

# Adsorption Behavior of Cocamidopropyl Betaine Under Conditions of High Temperature and High Salinity

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**ABSTRACT**: The adsorption behavior of cocamidopropyl betaine (CAB) in aqueous solutions and on sandstone surfaces was studied under the conditions of high temperature and high salinity. In aqueous solutions, as temperature increased from 25 to 90°C and salinity increased from 0 to 115,200 ppm, surface tension and the critical micelle concentration (cmc) of CAB both decreased. In the solid/liquid system, when the CAB concentration of salt solutions reached 0.30 wt %, the static saturation adsorption amount on the surface of clean sands was 14.77 mg g<sup>-1</sup> at 90°C. Because of its noticeable saturation adsorption capacity, the adsorption of CAB on the solid/liquid interface agreed with multilayer adsorption. Also, the adsorption amount on the surface of oil sands was greater than on clean sands. Besides, the dynamic saturation adsorption amount and retention amount of 0.07 wt % CAB solution were less than the static adsorption amount. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40424.

**KEYWORDS:** adsorption; surfaces and interfaces; surfactants

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#### INTRODUCTION

Among the chemical enhanced oil recovery (EOR) techniques, surfactant flooding can displace residual oil through reducing interfacial tensions (IFTs) between oil and water, emulsifying crude oil and altering the reservoir wettability. Surfactants usually used are petroleum sulfonate, alpha olefin sulfonate (AOS), internal olefin sulfonate (IOS), alkylbenzene sulfonate (ABS), and sodium dodecyl benzene sulfonate (SDBS).<sup>1-5</sup> It is difficult for these surfactants to act at high temperature over 90°C and high salinity over 100,000 ppm. To increase the temperature tolerance and salt resistance of surfactant flooding systems, two or more surfactants are usually combined.<sup>6,7</sup> Although the synergism of these surfactants can reduce IFTs between oil and water, chromatographic separation owing to different adsorption amounts is inevitable, which brings about huge surfactants loss in porous media and weakens displacement efficiency.<sup>8-10</sup> Thus, it is difficult to select surfactants suitable for these harsh reservoir conditions.

Recently, much focus has been placed on amphoteric surfactants which have many advantages, such as excellent tolerance of high temperature and salinity, high interfacial activity at low concentrations, and insensitivity to divalent ions. The most widely used amphoteric surfactants in China are sulfobetaine and hydroxysulfobetaine.<sup>11,12</sup> It was found that IFTs between oil and

water can be reduced to an ultralow value by a betaine-type surfactant alone within a wide concentration range (0.005–0.3 wt %).<sup>13,14</sup> But its excess adsorption on rock surface was caused by the interaction of the positively charged groups and negatively charged surface of reservoirs rocks. The resulting increase of the surfactant cost limited its application in EOR.

In China, there are many oilfields with the characteristic of high temperatures and salinities, such as the Huabei Oilfield, the Zhongyuan Oilfield, the Tahe Oilfield, and the Tarim Oilfield.<sup>15-18</sup> The reservoir temperatures range from 97.5 to 150°C and the salinity of reservoir water from 84,000 to 284,000 ppm with high concentrations of divalent metal cations. These harsh conditions can greatly affect surfactant adsorption on the surface of reservoir rocks. Because of increasing internal freedom energy at elevated temperatures, surfactant molecules move to the phase interface by thermodiffusion, which causes surfactant density to increase along the interface boundary layer. The effect of salt ions on the adsorption behavior of surfactants is more complicated. There are three modes. First, a screen of salt ions reduces the van der Waals interactions by decreasing the coulomb interaction between the adsorbing head groups of surfactants and rock surface.<sup>19</sup> Second, salt ions screen the electrostatic repulsions between head groups and improve surface coverage.<sup>20</sup> Third, the strong adsorption of salt ions on the

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Figure 1. Molecular structure of cocamidopropyl betaine (CAB).

