In addition to this, the importance of wavelength range is seeming to be one of the criteria that need to be considered as the peak of the spectrum cannot be clearly recognized when shorter wavelength is displayed. For example, from FTIR Figure presented by [77]. Although the peak is clearly observed at expanded wavelength, but it is nearly impossible to appear as the authors present shorten wavelength range. By employing FTIR, the spectrum of the unmodified and modified membrane must be crucially presented

in the arguments. The results of the FTIR would be adequate to acknowledge the existence of the certain functional group, only if when the peak can be clearly identified.

#### **4. Characterization of chemical composition (XPS)**

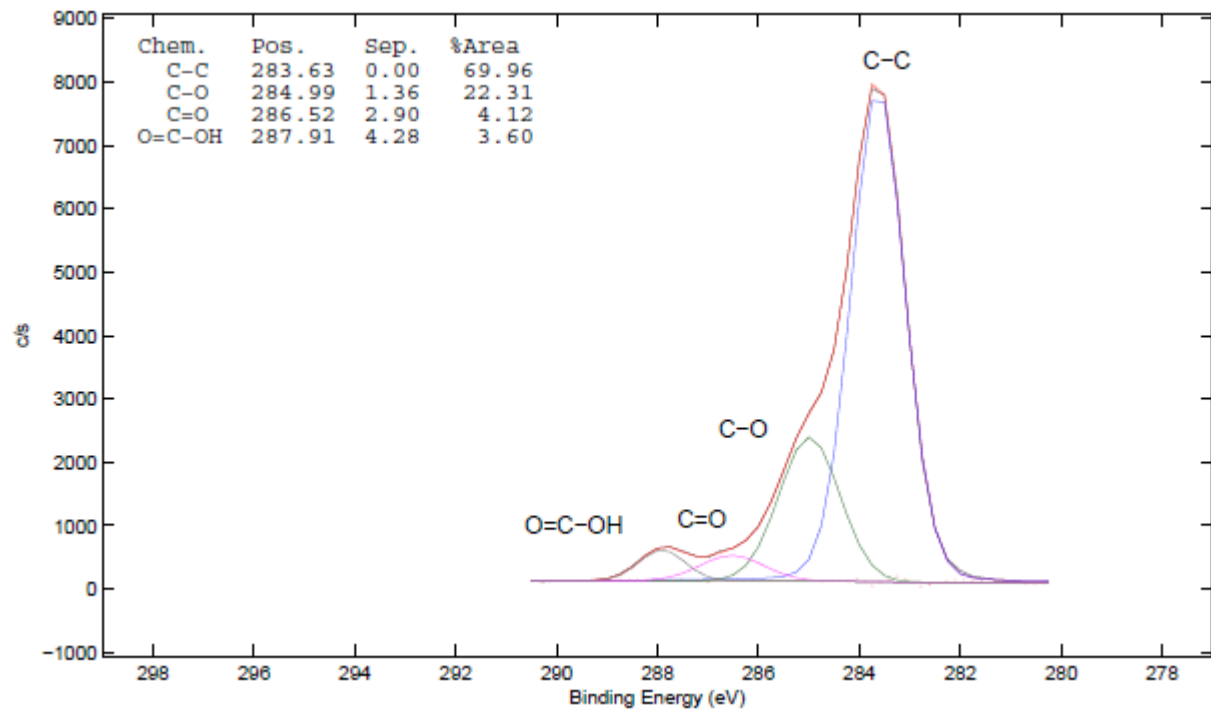
The X-ray photoelectron spectroscopy (XPS) is spectroscopic technique capable of providing atomic and molecular information about the surface of a solid material [78]. Commonly, it is used to obtain surface composition of homopolymers, copolymers, polymer blends, and grafting [79, 80]. The used of it were also discussed in varies others application. XPS is a high degree of chemical specificity application in terms of composition and structure. The graph resulted from XPS will be interpreted by the database and peaks of all elements will be identified. As the chemical bonds of the chemicals used in the membrane fabrication process will be included in the XPS results including its intensity, thus it is appropriate to mention the chemical bonds that exist in any particular chemicals for the case of membrane fabrication.

