Effects of surfactants on rheological properties of a dispersed viscoelastic microsphere

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ABSTRACT: The swelling ratio is an important performance for the application of viscoelastic microsphere. The reduction of swelling ratio can affect the particle size. The compatibility between particle size and formation pore can affect the plugging performance and EOR capability. Adsorption characteristics of cationic surfactant cetyltriethylammnonium bromide (CTAB), anionic surfactant petroleum sulfonate applied in GangXi oilfield (GXPS), and nonionic surfactant nonylphenol ether (TX-10) onto viscoelastic microspheres and the effect of the three types of surfactant on swelling ratio of viscoelastic microspheres were investigated. Effects of surfactants on rheological properties of viscoelastic microspheres were researched in two different modes referring to steady shear and dynamic shear, respectively, using Physica MCR301 Rheometer. The results showed that the interactions between viscoelastic microsphere and surfactants CTAB were electrostatic attraction and hydrophobic association, that the interaction between viscoelastic microsphere and surfactants TX-10 was just hydrophobic association, and that the interactions between viscoelastic microsphere and GXPS were electrostatic repulsion and hydrophobic association. At the same initial surfactant concentration, all these three types of surfactant could be adsorbed onto the surface of viscoelastic microspheres and reduce its swelling ratio and storage modulus. Because of different amount of adsorption, the extent of reduction order on swelling ratio and storage modulus was CTAB>TX-10>GXPS. In addition, the yield stress of viscoelastic microspheres which was obtained from modeling the data to Herschel-Bulkley model decreases with the increase of surfactant adsorption. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42278.

INTRODUCTION

With the development of oil fields, the phenomenon of water breakthrough among oil wells and water wells becomes serious and the injected water is less effective, so that there is still a large amount of remaining oil in the reservoir. To increase the ultimate recovery of the reservoir, the in-depth flow direction needs to be changed.[1–4] Viscoelastic microsphere is a viscoelastic plugging agent with 3D structure, which can absorb much more water than its own mass, ranging from several to hundreds of times. Furthermore, this agent is hard to release the absorbed liquids even under high pressure.[5,6] Microsphere is prepared by different particle size based on pore characteristics of the target layer in underground conditions. Negative effect on treatment of wells that are caused by formation temperature, salinity, shearing, adsorption, diluted formation water, chromatographic separation effect or other environmental factors can be reduced by using viscoelastic microspheres.[7,8] Viscoelastic microsphere and preformed particle gel are two similar plugging agents. Both of them have the property of swelling and plugging; however, the preparation method, initial size, and appearance are different. Viscoelastic microsphere is a kind of spherical particle prepared by inverse suspension polymerization and the initial size is in the nanometer to micrometer magnitude, whereas preformed particle gel is a kind of irregular blocky-shaped particle prepared by solution polymerization and the initial size is in the micrometer to millimeter magnitude.[9–13] Because of different initial size and sphericity between viscoelastic microsphere and preformed particle gel, viscoelastic microsphere is not easy to be broken after swelling and is prone to deformation under certain pressure, whereas preformed particle gel does not have such a good performance. Therefore, the rheological properties are different between them.

Viscoelastic microsphere and surfactant combination flooding technology has been successfully applied in oil fields. The surfactant can alter the wettability of rock, reduce the interfacial tension between oil and water, and emulsify oil to improve oil recovery. Viscoelastic microspheres not only take in water, but also absorb surfactant molecules. The surfactant molecules can

Table I. Dagang Oilfield Simulated Formation Water

Composition	Na$^+$/K$^+$	Ca^{2+}	Mg^{2+}	Cl$^-$	CO$_3$$^{2-}$	HCO$_3$$^-$	SO$_4$$^{2-}$	Total
Concentration /(mg/L)	2043	39	36	1337	135	3126	10	6726

affect the swelling ratio and rheological properties of the viscoelastic microspheres. The swelling ratio and rheological properties of swelling viscoelastic microsphere are important factors for the deformation of viscoelastic microsphere in the formation, which can affect the EOR performance of viscoelastic microsphere further. The interaction between surfactant and polymer hydrogel has become a subject of considerable theoretical and practical interest and has been extensively studied.[14–17] Kokufuta et al.[14] studied the effect of charge inhomogeneity of polyelectrolyte gels on their swelling behavior. Wu et al.[18] studied the interactions between polyacrylamide particle gel and cationic, anionic and nonionic surfactants. It was found that surfactant concentration showed a substantial increase after swelling of the dry gel particles and the dynamic modulus of the gels showed a significant decrease in the surfactant solutions. They indicate that the surfactant can aggregate to form the micelles and are absorbed onto the surface of particle gels. In the measurement of gel dynamic modulus, these micelles may act as a kind of lubricant between the surfaces of particle gels, the sensor plate and the glass plate. Hence, this will markedly reduce the frictional coefficient between the surfaces of particle gels, the sensor plate, and the glass plate. As a result, the dynamic modulus of G' and G" are decreased comparing with those measured without surfactant added.

