



## Interpreting contact angles of surfactant solutions on microporous hydrophobic membranes



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### ABSTRACT

Almost every single experimental study regarding membranes involves the measurement of contact angle (CA) to quantify the membrane wetting property. However, the interpretation of CA can sometimes be tricky. In this study, we investigate an interesting phenomenon about the CA of a surfactant solution on a microporous hydrophobic membrane. Specifically, a surfactant solution with a very low surface tension can have an unexpectedly high CA on a microporous hydrophobic membrane. In contrast, a water/ethanol mixture with the same surface tension completely wicks the membrane (i.e., zero CA). The drastic difference in CA between the two types of liquid of the same surface tension results from the rapid adsorption of surfactants at the wetting frontier which substantially reduces the local surfactant concentration and increases the local surface tension. The same theory can also be applied to explain the striking difference between the two liquids in capillary rise and liquid entry pressure. The results from this study cast significant doubt on the role of surface tension in understanding the wetting behavior of surfactant solutions when they are in contact with solid with large specific area and raise important questions regarding the utility of measuring CA for surfactant solutions on microporous hydrophobic membranes.

### 1. Introduction

Contact angle (CA) measurements have been performed in almost every single study for membrane fabrication and application. While CA measurements provide valuable information regarding membrane wetting properties, the interpretation of measurement data is not always straightforward. For example, the intrinsic CA is required in many theoretical models for membrane-related phenomena, such as wetting, (Franken et al., 1987; Lu et al., 2008; Wang et al., 2018) scaling, (Tong et al., 2017; Su et al., 2018; Xiao et al., 2019; Tong et al., 2019) and fouling. (Boo et al., 2018) However, it cannot be emphasized more that the CA measured on a microporous membrane is not the intrinsic CA when the system is in a Cassie-Baxter state or when the membrane has a large roughness. (Bormashenko, 2015; Rezaei et al., 2018; Horseman et al., 2021)

In many recent studies for making wetting resistant membrane for robust membrane distillation, surfactant solutions have been used as feed solutions to challenge the fabricated or reference membranes. (Lin et al., 2014; C. Boo et al., 2016; Lee et al., 2016; Huang et al., 2017; Lu et al.,

2018; Zheng et al., 2018; Chen et al., 2018; Chew et al., 2019) In nearly all cases, the CAs of the feed solutions on the tested membranes were substantially higher than that measured with homogeneous liquids (e.g., organic solvent or mixture of miscible liquids) with similar surface tensions. For example, a commercial polyvinylidene fluoride (PVDF) membrane that was wicked by mineral oil (surface tension  $\gamma = 30.8 \text{ mN m}^{-1}$ ) can have a very high CA with a surfactant solution of a similar surface tension. (Huang et al., 2017) It is the goal of this study to explain this interesting phenomenon.

In this work, we comparatively investigate the wetting behaviors of sodium dodecyl sulfate (SDS) solutions and water/ethanol mixtures. We hypothesize that surfactant adsorption near the wetting frontier renders the wetting behaviors of a surfactant solution fundamentally different from that of a homogeneous liquid of the same surface tension. We first measure the CAs of both liquids on non-porous PVDF films and porous PVDF membranes. We then evaluate the adsorption of SDS onto the surface of membrane and membrane pores. Finally, we compare the capillary rise and liquid entry pressure (LEP) with both liquids.

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## 2. Materials and methods

### 2.1. Chemicals and materials

Sodium dodecyl sulfate (SDS, 99%), ethanol (99.5%), Stains-All (CAS: 7423-31-6, 95%), and *N,N*-dimethylformamide (DMF, 99.8%) were purchased from Sigma-Aldrich. PVDF membrane with a nominal pore size of 0.45  $\mu\text{m}$  was purchased from GE Healthcare Life Sciences. PVDF flat sheets were purchased from McMaster-Carr. The received PVDF sheets were polished by SiC sandpapers with grits from 120 to 7000, and then washed by deionized (DI) water. Precision glass capillary tubes were provided by Accu-Glass with a nominal inner diameter of 0.58 mm.