polar surface leads to a displacement of surfactant molecules.<sup>21</sup> These reduce the adsorption amount of surfactants. Divalent cations, mainly Ca<sup>2+</sup> and Mg<sup>2+</sup>, can weaken the adsorption of betaine-type amphoteric surfactant DSB on a silica surface through lowering multilayer adsorption by an electrical mechanism in addition to the complexation mechanism.<sup>22</sup>

At present, the study of surfactant flooding technology for hightemperature and high-salinity reservoirs is an important and challenging topic. In our laboratory, the amphoteric surfactant cocamidopropyl betaine (CAB) was found, by a large number of selective preference experiments, to be stable under high temperature and high salinity conditions of the Tarim Oilfield. Also, the selected surfactant can reach ultralow IFTs with crude oil at the harsh conditions, as found from indoor physical simulation displacement experiments. To better understand the role of surfactants in surfactant flooding, it is necessary to study systematically the adsorption behavior of the surfactant from the viewpoint of fundamental and applied aspects.

Because of both positively and negatively charged hydrophilic groups in one molecule, amphoteric surfactant has complicated adsorption behavior on rock surface at high temperature, especially in reservoir water with high concentration of inorganic ions. This study shows the harsh conditions of high temperature, high salinity and hardness have a stimulating effect on the adsorption behavior of amphoteric surfactants in aqueous solutions and on the surface of oilfield sandstone.

#### EXPERIMENTAL

#### Materials

**Surfactant.** Cocamidopropyl betaine (CAB) is an industrial product of the Ho Tung Chemical Corporation (Taiwan). It was used as received and without any purification. The molecular formula is shown in Figure 1.

Water. Reservoir water was obtained from the Tarim oilfield located in northwest China. The total dissolved solid (TDS) was 115,200 ppm and the density was 1.05 g mL<sup>-1</sup> with a high hardness of 7040 ppm Ca<sup>2+</sup> and 614 ppm Mg<sup>2+</sup>. The composition is shown in Table I.

**Oil.** Crude oil was obtained from the Tarim oilfield and treated by dehydration and degassing. Its viscosity was 7.8 cP and density was  $0.825 \text{ g mL}^{-1}$  at  $90^{\circ}$ C.

Table I. Con	nposition o	f the	Reservoir	Water	of the	Tarim	Oilfield
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lons	Total Na <sup>+</sup>	Ca <sup>2+</sup>	${\rm Mg}^{2+}$	$CI^-$	$SO_{4}^{2-}$	$HCO_3^-$
Concentration	36,660	7040	614	70,560	245.3	103
(ppm)						

Adsorption Media. Sandstones were obtained from the Tarim oilfield, including a natural core and oil sands, and their mineral composition as determined by X-ray diffraction (XRD) is shown in Table II. The natural core was with a length of 6.88 cm and a diameter of 2.51 cm, used to obtain the dynamic adsorption amount during surfactant flooding. Oil sands were sieved to obtain particles with a size in the range of 180–125  $\mu$ m. Some oil sands were cleaned by a toluene/alcohol/ether mixture (2 : 1 : 1 by volume) then became clean sands. Both two types of sands were used to determine the static adsorption amount of the surfactant aqueous solution under different experimental conditions.

## **Experimental Methods**

**Surface Tension Measurement.** Aqueous solutions with different CAB concentrations in a range from 0.005 to 0.5 wt % were separately prepared with distilled water and reservoir water. The surface tensions of these solutions were measured by a Krüss DCAT41 Surface Tension Meter (the German Dataphysics Company, Germany) at 25 and 90°C. Each measurement was repeated three times, and the average value was taken.

Adsorption Experiments. The measurements of surfactant adsorption included the static adsorption and the dynamic core flooding adsorption experiments.

