

Adsorption Properties of Gemini and Monomeric Cationic Surfactants on Sandstone and Silica Nanoparticles

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Abstract

The surface modification of pore throat by adsorption of surfactants is thought to have a positive effect on water flooding in low and ultralow permeability reservoirs. In this paper, Gemini cationic surfactants, containing 12 and 16 carbon alkyl chains (ethanediyl-1,2-bis(dimethyl dodecyl ammonium bromide) and ethanediyl-1,2-bis(dimethyl cetyl ammonium bromide), referred to as GC₁₂ and GC₁₆) and hexadecyl trimethyl ammonium bromide (CTAB) were used as modifying agents to investigate the effects of the surfactant concentration, adsorption time and temperature on static adsorption onto the surface of sandstone and silica nano particles (NPS). The results show that the equilibrium adsorption amount of GC₁₆ on sandstone and NPS is higher than that of GC₁₂ on sandstone and NPS with the same initial concentration of 0.225 mmol/L in solution at 45°C. It is found that the adsorption amounts of GC₁₂ and GC₁₆ decrease as the raise of temperature. The adsorption rate of surfactant on sandstone surface is slower than that of NPS. The equilibrium adsorption time of these surfactants on sandstone is 20 h, while the time of NPS is only 2 h. At 55°C, the static saturation absorption amount of GC₁₂ is 210.56 μmol/g on NPS and 117.67 μmol/g on sandstone, while the amounts of CTAB on sandstone and NPS under static conditions are 1.18 times and 1.46 times of GC₁₂, respectively. Considering the number of tail chain in a molecule of surfactant, the packing densities of Gemini surfactants on solid surface are higher than that of the single-tail surfactant (CTAB). Therefore, the adsorption rate and amount of surfactant are affected by the specific surface of solid particles, charged density, tail chain number and length of the cationic group.

Keywords

Cationic Surfactants, Gemini Surfactants, Adsorption, Silica Nanoparticles, Sandstone

1. Introduction

The chemical recovery technology for enhanced oil recovery has been developed rapidly due to the depletion of oil resources. Water flooding is one of the main methods in low permeability reservoirs. Surfactant injection as a method of a chemical flooding can enhance oil recovery by reducing the interfacial tension (IFT) between oil and water, forming O/W emulsions, altering the rock wettability and thus resulting from the adsorption of surfactants on reservoir rock [1] [2] [3]. The adsorption of different surfactants, including anionic surfactant, nonionic surfactant, and zwitterionic surfactant on various solid/water interfaces, has been extensively studied by many authors [4]-[12].

In view of percolation theory of reservoirs, if interfacial tension (IFT) between oil and water is reduced to lower than 0.01 mN/m, the effect of capillary force would become negligible and the trapped oil droplets can mobilize in pore channels [13]. But for IFT between injection fluid and oil beyond this scope, capillary force is not neglected and will seriously affect the flow of oil. In recent years, surfactants and nano composites have been used to alter wettability of reservoirs for augmented injection to improve oil recovery [14] [15] [16] [17]. Hydrophobicity modification on the surface of sandstone and nano materials used to treat pore throat of stratum are favorable for changing the wetting stratum into nearly neutral or neutral wetting and reducing the capillary resistance to achieve the purpose of reducing the injection pressure [18] [19]. William [20] used XPS to identify the adsorption density of CTAB and the shape of the aggregates of the cationic surfactant adsorbed to the muscovite mica/aqueous solution interface to investigate the role of electrostatic interactions on surface aggregation. Rowaida KSK [21] prepared new nano composites as disinfectants and antiseptics in food-processing environments through surface modification of montmorillonite by cationic surfactants. Hou [22] used SEM and microscopic displacement to study mechanisms for wettability alteration of oil-wet sandstone surfaces by different surfactants and the effect of reservoir wettability on oil recovery. Asphaltenes adsorbed on sandstone was greatly reduced by the formation of ion pairs between negatively charged carboxylates of crude oil and quaternary ammonium groups of CTAB. CTAB is more effective than polyoxyethylene octyl phenyl ether and sodium lauryl mono-ether sulfate in altering the wettability of oil wet sandstone surfaces. Cationic surfactants could interact with carboxylic acid groups from crude oil to form ion pairs as a result of the electrostatic attraction [23] [24] [25].

