CHAPTER V
SURFACTANT ADSORPTION ON HYDROPHOBIC SURFACES: THE EFFECT OF SURFACTANT STRUCTURE AND SOLUTION pH

5.1 Abstract

The adsorption isotherms of cationic surfactants with various tail lengths and anionic surfactants with various headgroups on eight hydrophobic surfaces of various plastics – polytetrafluoroetylene (PTFE), high density polyethylene (HDPE), polycarbonate (PC), polyvinylchloride (PVC), acrylonitrile butadiene styrene (ABS), polymethyl methacrylate (PMMA), polyhexamethylene adipamide (PA66), and polycapolactone (PCL) – were measured. The effects of pH on surfactant adsorption also were examined. For cationic surfactants, homologous series of ammonium bromide series of surfactants were studied; while for anionic surfactants, three different headgroups (sulfate, sulfonate and carboxylate) were studied. The sulphate and sulfonate surfactants had approximately the same CMC ($\sim 1 \times 10^{-3}$ M) while that of the carboxylate surfactant was significantly higher and had a significantly smaller hydrophobic chain length. At a low surfactant concentration, an increase in tail length causes an increase in surfactant adsorption for both anionic and cationic surfactants. For cationic surfactants, the amount of adsorption at the CMC is almost independent of the surfactant tail length. In case of anionic adsorption, the carboxylate headgroup has the highest adsorption. In addition, pH does not significantly affect surfactant adsorption for all studied systems.

Keywords: Surfactant adsorption/Surface tension/Tail length/Headgroup/ Hydrophobicity

5.2 Introduction

The adsorption of surfactant onto solid/liquid interfaces has many applications, including processes with hydrophobic surfaces such as printing and deinking of plastics. The surfactant acts as a wetting agent to enhance the ability of

pigment to wet and spread over hydrophobic surface, or in deinking process causes a charge repulsion between the pigment and the polymer so as to allow the ink to detach from the polymer [1]. The surfactant adsorption capacity and aggregation morphology on a surface depend on several factors including surface charge, molecular structure of surfactant, solution pH, ionic strength, and temperature [2-4]. Surfactant adsorption has been studied extensively; one typical study is to measure a surfactant adsorption isotherm that is a plot of the amount adsorbed vs. surfactant concentration in solution. For particulate surfaces, surfactant adsorption is typically determined using a solution depletion method, i.e. measure the surfactant concentrations before and after adsorption with the difference being related to the amount adsorbed at different initial or equilibrium surfactant concentration in bulk liquid.

For hydrophobic surfaces, surfactant aggregates tend to form either monolayer or hemimicellar structure, i.e. hemispherical or hemicylindrical, with the charged or polar group oriented towards the aqueous solution [5]. Jodar-Reyes *et al.* [6] studied the adsorption of different amphiphilic molecules onto polystyrene by using the solution depletion method. Their surfactant adsorption isotherms indicated that the interaction between the surfactant tail group and non-polar part of surface is the main mechanism involved in the adsorption. Hoeft and Zollars [7] studied the adsorption of anionic surfactants on hydrophobic surfaces (sulfonated polystyrene latexes) and their results demonstrated that the less polar linear alkyl sulfonate (SLSN) provided lower amount of adsorption than the linear alkyl sulfates of the same chain length (SLS). Ali *et al.* [8] concluded for above results that the electrostatic repulsion between the negatively charged surface of sulfonated polystyrene latexes and the anionic headgroups of either SLS or SLSN caused the surfactant molecules to adsorb in a more extended conformation, thus allowing more molecules to contact the surface.

Higher or lower solution pH promotes surface charging which can lead to more association or dissociation between solid surface and surfactant molecule, depending on the relative charges of the two materials [9]. The addition of neutral electrolyte also tends to increase the amount of adsorption of ionic surfactants. This increase is due to the shielding of the electric field around the headgroups, which

results in decreasing the electrostatic repulsion between the similarly charged headgroups [2,10].

The aim in this research was to study the surfactant adsorption behavior on several hydrophobic surfaces (plastics) by using the solution depletion method. The surfactants studied in this work were the cationic surfactants with various hydrophobic tail lengths and anionic surfactants with various headgroups. In addition, the effect of solution pH was also determined on hexadecyl trimethyl ammonium bromide (CTAB) and sodium dodecyl sulfate (SDS) adsorption onto polytetrafluoroetylene (PTFE) and polyvinylchloride (PVC).