In most cases, XPS results is used to confirm the outcomes of the FTIR especially when it is debatable. For instance, in the case of membrane modification, Rahman and Seman [77] conduct a surface modification study on NF PES membrane to enhance the water permeability in FO via UV grafting using acrylic acid as the monomers. They claim that they found the new peak of carboxyl on the wavelengths of the membrane though it is very unclear, hence the XPS data were provided to confirm the presence of this functional group. Figure 4 a) and b) are the spectra obtained by XPS for both unmodified NF PES membrane and modified NF PES membrane respectively. These clearly confirmed the existence of the carboxyl group on the modified NF PES membrane although it is hardly appeared on the FTIR results. Besides, since the results of XPS spectra is summarizing the whole compositions, therefore, accurate conformation can be accomplished. Providing supportive evidences or references for similar cases are very convincing and sensible as for this case, similar trends of XPS results were too observed by [81].

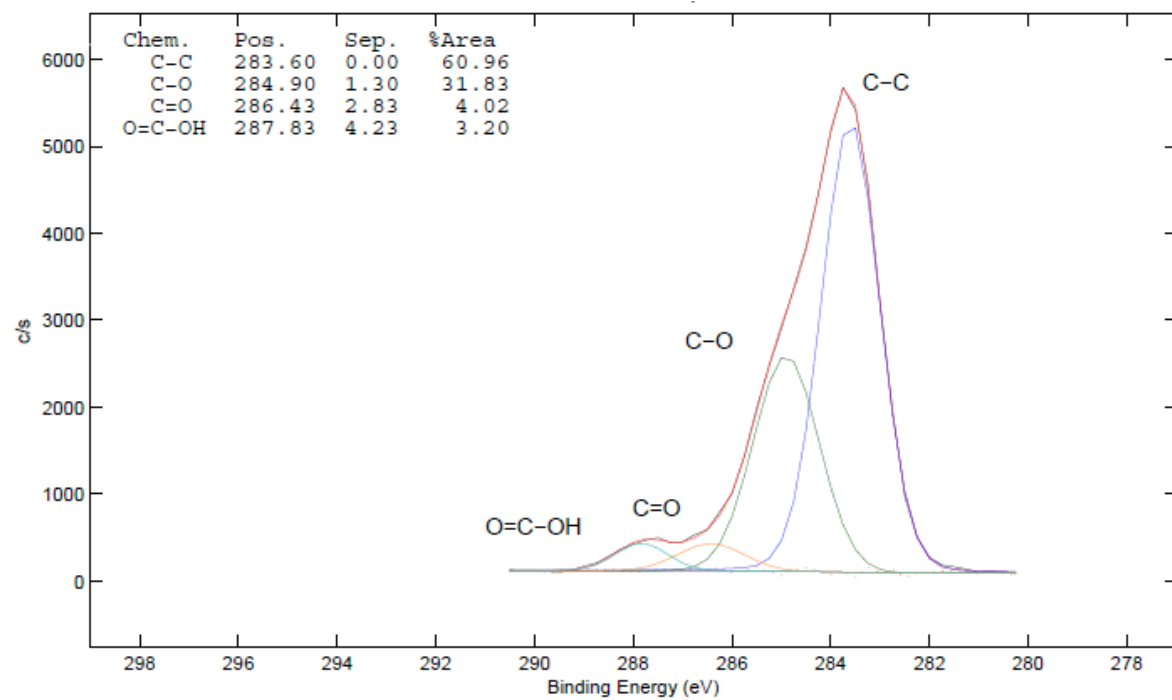
Moreover, the XPS also providing the information regarding the intensity of the newly formed chemical bonds on the surface where the preliminary suggestion on the thickness of new layer can be presumed. A study which set out to modify the surface of polysulfone (PSf) with polydopamine (PDA) at different coating times have shown that the intensity of the PDA is increases with time. These suggested that, different thickness of modified layer was attained and since the spectra due to PSf were also detected, this concise that the modified layer was thinly present. The authors set 10nm limitation for XPS penetration, therefore, the thickness of a new layer are less than 10nm [82]. To determine the effects of the modified layer, it is necessary to include both unmodified and modified XPS spectra over the discussion.