Static Adsorption. Static adsorption of surfactant on sand surface was investigated with three influencing factors, including surfactant concentrations, solid-to-liquid weight ratios and the type of sands, referring to oil sands or clean sands. The above typical adsorption experiment was carried out through mixing the sands of about 5 g and surfactant solutions with different concentrations at a solid-to-liquid weight ratio of 1 : 10. Another adsorption experiment was carried out through mixing the sands of about 5 g and the surfactant solution with a certain concentration at different solid-to-liquid weight ratios of 1 : 2, 1:5, 1:10, and 1:20. They are mixed in 100 mL conical flasks and shook for 24 h in a thermostatic oscillator at 90°C. Then the upper liquid was separated and centrifuged for 10 min at 4000 rpm. After centrifugation, the residual surfactant concentration in the supernatant was analyzed by a two-phase titration method. Static adsorption was calculated according to eq. (1):

$$\Gamma = (C_0 - C)V/m \tag{1}$$

where  $\Gamma$  is the amount of surfactant adsorbed on the sands (mg g<sup>-1</sup>),  $C_0$  and C are the initial and residual concentration of surfactant before and after the adsorption respectively (mg g<sup>-1</sup>), V is the volume of surfactant solution added to the conical flask (mL), and m is the mass of the sands (g).

**Dynamic Adsorption.** After being saturated by reservoir water, the natural core was placed in the core holder and then aged for 2 h at 90°C in a thermostatic water bath. Prior to the surfactant flooding experiment, reservoir water was injected into the core and the injection pressure was monitored until it remained stable. Surfactant solution was then continuously injected until the surfactant concentration in the effluent was close to the initial injection concentration. Then reservoir water was injected until the surfactant concentration in the effluent

Table II. Mineral Composition (wt %) of the Rock Core by XRD

Mineral composition	Quartz	Potassium feldspar	Clay	Ferrodolomite	Iron pyrite	Plagioclase
Percentage (wt %)	81	13	3	1	1	1

was reduced to zero. During the whole process, the injection rate of displacing fluids was 0.5 mL min<sup>-1</sup>. Dynamic adsorption was calculated according to eq. (2) as:

$$\Gamma_r = \frac{C_0 V - \sum_{i=1}^n C_i V_i}{m} \tag{2}$$

where  $\Gamma_r$  is the amount of surfactant retention on the core surface per gram of rock (mg g<sup>-1</sup>),  $C_0$  is the initial concentration of the surfactant before adsorption (mg g<sup>-1</sup>), V is the total volume of injected surfactant solution when the surfactant concentration in the effluents was close to the initial injection concentration (mL),  $C_i$  is the surfactant concentration in the effluents,  $V_i$  is the volume of every effluent sample (mL), m is the mass of the natural rock (g), and n is the total of effluent samples until the surfactant concentration was reduced to 0.

#### **Determination of Surfactant Concentration**

The CAB concentration was determined by the colorimetric method<sup>23</sup> through UV–vis measurement. This method was based on the precipitation which was reacted on by CAB and reineckate salts. The typical analytical process was as follows:

The reineckate salt solution was freshly prepared by adding 1.50 g reineckate salt into 100 mL distilled water and adjusting the pH value to 1.0 using an HCl solution. The solution was stirred at room temperature for 45 min and filtered. This reagent must be prepared just prior to use, because it would gradually decomposed leading to huge measurement errors.

About 5.0 mL of standard betaine solution with concentrations of 0.02, 0.04, 0.06, 0.08, and 0.10 wt % was separately pipetted into 50 mL conical flasks. These flasks were placed in an ice bath for 15 min. Then 15 mL of fresh reineckate salt solution was added into each flask. The flasks were returned to the ice



Figure 2. Standard curve of CAB solutions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

bath for 3 h. After that, the precipitations were filtered in the mother liquor by leaching and washed by ether until the washing solvent was colorless. The betaine reineckate precipitations were dissolved in 70% acetone solution, the filtrate was collected in 25 mL volumetric flasks, and then diluted to a volume with 70% acetone.

A portion of this solution was transferred to the adsorption cell and the absorbance at a wavelength of 525 nm was measured, using 70% acetone as a reference solution. The Lambert Beers Law was observed and the CAB concentration obtained was in the range of 0 to 0.10 wt % (Figure 2).

