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Measuring hydrophilicity of RO membranes by contact angles via sessile drop and captive bubble method: A comparative study

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HIGHLIGHTS

► Hydrophilicities of RO membranes are measured by contact angle analysis.
► Contact angles are compared by the sessile drop method and captive bubble method.
► Sessile drop method shows inconsistent contact angles with various conditions.
► In contrast, captive bubble method shows reliable and reproducible contact angles.

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ABSTRACT

Contact angle measurement is conducted to determine surface hydrophilicity of RO membrane with the intention of predicting membrane performance or fouling potential. Most researchers of membrane technology have used contact angles without considering effective factors for contact angle measurement such as measurement time, drop volume and membrane sample preparation. In fact, significantly different contact angles are reported even on the same RO membrane. In this study, contact angles were measured by the sessile drop method and captive bubble method and were compared with various conditions such as measurement time, drop volume, membrane sample preparation and liquid type in the commercialized RO membranes. As a result, sessile drop method showed unreliable contact angles, which varied dramatically with the preparation conditions. On the other hand, the captive bubble method, which represents conditions closer to the real RO membrane process, showed reproducible contact angles. For the different commercially available RO membranes, similar hydrophilicity was observed. Overall, the captive bubble method appears to be a more adequate method for measuring contact angle of RO membranes.

1. Introduction

Surface analysis of reverse osmosis (RO) membrane is widely used for studying membrane performance or membrane fouling. In terms of membrane performance, it is well known that a rougher membrane, whose larger effective surface area and/or more hydrophilic membrane provides a better wettability, possess a higher permeate flux under otherwise identical surface properties [1,2]. In terms of membrane fouling, a more hydrophilic, softer and neutrally charged membrane results in a low fouling potential [3,4]. Typically, the surface properties of RO membrane are characterized by its hydrophilicity, roughness, and surface charge. These properties can usually be measured by contact angle analyzer, atomic force microscopy and zeta potential analyzer, respectively.

However, it is unclear if these analytical techniques can exactly measure the heterogeneous membrane surface of polyamide RO membranes during its application. It is reported that atomic force microscopy has limitations to measure the surface roughness of RO membranes because the roughness increases with increasing analysis area and is limited to a maximum analysis area of only $10 \times 10\ \mu m^2$ [5]. Also, the zeta-potential analysis of RO membranes is utilized, and it is generally agreed that the streaming potential method is more useful than other methods, which are inadequate to measure extremely small pores in RO membranes compared to microfiltration (MF) or ultrafiltration (UF) membranes [6].

Interestingly, the contact angle measurements of RO membranes have rarely been studied. Even though some studies tried to clarify the characterization of new membranes or modified membranes and to predict membrane fouling based on contact angle measurements, most of them did not follow an accurate protocol, making it hard to compare the individual data. Table 1 summarizes the reported contact angles on various models of RO membrane. In general, higher contact angles indicate a more hydrophobic surface [7]. As shown in Table 1, the contact angles differ significantly even when measured

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Table 1
Literature summary of contact angles of RO membranes measured by the sessile drop method.

RO membrane type	Model	Contact angle (°) in [Ref.]
SWRO	SW30HR	17.2–32.0 [7–9]
	SWC3+	70.1–88.0 [8,10]
eBWRO	LFC1	20.1–78.0 [7,11–13]
	XLE	39.8–67.6 [7,14–17]
	BW30	25.9–76.0 [7,18–21]
	HL	27.5–51.9 [7,11,22–24]

on the same RO membrane. These differences might result from various factors, such as measurement time, drop (or bubble) volume, liquid types, temperature and humidity. Therefore, it is necessary to establish a measurement protocol that can measure reproducible and reliable contact angles, which will easily compare the quality of RO membranes.

The objective of this study is to investigate an adequate contact angle measurement for RO membranes. Two popular methods of contact angle measurements – sessile drop method and captive bubble method – are used in commercially available thin film composite polyamide RO membranes. The consistency of contact angle is evaluated by different measurement time, drop (or air bubble) volumes and membrane sample preparation or liquid types.