### 2.2. Wetting property characterizations

Surface tensions of SDS and ethanol solutions were measured using the Du Noüy ring method on a Sigma 700 tensiometer coupled with a Pt-Ir ring (Attension, Biolin Scientific) at 20 °C. Ten measurements were conducted for each solution. In-air contact angles (CAs) were measured using an optical tensiometer (Theta Lite, Biolin Scientific). Capillary rises were measured using a reported method. (Extrand and Moon, 2012)

Liquid entry pressure (LEP) of the PVDF membrane with DI water, SDS solution and water/ethanol mixture were measured using an Amicon® cell (Millipore Sigma) with an effective membrane area of 4.1  $\text{cm}^2$ . All solutions used in the test were freshly prepared then allowed to stabilize for 2 h before tests. The pressure was supplied by compressed  $\text{N}_2$  and controlled by a precision digital pressure regulator. The LEP was recorded as the applied pressure at which continuous liquid flow started to emerge.

### 2.3. SDS adsorption

In the SDS adsorption experiments, a 5  $\mu\text{L}$  SDS droplet was carefully placed onto the PVDF membrane surface. The weight of the droplet was measured by a microbalance with a resolution of 1  $\mu\text{g}$ . To quantify SDS adsorption, the sessile drop was withdrawn by a pipette and the concentration of the recovered solution was measured by a spectrophotometric method reported in literature (also section S.1). (Rupprecht et al., 2015) The SDS retained by the membrane was mostly due to adsorption as the mass of the solution retained by the membrane was below 0.5% of the original mass. Therefore, we can estimate the SDS concentration in the withdrawn solution with a reasonable accuracy.

## 3. Results and discussion

By tuning the concentration of SDS and the weight percentage of ethanol in the water/ethanol mixture, we achieved different surface tensions for both liquids (Fig. 1A and B). As surfactants are very effective in reducing the liquid surface tension, it takes only millimolar concentrations of SDS to attain a surface tension similar to that of a water/ethanol mixture with a high weight percentage of ethanol. The minimum surface tension of an SDS solution was slightly higher than 30  $\text{mN m}^{-1}$ , consistent with literature. (Fainerman et al., 2010; Mysels, 1986) At the concentration of 4~5 mM, the surface tension of the SDS solution already approached the minimum even though the critical micelle concentration is ~ 8 mM. For water/ethanol mixture, the surface tension gradually dropped to ~22  $\text{mN m}^{-1}$  as the ethanol weight percentage approached 100%.

The CAs of SDS solution or water/ethanol mixture on a smooth PVDF sheet continued to drop as the liquid surface tension decreased, which is qualitatively congruent with Young's Equation (Fig. 1C). However, the CAs of the SDS solution (on a PVDF sheet) appeared to be systematically greater than that of the water/ethanol mixture, which may be explained in two possible ways. First, Young's equation (eqn. (1)) suggests that the

CA ( $\theta$ ) depends not only on the solid surface tension ( $\gamma_s$ ) and liquid surface tension ( $\gamma_L$ ), but also on the interfacial tension between the liquid and the solid surface ( $\gamma_{sL}$ ). (Israelachvili, 2011)