Recently, cationic Gemini surfactant, which is composed of two traditional single-alkyl and mono-hydrophilic surfactants that are connected by a spacer, has been paid much attention to its special structure. Each cationic Gemini surfactant has two positive charges in the molecule, rendering superior properties than traditional surfactants, such as the ability of reducing the interfacial tensions, and altering the surface electrical properties of the solid. In recent years, the adsorption of surfactants on solid surfaces, such as soil, silica, montmorillonite, kaolinite, has been reported [26] [27] [28] [29]. Grosmaire *et al.* [26] used

calorimetric methods to investigate adsorption process of dodecyl trimethyl ammonium bromide (DTAB) and dimeric surfactants. Their adsorption onto amorphous silica is exothermic, and then the exothermic effect rises during the formation of surfactant aggregates. Especially the variation of enthalpy is more exothermic for the Gemini surfactants than for single tail surfactant DTAB, which indicates that the interfacial aggregate is more compact by the Gemini surfactant. Zana [27] investigated the adsorption of DTAB and cationic Gemini surfactants at silica/solution interfaces. The adsorption amounts of DTA-Band1,2-bis (dodecyl dimethyl ammonio)-ethane dibromide are 3.5 and 5.4 mol chain/m², respectively, in a word, the adsorption density of dimeric surfactant is 1.54 times that of the single tail surfactant. Qi [28] compared the equilibrium concentration at the maximum adsorption of 1,2-bis(cetyldimethylammonio)-ethane dibromide (16-2-16, or GC₁₆) and CTAB at 25°C, The result indicates that the Gemini surfactant 16-2-16 is much more efficient than the corresponding conventional surfactant CTAB at achieving the maximum adsorption on both kaolinite and Na-montmorillonit. Adsorption of 16-2-16 with two quaternary ammonium head groups onto negative sites of the clay is almost identical to that of conventional surfactants CTAB. However, there are very few reports dealing with the comparative study on adsorption behavior of Gemini surfactants and single chain traditional surfactants on silica nano particles (NPS).

In this study, we investigated the adsorption behaviors of Gemini cationic surfactants and single-head cationic surfactant on solid surface with different specific surface area, such as crushed sandstone and nanometer materials. Single-tailed cationic surfactants (CTAB) and Gemini surfactants (GC₁₂, GC₁₆) were used as the modifying agents to make a contrastive investigated adsorption rate and adsorption amount on sandstone and NPS surface.

2. Experimental Section

2.1. Materials and Instruments

Hexadecyl trimethyl ammonium bromide (CTAB), dodecyl dimethyl tertiary amine, cetyl dimethyl tertiary amine, sodium dodecyl sulfate (SDS), ethyl acetate, methine Blue, thymol blue, bromocresol green were obtained from Aladdin. Sandstone provided by Henan Oilfield was treated by washing, drying, grinding and sieving, with 60 to 100 meshes (0.25 mm - 0.15 mm) and a BET surface area of 5.812 m²/g, to determine the adsorption of surfactants. Nano-silica (NPS) with a BET surface area of 128.896 m²/g was purchased from Nanjing Haitai Nano-materials Co., Ltd, and used as received. The water used in laboratory was twice the distilled water. THZ-82 water bath thermostat oscillator was used to control the temperature in each test; UV-1100 spectrometer was employed to determine the concentrations of surfactants in the solutions.

The series of cationic Gemini surfactants were synthesized according to our previous study [30]. The CMC values of GC₁₆, GC₁₂ and CTAB at 25°C are 0.03 mmol/L, 1.0 mmol/L and 0.8 mmol/L, respectively. **Figure 1** shows the chemical structure of these Gemini surfactants.