5.3 Experimental

5.3.1 Materials

Polytetrafluoroetylene (PTFE) was purchased from Chemical Innovation Co., Ltd. High density polyethylene (HDPE) was obtained from Thai Polyethylene Co., Ltd. Polycarbonate (PC), grade Makrolon®PC, was obtained from Bayer Thai Co., Ltd. Polyvinylchloride (PVC), grade SG580, was obtained from Thai Plastic and Chemicals Public Co., Ltd. Acrylonitrile butadiene styrene (ABS) was obtained from IRPC Public Co., Ltd. Polymethyl methacrylate (PMMA) was obtained from Diapolyacrylate Co., Ltd. Polyhexamethylene adipamide (PA66), grade A31, was obtained from SY Smile Co., Ltd. And polycapolactone (PCL) was purchased from Sigma-Aldrich Co., Ltd. The provided pellets were ground into a powder by a hammer mill. The powder was then sieved to obtain particles sizes in the range of 45-125 μm. Finally, the sieved powders were rinsed with distilled water 3-4 times and kept at room temperature until dried. Two types of surfactants were used in this work: cationic surfactants – hexadecyl trimethyl ammonium bromide (CTAB, $C_{19}H_{42}NBr$), tetradecyl trimethyl ammonium bromide (TTAB, $C_{17}H_{38}NBr$) and dodecyl trimethyl ammonium bromide (DTAB, $C_{15}H_{34}NBr$) supplied by Fluka Co., Ltd. (Switzerland) with a purity of 98% and the anionic surfactants – sodium dodecylsulphate (SDS or $C_{12}H_{25}SO_4Na$, $\geq$99% purity), 4-octyl benzene sulfonate sodium salt (SOBS or $C_{14}H_{21}SO_3Na$, 97% purity) and sodium octanoate (C8 or $C_8H_{15}O_2Na$, >99% purity) purchased from Sigma Chemical Company (St. Louis,

MO). AR grade hydrobromic acid (HBr, 48% purity), purchased from Farmitalia Carlo Erba (Thailand Br) Co., Ltd. (BKK, Thailand) and sodium hydroxide (NaOH, 99% purity), purchased from RCI Labscan Co., Ltd. (BKK, Thailand) were used to adjust pH. All chemicals were used without further purification.

5.3.2 Surfactant Adsorption Experiments

Adsorption experiments were carried out using the solution depletion method in a series of vials with screw caps. 0.25 g of each powdered plastic sample was added into 20 mL of a surfactant solution with different surfactant concentrations. NaOH or HBr were added if necessary to adjust the solution pH at 9 or 3, respectively. Solutions were allowed to equilibrate in an incubator at 30°C and shaken by hand twice a day for 5 d. The supernatants were filtered using a Nylon syringe filter with 0.45 μm pore size and the filtrates were analyzed for the amount of surfactant in the bulk phase by using a total organic carbon analyzer (Shimadzu, TOC-V CSH).

The surfactant adsorption was calculated by the concentration difference method shown in Equation 5.1 [11], and the adsorption isotherm was plotted on log-log scale between the amount of adsorbed surfactant at interface and equilibrium concentration in the bulk solution.

$$\Gamma_{SL} = \frac{(C_0 - C)V}{W_{plastic}a_s} \tag{5.1}$$

The inversion of surfactant adsorption was used to calculate the occupied surface area per molecule (A):

$$A = \frac{10^{26}}{N_A \Gamma_{SL}} \tag{5.2}$$

where Γ_{SL} is the surfactant adsorption at solid/liquid interface; μmole/m², C_0 is the initial surfactant concentration (measured weight of surfactant/molecular weight, μM); C is the equilibrium surfactant concentration (measured weight of surfactant

via TOC/molecular weight) (μM); V is the volume of a surfactant solution, L; $W_{plastic}$ is the weight of a powdered plastic sample, g; a_s is the BET surface area of a powdered plastic sample; m^2/g, and N_A is Avogadro's number (6.02×10^{23}).

5.3.3 Other Measurements

The surface tensions of surfactant solutions were measured by the pendant drop technique using a drop shape analysis instrument (Krüss, DSA10) at room temperature (30°C). The critical micelle concentration (CMC) of studied surfactants was obtained from the break point of the semi-logarithmic plot of the surface tension (γ_{LV}) vs. initial surfactant concentration.

The specific surface areas of the powdered plastics were measured by a surface area analyzer (Quantachrome, Autosorb-1). The measurement technique was carried out by measuring the quantities of nitrogen adsorbed onto the solid surface at liquid nitrogen temperature (-196°C). The powdered plastic was dried and out-gassed in the sample cell at 80°C for at least 24 h before the nitrogen adsorption step.

Zeta potentials of studied plastic powders were determined by using a zeta meter (Zeta-meter, 3.0+). A quantity of 0.1 g of each studied powdered plastic was added to 40 mL of distilled water. The pH was adjusted by addition of a concentrated NaOH or HBr solution. The samples were then placed in an electrophoresis cell maintained at 30°C. The two electrodes placed at the ends of the cell were connected to a power supply, which created an electric field, causing the charged colloids to move. Velocities of individual particles were tracked via a grid in the eyepiece of the microscope. The pH was adjusted so that the particles showed no net movement, indicating the point of zero charge (PZC).