$$\cos \theta = \frac{\gamma_s - \gamma_{sL}}{\gamma_L} \quad (1)$$

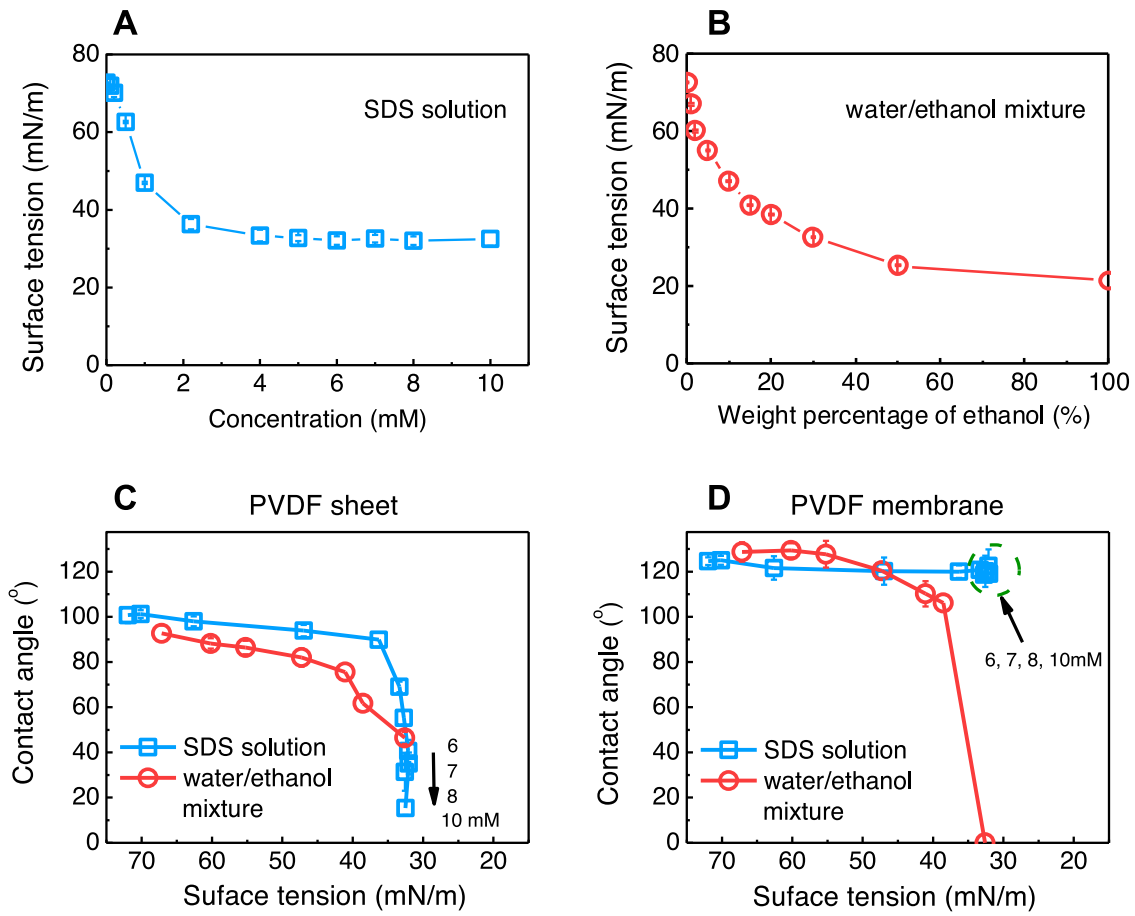
For a given surface (PVDF) and two liquids with the same surface tension,  $\gamma_L$ ,  $\theta$  may differ because the interfacial tension,  $\gamma_{sL}$ , can vary depending on the liquid-solid interaction. The liquid-solid interfacial tension,  $\gamma_{sL}$ , likely differs between an SDS solution and a water/ethanol mixture (interacting with a PVDF surface) even if they have the same liquid surface tension. Because of the different molecular structures of SDS and ethanol and the vastly different mole fractions of aqueous solutions at a similar  $\gamma_L$ , their liquid-solid interfacial tensions are expected to be different according to van Oss' theory that considers both the Lifshitz-van der Waals (LW) and Lewis acid-base (AB) contributions to the interfacial tension. (Van Oss et al., 1988)  $\gamma_{sL}$  has contributions from the LW interaction and the polar, AB interaction.

The second possible explanation is that the adsorption of SDS onto the PVDF membrane, which may dramatically change characteristics of the substrate surface and in turn affect the LW and AB contributions. It was observed that the CA of the SDS solution continued to decrease with increasing SDS concentration beyond 5 mM (Fig. 1C) even if the liquid surface tension already reached a minimum (Fig. 1A); Additionally, dynamic wetting was observed with the SDS solution, i.e., the CA slowly decreased over time. With an SDS concentration above 5 mM, the PVDF sheet was fully wetted (i.e., CA not detectable) in about 5 to 10 min. Both observations suggest that surfactant adsorption plays an important role that distinguishes the wetting behavior of a surfactant solution vs. a homogeneous liquid such as water/ethanol mixture.