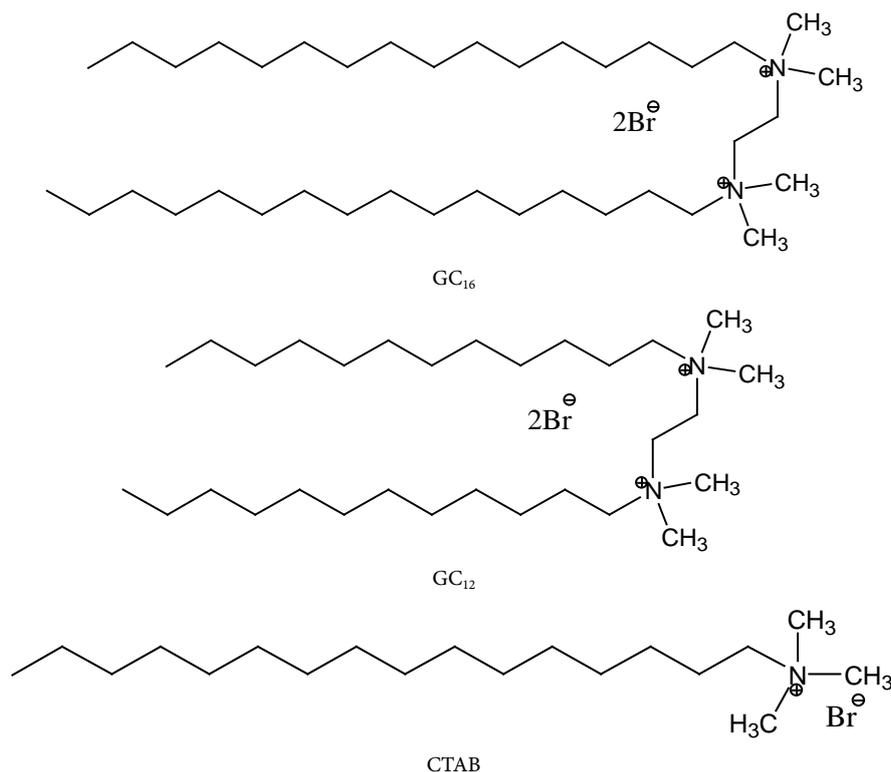


Figure 1. Molecular structure of GC₁₆, GC₁₂ and CTAB.

2.2. Determination of Surfactant Concentration

2.2.1. Two-Phase Titration Method

Thymol blue (TB) and bromothymol blue (MB) were equivalently mixed as the compound indicator of water-organic mixtures, then the sample solution of cationic surfactants was titrated with known concentrations of standard anionic surfactant solution were measured to obtain the standard calibration curve [31].

5 mL of the standard sodium lauryl sulfate (SDS) solution with the concentration of 1.8×10^{-2} mmol/L, 3.6×10^{-2} mmol/L and 9×10^{-3} mmol/L, respectively, was added into a 100 mL cylinder with a stopper. 5 mL of mixed indicator and 5 mL of sodium sulfate acidic solution were successively introduced into the cylinder, distilled water was then added to obtain 30 mL of a water phase, followed by 5 mL of dichloromethane. The mixture in the cylinder was homogenized by vigorous hand shaking. The mixture was titrated with the cationic surfactant solution to be measured till the color of the bottom phase changed from light purple gray to bright yellow green. The color of the upper layer gradually shallowed from pink to colorless. A blank test was conducted to facilitate the comparison. The consuming volume of the cationic surfactant solution was noted to determine the surfactant concentration.

2.2.2. Spectrometric Method

The concentrations of surfactant solution lower than 0.01×10^{-3} mmol/L were determined by the UV spectrometer. The mixture of cationic surfactants with bromocresol green indicator is able to form complexes which can easily dissolve

in chloroform phase and appears blue. The absorbance of the surfactant solution was determined at a wavelength of 630 nm when dichloromethane was used as a reference. The linear relationship was drawn between the concentrations of the cationic surfactant and the absorbance of solution as a standard curve. The concentration of sample solution can be obtained from the standard curve of plotting absorbance with concentration of the cationic surfactant.

2.3. Adsorption Test of Cationic Surfactants

0.4 g (accurate to ± 0.0001 g) of crushed sandstone or NPS was weighed into a glass tube, followed by adding 8 mL of surfactant solution. The mixtures were mixed and then kept in the water bath oscillator at 120 r/min for a period of time at a constant temperature. The tube was taken out and then centrifuged at 3500 r/min for 10 min. The supernatant was taken to determine the concentrations of surfactants. The adsorption amount of surfactant on solid particles can be obtained from the surfactant solution concentration change between the initial solution and terminated solution using the following Equation (1).

$$\Gamma = \frac{(c_0 - c_e)V}{m} \quad (1)$$

where V is the volume (8 mL) of the solution; Γ is the adsorbed surfactants on NPS or sandstone per unit mass, $\mu\text{mol/g}$; c_0 and c_e are the concentration of the surfactant before and after the adsorption, mmol/L ; and m is the weight of solid particles, g .