5.4 Results and Discussion

BET surface areas for all studied plastics are shown in Table 5.1. All of the plastics possess very low specific surface areas. The CMC values of all studied

surfactant solutions obtained from the surface tension isotherms (Figure 5.1) also were shown in Table 5.2, which is similar to literature values [12].

Table 5.1 Specific surface areas of all studied plastics

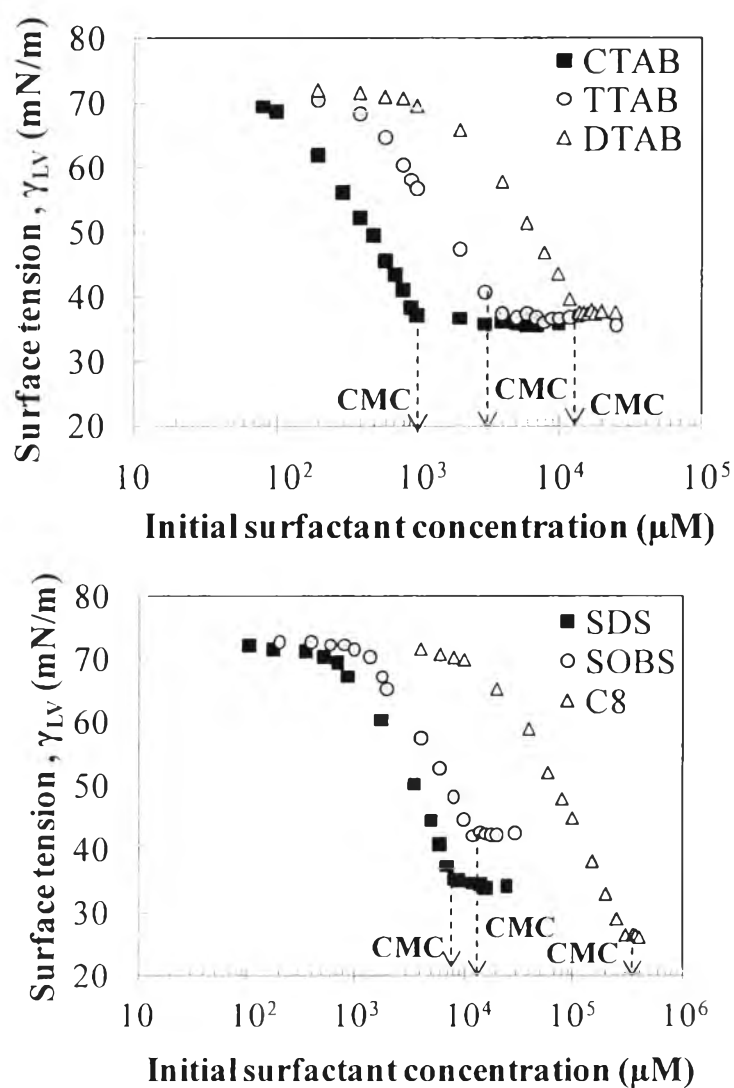| Plastic	HDPE	PTFE	ABS	PMMA	PC	PVC	PA66	PCL
Specific surface area (m^2/g)	22.25	4.712	9.674	7.436	5.607	4.266	18.73	4.983

Figure 5.1 Surface tension isotherms of cationic and anionic surfactants without adjusted pH.

Table 5.2 CMC values of all studied surfactants

Surfactants	CMC (μM)
CTAB	1.0×10^3
TTAB	4.0×10^3
DTAB	1.4×10^4
SDS	8.3×10^3
SOBS	1.2×10^4
C8	3.5×10^5