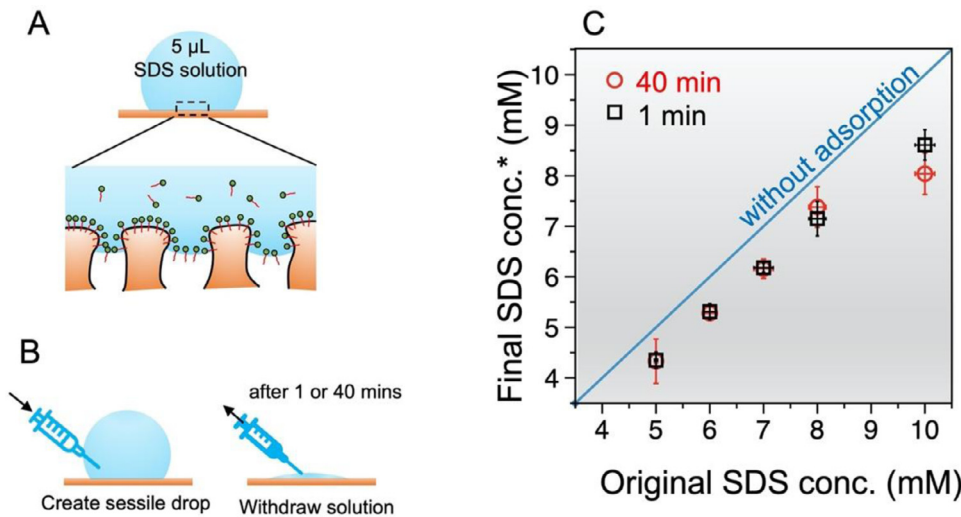
The behavior of dynamic wetting has been investigated in previous studies but not fully understood. (Lee et al., 2008; Milne and Amirfazli, 2010) One possible mechanism is the autophilic effect, i.e., adsorption of surfactants onto the unwetted solid surface near the triple-phase boundary (TPB). (Kumar et al., 2003) However, it may also be explained by the adsorption-induced reduction of SDS concentration and increase of  $\gamma_L$  in the solution near the TPB, which results in a higher CA than prediction based on bulk  $\gamma_L$ . The TPB expands when the wetted surface near the boundary is saturated and can no longer adsorb more surfactants.

The liquid droplets behaved very differently on a porous PVDF membrane surface (Fig. 1D) as compared to a smooth PVDF flat sheet. With increasing ethanol concentration, the CA of the droplets decreased slowly (from 129° to 106°) as  $\gamma_L$  decreased from 67.2 to 38.5  $\text{mN m}^{-1}$  before it exceeded a critical value. Above a critical ethanol concentration, spontaneous wicking (or imbibition) into the porous membrane occurred (i.e., CA=0 in Fig. 1D). Below the that critical concentration, the apparent CA of SDS solution droplets on a porous PVDF membrane were consistently higher than that on a smooth PVDF sheet, which can be readily explained by the Cassie-Baxter theory. (Wang et al., 2016) Without spontaneous wicking, the sessile drop of water/ethanol mixture was supported by a composite surface of the PVDF solid and air in the pore, which, according to the Cassie-Baxter theory, would result in a higher apparent CA. Beyond the threshold  $\gamma_L$ , the system transitioned spontaneously from the Cassie-Baxter state to the Wenzel state.

With droplets of SDS solution on a porous PVDF membrane, the apparent CA remained almost unchanged throughout the entire tested range of concentration and surface tension (Fig. 1D). On one hand, the behavior of SDS solution on a PVDF membrane is strikingly different compared to that of water/ethanol mixture on a PVDF membrane or that of SDS solution on a smooth non-porous PVDF flat sheet. On the other hand, this observation is not too surprising as it has been observed, but not elucidated, in previous studies. (Lee et al., 2016; Huang et al., 2017; Zheng et al., 2018; C. Boo et al., 2016) Compared to CA measured with a non-porous flat sheet, the CAs measured with a porous membrane were consistently higher, again due to the system being in a Cassie-Baxter state.