2. Materials and methods

2.1. RO membranes

Table 2 describes seven commercially available polyamide RO membranes chosen in this study and their performances supported by each manufacturer. As shown in Table 2, the RO membranes can be classified by seawater RO (SWRO; SWC5, SW30HR, TM820, SHN, and SHF) membranes and brackish water RO (BWRO; LFC1 and FLR) membranes. The RO membranes SWC5, SW30HR, TM820, and LFC1 were selected since they are widely used in both industry and academia [25–28]. Additionally, SHF was equipped on a pilot scale in Kijang, Republic of Korea. SHF, SHN and FRN were kindly received from Woongjin Chemical Co., Republic of Korea, while other RO membranes were purchased from the respective manufacturer. The RO membranes were stored as flat sheets in deionized (DI) water (Barnsted NANO Pure, USA) at 4 °C, as described in detail in our previous researches [29,30].

Table 2
Commercially available polyamide RO membranes chosen in this study and their performances (provided by each manufacturer).

Type	Model	Manufacturer	Permeate flow[c] (m^3/d)	Salt rejection[c] (%)	Max. operation temp. (°C)	pH range
SWRO[a]	SWC5	Hydranautics Nitto denko	34.1 (37.2 m^2)[d]	99.7–99.8	45	3–10
	SW30HR	Dow Filmtec	23 (30 m^2)[d]	99.6–99.75	45	2–11
	TM820	Toray	23 (34 m^2)[d]	99.75	45	2–11
	SHN	Woongjin Chemical	24.6 (37.2 m^2)[d]	99.75	45	3–10
	SHF	Woongjin Chemical	34.1 (34.4 m^2)[d]	99.7	45	3–10
BWRO[b]	LFC1	Hydranautics Nitto denko	41.6 (37.2 m^2)[d]	99.5	45	3–10
	FLR	Woongjin Chemical	34 (37.2 m^2)[d]	99.6	45	3–10

[a] SWRO: seawater RO membrane.
[b] BWRO: brackish RO membrane.
[c] SWRO: under 800 psi of pressure with 32,000 mg/L of NaCl solution; BWRO: under 225 psi of pressure with 2,000 mg/L of NaCl solution.
[d] Effective membrane area (m^2) for 8 inch module.

Two RO membranes, LFC1 and SWC5, as representative fully coated and uncoated RO membrane in this study were used in order to measure the contact angle by two methods at various conditions.

2.2. Membrane characterization

Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR; Nicolet spectrophotometer 5700, Thermo Electron Corp., MA) was used for obtaining chemical and elementary information of membrane surface. The analysis of high wave-number region (3800–2600 cm^{-1}) of FTIR was used for analyzing thin (thickness<200 nm) polyamide top layers [31] since polyamide top layer (thickness: ~200 nm) mainly influences the contact angle. This analysis informs whether seven RO membranes are fully coated or uncoated.

2.3. Contact angle measurement

2.3.1. Basic information

Typical methods of contact angle measurement are the sessile drop method (including captive bubble method), the Wilhelmy plate method, and the capillary rise method. Among them, the sessile drop method and captive bubble method are used most frequently in the literature. For both methods, a contact angle analyzer (KRÜSS, DSA100, Germany) was used. Fig. 1 illustrates the schemes of sessile drop method and captive bubble method. Sessile drop method was measured by deposition of a liquid droplet onto the membrane surface (Fig. 1(a)) by using an 'I'-shaped needle, while the captive bubble method was conducted by forming an air bubble onto the immersed RO membrane surface in the liquid by using a 'J'-shaped needle (Fig. 1(b)). DI water was used as a liquid for both the methods. Theoretically, contact angles measured by both sessile drop method and captive bubble method are described by the Young–Dupré equation (Eq. (1)), assuming that surface is smooth and homogeneous [32].

