

Viscoelastic Properties of Anionic Brominated Surfactants (Sifat Viskokenyal Surfaktan Anionik Terbromida)

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ABSTRACT

The scarcity of land based oil reserves has necessitated the exploration of off shore oil. This exploration is often carried out in pristine waters and the use of green chemicals is essential to reduce environmental degradation. In the recovery of oil and gas from rocky formations, well bore fluids such as packer fluids, fracturing fluids, conformance and permeability control fluids are extensively employed. Potassium oleate as viscoelastic surfactants gives a low partition coefficient when in contact with hydrocarbon. Bromination of the oleate chain has been shown in this work to increase the partition coefficient and still maintaining its viscoelasticity. The partition coefficient increased to ca 8% compared to negligible for potassium oleate. A gel was formed when a 20% solution of potassium 9-bromo stearate was mixed with 8% KCl. Contacting this gel with hydrocarbon resulted in a loss of viscosity due to the improved solubility of the brominated compound in hydrocarbon. This facilitates the removal of the surfactants after its use as a fracturing fluid. The viscoelastic properties were demonstrated using a Bohlin rheometer. The graph of viscosity vs shear rate shows at first a shear rate independence up to a shear rate of 0.2 s^{-1} and then falling with shear rate typical of a viscoelastic fluid. The zero shear viscosity η_0 varied from 18 Pa s to 220 Pa s whilst the shear viscosity at 100 s^{-1} ranged from 0.16 Pa s to 1.5 Pa s for surfactant concentration from 5 to 20%.

Keywords: Brominated oleate; green chemistry; oil and gas; surfactants; viscoelastic

ABSTRAK

Kekurangan simpanan takungan minyak mentah di daratan memerlukan carigali dilakukan di bahagian lautan. Pencarian minyak mentah di lautan biasanya dilakukan di perairan bersih dan penggunaan bahan kimia hijau adalah perlu bagi mengurangkan kerosakkan alam sekitar. Bagi memperolehi minyak dan gas dari kawasan berbatu, penggunaan bendalir pengerudi lubang seperti bendalir pepadat, bendalir pemecah, pembentuk dan bendalir pengawal ketelapan digunakan. Kalium oleat boleh digunakan sebagai surfaktan viskokenyal memberikan pemalar partisi yang rendah bila bersentuhan dengan hidrokarbon. Dalam kajian ini rantai oleat terbromida menunjukkan peningkatan pemalar partisi disamping mengekalkan sifat viskokenyalnya. Gel terbentuk apabila 20% larutan kalium 9-bromo sterat yang dicampurkan dengan 8% KCl. Penyentuhan gel ini dengan hidrokarbon akan menurunkan kelikatan yang disebabkan penambah baikan kelarutan sebatian terbromida dalam hidrokarbon. Ini akan menggalakkan penyingkiran surfaktan bendalir pemecah selepas digunakan. Sifat viskokenyal ditunjukkan dengan menggunakan reometer Bohlin. Graf kelikatan terhadap kadar ricih menunjukkan pada permulaanya kadar ricih tidak bergantung kepada kadar ricihan pada 0.2 s^{-1} dan kemudian jatuh dengan kadar ricih yang biasanya sebagai bendalir viskokenyal. Kelikatan ricih pada sifar η_0 daripada 18 Pa s ke 220 Pa s dengan nilai kelikatan ricih pada julat 100 s^{-1} daripada 0.16 Pa s ke 1.5 Pa s untuk kepekatan surfaktan antara 5 ke 20%.

Kata kunci: kimia hijau; minyak dan gas surfaktan; oleat terbromida; viskokenyal

INTRODUCTION

The oil and gas industries has been using fracturing as a technique to increase well productivity. High pressure is applied using positive displacement pumps against the reservoir rock formations to create fissures. A proppant, which is usually sand at 20-40 mesh, is used in conjunction with the fracturing fluid in order to keep the fracture open after the fracture fluid has been removed. Traditionally, low cost high molecular weight polymers such as guar gum have been used. After its use, the gels are degraded by a delayed oxidative or enzymatic breaker which is pumped in together with the fracture fluid. Despite the polymer

cleavage, the process was often left with portions of high molecular weight polymers which reduces the conductivity of the well (Jones et al. 2007).

The use of green surfactants made from renewable sources that can form worm like micelles upon the addition of activators such as brine have recently been used. These surfactants do not leave any residue in the formations as the gel breaks after sustained exposure to hydrocarbons.