## **RESULTS AND DISCUSSION**

#### Surface Properties

The surface tension measurements were carried out to investigate the surface properties of CAB in aqueous solutions. These solutions were prepared with distilled water and reservoir water, and measured at 25 and 90°C separately. The experimental results are shown in Figure 3. As can be seen from Figure 3, under the two experimental conditions, the surface tension sharply decreased as the CAB concentration increased. With a further increase of CAB concentration, the surface tension gradually decreased and finally almost remained constant. It was demonstrated that the adsorption of surfactant molecules at the air/water interface achieved saturation and micelles were formed. The cmc value of CAB in distilled water solutions at  $25^\circ C$  was 2.92 mmol  $L^{-1}$  (about 0.101 wt %). In general, the cmc value of surfactants follows the principle: the smaller the cmc value, the greater the surface activity. Compared with the cmc values of the typical conventional commercial surfactants,



Figure 3. Surface tension isotherms as a function of CAB concentration in aqueous solutions under the two experimental conditions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary. com.]

Table III. The Surface Properties of CAB in Aqueous Solutions

	Experimental conditions		
	1#	2#	
cmc (wt %)	0.101	0.058	
cmc (mmol L <sup>-1</sup> )	2.92	1.69	
$\gamma_{cmc}$ (mN m <sup>-1</sup> )	34.79	22.86	
$\Pi_{\rm cmc}$ (mN m <sup>-1</sup> )	37.21	47.46	
$\Gamma_{max}$ ( $\mu$ mol m <sup>-2</sup> )	4.746	1.610	
A <sub>min</sub> (Ų)	34.99	103.17	

Note: 1# is measured in distilled water at 25°C; 2# is measured in reservoir water at 90°C.

such as sodium dodecyl sulfate (SDS),<sup>24</sup> alpha olefin sulfonate (AOS),<sup>25</sup> and sodium dodecyl benzene sulfonate (SDBS),<sup>26</sup> which are 7.8 mmol  $L^{-1}$ , 0.100 wt %, and 2.764 mmol  $L^{-1}$ , respectively, the cmc value of CAB is lower or similar.

From Figure 3, it was found that a simultaneous increase in temperature and salinity had a significant effect on the surface tensions of CAB. With increase in temperature and salinity, the surface tension and cmc values of CAB further decreased. To further study the effect of temperature and salinity on the surface activity, the effectiveness of surface tension reduction  $\Pi_{\rm cmc}$  the maximum surface excess concentration  $\Gamma_{\rm max}$  and the minimum area  $A_{\rm min}$  occupied per surfactant molecule at the air/water interface were proposed and determined as follows:

$$\Pi_{\rm cmc} = \gamma_0 - \gamma_{\rm cmc} \tag{3}$$

where  $\gamma_0$  is the surface tension of the solvent and  $\gamma_{cmc}$  is the surface tension of solutions when the surfactant concentration is above the cmc.

$$\Gamma_{\max} = -\frac{1}{nRT} \left(\frac{d\gamma}{d\ln c}\right)_T \tag{4}$$

where *n* is the number of solute species whose concentration at the interface changes with the surfactant concentration *c*, the value of *n* is taken as 1 for an amphoteric surfactant in aqueous solution,<sup>27</sup> *R* is the gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>), *T* is the absolute temperature,  $\gamma$  represents the surface tension, and  $d\gamma/d(\ln c)$  is the slope of surface tension  $\gamma$  vs. ln c dependence when the concentration is near cmc.

$$A_{\min} = \frac{1}{N_A \Gamma_{\max}} (\times 10^{23}) \tag{5}$$

where  $N_A$  is Avogadro's constant (6.022 × 10<sup>23</sup> mol<sup>-1</sup>).

For the adsorption of CAB at the air/water interface under both experimental conditions, the values of the previously mentioned parameters are summarized in Table III. It can be seen that compared to these parameters, the cmc values of CAB solutions decreased from 0.101 wt % to 0.058 wt % (2.92 and 1.69 mmol  $L^{-1}$ , respectively) and  $\gamma_{\rm cmc}$  reduced from 34.79 to 22.86 mN m<sup>-1</sup>, with the resulting reduction of  $\Gamma_{\rm max}$  and increment of  $\Pi_{\rm cmc}$  and  $A_{\rm min}$ .