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG}\cos\theta_c \tag{1}$$

(γ_{SL}: solid–liquid interfacial energy, γ_{LG}: liquid–gas interfacial energy, γ_{SG}: solid–gas interfacial energy, θ_c: equilibrium contact angle).

2.3.2. Sessile drop method

Since membrane sample preparation is an essential prerequisite to measure contact angle by sessile drop method, the membrane sample was kept dry in contrast to the captive bubble method. Membrane samples were dried at 40 °C to be below 45 °C which is the maximum operation temperature of RO membranes (Table 2) in the vacuum oven. The contact angle measurement was then performed as follows. A dangling droplet of 6 μL of DI water at the end of 'I'-shaped needle was carefully deposited to membrane surface to avoid the effect of falling force by gravity (Fig. 1(a)). From this moment, all processes were recorded for 500 frames (approximately 16 s) at least. Contact angle values, calculated by DSA100 software (KRÜSS, DSA100, Germany), were determined as the averaged values during the periods of 300–500 frames (about 10–16 s).

For assessing the effect of measurement conditions onto contact angle value, first, membrane samples were compared when dried under atmospheric conditions and dried at 40 °C in the vacuum oven to investigate the effect of membrane sample preparation. Second, contact angle values were recorded as a function of time to find out the effect of measurement time. Finally, droplet volumes of DI water were varied between 1 and 8 μL. Droplets larger than 8 μL of volumes of DI water fell easily from the needle which might affect contact angle value because they were too heavy. To guarantee a statistical integrity, at least three samples were used for each RO membranes and contact angle measurements were performed on at least five positions

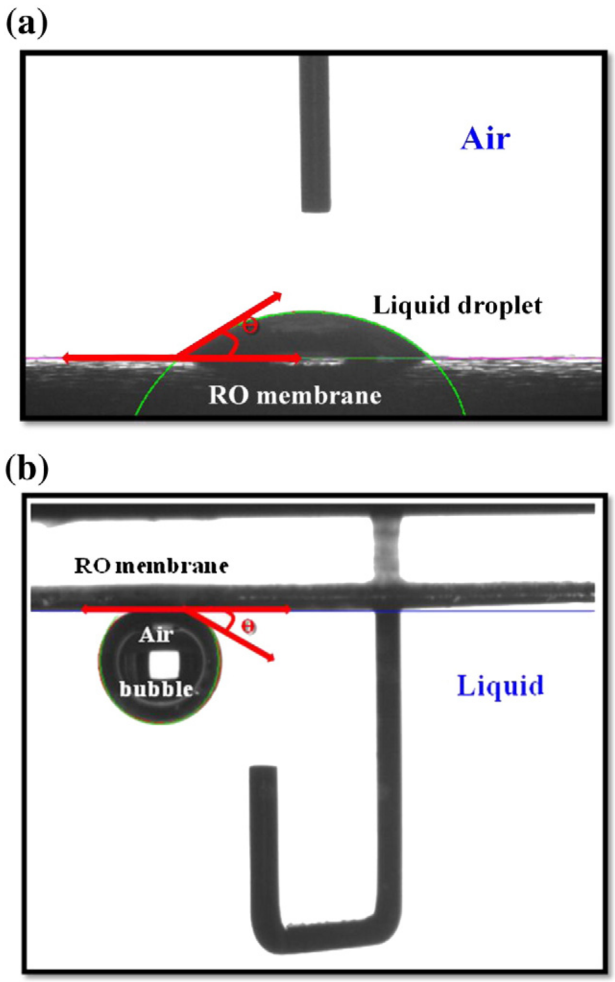

Fig. 1. Schemes of (a) sessile drop method with 'I'-shaped needle and (b) captive bubble method with 'J'-shaped needle (θ: contact angle and green line: analyzed water drop (or air bubble) surface).

on each sample. Each experiment was performed at the same time in order to minimize other effects on contact angle values such as temperature (24 °C ± 1 °C) and humidity (60% ± 10%). As a control, contact angle of indium tin oxide (ITO) glass (Samsung Corning Ltd., Republic of Korea) was measured.