3. Results and Discussion

3.1. Standard Working Curve

Standard solutions with different concentrations of cationic CTAB and Gemini surfactant solutions were prepared to measure the absorbance (A). The absorbance A was plotted against the concentration c to obtain the standard curves shown in **Figure 2** and **Figure 3**.

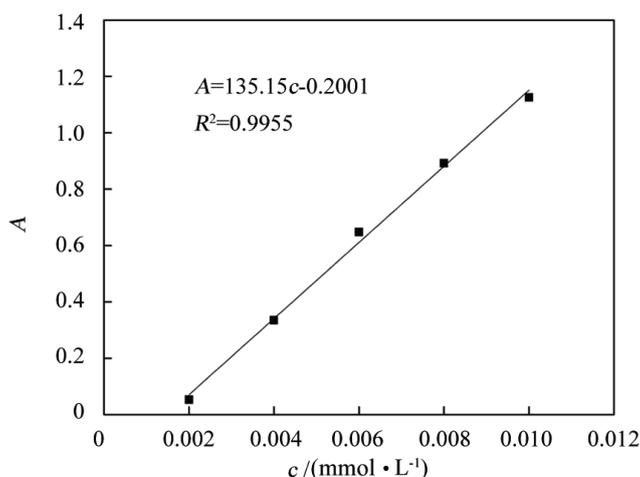


Figure 2. Relation of the absorbance and concentration of CTAB solution.

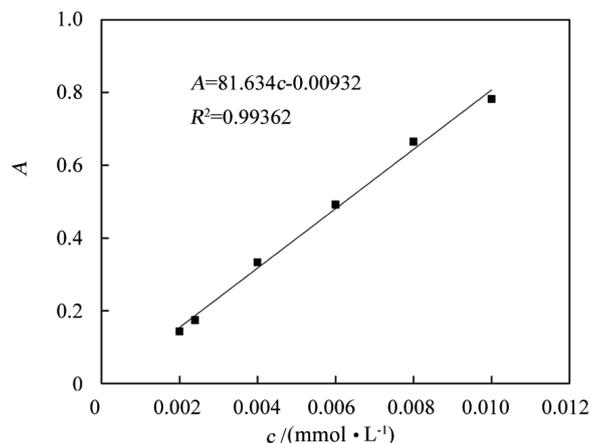


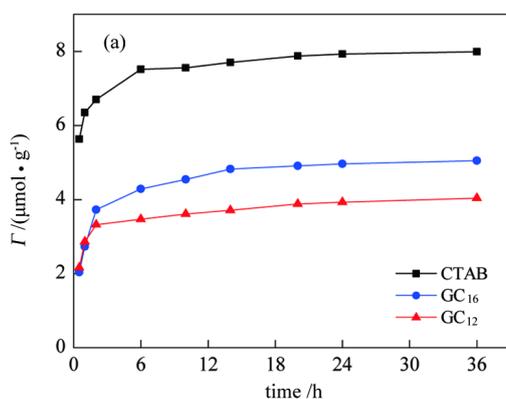
Figure 3. Relation of the absorbance and concentration of GC_{12} solution.

The results demonstrate that under the experimental conditions, there is a good linear relationship between absorbance A and CTAB and GC_{12} whose concentrations are lower than 0.012 mmol/L.

3.2. Determination of Adsorption Equilibrium Time

The highest concentration of GC_{16} solution that maintained clarification was 0.225 mmol/L at 25 °C, acting as the initial concentration of the three surfactants for adsorption experiments. This concentration is equivalent to 7.5 times CMC of GC_{16} , 0.225 times that of CTAB, and 0.281 times that of GC_{12} , indicating the initial concentration measured in the experiments is below the CMC values of CTAB and GC_{12} , and above that of GC_{16} . The adsorption of surfactants in different adsorption time was determined to obtain plots of the adsorption amount of the solution of CTAB, GC_{16} and GC_{12} on solid sandstone and NPS surface versus soaked time at 45 °C, as is shown in **Figure 4**.