Single chain surfactants when solubilized in water forms spherical micelles. For ionic surfactants, the presence of excess counterions reduces the repulsive interaction between the charged head groups thereby decreases the spontaneous curvature of the aggregates. The reduction

in the spontaneous curvature favours the formation of cylindrical aggregates. The surfactant molecules at the both ends of these cylindrical aggregates have excess free energy in comparison with the molecules in the cylindrical part. This excess energy is called end cap energy and is the driving force for the linear growth of cylindrical micelles. These cylindrical micelles entangle with each other causing the surfactants to exhibit viscoelastic behaviour with a low shear Newtonian region and, a shear sensitive non Newtonian region beyond a critical shear rate. This shear thinning behaviour also allows superior penetration of porous formations (Brown et al. 2003). A more important function of the viscoelastic surfactant is that they act as reversible thickening agents so that their viscosity varies significantly between water and hydrocarbon bearing zones. In this way, the fluid is able to preferentially penetrate hydrocarbon bearing zones.

One of the problems associated with the use of surfactants is that they form stable oil in water emulsions during clean up operations. Such emulsions form because conventional viscoelastic surfactants have limited solubility in hydrocarbon. Aqueous phase viscoelastic surfactants when in contact with hydrocarbons will distribute itself between the two phases according to the partition coefficient. Potassium oleate, a palm oil derivative, has been used widely as viscoelastic surfactants. However its partition coefficient with hydrocarbon is low. Recently, a patent has been issued which reported that the partition coefficient of a surfactant can be improved by the addition of a short alkyl chain to the alpha carbon of a surfactant molecule (Jones et al. 2007). This work showed that bromination of the alkyl chain can also improve the partition coefficient of the viscoelastic surfactant.

MATERIALS AND METHODS

MATERIALS

Oleic acid (>90%), potassium oleate and potassium chloride were purchased from Sigma. Hydrogen bromide was supplied from Merck as 33% dissolved in acetic acid.

Bromination of Oleic Acid 80 mL of hexane and 20 g of oleic acid was mixed into a 500 mL conical flask with stopper. Eighty milliliter of 33% HBr in CH_3COOH was then added into the flask and immediately closed with the stopper. The mixture was stirred vigorously for 60 minutes after which 250 mL of deionised water was added into the flask to extract the unreacted acid. The water layer was removed and the organic layer was washed with deionised water until it is neutral. The organic layer was then evaporated to dryness and further dried in the vacuum oven at 60°C. The brominated methyl oleate was analysed using H-NMR.

Nuclear Magnetic Resonance H-NMR spectras were recorded using a 275 MHz JOEL NMR spectrophotometer operating at 270.05 MHz. Each sample of 0.2 g was

mixed with 400 μL of deuterated chloroform and a small proportion of TMS as internal reference was added. This mixture was introduced into a 5 mm diameter NMR tube. The acquisition parameters were as follows: spectral width, 5800Hz, relaxation delay, 3.9 second, number of scans 16, acquisition time 2.7 seconds, pulse width 90°. The intensities of the peaks were determined by the integration of the areas by the equipment software. Chemical shifts are reported in parts per million downfield from TMS.

Saponification of 9-bromo Stearic Acid Two grams of sample was accurately weighed into a round bottom flask equipped with a reflux condenser. After that, 50 mL of potassium hydroxide in ethanol-water mixture (3:1 by volume) was added into the flask. The mixture was allowed to boil gently until the sample is completely saponified. After the saponification process was completed, the ethanol and water were removed under vacuum and the product was further dried in a vacuum oven at 60°C. The resulting anhydrous potassium soap was recrystallized from absolute ethanol to remove unreacted fatty acid and excess salt. After drying, a fine white to light yellowish powder was obtained.

Partition Coefficient The partition coefficient was determined using the shake flask method. Various quantities of surfactant were dissolved in 50 mL of water in a test tube after which 50 mL of hexane was added. The mixture was vigorously shaken and the concentration of surfactant in each of the liquid layers determined gravimetrically. The mixtures were stabilized at various temperatures using a constant temperature bath.

Gel Break Test Five milliliter of surfactant of the designated concentration in 8% KCl was prepared. Five milliliter of hexane as solvent was added to the high viscosity solution. An interface layer can be observed and the samples were left to stand. The time required for the gel to completely break was recorded.