5.4.1 The Effect of Surfactant Tail Group on Surfactant Adsorption

CTAB, TTAB and DTAB were chosen to study the effect of tail length on adsorption which are the members of homologous series to avoid the effect of polar headgroup. The adsorption isotherms of them on eight plastics without adjusted pH are shown in Figure 5.2. Adsorption isotherms are said to have four features: Region I represents adsorption where the monomers are isolated on the surface and often involves attraction between opposite charges of the surface and the headgroup. That is the adsorption by having the cationic head groups onto the negatively charged sites on the plastic surface and the hydrophobic alkyl chains orient in horizontal plane or slightly tilt or L-shape onto the uncharged sites of the surface or the backbone of polymer (parallel to the surface of the solid). As the tail length of surfactant decreased, higher equilibrium surfactant concentration is required to achieve Region I [13]. This is because the shorter tail length has less hydrophobic interaction on hydrophobic sites of plastic surfaces. Region II is the steepest sloping region where adsorption is cooperative as the equilibrium surfactant concentration increases. The hydrophobic tail portion can adsorb either on vacant uncharged sites or toward the aqueous phase or in perpendicular to the surface. Region III is where packing on the surface becomes tight defined a close-packed monolayer and adsorption slows down (as seen in Figure 5.3) and Region IV is where no more adsorption occurs, corresponding to the saturated surfactant adsorption when an equilibrium surfactant concentration is greater than the CMC and the maximum adsorption can be determined in this region.
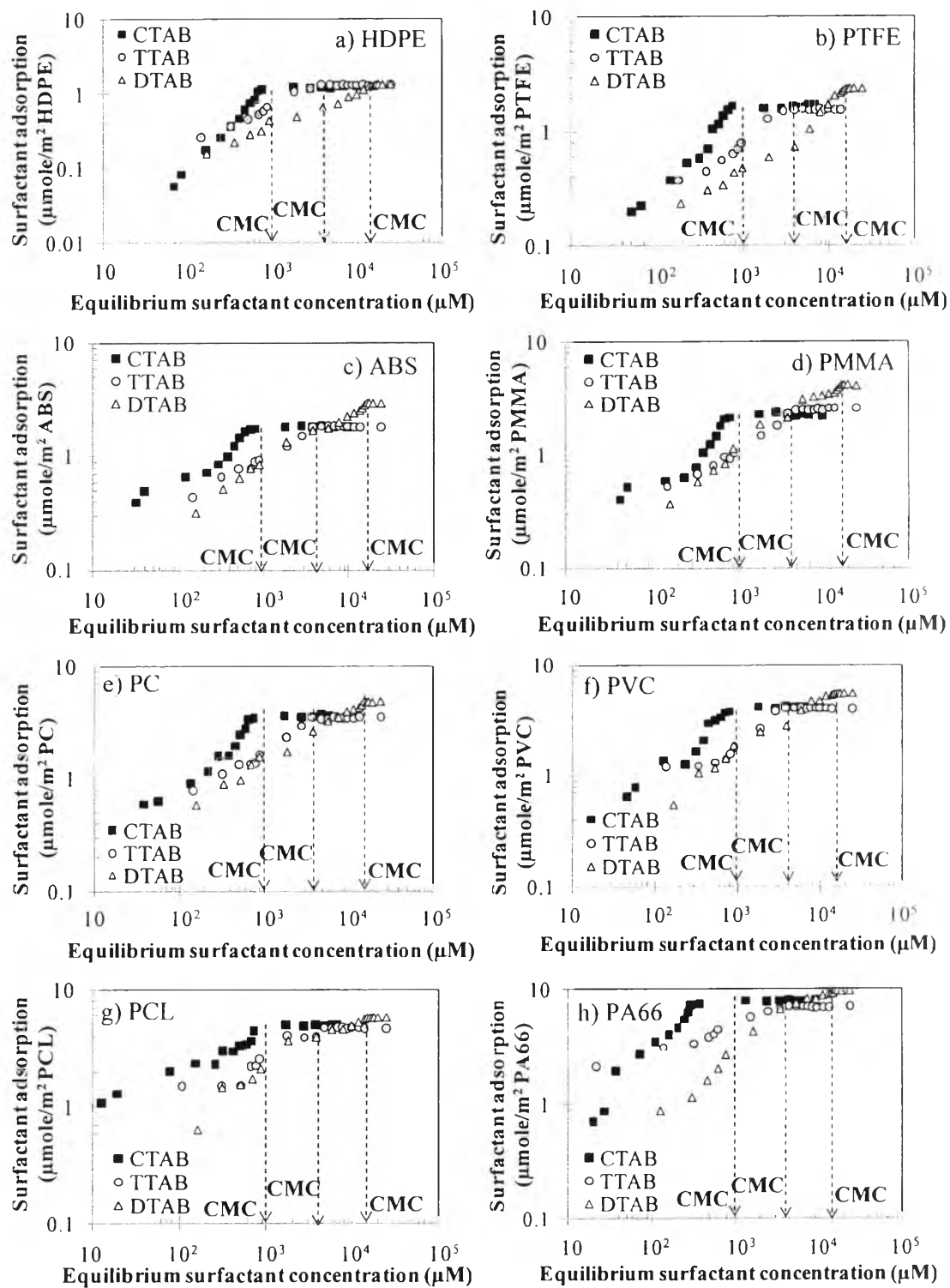
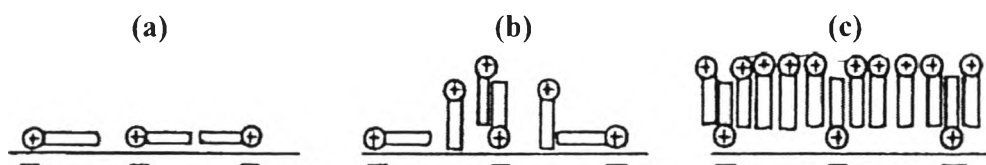**Figure 5.2** Adsorption isotherms of cationic surfactants on different hydrophobic surfaces at 30°C.

Figure 5.3 Schematic illustration of the adsorption of cationic surfactant on low-charged hydrophobic surfaces; (a) neutralization of surface charge; (b) partial surface coverage; (c) monolayer coverage [14].