2.3.3. Captive bubble method

Membrane samples for the captive bubble method were used as taken out from the preservation condition without any further preparation. Membrane samples were attached to a glass support and immersed into DI water at room temperature. The effect of measurement time (up to 16 s) and the effect of various volumes of air bubble (6–20 μL) in captive bubble method were compared with sessile drop method. 6 μL of air bubble was the smallest volume that could be attached onto the membrane surface. A 'J' shaped syringe needle was used to dispense an air bubble from beneath the membrane sample (Fig. 1(b)). Other processes such as recording and calculating contact angle were the same as described for the sessile drop method. In addition, for investigating the effect of liquid types, membrane samples were also immersed into the 3.5% sodium chloride (Aldrich, St. Louis, MO) solution and the contact angle was compared to those measured in DI water. 10 μL volume of air bubble was employed when the effect of measurement time and liquid types was investigated with the averaged values during the periods of 300–500 frames (about 10–16 s). Again, ITO glass was used as a control substrate.

3. Results and discussion

3.1. Characteristics of RO membranes

Fig. 2 shows the ATR FT-IR spectra of different polyamide RO membranes in the high wavenumber region between 3800 cm^{-1} and 2600 cm^{-1}. The peak in the 3300–3400 cm^{-1} region indicates a hydroxyl (−OH) group, which is characteristic for fully coated RO membranes. While uncoated RO membrane consists only of a pure polyamide layer, fully coated RO membranes are usually coated with other substances such as polyethylene glycol or polyvinyl alcohol [33]. Therefore, SW30HR (SWRO) and LFC1 (BWRO) were identified as fully coated RO membranes while the other RO membranes were uncoated RO membrane in this study.

3.2. Limitation of sessile drop method for measuring RO membrane hydrophilicity

Fig. 3 illustrates the irregular contact angles on sessile drop method as a function of measurement time, liquid drop volume, and membrane sample preparations. Fig. 3(a) shows that the contact angles measured by sessile drop method decreased with time. While contact angles on ITO glass were constant over time, contact angles on RO membranes significantly decreased in the beginning of the measurement and gradually decreased afterwards. This reduction can be explained by various reasons. First, the liquid drop is likely to be adsorbed to the dried membrane surface, which essentially altered the surface nature be exposing more hydrophilic components. Next, the slight reduction of contact angle was probably due to the continuous spreading on the membrane surface and hence enhanced evaporation. Noteworthy, these effects occurred for both fully coated and uncoated RO membranes.

Fig. 3(b) shows that somewhat arbitrary contact angles were measured with different drop volumes from 1 μL to 8 μL. In contrast, contact angles on ITO glass were only slightly decreased due to an increased gravitational force. On the other hand, contact angles on LFC-1 and SWC5 RO membranes showed completely opposite behavior. First, the contact angle of LFC1 was in general higher than the contact angle of SWC5, which indicates that LFC1 consists of a more hydrophobic surface. However, when 4 μL of liquid drop was used, a similar contact angle was measured for LFC1 and SWC5. Next, while contact angles for LFC1 seemed to decrease with increasing drop volume, they increased for the SWC5 with increasing drop volume.

In addition, the variations of the contact angles depending on the different membrane sample preparations are compiled in Fig. 3(c).

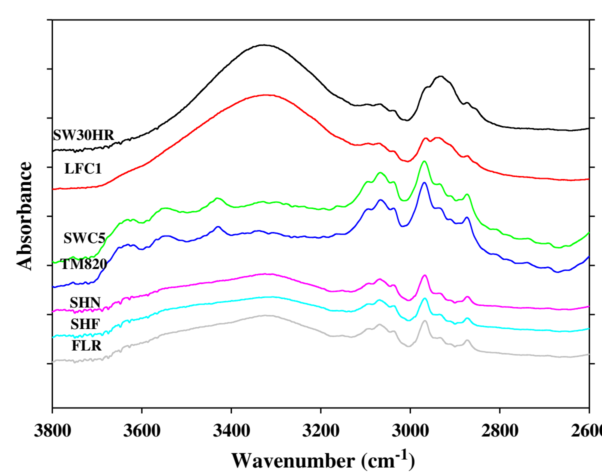

Fig. 2. The wavenumber region of 3800–2600 cm^{-1} in ATR FT-IR results for commercially available polyamide RO membranes.