Rheology Samples for rheological measurements were prepared by dissolving the designated amount of surfactant in DI water and then diluting it with the required concentration of KCl. The mixtures were homogenized and left to equilibrate for at least 1 day to ensure that the samples were bubble free. The rheology profiles were measured at 25°C using Bohlin VOR Rheometer. A cone and plate geometry of 4° cone angle and with a diameter of 40 mm was used. This rheometer consist of a rotating lower plate and a fixed upper cone with a measuring gap of 70 μm . The measurements were taken with a controlled shear rate being applied to the sample whilst the resulting shear stress is monitored with a force transducer. The temperature of the system was controlled with an oil bath. A sample cover was used to minimize any change in sample composition by evaporation during the measurements. Frequency sweep measurements were performed in the linear viscoelastic regime of the samples as determined by dynamic strain sweep measurements.

RESULTS AND DISCUSSION

The starting material for the synthesis of brominated fatty acids were oleic acid. The oleic acid was analysed by using GC to contain predominantly 96.6% of C18:1 and 3.4% of C18:2. Oleic acid was reacted with hydrogen bromide to give the alkyl halide. The double bond at position 9 of the hydrocarbon backbone is a source that is rich in π electrons and therefore acts as a base. The proton of the hydrogen bromide being electrophilic, adds to the double bond producing a carbocation intermediate. The reaction is completed by the subsequent addition of the bromide to the carbocation yielding the product. Samples were drawn periodically, washed, neutralized, dried and analyzed using H-NMR. The spectrum indicates the existence of brominated carbon at $\delta = 24$ with

H-NMR: 0.9 (t, 3H, $\text{CH}_3\text{-CH}_2\text{-}$), 1.4-1.5 (m, 2H, $\text{CH}_3\text{-CH}_2\text{-CH-}$), 2.1-2.2 (m, 2H, $\text{-CH}_2\text{COO-}$), 5.35(t, 1H, $\text{-CH}_2\text{=CH-}$), 4.0(m, 1H, -CHBr).

After reacting hydrogen bromide with the oleic acid for an hour, the reaction was substantially complete as indicated by the disappearance of the peak at $\delta 5.35$ representing the double bond of the oleic acid (Figure 1).

This was replaced by the peak at $\delta 4.0$ representing -CHBr (Figure 2). The brominated oleic acid was saponified with potassium chloride to give potassium 9-bromo stearate. This is essentially a brominated anionic surfactant.

Anionic surfactants dissolve in aqueous media to form a surfactant solution. At concentrations above the critical micelle concentration (CMC) the surfactant molecules self assemble into spherical micelles. This formation is controlled by two competing energies: the repulsive interactions between the ionic head groups on the micelle surface and the attractive van der Waals interactions between the tails. Surfactant morphologies beyond the CMC are controlled by the packing parameter P which is defined as the ratio of the volume occupied by the tail group to the volume of a cylinder, whose cross sectional area is equal to the area of the head group and the length which is equivalent to that of the tail group (Raghavan 2001).

P is given as

$$P = \frac{v}{Al}, \quad (1)$$

where, v is the volume occupied by tail group, A is the area of head group, l is the length of tail group