Temperature plays an important role in surfactant molecular thermodynamic behavior. Increasing temperature weakens the interactions between the head groups of surfactant molecules at the surface.<sup>28</sup> Moreover, some of the existing hydrogen bonds rupture caused by thermal fluctuation, which causes a decrease of hydration of hydrophilic groups and makes the surfactant molecules more hydrophobic.<sup>29</sup> Thus, surfactant molecules are easier to transfer from the bulk solution onto the interface and be rearranged in the solution to assemble the hydrophobic core of aggregates. Therefore, as temperature increases, the surface tension decreases. In addition to temperature, salt also has a significant effect on the surface tension of surfactants. In surfactant solutions with high ionic strengths, the repulsive electrostatic interactions between the surfactant head groups at the micelle surfaces are weakened.<sup>24</sup> Also, significant amounts of electrolyte ions binds to the micelle surfaces, alters the average surface charge density of the surfactant adsorption layer<sup>30</sup> and reduces the overlap between the electrical double layers.<sup>31</sup> An interaction of a van der Waals type overcomes the repulsive interaction and there is a net attractive force between similarly charged surfaces, so the micelles are able to form at lower surfactant concentrations. Accordingly, surface tension and the cmc value decrease.

#### Static Adsorption Process

To study static adsorption, the effect of three factors, surfactant concentration, the solid-to-liquid weight ratio and the type of sands on the adsorption of CAB aqueous solutions were investigated.

#### Surfactant Concentration

The effect of the CAB concentration on the static adsorption behavior on the surface of clean sands was investigated at 90°C where the solid-to-liquid weight ratio was 1 : 10 as shown in Figure 4. As Figure 4 shown, an increasing CAB concentration led to an improvement in the adsorption capacity on the surfaces of clean sands. The adsorption isothermal of solutions was "*L*" curves, matching the Langmuir isotherm.<sup>32</sup> The concentration at adsorption equilibrium was much higher than the cmc value (shown as the red dot on the graph). It indicated that amphoteric surfactants had very strong adsorption on the



**Figure 4.** Static adsorption of CAB as a function of concentration at 90°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]





surface of sandstone and this adsorption behavior was not homogeneous monomolecular adsorption. Because CAB is an amphoteric surfactant with cationic and anionic centers, the adsorption on the solid/liquid interface could be multilayer adsorption through the attraction of positive charges and negative charges between molecular adsorption layers. When the CAB concentration of the aqueous solutions reached 0.30 wt %, the adsorption reached the saturation point and the adsorption amount was 14.77 mg g<sup>-1</sup>.

As previously reported, the static adsorption of 0.30 wt % sulfobetaine surfactant solution prepared at 10-20 wt % NaCl on the quartz sand surface was only about 1 mg  $g^{-1}$  at  $70^\circ \text{C.}^{33}$  But in a recent study, the adsorption of CAB on the sandstone surface at 90°C was obviously larger. There are possible reasons to explain this phenomenon. First, it was proved from the experimental results of surface tension that amphoteric surfactants diffuse more easily to the interface boundary layer and aggregate at the solid/liquid interface as the temperature rises. Second, due to the negative charges on sand surfaces, the electrostatic attraction between positive sites of amphoteric surfactants and negative sites of sand strengthens the adsorption of amphoteric surfactants on sand surface. Also, surfactant dissolution was decreased because of the high salinity in reservoir water, which caused surfactant molecules to adsorb on the solid/liquid interface.34

# Solid-to-Liquid Weight Ratio and the Type of Sands

The effect of the solid-to-liquid weight ratio on the static adsorption behavior of CAB on the surface of the oil sands and the clean sands was investigated at 90°C as shown in Figure 5. From Figure 5, it pointed out the adsorption capacity of CAB was remarkably enhanced with an increase in the solid-to-liquid weight ratio. It also showed that the adsorption capacity was obviously greater on the surface of the oil sands than on that of the clean sands. According to the rule of "like dissolves like," a solute will dissolve best in a solvent that has a similar chemical structure. Crude oil is composed of nonpolar components which have a structure similar to the lipophilic groups of surfactants. The lipophilic groups stretched into the crude oil that covered the oil sand surface, and formed hydrogen bonds which helped to increase the amount of surfactant adsorption. So the adsorption amount on the oil sands was larger than on the clean sands.