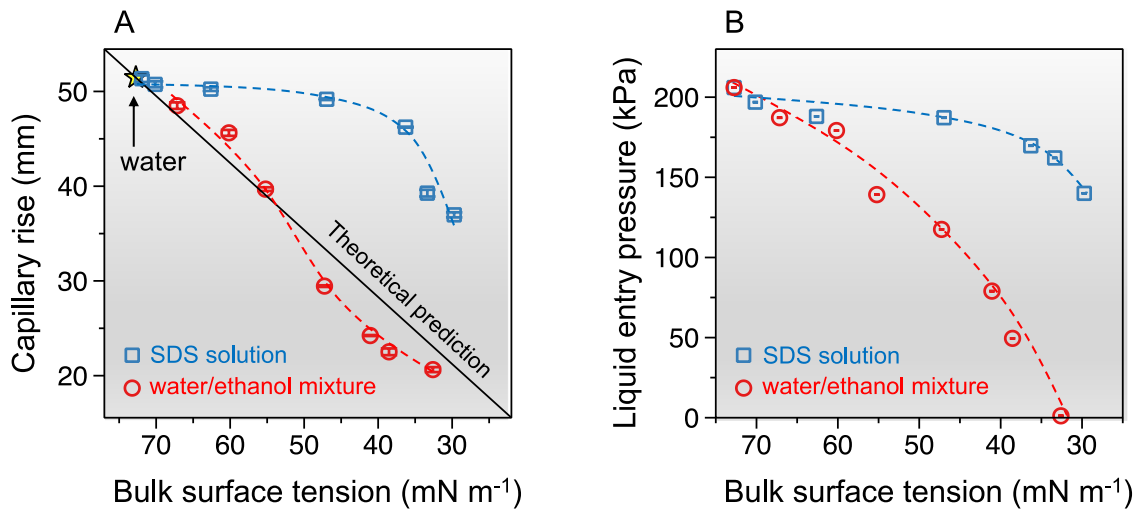
**Fig. 1.** Surface tensions of (A) SDS solutions at different concentrations and (B) water/ethanol mixture with different ethanol weight fraction. Sessile drop in-air CAs for liquids of different surface tensions on (C) non-porous PVDF sheet, and (D) porous PVDF membrane. All CAs were measured within 30 s after the droplets were placed on the surface of the PVDF sheet or membranes and the CAs were quasi-stable during measurements (i.e., no rapid change of CA was observed). However, when the SDS concentration exceeded 5 mM, the CA of an SDS solution on a PVDF sheet (C) dropped to zero in 5–10 mins. In all panels, the error bars represent standard deviations.



**Fig. 2.** (A) Schematic illustration of surfactant adsorption to the membrane surface and pore surface. (B) Illustration of the adsorption experiment including the creation of a sessile drop and the subsequent extraction of SDS solution from the sessile drop. The mass of SDS solution retained by the membrane is always less than 0.5% of the original mass. (C) Final SDS concentration in the withdrawn solution vs. the original SDS concentration for two different adsorption time scales. The error bars represent standard deviations. \*We note that the final SDS concentrations in (C) were calculated based on the initial volume of the droplet and serve as a proxy measure of the SDS mass in the droplet to demonstrate adsorption. The actual SDS concentrations, especially in the droplets after 40 mins, were likely higher due to reduction of the droplet volume due to evaporation.

The most interesting feature observed in Fig. 1D is that an SDS solution never wicked a porous PVDF membrane even when its surface tension dropped below the critical  $\gamma_L$  for water/ethanol mixture. This phenomenon is most likely caused by the adsorption of SDS onto the membrane surface and pore surface (Fig. 2A). The surface tension of the SDS solution in the wetting frontier (i.e., the liquid-air interface)

is substantially higher than that in the bulk solution due to the fast adsorption of SDS onto the pore surface. While the surfactants in the bulk can transport to the wetting frontier, the surface tension of the wetting frontier will not decrease sufficiently until the wetted pore surface near the wetting frontier is saturated and can no longer adsorb any surfactant.