However, the systematic study on the adsorption of surfactants onto viscoelastic microspheres has not been studied yet. The purpose of this article is to research the effects of three types of surfactant on rheological properties of a dispersed viscoelastic microsphere. Viscoelastic microspheres were prepared by inverse suspension polymerization, and we systematically studied the adsorption isotherm of anionic surfactant petroleum sulfonate applied in GangXi oilfield (GXPS), nonionic surfactant nonylphenol ether (TX-10), and cationic surfactant cetyltrimethylammonium bromide (CTAB) onto viscoelastic microspheres that were immersed into the surfactant solutions. In addition, the effects of surfactants on the swelling ratio of viscoelastic microspheres were studied. On the basis of the adsorption of surfactants on viscoelastic microspheres and swelling ratio of microspheres, both steady-state shearing and dynamic oscillatory measurements were performed to study the effect of surfactants on rheological properties of viscoelastic microspheres by MCR301 rheometer.

EXPERIMENTAL

Materials
Acrylamide(AM), ammonium persulfate(APS), cetyltriethylammnonium bromide (CTAB), petroleum sulfonate applied in GangXi oilfield(GXPS), nonylphenol ether (TX-10), sodium chloride(NaCl), ammonium chloride (NH$_4$Cl), polyethylene glycol (PEG-200), sodium hydroxide (NaOH), and dispersants (Span80 & Tween60) were all purchased from Sinopharm

Chemical Reagent Co. (China). N,N'-methylenebisacrylamide (MBA) was obtained from Tianjin Kemi'ou Chemical Reagent Co. (China). Anhydrous ethanol was obtained from Xilong Chemical Co. (China). Aviation kerosene was purchased from the market. Deionized water was used for the preparation of all aqueous. The composition of simulated formation water used in this article was shown in Table I.

Preparation
Viscoelastic microspheres were developed by inverse suspension polymerization used in this artilce.[19] Typically, 0.6 g of dispersants [m(Span80) : m(Tween60) = 1 : 1] and 60 g of aviation kerosene were put in a 250-mL four-necked flask equipped with a mechanical stirrer, a dropping funnel, a reflux condenser, and a nitrogen catheter. Meanwhile, the mechanical stirrer was started at a speed of 380 rpm under nitrogen atmosphere to make the dispersants dissolved. Then, the mixed solution with AM, APS, MBA, NH$_4$Cl, PEG-200, and NaOH (6 g of AM, 0.006 g of MBA, 0.2 g of 15 wt % APS, 0.34 g of 15 wt % NH$_4$Cl, 0.005 g of PEG-200, and 0.254 g of 15 wt % NaOH) was added drop wise into the oil phase at the speed of 2 mL/min. The temperature of the thermostat water bath was raised up to 69°C after dripping off. After reacting for 6 h at the certain temperature above, the resulting product was washed several times with anhydrous ethanol and then dried at 80°C for 48 h. Thus, viscoelastic microspheres were obtained.

Microscopic Characterization
Optical microscope (OM) images were taken by XSJ-2 optical microscope (Chongqing Optical Instrument Co., China) to analyze the morphology of viscoelastic microspheres before or after swelling.

Environment Scanning Electron Microscope (ESEM) images were taken by FEI Quanta 200 FEG (FEI Company, Holland) to observe the structure of viscoelastic microspheres after swelling.

A certain quality of viscoelastic microspheres was dispersed in anhydrous ethanol solution or simulated formation water. The particle size distribution curves were measured by Rize2006 laser particle size analyzer (Jinan Runzhi Technology Co., China) to analyze the particle size and distribution. D_{50} was used to express the average particle size of microspheres before or after swelling where D_{50} is the particle size of cumulative distribution curve on probability of 50%.

Adsorption Measurement of Surfactants
An amount of 0.1 g dry viscoelastic microspheres was swollen in 100 mL surfactant solutions with different concentrations (C_0) prepared using simulated formation water in a 250-mL Erlenmeyer flask. Erlenmeyer flask was shaken in SHA-C digital water bath oscillator (Changzhou Guohua Electric Appliance Co., China) at 50°C for 48 h. After reaching adsorption equilibrium, the supernatant fluid was taken out and separated by
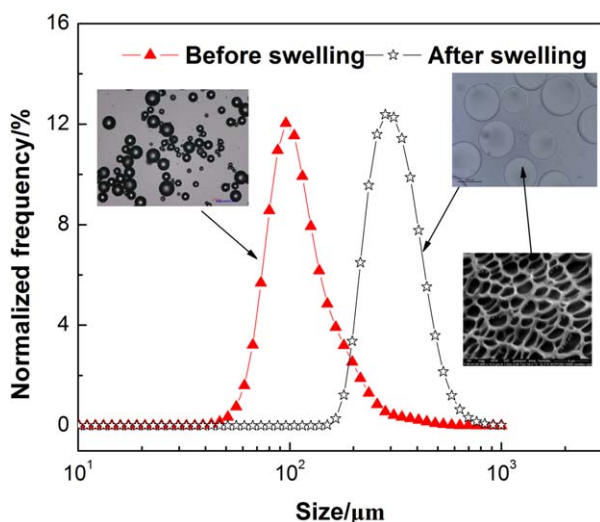
Figure 1. Microstructure of viscoelastic microspheres. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