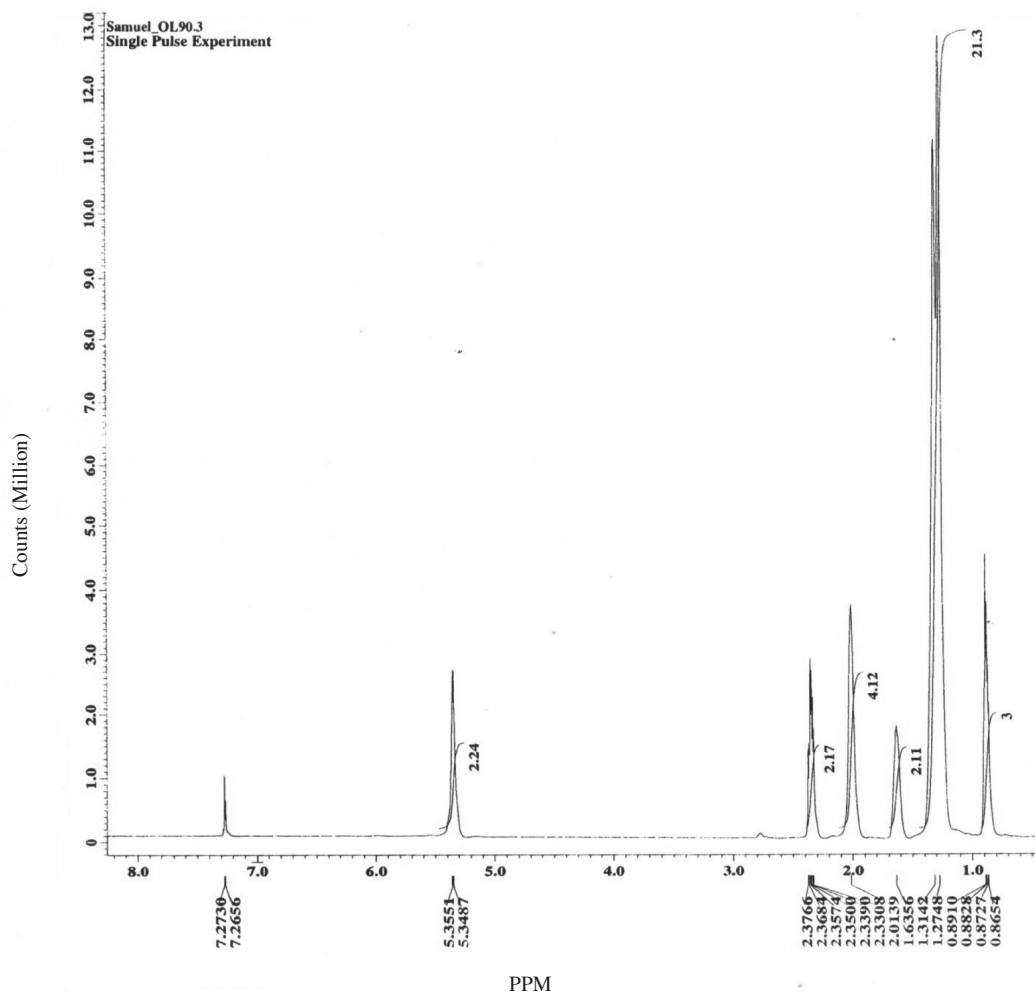


FIGURE 1. H-NMR spectrum of oleic acid

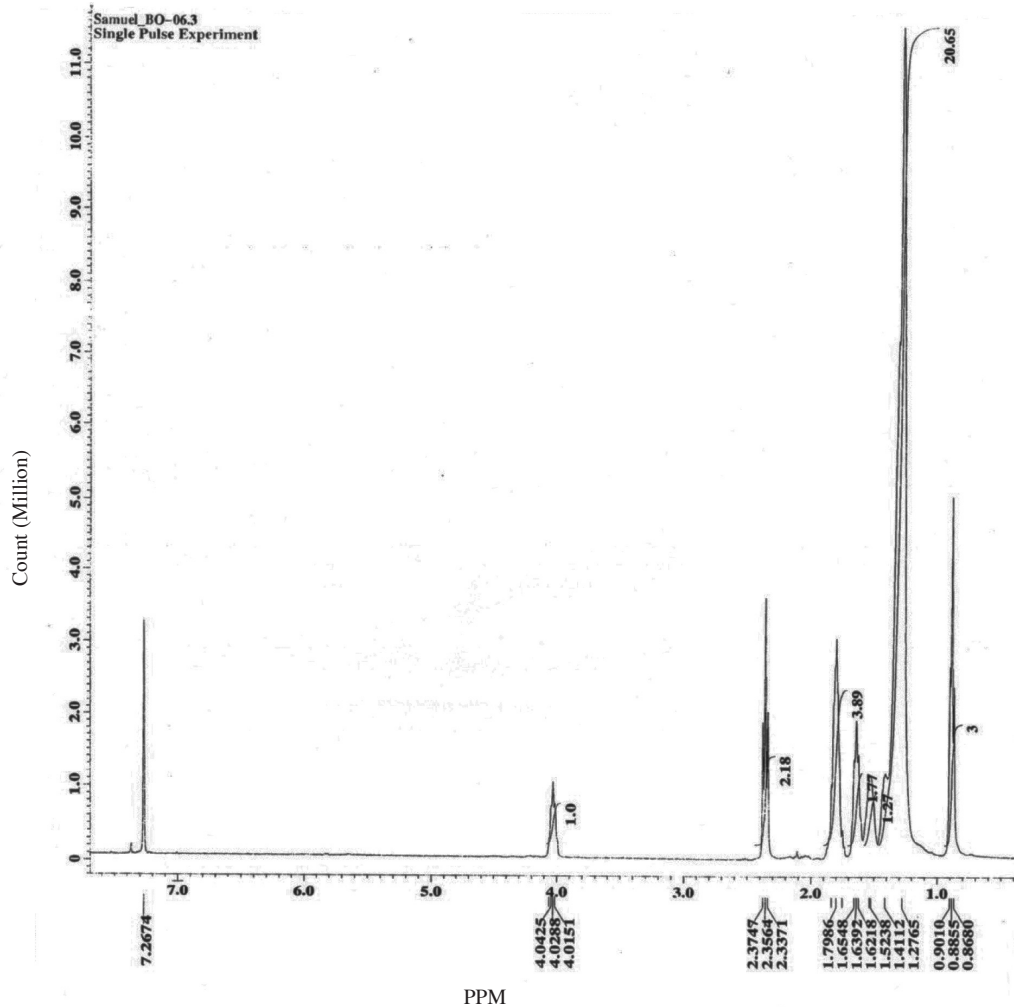


FIGURE 2. H-NMR spectrum brominated oleic acid

Surfactant molecules with packing parameter $P > \frac{1}{3}$ self-assemble into spherical micelles. When small ionic species are added to the micellar solution, the electrostatic repulsion between the surfactant headgroups is reduced and the packing parameter increases. Consequently, the surfactant molecules go under a phase transition to form rod-like structures. This phenomenon can be seen in the gentle rise of viscosity in Figure 3 from 2% to 6%. At these low salt content these rod like micelles will grow and increase the viscosity by a small amount (Magid & Li 2000). When the length of the rod like micelles has increased further so that they are flexible and can curve freely, the micelles become wormlike (Driess 2007) and this resulted in the steep rise in viscosity from 6 to 8% salt concentration. Finally, at very high concentrations above 8%, the worm-like micelles break into several pieces (Ali & Makhloufi 1999) and at the same time salting out occurs and the viscosities drop rapidly. The existence of worm-like micelles at high surfactant and salt concentrations for anionic surfactants have been proven by cryo TEM (Lin 1996).

The viscosity as a function of shear rate is presented in Figure 3 for various concentrations of potassium 9-bromo

stearate in 8% KCl. The fluid rheogram is characterized by a high viscosity region at low shear rates, in which the surfactant solutions display a Newtonian behaviour and a shear thinning region where a drop in viscosity is observed. The high viscosity observed in the Newtonian region can be interpreted as the result of