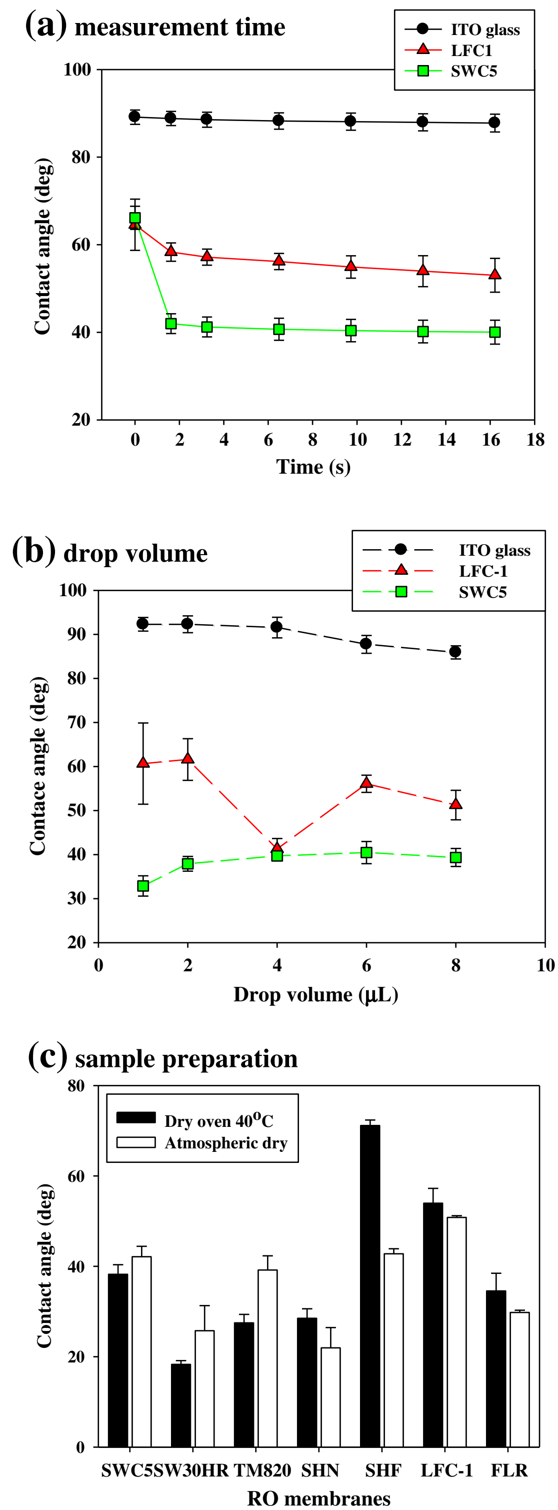

Fig. 3. Limitation of the sessile drop method for measuring RO membrane hydrophilicity on (a) measurement time for 16 s approximately (drop volume: 6 μL), (b) drop volume (1–8 μL), and (c) membrane sample preparation (dry oven at 40 °C and atmospheric dry).

The result showed that contact angle values can differ up to 30° between the dried membrane at atmospheric condition and the dried membrane at 40 °C in the vacuum oven. Note that so far only differences up to 4° were reported for different drying conditions in the temperature range of 40–80 °C [34]. From these results, it can be concluded that membrane sample preparation for characterization of RO membranes by the sessile drop method is one of the most important factors considered in contact angle measurement, although it was mostly omitted in the literature up to now.