In a recent study conduct by Song et al. [83], they provide both C1s and wide scans spectra to distinguish the intensity of the elements for each CA and its modified substrate. Based on the analysis, it is clearly indicating that the presence of N1s signal only in the CA-polyvinyl and polydopamine modified. Knowing the chemical compositions through XPS promote a good suggestion to whether chemical crosslinking did happen or otherwise in the case of interfacial polymerization. This theory was described by Zheng et al. [84] where fewer crosslinked polyamide structure were observed based on the XPS analysis due to higher concentration of sulfonated polysulfone in the substrate ratio that leads to competitive reaction between water and M-phenylenediamine to react with trimesoyl chloride. In contrast the intensity of presented elements was used to described the changes of the modified surface [15].





a)



b)

**Figure 4** XPS spectra for a) unmodified NF PES membrane and b) modified NF PES membrane. (Adapted with permission from the corresponding author) [77]

## 5. Characterization of the membrane surface roughness (AFM)

Atomic force microscopy (AFM) provides capability of imaging the variety of material properties through mechanical interaction between the tip and the membranes [85]. These advantages allow AFM to map, differentiate and classify the multicomponent material on the membrane surface easily which hardly obtained when using electron or photon scanning. In FO, the roughness is associated to fouling wherein higher fouling tendencies were found on rougher surfaces [86]. Many researchers agree that higher surface roughness offers more spaces for organics foulants to attach. When the roughness is observed, the image of ridge and valley can be clearly identified with brighter color represent the ridge and darker color is the valley [87].

Upon interpreting the AFM results, all the parameters in characterizing the surface topography of membrane surface must be understood and clearly justified. The mean roughness ( $R_a$ ) and the root means square roughness ( $R_q$ ) are the most used parameters [88]. Meanwhile some prefer to include the maximum profile peak height ( $R_p$ ), maximum profile valley depth ( $R_v$ ) and maximum height of the profile ( $R_T$ ) [82].  $R_a$  and  $R_q$  can be calculated in the Eq. 1 [89] and Eq. 2 [90], respectively.

$$R_a = \frac{1}{L} \int_0^L |Z(x)| dx \quad (1)$$

$$R_q = \sqrt{\frac{1}{L} \int_0^L |Z^2(x)| dx} \quad (2)$$

where  $L$  is define as the surface profile measure in terms of height ( $Z$ ) and position ( $x$ ) of the sample over the evaluation length ( $L$ ). Rather, the parameters can be easily and quickly analyzed via many available software [91].

Theoretically, both  $R_a$  and  $R_q$  only showing the mean absolute profile of the surface in which that many surfaces profiles might have the same  $R_a$  and  $R_q$  values although at different surface roughness. Because both parameters only depend on the average profile of the heights, the fluctuation on the surfaces cannot be distinguish accordingly. Therefore it is not relevant to say one surface is rougher than the other based on  $R_a$  and  $R_q$ . Though, many previous works stresses on these parameters probably because it is the most common parameters discussed and regardless of the roughness distribution, roughness can be measured by these two parameters [92, 93]. Whereas some authors restrained to compare the roughness of the surfaces using any of the parameters, provided that in most cases it can be clearly seen through AFM images particularly the peak and valley [94]. Nonetheless, parameters like  $R_p$ ,  $R_v$  and  $R_T$  can be considered to study on the undulation of the surface roughness. Especially when the height of both peaks and valley need to be known.

In the case of FO, the AFM characterization were accomplished to determine the surface roughness of fabricated membrane or to distinguish the surface of the membrane before and after modification and/or to differentiate the roughness levels after the modifications at varieties conditions and limitations. Given that, the increase in surface roughness would provide larger surface area which should lead to more feed water contact, however, due to the valleys structure on the rougher surface, fouling tendency are more significant [95, 96].

## 6. Characterization of the membrane structure (SEM/FESEM)

The scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM) are the most dominant membrane characterization instruments among others that dealt with electron microscopy. This might be due to advantages that analysis works can be done with the bulk sample. Similar to other characterization techniques, if the membrane is fabricated and/or modified, scanned

image of the membrane must be crucially embraced and both unmodified membrane and modified membrane should be presented in the case of membrane modification.