entanglement of the worm-like micelles. Shear thinning is believed to be due the alignment of the aggregates as the shear rate increases (Goel et al. 2002). It is observed that the critical shear rate for shear thinning remained essentially constant at around 0.3 s^{-1} for all concentrations of the viscoelastic surfactant (VES). Total shear thinning is more than 2 orders of magnitude. The zero shear viscosity, η_0 , varied from 220 Pa s for 20% VES concentration to 18 Pa s for 5% concentration. As the concentration of the VES increases it is expected that the viscosity also increases as the network density increases in tandem. The surfactant gel shear thin to 1.5 Pa s (20%) and to 0.16 Pa s (5%) at 100 s^{-1} .

Well bore fluids for suspension of particulates (Goel et al. 2002) is acceptable if the shear viscosity is more than 0.1 Pa s at a shear rate of 100 s^{-1} . At a viscosity of 0.1 Pa s, the settling rate of oil field proppant is 1 cm min^{-1} which

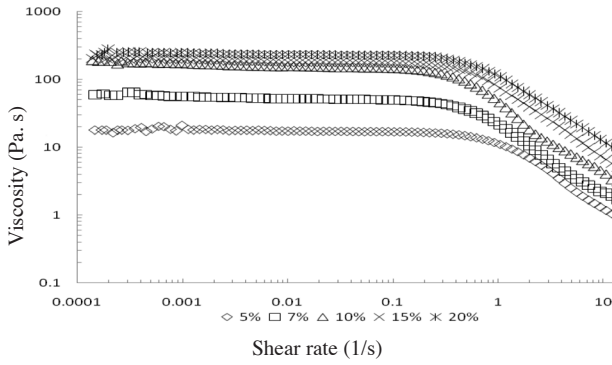


FIGURE 3. Rheogram of viscosity as a function of shear rate for 5%, 7%, 10%, 15%, 20% of potassium 9-bromo stearate in 8% KCl

is less than the 5 cm/min deemed acceptable. The shear rate of 100 s^{-1} is commonly used as the shear rate found in proppant transport.

Figure 4 shows the variation of shear stress for the brominated surfactant as the shear rate is increased. The shear stress increases linearly until $\sim 0.8 \text{ s}^{-1}$ and rapidly plateau. This stress plateau is believed to be caused by shear banding (Lerouge 2000). Such bands are believed to be associated with the coexistence of different regions of different shear rates within the sample. The critical shear rate is constant at 0.8 s^{-1} for increasing concentrations of VES.