Overall, the results indicate that sessile drop method has a weakness due to membrane damage caused by the drying process [35]. In contact angle measured by sessile drop method, it was expected that contact angles of fully coated RO membranes were lower than those of uncoated RO membrane since fully coated RO membranes have more hydrophilic functional group (i.e. –OH) (Fig. 2). However,

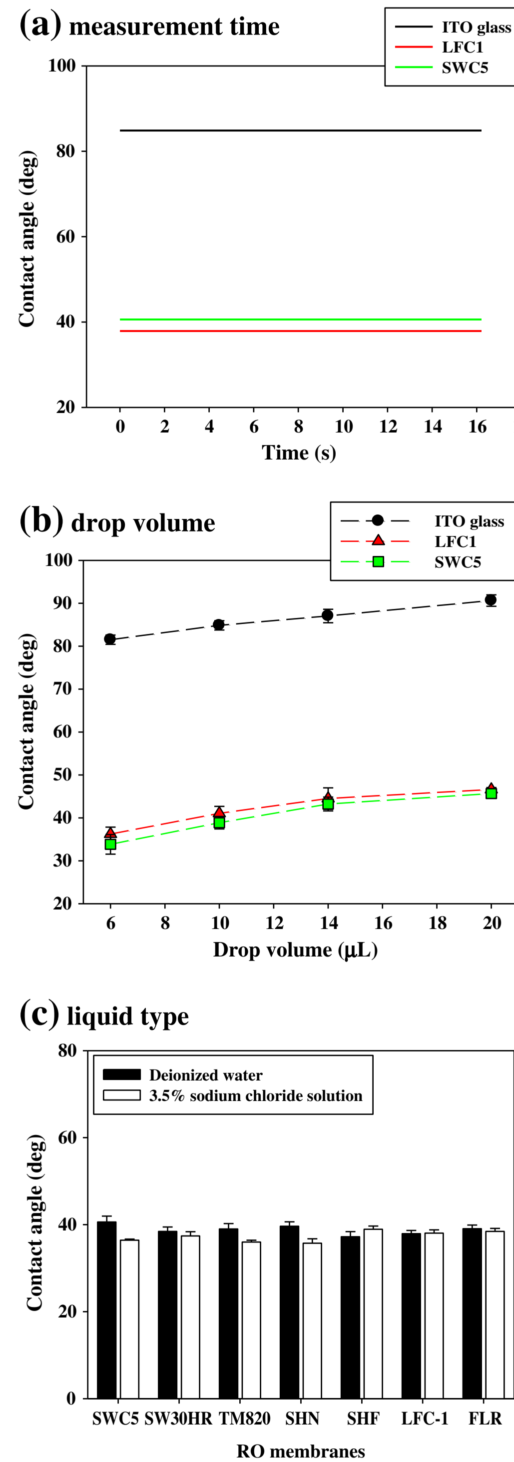

Fig. 4. Contact angle values on the captive bubble method; (a) measurement time for 16 s approximately (drop volume: 10 μL), (b) drop volume (6–20 μL) and (c) liquid type (deionized water and 3.5% sodium chloride solution).

Table 3
Summary of contact angles measurement by sessile drop and captive bubble methods in this study.

	SWC5	SW30HR	TM820	SHN	SHF	LFC-1	FLR
Sessile drop method[a]	38.2 ± 2.1	18.3 ± 0.8	27.5 ± 1.9	28.5 ± 2.1	71.2 ± 1.2	54.0 ± 3.3	34.6 ± 3.9
Captive bubble method[b]	40.6 ± 1.4	38.5 ± 1.0	39.0 ± 1.2	39.7 ± 1.0	37.2 ± 1.2	37.9 ± 0.8	39.1 ± 0.8

[a] Sessile drop method: measured with 6 μL of deionized water.
[b] Captive bubble method: measured with 10 μL of air bubble.

opposite results were shown in Fig. 3, in which fully coated RO membrane LFC1 was more hydrophobic than uncoated RO membrane SWC5. It is also presumed that the drying process can damage the surface and result irreversibly in more hydrophobic surfaces. These uncertainties in contact angle measurements of RO membranes with measurement time, drop volume and membrane sample preparation suggest that the sessile drop method is somewhat limited for routine characterization method of RO membranes.