The transition point between Region I and II is often called the critical aggregate concentration (CAC) and is typically about 0.1 CMC. The Region II to Region III transition is often not distinguishable. The Region III to Region IV transition occurs at the CMC.

For any given plastic below the CMC, CTAB provided the highest adsorption and followed by TTAB and DTAB, respectively, confirming that an increase in the alkyl chain length can increase the efficiency of surfactant adsorption [2,14,15]. This is observed in Figure 5.2, as the slopes of regions II are clearly steepest for the CTAB that is the CTAB adsorption increases most rapidly with concentration.

A higher alkyl chain length will have a higher driving force to not be an isolated monomer in solution; the same driving force is responsible for the lower CMC. At the CMCs, the maximum adsorptions of all studied surfactant on all given plastic surfaces also are shown in Table 5.3. The values are similar for all three different tail length surfactants on all plastics, suggesting that the surfaces are saturated with surfactant. The adsorption of DTAB on all plastics is slightly higher, suggesting that more sites are available to this shorter alkyl chain surfactant.

The adsorption areas calculated for each plastic are shown in Table 5.4, which can be compared to the close-packed monolayer at the water/air interface of 54 Å^2/CTAB molecule, 59 Å^2/TTAB molecule and 51 Å^2/DTAB molecule calculated using the Gibbs adsorption equation applied to surface tension data [2]. Almost none of the data matches these numbers. For areas per headgroup that are greater than water/air interface values, BET surface areas are likely greater than those available to a surfactant. For those less than water/air interface values, then

possibly a bilayer was forming. It is interesting that the degree of hydrophobicity of studied plastics revealed in Chapter IV (PTFE > HDPE > PC > PVC > ABS > PMMA > PA66 > PCL) shows slightly different order from the order of maximum adsorption, (HDPE < PTFE < ABS < PMMA < PC < PVC < PCL < PA66) which is likely due to differences in surface morphology (i.e. the surface roughness) [3,16].

A rougher surface, corresponding to lower the gap between the peaks of surface will reduce adsorption relative to the available surface area due to a reduction in tail-tail interactions which favor surfactant adsorption [3]. If the occupied surface area per surfactant molecule is higher than this gap, the surfactant molecule will not adsorb on this site. A shift from monolayer to bilayer adsorption would be expected in the case of decreasing hydrophobicity as was found. Such a shift is not strictly a function of surface chemistry and these other factors may explain some of the difference between the two lists.

Table 5.3 Maximum adsorption of all studied surfactants on eight plastics at 30°C

Surfactant	pH	The maximum adsorption (μmole/m^2)							
		HDPE	PTFE	ABS	PMMA	PC	PVC	PCL	PA66
CTAB	3	-	1.64	-	-	-	4.70	-	-
	N*	1.25	1.62	1.83	2.27	3.63	4.13	4.93	7.91
	9	-	1.82	-	-	-	3.57	-	-
TTAB	N	1.32	1.54	1.84	2.53	3.47	4.06	4.67	7.02
DTAB	N	1.34	2.37	2.91	4.15	4.81	5.46	5.70	9.78
SDS	3	-	2.48	-	-	-	5.61	-	-
	N	0.82	1.95	1.17	2.03	2.25	2.41	7.26	5.79
	9	-	1.65	-	-	-	2.72	-	-
SOBS	N	2.08	1.37	3.80	10.2	4.72	7.56	12.1	14.9
C8	N	2.08	5.29	3.93	13.6	6.67	10.5	14.2	15.6

*Note N stands for the neutral solution (pH of 5.5-6).

Table 5.4 The occupied surface areas of all studied surfactants on eight plastics at different solution pHs

Surfactant	pH	The occupied surface area (Å^2/molecule)							
		HDPE	PTFE	ABS	PMMA	PC	PVC	PCL	PA66
CTAB	3	-	101	-	-	-	35.4	-	-
	N	133	101	90.8	73.3	45.8	40.2	33.7	21.0
	9	-	91.3	-	-	-	46.6	-	-
TTAB	N	126	108	90.4	65.6	47.9	40.9	35.6	23.7
DTAB	N	124	70.2	57.0	40.1	34.5	30.4	29.1	17.0
SDS	3	-	67.0	-	-	-	29.6	-	-
	N	203	85.3	142	81.8	73.9	69.0	22.9	28.7
	9	-	101	-	-	-	61.0	-	-
SOBS	N	80.0	121	43.7	16.3	35.2	22.0	13.7	11.2
C8	N	80.0	31.4	42.3	12.2	24.9	15.8	11.7	10.6