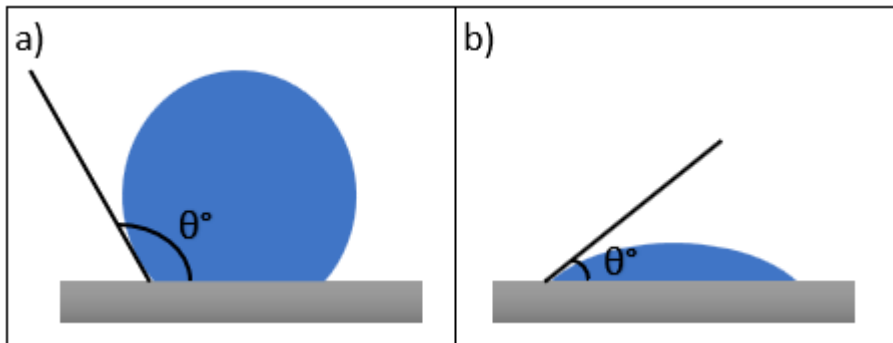
Typically SEM is used to present either any of the top surface structure, bottom surface structure, cross-sectional image of the whole membrane structure or cross-sectional image of the dense and porous layer structure separately. Equivalent to what have been reported by [97] where investigators examined the post-treatment induced effect on the TFC-FO membrane microscopic structure. They show the SEM imaging of the top and bottom surface each of modified membrane as well as comprehensive cross section of the samples. Else, one can simply offer the whole cross-sectional area of the membrane and pin point the area that will be magnified for further imaging details [83]. Additionally, the thickness of the membrane also can be retrieved from the same images.

It is interesting to note that the visibility of the newly induced substances on the membrane are not always observable from the SEM images although the chemicals functional group or bonds might have been clearly spotted in FTIR or XPS. This is similar to the recent investigation reported by [21] where they found that similar images were formed via SEM for both commercial TFC FO membrane and Sulfobetaine Methacrylate (SBMA) polymerized-TFC FO membrane. However the presence of Poly-SBMA was clearly confirmed by FTIR. Somewhat, in most cases it is comparatively visible in both surface chemistry and morphological characterizations techniques. Al<sub>2</sub>O<sub>3</sub> nanoparticles were added to the thin film nanocomposite (TFN) membranes and show large “leaf-like” morphological structures while certain peaks observed via FTIR analysis indicate the successful integration of the nanoparticles [98]. These are because FO membrane is typically very thin, and any modification applied onto the fabricated membranes were driven to create a finely layer. Hence the unclear images of FESEM/SEM.

## **7. Characterization of the membrane wettability (Contact angle)**

Typically, contact angle (CA) is the simplest experiment to test a hydrophilicity or hydrophobicity of a membrane surface by simply placing a drop of liquid on the membrane surface, hence leads to the formation of an interface between two phases [99]. The angle known as contact angle is defined as the tangent line drawn at the droplet curve to the points where it intersects the membrane surface based on three contact points. Figure 5 clarify the angle mentioned for measuring wettability. The full range angle of hydrophilicity/hydrophobicity are as presented in Table 3.

Out of many methodologies that have been reported earlier for CA measurements, the most common adopted way is the sessile drop method. However, the measurement of the CA for the porous surfaces such as membrane would be very difficult, and the result is questionable. This is because of the two phenomenon that might occurred after the water is dropped to the membrane surface; absorption and spreading. Both are described in Figure 6. In addition to that, the evaporation of the liquid also contributes to the inclined volume of liquid drop. Therefore, the protocol in handling the wettability test must be standardize for all samples compare with control sample. In one known recent research work, it was observed that the water contact angle (WCA) of all membranes reduces expressively in the first 10 seconds and became slowly decreases after. The WCA were recorded at 2 minutes for each where they noticed the declination getting stable [83]. The WCA can be measured from the averages of WCA taken at least at three different points on each membrane surfaces to enhanced the accuracy of the outcomes [19].