centrifugation at 2000 RPM for 10 min by means of 80-1 Tabletop Low speed Centrifuge (Jintan Medical Instrument Factory, China). The TX-10 concentration C_e was determined by uvspectrophotometry,[20] and the concentration C_e of CTAB and GXPS was determined by two-phase titration method.[21,22] The equilibrium of adsorption of surfactant on viscoelastic microspheres was calculated by the expression:[23–26]

$$Q_e = \frac{(C_0 - C_e)V}{m_0} \qquad (1)$$

where Q_e is equilibrium adsorption of surfactant on viscoelastic microspheres, mg/g; C_0 and C_e are the initial and equilibrium concentrations of surfactant, mg/L; V is volume of aqueous phase, L; m_0 is the weight of dry viscoelastic microspheres, g.

Swelling Ratio

The swelling ratio of viscoelastic microsphere was evaluated by weighing method.[27] Dry viscoelastic microspheres weighed m_0 were put in a 20-mL test tube. And then 15 mL different concentrations of three types of surfactant solutions were put into the test tubes. Finally, the viscoelastic microspheres were weighed again after they reached swelling equilibrium under temperature of 50°C water bath for 48 h. The swelling ratio of viscoelastic microspheres is:

$$A = \frac{m_1 - m_0}{m_0} \qquad (2)$$

where A is the swelling ratio of the viscoelastic microspheres, and m_1 represents the viscoelastic microspheres weight after they reached swelling equilibrium.

Rheological Properties

The temperature was controlled constantly at 50°C Rheological properties of swelling viscoelastic microspheres after adsorbing surfactants were measured in two different modes named steady shear and dynamic shear mode, respectively, using Physica MCR301 Rheometer (Anton Paar, Austria). A cone-plate geometry measuring system with a diameter of 49.959 mm and a gap
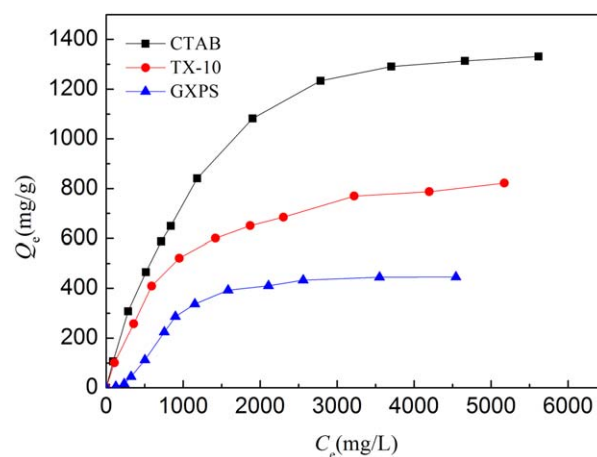
of 0.047 mm was used. The steady shear measurement was conducted with the shear rate from 0.01 to 1000 s^{-1}. Dynamic shear measurement was performed in the linear viscoelastic regime. First, a dynamic measurement was conducted with the strain amplitude from 0.01 to 100% at a constant frequency of 1 Hz to determine the linear viscoelastic regime. Then, the frequency sweep measurement was performed in the linear viscoelastic regime with the frequency from 0.01 to 100 Hz to determine the storage modulus G' and loss modulus G''.

Figure 2. The adsorption isotherm of different types of surfactant onto viscoelastic microspheres. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