3.3. Reliability of captive bubble method

Fig. 4 describes the results of the corresponding contact angle measurements by the captive bubble method. Fig. 4(a) shows that contact angles on both RO membranes and ITO glass by captive bubble method were constant compared to contact angles by sessile drop method (Fig. 3(a)). As the measurement is conducted under water, any effects of swelling or evaporation could be eliminated and it thus represents more realistic RO membrane in the process.

Fig. 4(b) shows that linearly increasing contact angles were observed with different air bubble volumes from 6 μL to 20 μL. This increase can be explained by increased buoyancy force. As the air bubble was forced stronger onto the membrane surface due to a higher buoyancy force, larger contact angles were observed. The results showed more stable experimental data rather than the unreliable contact angles that were obtained with different drop volumes by the sessile drop method (Fig. 3(b)). Thus, unlike the sessile drop method, the captive bubble method is more capable to assess the hydrophilicity of RO membrane surface. However, the possibility cannot be excluded that the surface of the membrane is fully wetted by water and the air bubble might be in touch with water layer, but not with the membrane surface, rendering the measurement of contact angle of membrane less conventional, requiring further investigation.

In addition, the contact angles were measured using two different liquids (DI water and 3.5% sodium chloride solution) and the results are depicted in Fig. 4(c). Generally, the contact angles measured in DI water were slightly higher than the contact angles measured in sodium chloride solution. The only exception was observed for the SHF membrane. This indicates that the ionic strength affects the contact angles not as much as previous reports suggested which utilized only the sessile drop method. They observed much lower contact angles for 3.5% sodium chloride solution rather than DI water [16].

The data for contact angle at identical conditions for both methods (sessile drop and captive bubble method) were presented in tabular form along with statistical analysis (Table 3). As shown in Table 3, the contact angles measured by captive bubble method were similar for all RO membrane with values at around 40°, while the contact angles measured by the sessile drop method resulted in fluctuating values from 18.3° to 71.2° for the different RO membranes. As mentioned already above, the captive bubble method is more suitable to obtain reliable and reproducible contact angles of RO membranes. Moreover, the captive bubble method represents a measurement that is much closer to the real RO membrane process. For example, a drying process is a necessary step in the membrane sample preparation for the sessile drop method, which might alter the polyamide RO membrane dramatically and differently for different RO membranes. In contrast, RO membrane samples that are analyzed by the captive bubble method are already wetted in DI water during membrane storage. Therefore, the influence of swelling during the contact angle measurement can be neglected, because the RO membrane samples are already completely hydrated [35–37]. In addition, similar and time independent contact angles by the captive bubble method (Table 3 and Fig. 4) might be explained that the air bubble is mostly in contact with water molecules from the fully hydrated polyamide layer [38,39].

Overall, the captive bubble method appears to be a more representative characterization technique allowing a fast measurement of the membrane hydrophilicity in real RO membrane process, because the RO membrane is never dried in the full process ranging from its manufacture step to the autopsy step at the end of membrane lifetime. All RO membranes in this study show a similar performance in terms of salt rejection and only varied in the permeate flow (Table 2). Therefore, the surfaces of commercially available polyamide RO membranes seem to have similar hydrophilicities in operation. As such, it is easy to understand that all RO membranes possess similar contact angles.

4. Conclusion

We observed that contact angles obtained by the sessile drop method, even though mostly used in the literatures, showed inconsistent values with different measurement times and drop volumes. Further, the membrane sample preparation had a strong effect on the contact angles. In contrast, we could show that reliable and reproducible contact angles can be obtained by the captive bubble method, which are essentially independent of measurement times, drop volumes and liquid types. Further, captive bubble method represents measuring conditions that are likely more similar to the real RO membrane process, since the contact angle is measured under wet conditions. As such, it represents an important step forward in studying and detecting fouling behavior of RO membranes.

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