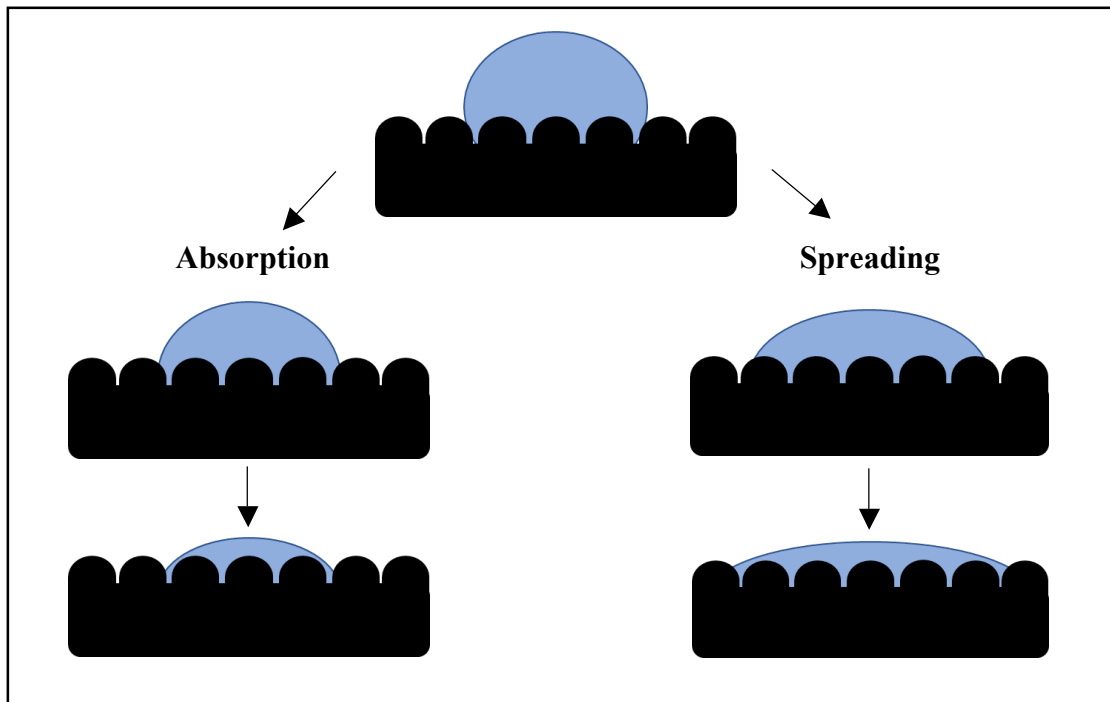


**Figure 5** Scheme of the determination of angle for a) hydrophobic and b) hydrophilic [100]

**Table 3** Full range of contact angle ( $\theta^\circ$ ) with defined level of wettability [101].

Wettability	Angle ( $^\circ$ )
Super hydrophobic	150°-180°
Hydrophobic	90°-150°
Hydrophilic	5°-90°
Super hydrophilic	0°-5°

One of the important things to know when dealing with the CA is that the active and support layer are usually have different WCA values. This is due to the hydrophilicity level of the FO membrane itself where support layer often less hydrophilic than the active layer [102]. To date, as various methods have been developed and introduced to enhance the performance of FO membrane while following its ideal standard, [19] found themselves a different type of FO membrane comes with no support layer. The polytriazole-co-oxadiazole-co-hydrazide (PTAODH) membrane simply having two surfaces of similar material, pore size and hydrophilicity yet the robustness of the membranes is not convincing [19]. The length of the period within when the liquid is dropped to the membrane surface and when the measurement of the angle was taken should have been standardized for each sample measured.



**Figure 6** The two phenomenon that occur on the porous surface after a drop of liquid (mostly water) is placed. (Adapted from books edited by [103])

## 8. Conclusions

This paper presents an insight review on surface characterization of FO membranes. Regardless the types of FO membranes and the methods of producing it, the surface characterization comprises of FTIR, XPS, FESEM/SEM, AFM and Contact angle were discussed in the way that how the findings should be reported. This work can't be considered as the guidelines to interpret the characterization analysis, rather it is showing the relevant discussion that should be measured and included when deliberating the analysis result for FO membranes. Generally, as the issues with FTIR have been address earlier, the spectrum values should be presented within relevant wavelength. Although characterization using XPS is automatically interpreted by the equipment itself, a figure shown the XPS spectra is highly reassured to be included in the report. Similarly, it is also preferred in presenting FESEM/SEM and AFM results. As the contact angle measurement is usually not suitable for porous surface like membrane, more control should have been taken if one wants to consider using it as to measure the wettability properties. Insufficient and misinterpreted characterization data might have not changed the novelty fact of the research, yet it may lead to lower its impact. Appropriate characteristic evidence and discussions of the fabricated or modified membrane would be very convincing for the research.

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