5.4.2 The Effect of Surfactant Headgroup on Surfactant Adsorption

Headgroups in this study were sulfate (SDS), sulfonate (SOBS), and carboxylate (C8). Adsorption isotherms shown in Figure 5.4 represent the adsorption of SDS, SOBS and C8 on various hydrophobic surfaces. The SDS adsorption isotherms for all studied plastics showed a slightly sigmoidal shape which indicates the strong interaction between the adsorbed surfactant molecules themselves with hydrophobic chain adsorption [17]. However, the adsorption isotherms also do not reveal the first horizontal region of S-shape isotherm which is attributed to the electrostatic repulsion between negatively charged site of plastic surface and anionic surfactant head group. So, the adsorption isotherm is appeared in Region II which corresponds to more hydrophobic interaction with Van der Waals and dispersion forces between the surfactant hydrocarbon chain and hydrocarbon backbone of polymer than anionic headgroup interaction with slightly positively charged sites of polymer surface. Therefore, SDS with the longest hydrocarbon chain can more

adsorb on these surfaces. When the concentration closes to the CMC, the slopes gradually decrease due to closely-packed formation, leading to the electrostatic repulsion between coming and adsorbed anionic head group. The concentration above CMC, the isotherm is leveled off and the saturated polymer surfaced by surfactant coverage is occurred which corresponds to Region IV, indicating to the maximum adsorption. For SOBS and C8, there is only one slope prior to Region IV which is not unexpected due to the smaller alkyl chain length [18]. Similar to the cationic surfactants, the smallest alkyl chain length C8 has the highest adsorption at the CMC. The slopes of the isotherms below CMC of 0.7 for SOBS and of 0.9 for C8 are higher than that of SDS (0.3). Such a result is somewhat surprising since the longer alkyl chain surfactant would be expected to have a higher slope; however more polar of sulfate group provides more headgroup repulsion than benzenesulfonate and carboxylate groups that causes the lower adsorption level [19]. The area per molecule assuming close-packed monolayers at the water/air interface are 51 Å^2/SDS molecule, 66 Å^2/SOBS molecule and 57 Å^2/C8 molecule by using the Gibbs equation [20] whereas the areas per molecule calculated are shown in Table 5.4. Again, as previously a decrease in hydrophobicity roughly scales with an increase in adsorption. Table 5.4 indicates bilayer formation on PCL and PA66 for all three anionic surfactants and additionally on PMMA, PC and PVC for only SOBS and C8.
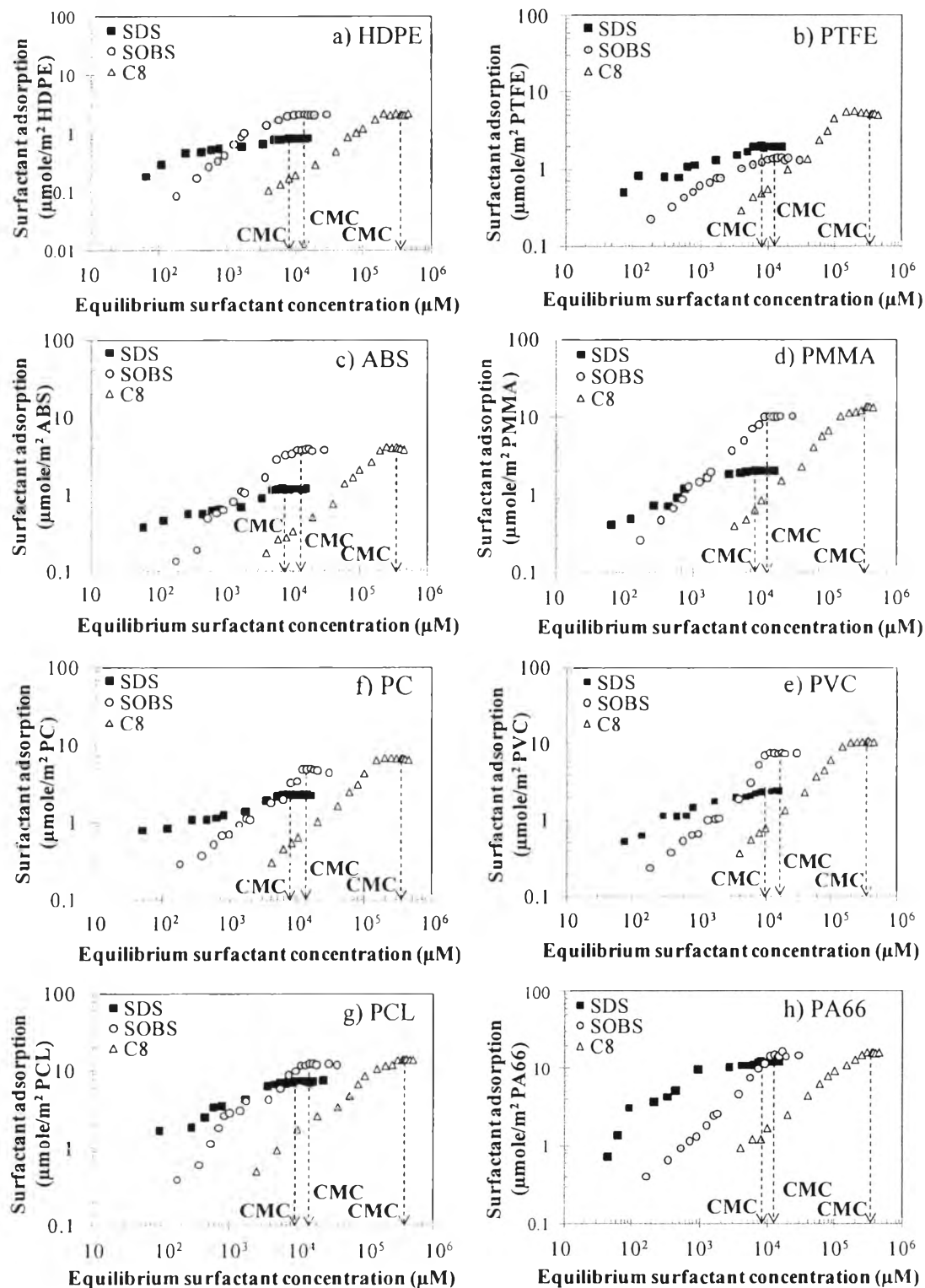
Figure 5.4 Adsorption isotherms of anionic surfactants on different hydrophobic surfaces at 30°C.