Microstructure

Figure 1 gives the particle size analysis of viscoelastic microspheres before or after swelling at room temperature. It can be observed that the average particle sizes of viscoelastic microspheres before or after swelling are 100.5 and 295.8 μm, respectively. As can be seen from Figure 1, the viscoelastic
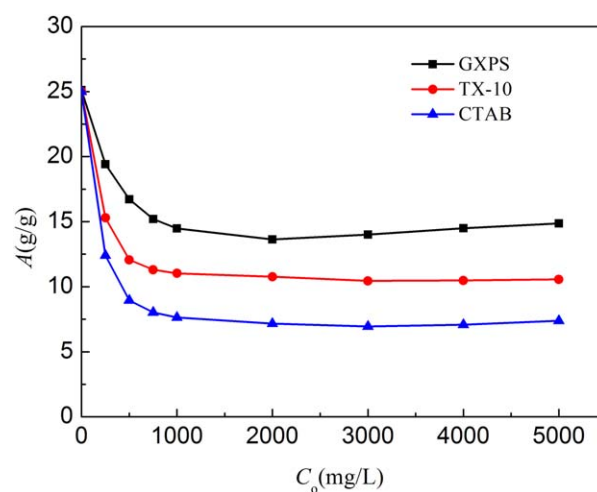

Figure 3. Dependence of swelling ratio of viscoelastic microspheres on equilibrium concentration of different types of surfactant. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
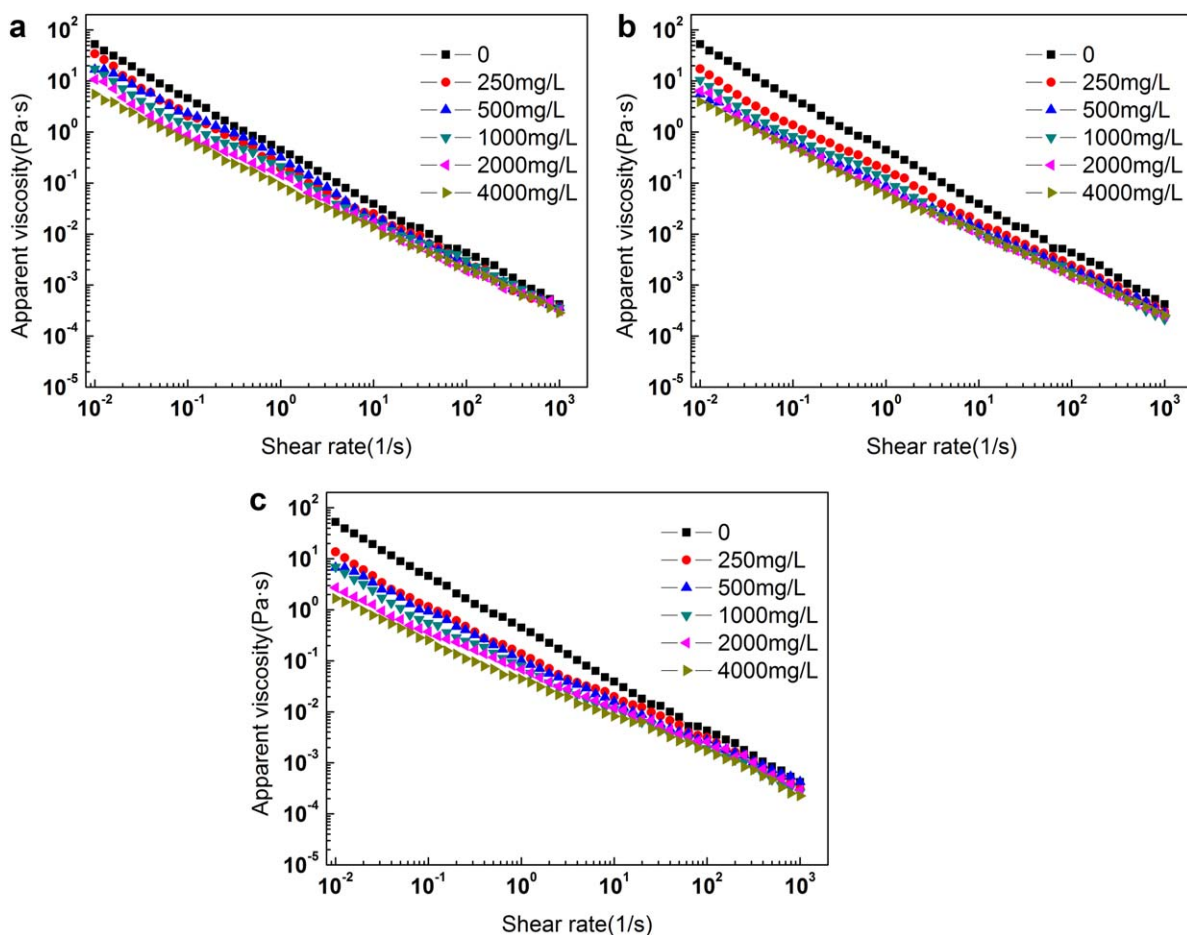
Figure 4. Dependence of apparent viscosity of viscoelastic microspheres as a function of shear rate at different surfactant concentrations for the three types of surfactant (a) GXPS (b) TX-10 (c) CTAB. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

microspheres are spherical particles and can swell many times because of the absorption of water. As the viscoelastic microspheres contact with water, the water molecules can go into the internal network under the action of inside and outside osmotic pressure, stretching the molecular chain of the network structure, enlarging the volume of viscoelastic microspheres. The three-dimensional network structure with pore sizes ranging from 1.29 to 2.95 μm can be seen clearly from the ESEM image (Figure 1). The investigated microstructure confirms the swelling mechanism of the viscoelastic microspheres.[28]