It can be concluded from **Figure 3** that the adsorption amount of CTAB, GC_{16} and GC_{12} on sandstone surface rapidly increases at 45 °C with the increase of adsorption time, and then remains constant with time and reach equilibrium after 20 h of the adsorption on sandstone, while the adsorption equilibrium time of surfactant solution on NPS surface is only 2 h, which indicates that the rate of adsorption of surfactants on NPS is faster to reach equilibrium on the surface of



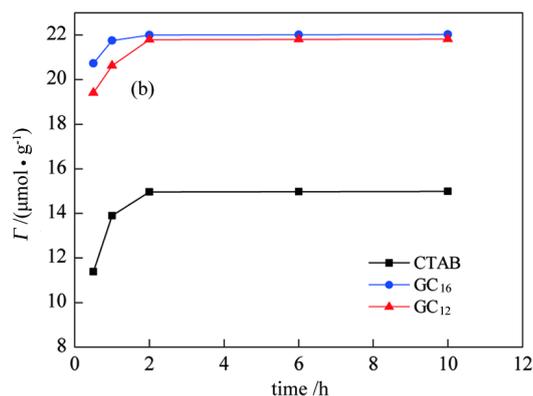


Figure 4. Static adsorption of 3 surfactants on sandstone (a) and NPS (b) vs. time at 45°C.

sandstone as a result of large specific surface area of NPS.

The adsorption amount of surfactant solution with an initial concentration of 0.225 mmol/L on both solid surfaces at 45°C can be drawn from **Figure 3**, as shown in **Table 1**. The equilibrium adsorption amount of GC₁₆ with double chains and double heads on NPS is 22.01 μmol/g, while that of CTAB is 14.96 μmol/g. In a word, the adsorption amount of GC₁₆ is 1.5 times that of single-alkyl CTAB. For crushed sandstone, the adsorption amounts of GC₁₆ and CTAB are 4.91 μmol/g and 7.87 μmol/g, respectively, which shows that the adsorption amount of Gemini surfactant GC₁₆ on solid surface is greater than that of the single-chain surfactant CTAB. In addition, it is seen that the maximum adsorption of GC₁₆ on the solid surface including sandstone and NPS is slightly larger than that of GC₁₂, which indicates that the length of hydrophobic alkyl affects the equilibrium adsorption amount, in a word, the adsorption increases with the increase of alkyl carbon number of cationic surfactant molecules with the similarity structure.

Table 1. Equilibrium adsorption amount on sandstone and NPS.

surfactant	CTAB/(μmol·g ⁻¹)	GC ₁₆ /(μmol·g ⁻¹)	GC ₁₂ /(μmol·g ⁻¹)
NPS	14.96	22.01	21.79
Sandstone	7.87	4.91	3.88

3.3. Effect of Temperature on the Adsorption

The surfactant solution with the initial concentration of 0.225 mmol/L was mixed with a specified mass of particle solid, and immersed in oscillating baths at different temperature for some time, the residual concentration of solution at each time step was determined. When the concentrations of surfactants remain constant with the adsorption time, the adsorption process reaches the equilibrium state. The equilibrium adsorption amounts of the surfactant at 35°C - 55°C were obtained from the trend of the adsorption amount with time, as shown in **Table 2**.

Table 2. Equilibrium adsorption amount on sandstone and NPS at different temperatures.

$t/^\circ\text{C}$	CTAB/ $(\mu\text{mol}\cdot\text{g}^{-1})$		GC ₁₆ / $(\mu\text{mol}\cdot\text{g}^{-1})$		GC ₁₂ / $(\mu\text{mol}\cdot\text{g}^{-1})$	
	sandstone	NPS	sandstone	NPS	sandstone	NPS
35	9.06	15.40	7.29	22.36	5.49	22.16
45	7.87	14.96	4.91	22.01	3.88	21.79
55	7.48	14.52	4.70	21.76	3.51	20.98

It can be seen from the result in **Table 2** that the adsorption of surfactant solution with fixed concentration on both solid surfaces decreases with the increasing temperature. At the temperature ranging from 35°C to 45°C, the adsorption amounts of GC₁₆ and GC₁₂ on the sandstone surface decrease by 2.38 $\mu\text{mol/g}$ and 1.61 $\mu\text{mol/g}$, respectively, while the adsorption amounts on the NPS surface decrease by 0.35 $\mu\text{mol/g}$ and 0.37 $\mu\text{mol/g}$, respectively. This is attributed to the fact that the solubility of the ionic surfactant in water increases with the increasing temperature, causing that the affinity of the surfactant with water is enhanced and the tendency to adsorb onto the solid surface is reduced at higher temperature, and thus the adsorption amount is reduced [32] [33]. At the same time, it reveals that the adsorption is an exothermic process and the increasing temperature is favorable for the desorption of surfactants from solid surface. It can also be seen that the adsorption amount of Gemini surfactant GC₁₆ with longer tail is higher than that of GC₁₂.