**Fig. 3.** (A) Capillary rise as a function of bulk surface tension for water/ethanol mixture and SDS solution. The line of theoretical prediction is obtained from eqn. 3 by assuming complete wetting throughout the range of bulk surface tension. (B) LEP as a function of bulk surface tension for water/ethanol mixture and SDS solution. The error bars represent standard deviations.

This theory has been applied successfully in modeling the kinetics of wetting by surfactants in membrane distillation (MD). (Wang et al., 2018) In such a model, the surface tension in the wetting frontier must drop until the LEP is lower than the hydraulic pressure (difference) for the liquid-air interface to propagate forward. The two primary differences between membrane wetting in MD and wetting by a sessile drop on a membrane are: (1) the absence of applied pressure in CA measurement except for the negligibly small pressure exerted by the weight of the sessile drop; and (2) the absence of advective surfactant transport in a sessile drop on a porous membrane. These differences lead to the metastable Cassie-Baxter state with a high CA as shown in Fig. 1D.

To confirm the presence of significant adsorption of SDS onto the membrane surface and pore surface, we performed experiments in which we withdraw the solution from a sessile drop and measure the amount of SDS remaining in the withdrawn solution (Fig. 2B). The results from the adsorption experiments clearly suggest the presence of SDS adsorption which reduces the SDS concentration in the withdrawn solution (Fig. 2C). In most cases, the SDS concentration was reduced by at least 10%. In general, a higher reduction in SDS concentration was observed with a higher starting concentration, which suggests the dependence of the degree of adsorption on the bulk SDS concentration.

When the SDS concentration was below 10 mM, there was no observable difference in the SDS concentration of the solutions withdrawn after 1 min vs. 40 mins, which implies that (1) SDS adsorption onto the membrane surface and the surface near the pore entrance was very fast; and (2) the SDS solution mostly remained on the surface without further wicking deeper into the pores. At 10 mM, however, considerably more SDS was retained by the PVDF membrane after 40 mins as compared to that after 1 min. Because the initial adsorption of SDS was fast, we believe that the additional SDS adsorption between the 1st and the 40th mins was caused by further wicking of the solution. The higher bulk SDS concentration promoted further liquid penetration into the pores due to the faster diffusion of SDS to the wetting frontier resulting from a larger concentration gradient. Based on a mass transport model developed previously, we estimate that the sessile drop containing 10 mM SDS would increase its wetting depth by  $\sim 77 \mu\text{m}$  in 40 mins (section S.2). Such an increase in wetting depth is  $\sim 4.5\%$  of the height of the sessile drop ( $\sim 1.7 \text{ mm}$ , section S.3) and would not lead to drastic change in the shape of the sessile drop or the CA. However, the CA did slightly decrease over the 40 mins of experiments regardless of SDS concentration due to water evaporation (even with DI water, see Fig. S3).

To further corroborate the theory of adsorption-induced wetting mitigation, we performed two additional sets of experiments. First, we measured the capillary rise of water/ethanol mixture and SDS solution with a series of surface tensions. The capillary rise,  $h$ , can in theory be predicted by the following equation:

$$h = \frac{2\gamma_L \cos \theta}{\rho g r} \quad (2)$$

where  $\theta$  is the intrinsic CA between the liquid and the capillary wall,  $\rho$  is the liquid density,  $r$  is the capillary radius, and  $g$  is the acceleration of gravity. For a clean glass surface,  $\theta$  is zero for water (and can thus be even lower for liquids with lower surface tensions) and eqn. (2) can thus be reduced to