Adsorption of Surfactants

The surface of viscoelastic microspheres is negatively charged because of the existence of carboxyl. The zeta potential of viscoelastic microspheres is -5.8 mV and the surface electrical is an important cause for the different adsorption capacities between surfactants and viscoelastic microspheres, especially for those of cationic surfactant (CTAB) and anionic surfactant (GXPS). The interaction force between CTAB and viscoelastic microspheres is electrostatic attraction, whereas the interaction force between GXPS and viscoelastic microspheres is electrostatic repulsion. The adsorption isotherm of different types of surfactant (CTAB, TX-10 and GXPS) on viscoelastic microspheres is shown in Figure 2. the figure shows that the order of adsorption equilib-

rium of three types of surfactant onto viscoelastic microspheres is CTAB> TX-10>GXPS under the same surfactant concentration.

CTAB can be hydrolyzed to produce quaternary ammonium salt ions in the simulated formation water. CTAB can be adsorbed onto viscoelastic microspheres by electrostatic attraction and hydrophobic association.[29] The equilibrium adsorption Q_e of CTAB onto viscoelastic microspheres increases with the increase of C_e. The increase of equilibrium adsorption slows down when it increases to a certain value. Here, the adsorbed CTAB molecules repel with the solution of CTAB molecules and Q_e almost remain the same. Therefore, CTAB adsorption capacity is the largest. TX-10 is adsorbed only by hydrophobic association with viscoelastic microspheres.[30] The equilibrium adsorption Q_e of TX-10 on viscoelastic microspheres also increases gradually with the increase of C_e, and when the adsorption potential is full, Q_e will no longer increase.

The adsorption isotherm of GXPS consists of three sections.[31] The first section is characterized by the increase of C_e, Q_e being small. This is because the negatively charged carboxyl ionized from the molecular side chain of the viscoelastic microsphere forms diffused electric double layer and has an electrostatic repulsion effect on sulfonic acid base group of GXPS. Therefore,
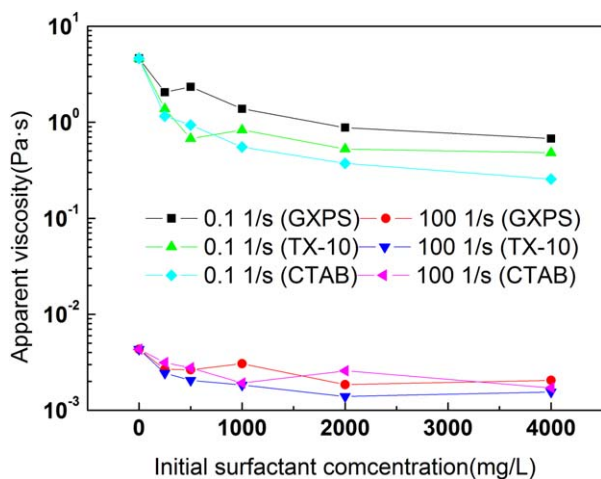
Figure 5. Dependence of apparent viscosity of viscoelastic microspheres as a function of initial surfactant concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the adsorption of GXPS on viscoelastic microspheres is insignificant at low GXPS concentration. With the increase of C_e, Na^+ hydrolyzed from GXPS compresses the diffused electric double layer, decreases the thickness of the electric double layer and

reduces the electrostatic repulsion force between the sulfonic acid base group of GXPS and the sulfonic acid base group of viscoelastic microsphere. As a result, hydrophobic association structure is gradually formed by hydrophobic interaction between the hydrophobic tail chain of GXPS and the molecular hydrophobic chain of viscoelastic microspheres. After that, the Q_e begins to increase and this is the second section. When Q_e reaches a certain degree, Q_e keeps constant because of the electrostatic repulsion effect between the sulfonic acid base groups of adsorption GXPS molecules and the sulfonic acid base groups of GXPS in the solution. This is the third section. Because of three different types of interaction mechanism between different surfactants and microspheres, the order of adsorption equilibrium of surfactant on viscoelastic microspheres is CTAB> TX-10>GXPS.