The adsorption depends on the specific surface area of adsorbent. In the whole range of experimental temperatures (35°C - 55°C), the weaker effect of temperature on the desorption. The adsorption amounts of GC₁₆, GC₁₂ and CTAB on NPS surface decrease by 1.11%, 1.58% and 2.86% respectively, while that on sandstone decrease 17.4%, 35.5% and 36.1%, respectively, indicating that adsorption amount of Gemini surfactants on the solid surface decreases slightly compared with that of conventional surfactants. It is well known that the charge density of Gemini cationic surfactants is larger than that of single-alkyl cationic surfactants and nanometer material possess larger specific surface area with more negatively charge on the surface than that of conventional surfactants, so the adsorption capacity of the double-alkyl cations was stronger than that of single-alkyl cations. Each chain of Gemini surfactant molecules adsorbs on silica much more closely than the monomeric surfactant with the same chain length, which is identical to the result reported by Esumi K [34].

3.4. Effect of Concentration of Surfactant on Adsorption

GC₁₂ and CTAB were used to discuss the effect of concentration of surfactants in distilled water on adsorption due to low solubility of GC₁₆ in water. The concentrations of surfactant solutions were measured after being adsorbed for 24 h on sandstone and NPS at 55°C, and the static adsorption amount was calculated based on Equation (1). The adsorption isotherms of the two surfactants were illustrated in **Figure 5** and **Figure 6**.

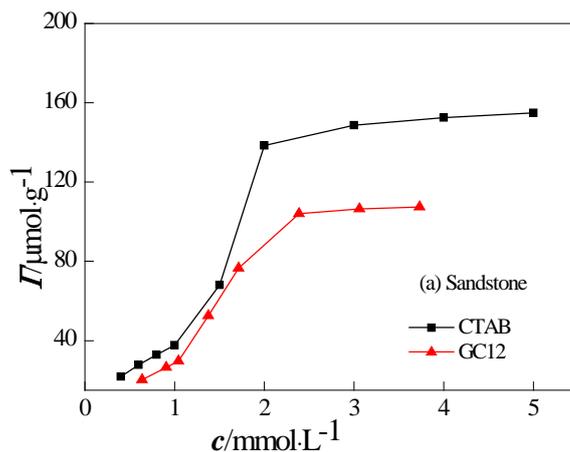


Figure 5. Static adsorption of surfactants on sandstone vs. equilibrium concentration.

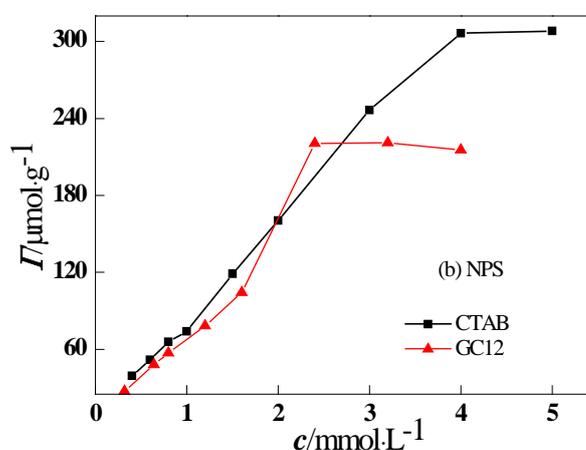


Figure 6. Static adsorption of surfactants on NPS vs. equilibrium of surfactants.

Figure 4 and Figure 5 shows the typical isotherm of adsorption of surfactants in solution on the solid surface in a rather wide range of concentration of surfactants going beyond the CMC. In the first region, the adsorption increases gradually with the concentration. The second region is a sudden increase region of the adsorption due to the lateral interaction between the adsorbed monomer, resulting in the surface aggregation of the surfactants, forming “micelle-like” aggregates or admicelles [7] [35] on the solid surface. The third region shows a slower rate of adsorption. The fourth region is the plateau region of adsorption of surfactants on solid surface basically reaching equilibrium.