$$h = \frac{2\gamma_L}{\rho g r} \quad (3)$$

Our experimental results suggest that the Eq. (3) can describe the trend of capillary rise measured using water/ethanol mixture with reasonable accuracy but poorly predicts the capillary rise of an SDS solution (Fig. 3A). An SDS solution has a higher capillary rise than that of a water/ethanol mixture of the same surface tension because of the adsorption of the SDS onto the capillary wall. The adsorption of SDS is substantially faster than the upward diffusion of SDS from the reservoir to the wetting frontier, as the capillary radius ( $\sim 0.29 \text{ mm}$ ) is more than two orders of magnitude smaller than the capillary rise. Therefore, the SDS solution inside the capillary had a significantly lower SDS concentration, and thus, a significantly higher surface tension, than that of the bulk solution in the reservoir. Consequently, the capillary rise was much higher than what would have been predicted using a homogeneous liquid with the same bulk surface tension.

As the SDS concentration continued to increase (which led to the reduction in bulk surface tension), the capillary rise eventually dropped. This is not only because of the lower bulk surface tension, but perhaps more important because of the higher bulk SDS concentration that reduced the relative impact of adsorption on the bulk concentration. In other words, assuming the capillary surface was always saturated with SDS with a constant aerial density of SDS, the percentage reduction of SDS concentration was considerably higher for an SDS solution with a lower starting concentration.

The liquid entry pressure (LEP) also differed substantially between water/ethanol mixtures and SDS solutions with the same bulk surface tension (Fig. 3B), especially when the concentration was high, and the



bulk surface tension was low. The dependence of  $\gamma_L$  on LEP is described by the following relation:

$$LEP \propto -\frac{\gamma_L \cos \theta}{r_{max}} \quad (4)$$

where  $r_{max}$  is the maximum pore radius, and  $\theta$  is the intrinsic CA between the liquid and the membrane material. Although LEP has the similar dependence on  $\gamma_L \cos \theta$  as capillary rise,  $\theta$  is non-zero for a hydrophobic membrane and thus the term  $\gamma_L \cos \theta$  cannot be reduced to  $\gamma_L$  as in Eq. (3). For an SDS solution and a water/ethanol mixture of similar  $\gamma_L$ , their intrinsic CAs are slightly different (Fig. 1C) but the difference was not sufficient to explain their difference in LEP (Fig. 3B).

The large difference in LEP between SDS solution and water/ethanol mixture with the same surface tension arises again from the SDS adsorption onto the membrane pore surface. When the SDS solution was partially forced into the pores under the applied hydraulic pressure, the adsorption of SDS onto the pore surface depleted the SDS and increased the liquid surface tension near the wetting frontier. Because the LEP is determined by the force balance at the TPB at the wetting frontier, the measured LEP was substantially higher than the LEP predicted using the bulk surface tension due to the adsorption-induced increase of surface tension.

#### 4. Conclusion

In this work, we elucidated an interesting phenomenon that has been commonly observed in previous studies, i.e., surfactant solutions with low surface tension can maintain a high CA on a porous hydrophobic membrane. Using SDS as an example, we demonstrated with multiple sets of experiments that this phenomenon is caused by the adsorption of surfactants onto the membrane pore surface that reduces the surfactant concentration and increases the liquid surface tension at the wetting frontier.

Since CA measurement is so prevalent as a membrane characterization technique, we need to be particularly careful when inferring membrane properties using results from CA measurements. Our analysis suggests that there are complications in interpreting CA of a solution of surface-active agents (i.e., surfactants) which (1) has a strong preference for interface and (2) can change surface tension with a low concentration. In general, CA should be measured using homogeneous liquid, such as a pure solvent or a mixture of miscible solvents, to avoid the complications encountered with surfactant solutions.

While this study focused on CA measurements, it broadly shows that the wetting behaviors of a surfactant solution differ substantially from that of a homogeneous liquid like a water/alcohol mixture because of surfactant adsorption near the wetting frontier. Such an observation may have broad and important implications on a wide variety of wetting phenomena involving surfactant solutions.

#### Declaration of Competing Interest

The authors declare no competing financial interest.

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#### Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.memlet.2022.100015](https://doi.org/10.1016/j.memlet.2022.100015).

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