Swelling Ratio

Viscoelastic microspheres can absorb much more water than their mass. After swelling, they do not easily release the absorbed water even under high pressure. The swelling ratio of viscoelastic microspheres was commonly characterized by the mass ratio of viscoelastic microspheres in an equilibrium state in surfactant solution to the dried particle.[32,33] The swelling ratio of viscoelastic microspheres as a function of equilibrium
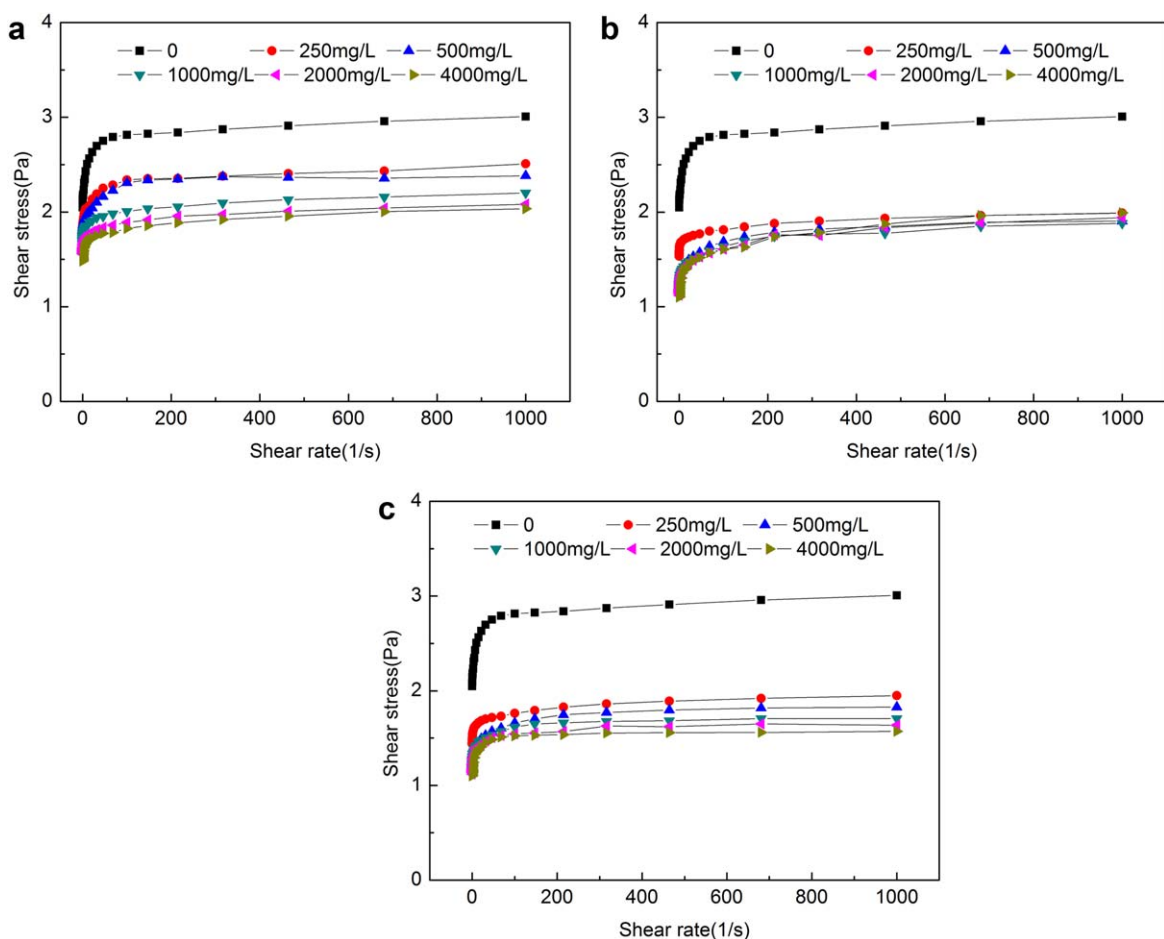

Figure 6. Dependence of shear stress of viscoelastic microspheres on shear rate at different surfactant concentrations for three types of surfactant (a) GXPS (b) TX-10 (c) CTAB. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table II. Analyses of Orthogonal Experiments

Type of surfactant	Surfactant concentration (mg/L)	τ_R (Pa)	K (Pa·s^n)	n	R^2
CTAB	0	1.55547	0.71166	0.11019	0.96400
	250	1.30390	0.23696	0.14678	0.99312
	500	1.10214	0.23676	0.17346	0.98989
	1000	0.87212	0.44727	0.09901	0.98915
	2000	0.71409	0.56383	0.07854	0.98991
	4000	0.54120	0.70773	0.06140	0.97639
TX-10	0	1.55547	0.71166	0.11019	0.96400
	250	1.43466	0.19342	0.15257	0.99730
	500	1.13172	0.20414	0.20372	0.99057
	1000	1.06246	0.24531	0.17921	0.99600
	2000	1.03564	0.22891	0.20137	0.99792
	4000	1.03124	0.20520	0.22707	0.99778
GXPS	0	1.55547	0.71166	0.11019	0.96400
	250	1.55422	0.37388	0.13938	0.98247
	500	1.55796	0.32409	0.15410	0.96511
	1000	1.53606	0.23454	0.15056	0.99940
	2000	1.45139	0.20920	0.15992	0.99968
	4000	1.40042	0.18921	0.17580	0.99952