It could be seen from the figures that within the experimental concentration range, all four adsorption curves were in consistent with the Langmuir adsorption rule. Due to the different adsorbent properties of each surfactant on sandstone and NPS, the solution concentration to achieve the equilibrium state is different from CTAB and GC_{12} . The saturated adsorption concentrations of CTAB on sandstone and NPS are 2 mmol/L and 4 mmol/L, respectively, and the corresponding saturated adsorption amounts are $138.43 \mu\text{mol/g}$ and $306.46 \mu\text{mol/g}$, respectively. The saturated adsorption concentration of GC_{12} on sandstone and

NPS are both 2.4 mmol/L, and the corresponding saturated adsorptions are 117.67 $\mu\text{mol/g}$ and 210.56 $\mu\text{mol/g}$, respectively, which shows that the adsorption amount in unit of $\mu\text{mol/g}$ of single-tailed CTAB on sandstone is 1.18 times that of double-tailed GC_{12} and 1.46 times that of GC_{12} on NPS.

The adsorption of Gemini surfactant GC_{12} and traditional cationic surfactant CTAB on the negatively charged solid surface are driven mainly by the electrostatic interaction between the positive charges in cationic surfactant molecules and negative charges on sandstone and NPS. One GC_{12} molecule has two cationic heads bonded by a linking group, which would occupy two adsorption sites, while the traditional cationic surfactant CTAB has only one ion head and occupies only one adsorption site. Therefore, the adsorption of CTAB on sandstone and NPS should be twice that of GC_{12} , but in fact it is less than twice that of GC_{12} , which indicates two tails in one molecule of GC_{12} packing more closely than the conventional single-head and single-tail cationic surfactants and more surfactant molecules detained on the solid surface with negative charge. For conventional cationic surfactants, there exists an electrostatic repulsion between two adjacent cations when packing on solid-liquid interface, less negative charged points of the solid surface are occupied. So, it is reasonable that the actual adsorption of CTAB on sandstone and NPS surface is less than twice that of GC_{12} . In addition, the saturated adsorption of GC_{12} on the NPS surface is 1.8 times that of GC_{12} on the sandstone surface, which is due to the fact that NPS has a larger specific surface area and a smaller particle size compared with the sandstone.

4. Conclusions

The adsorptions of two cationic Gemini surfactants (GC_{12} and GC_{16}) on Henan sandstone with BET specific surface area of 5.812 m^2/g and NPS with BET specific surface area of 128.896 m^2/g were studied and compared with that of typical cationic surfactant (hexadecyl trimethyl ammonium bromide, CTAB).

The equilibrium adsorption time depends on the specific surface area of adsorbents, the equilibrium time of CTAB, GC_{16} and GC_{12} in fixed concentration solution on NPS is 2 h at 45°C, while on the sandstone it is 20 h. The equilibrium adsorption amount of Gemini cationic surfactants with the same spacer group onto the solid surface increases with the increase of hydrophobic carbon number. At 45°C, the equilibrium adsorption amounts of GC_{16} and GC_{12} on the surface of sandstone are 4.91 $\mu\text{mol/g}$ and 3.88 $\mu\text{mol/g}$ respectively, while on the surface of NPS the amounts are 22.01 $\mu\text{mol/g}$ and 21.79 $\mu\text{mol/g}$, respectively, that is to say, cationic surfactants prefer to adsorb onto the solid particles with large specific surface area. The adsorption isotherms on the solid surface are in consistent with the Langmuir adsorption rule. The adsorption decreases weakly with the increasing temperature. The saturated adsorption of Gemini cationic surfactant GC_{12} on NPS surface is higher than that on the sandstone surface due to the smaller particle size and larger specific surface area of NPS. Although the saturated adsorption amounts of single-chain CTAB on sandstone and NPS surface are 1.18 times and 1.46 times that of GC_{12} , respectively, considering the number

of tail chain of surfactants, the two alky chains of Gemini surfactants GC₁₂ molecules pack on the solid surface more closely than the single-chain of CTAB molecules.

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