5.4.3 The Effect of pH on Surfactant Adsorption

Adsorption isotherms of CTAB and SDS on PVC and PTFE with different pHs are shown in Figure 5.5. For both cases (CTAB and SDS), the pH level did not change the CMC from the Region III-Region IV transition concentration or surface tension of the isotherm (Figures 5.5 and 5.6, respectively). Surprisingly, the surface tension of CTAB as shown in Figure 5.6a shows that the surface tension decrease for pH 3 and 9 is lower than for neutral pH below the CMC but the CMC does not change. Perhaps H^+ or OH^- from added acid or base in surfactant solutions may interfere the surfactant molecule at air/water interface. The areas per headgroup are therefore different as are shown in Table 5.4.

From the zeta potential measurement, the point of zero charge (PZC) of PVC and PTFE are 2.7 and 2.9, respectively. Thus, the surfaces of both plastics are net negatively charged over the studied pH (3 - 9). Hence, the expectation is that the adsorption increases for as pH increases and decreases for CTAB and SDS respectively without any large changes assuming that surface charge-surfactant headgroup charge plays a role in adsorption. From Figure 5.5, the isotherms do not show any difference in adsorption of CTAB with solution pH on PTFE, while that of CTAB on PVC increases with pH decreasing. Such a result suggests that the adsorption of CTAB is not charge driven; however the area per headgroup calculation suggests CTAB bilayer formation on PVC which therefore is contradictory. The trend for SDS is exactly as expected; the adsorption increases as the pH decreases. Surprisingly, for PVC the adsorption of SDS decreased as the pH changed from 9 to 6, which is opposite to what is expected.