concentration of different types of surfactant in solution was illustrated in Figure 3. As the curves show, under the low concentration of surfactant, the swelling ratio of viscoelastic microspheres after adsorption was smaller than that before adsorption. With the increase of surfactant concentration, surfactant molecules exist both in external aqueous phase and in the network of viscoelastic microspheres in the form of micelles and distribute evenly. Therefore, the volume of viscoelastic microspheres insignificantly changes. The effect of surfactant on the swelling ratio of viscoelastic microspheres is closely related to the adsorption of surfactant on viscoelastic microspheres. The more adsorption of surfactant, the smaller the swelling ratio of viscoelastic microspheres is, and hence, under the same surfactant concentration, the swelling ratio reduction order of viscoelastic microspheres is CTAB> TX-10>GXPS, which is similar to the order of adsorption equilibrium.

Rheological Properties
Steady Shear Rheological Property. Figure 4 shows the apparent viscosity of viscoelastic microspheres as a function of shear rate at different surfactant concentrations. For all the three types of surfactant, the apparent viscosity of viscoelastic microspheres at low shear rate exhibits rather high values, which is related to the three-dimensional network structure (Figure 1), and decreases with the increase of shear rate. This is called shear-thinning behavior, which has been observed in a dispersion of hematite in [C$_2$mim][EtSO$_4$] and in a dispersion of single-walled carbon nanotubes (SWCNTs) in [C$_4$mim][BF$_4$].[34,35] Figure 5 is a plot of shear viscosity versus initial concentration of different types of surfactant at the shear rate of 0.1 and 100 s^{-1}. As shown in the curves, after being immersed into surfactant solutions, the apparent viscosity of viscoelastic microspheres at

a lower shear rate decreases with the increase of surfactant concentration compared with the unimmersed one. The three-dimensional network structure of viscoelastic microspheres is destroyed at higher shear rate, and viscoelastic microspheres exhibit shear-thinning, too. Therefore, at higher shear rate, the apparent viscosity of viscoelastic microspheres immersed in surfactant solutions with different surfactant concentrations hardly changed.

A plot of shear stress, τ, versus shear rate, $\dot{\gamma}$, at different concentration for three types of surfactant (Figure 6) were fitted to Herschel-Bulkley mode (eq. (1)),[36–39]

$$\tau = \tau_R + K\dot{\gamma}^n \qquad (3)$$

where τ and τ_R are shear stress and yield stress, separately, (Pa), $\dot{\gamma}$ is shear rate, (1/s), K is consistency index (Pa·s^n), and n is power law index.

The fitting results are shown in Table II. From Table II, it can be seen that the yield stress decreases with the increase of initial surfactant concentration. Yield stress represents the minimum amount of force needed to be applied to the fluid before it would start to move.[40] Adsorption of surfactant molecules reduces the yield stress of viscoelastic microspheres and makes it easier for the gel to move. Because the adsorption of CTAB among the three types of surfactant is the biggest, the yield stress of CTAB solution is the smallest at the same concentration.