#### **Dynamic Adsorption**

The dynamic adsorption of CAB on the natural core was investigated at 90°C by the core flood test with an injection rate of 0.5 mL min<sup>-1</sup>. The pore volume  $(V_p)$  and permeability of the natural core are 7.304 cm<sup>3</sup> and 0.237 Dc, respectively. The experimental results are shown in Figure 6.

From Figure 6, it can be found that there was a longer adsorption and desorption process for the dynamic adsorption of CAB on the natural core. When the injection volume of 0.07 wt % CAB solution was over 40  $V_p$ , the dynamic adsorption reached saturation and the adsorption amount of CAB was 7.618 mg g<sup>-1</sup>. When the injection volume was more than 55  $V_p$  the retention amount of CAB was 1.590 mg g<sup>-1</sup>. These amounts are less than the static adsorption amount 7.799 mg g<sup>-1</sup> with the same concentration of CAB on oil sands at a solid-to-liquid weight ratio of 1 : 20.

Adsorption is usually due to following aspects: (i) electrical interactions, which are molecular interactions with opposite charges<sup>35</sup> and (ii) chemical bond interactions, such as hydrogen bonding and hydrophobic bonding.<sup>36</sup> The former is stronger. Except for these aspects, it revealed that the size of the surface area also affects the adsorption quantity. In the static adsorption experiments, the surfactant completely contacted the sand surface. But in the dynamic adsorption experiment, the displacing fluid could not spread to the pores with low permeability, which was directly responsible for the smaller swept area than the contact area of static adsorption. This is one reason that the dynamic saturation adsorption amount and the dynamic retention amount were less than the static adsorption amount. It is also because of simultaneous adsorption and desorption in the



Figure 6. Dynamic adsorption of 0.07 wt % CAB solution on the natural core at  $90^{\circ}$ C as a function of the injection volume. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

process of dynamic adsorption. Part of surfactant adsorbed was desorbed from the surface of the natural core during follow-up brine water flooding.

# CONCLUSIONS

The phase behavior of CAB in aqueous solutions and its adsorption behavior on a sandstone surface were investigated under the condition of high temperature and high salinity. The following conclusions can be drawn from this study.

As temperature and salt ion concentration simultaneously increased, the cmc value and  $\gamma_{cmc}$  of CAB aqueous solutions decreased at the air/water interface. When temperature rose from 25 to 90°C and salinity rose from zero to 115,200 ppm, the cmc value decreased from 2.92 mmol  $L^{-1}$  (0.101 wt %) to 1.69 mmol  $L^{-1}$  (0.058 wt %) and  $\gamma_{cmc}$  decreased from 34.946 to 23.249 mN m<sup>-1</sup>. In the solid/liquid system, when CAB concentrations of the solutions prepared with reservoir water reached 0.30 wt %, the static adsorption on the oil sand surface reached the saturation point which was 14.77 mg  $g^{-1}$  at 90°C. This large value was taken as proof that the adsorption of CAB at the solid/liquid interface was multilayer adsorption. Moreover, the adsorption capacity of CAB increased with the increasing solidto-liquid weight ratio, and the adsorption amount on the oil sand surface was much more than that on the clean sand surface. Also, the dynamic adsorption on the natural core reached a balance when the injection volume of 0.07 wt % CAB solution was over 40 Vp. Accordingly, it was a longer adsorption and desorption process for the dynamic adsorption of CAB. The dynamic saturation adsorption amount and the dynamic retention amount were 7.618 and 1.590 mg g<sup>-1</sup>, respectively, which were less than the static adsorption amount of 7.799 mg  $g^{-1}$ with the same CAB concentration on the oil sands at a solid-toliquid weight ratio of 1 : 20.

This work is expected to provide a scientific basis and technical support for surfactant selection and practical project design of surfactant flooding in high-temperature and high-salinity sandstone reservoirs.

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