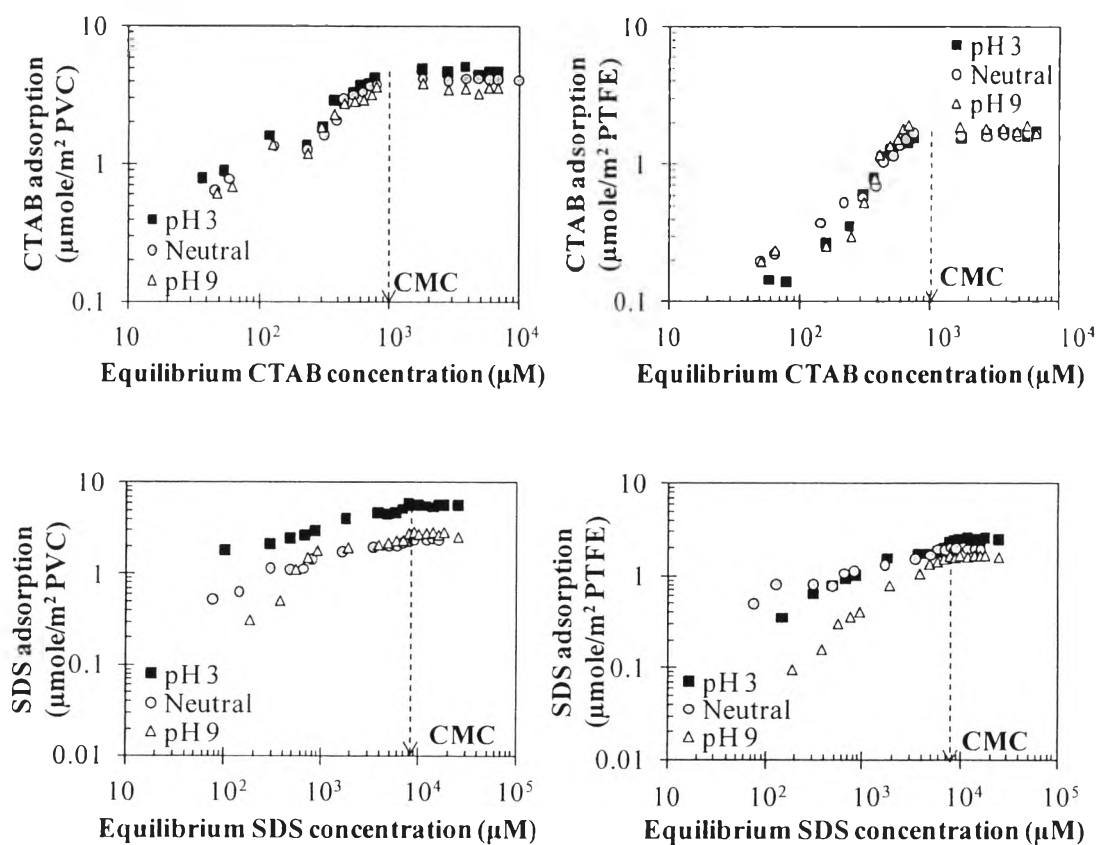
Figure 5.5 Adsorption isotherms of CTAB and SDS both on PVC and PTFE with various solution pHs at 30°C.
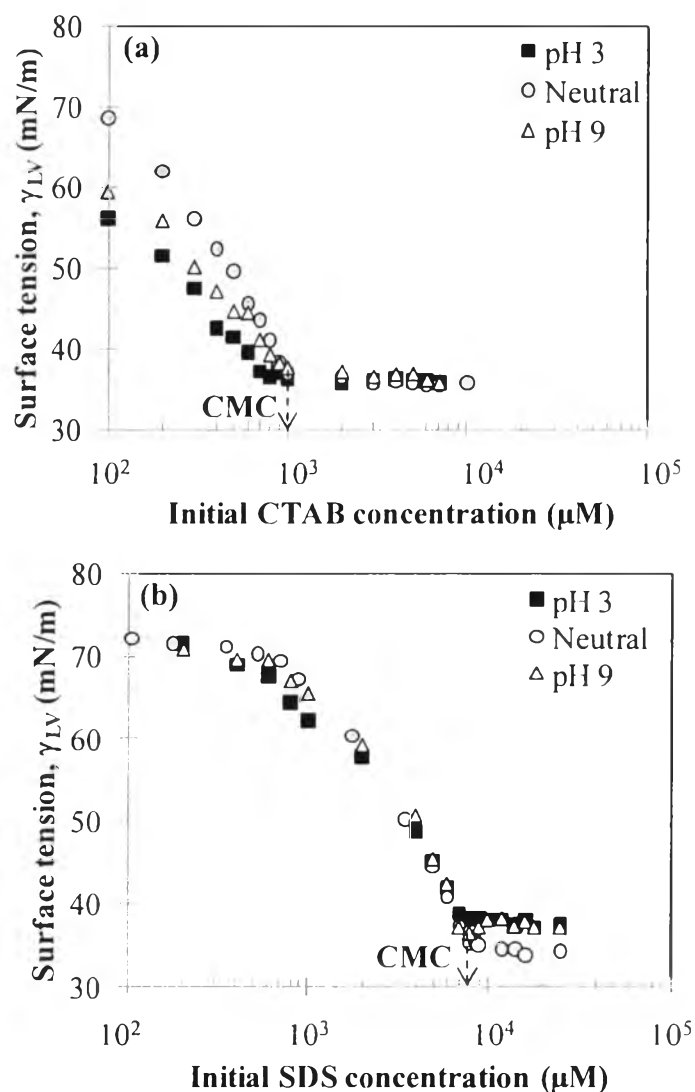
Figure 5.6 Surface tension isotherms of CTAB and SDS at various pHs.

5.5 Conclusions

Surfactant adsorption trends to increase with decreasing hydrophobicity of polymer even if the detailed order of adsorption is not clear correlation to the hydrophobicity of polymer due to the surface roughness of powdered polymer. For the effect of cationic tail group (a homologous series), CTAB (highest tail length) provides the highest adsorption below the CMC while the plateau adsorption does not different significantly for three cationic surfactants. The bilayer can be formed on lower hydrophobicity surfaces (PCL and PA66) and on moderate ones like PC and

PVC for all three cationic surfactants. For the effect of anionic surfactant with both different tail length and headgroup, the adsorption below CMC is higher for the longer alkyl tail group surfactant as well. Above the CMC, C8 provides the highest adsorption for all polymers possibly due to less polarity for carboxylate compared to benzenesulfonate and sulfate, respectively. The pH level only slightly affects the adsorption level for CTAB on both polymers and SDS on PTFE due to very low charged sites on hydrophobic surface (polymer). However, the SDS adsorption on PVC is quite high at pH=3.

5.6 Acknowledgements

5.7 References

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