Dynamic Oscillating Rheological Property
A frequency sweep (0.01–100 Hz) on viscoelastic microspheres both free and with different types of surfactant was performed with oscillatory strain amplitude of 0.1%. The strain amplitude
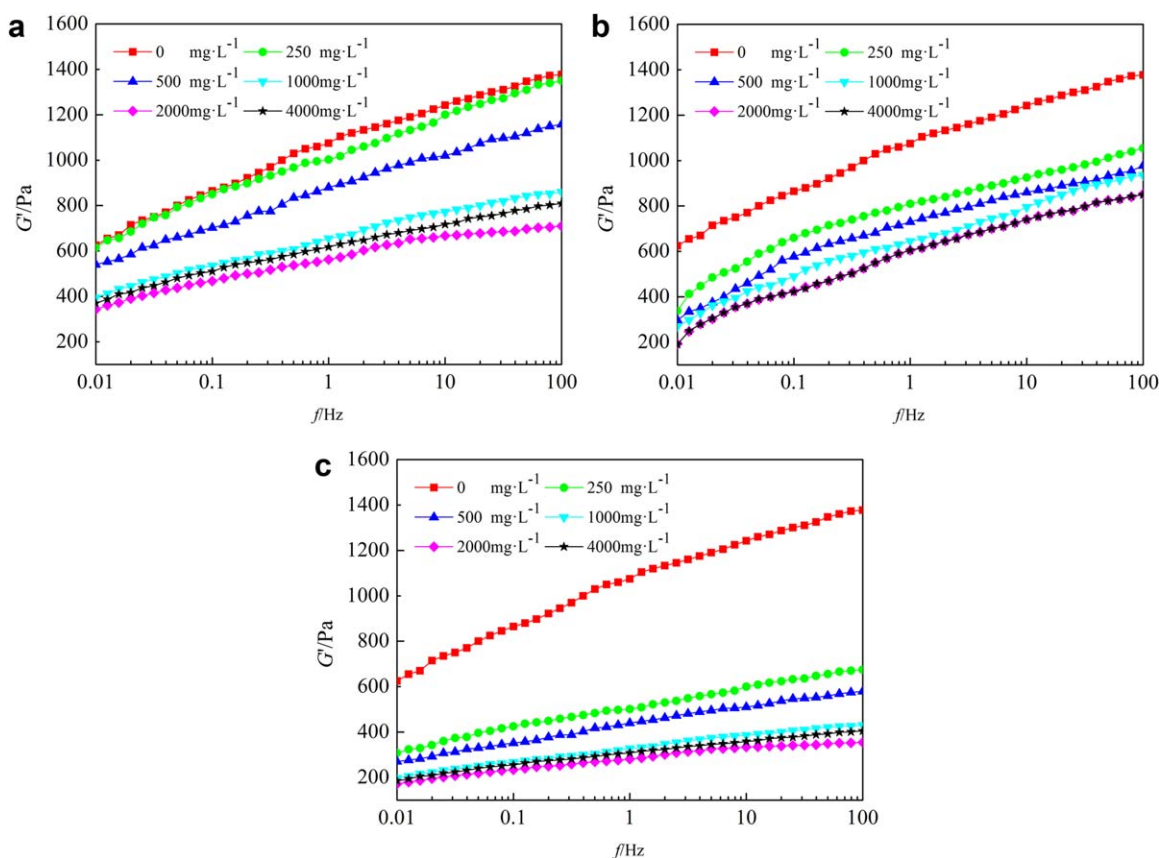
Figure 7. Dependence of storage modulus of viscoelastic microspheres on frequency at different surfactant concentration (a) GXPS, (b) TX-10, (c) CTAB. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was chosen based on strain sweep at constant frequency to determine the conditions within the linear viscoelastic region. Figure 7 shows the dependence of storage modulus of viscoelastic microspheres on frequency at different surfactant concentrations. The results show that the three types of surfactants are able to make the storage modulus lower, that the order of storage modulus reductions at the same concentration is: CTAB>TX-10>GXPS.

The interaction force between GXPS and viscoelastic microspheres is electrostatic repulsion at low concentration, but the adsorption on viscoelastic microspheres is small. The storage modulus of viscoelastic microspheres decreases slightly, simply because of salt effect which compresses the diffuse electric double layer. When concentration of GXPS increases to a certain extent, the interaction force is hydrophobic association, then more and more GXPS molecules are adsorbed on the surface of viscoelastic microspheres, the volume of it gradually reduces, the interaction forces are weaken, and storage modulus significantly decrease. After reaching the equilibrium concentration, storage modulus substantially remained steady.

The interaction force between TX-10 and viscoelastic microspheres is only hydrophobic association even at low concentration. When concentration of TX-10 is below the CMC, steric clash increases and the hydrophilic radical occurs orientation, the ability of the hydrophilic radical to resist elastic deformation

is weakened, so the storage modulus decreases. After reaching the equilibrium concentration, storage modulus changes slightly.

The interaction forces between CTAB and viscoelastic microspheres are hydrophobic association and electrostatic attraction. The viscoelastic microspheres are negatively charged, so a large number of cationic are adsorbed on their surface, and the adsorption increases with hydrophobic interaction, so the interaction forces weaken and storage modulus decrease significantly. After reaching the equilibrium concentration, storage modulus substantially remain unchanged.

CONCLUSIONS

GXPS molecules can adsorb onto viscoelastic microspheres because of the electrostatic repulsion and hydrophobic association. The interaction forces between CTAB and viscoelastic microspheres are electrostatic attraction and hydrophobic association. TX-10 molecules can be adsorbed onto viscoelastic microspheres via hydrophobic association. Because of the adsorption of surfactant, the swelling ratio and storage modulus of viscoelastic microspheres decrease to some extent, and the yield stress of viscoelastic microspheres which was obtained from modeling of the data to Herschel-Bulkley model decreases. Because of different amount of adsorption, the extent of reduction order on swelling ratio and storage modulus was CTAB>TX-10>GXPS. This study will significantly benefit the

application of viscoelastic microspheres and surfactant combination flooding technology in heterogeneous reservoirs.

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