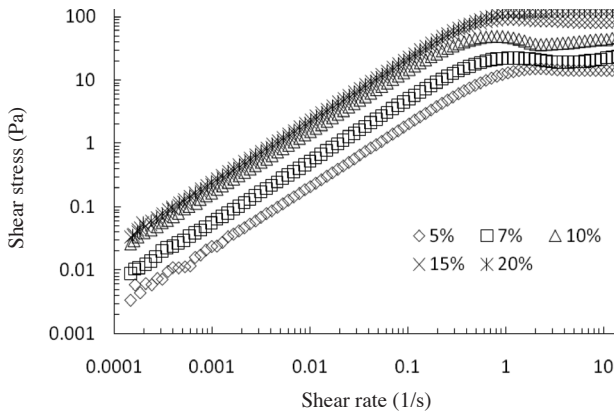


FIGURE 4. Shear stress as a function of shear rate for various concentrations of potassium 9-bromo stearate in 8% KCl solution

The behaviour of the elastic modulus against frequency is shown in Figure 5. The moduli increases as the frequency increases and reaches a plateau, G_0 at a frequency of around 0.2 Hz. The plateau modulus, G_0 , increases as the concentration increases. G_0 is a measure of the average length between entangled points or the mesh size and is usually proportional to the number density of the micelle aggregates (Kim & Yang 2000). As shown in Figure 7, G_0 increases monotonically with concentration of the brominated VES. At low frequencies, the elastic modulus

also increases with concentration but only to a maximum at around 15% concentration and then decreases. This shows that the samples are showing increasing gel-like properties. The gel-like character for increasing concentration is also apparent from the graph of Figure 4 where the apparent yield stress increases with concentration.

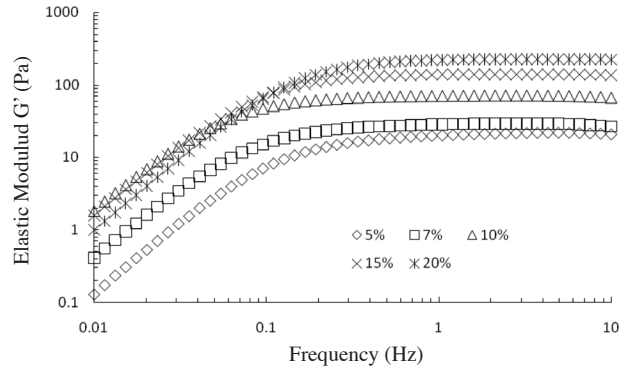


FIGURE 5. Elastic modulus versus frequency for various concentrations of potassium 9-bromo stearate in 8% KCl solution

Figure 6 shows the elastic response and Figure 7 shows the viscous response of the brominated VES with frequency. The oscillation response of a viscoelastic material is described by its complex modulus G^* (Goodwin & Hughes 2000) and this is defined as the ratio of maximum stress σ_0 , divided by maximum strain γ_0 . The complex modulus is constant for a given frequency. Expressing G^* in complex number notation gives:

$$G^*(\omega) = G \left(\frac{i\omega\tau_m}{1+i\omega\tau_m} \right), \quad (2)$$

where G is the shear modulus and τ_m is the time constant.

This expression describes the variation of complex modulus G^* with frequency for a Maxwell model. Separating the real and imaginary components of this

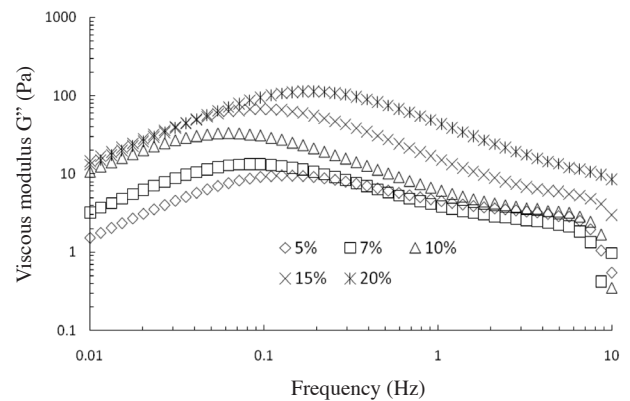


FIGURE 6. Viscous modulus, G'' versus frequency for various concentrations of potassium 9-bromo stearate in 8% KCl solution

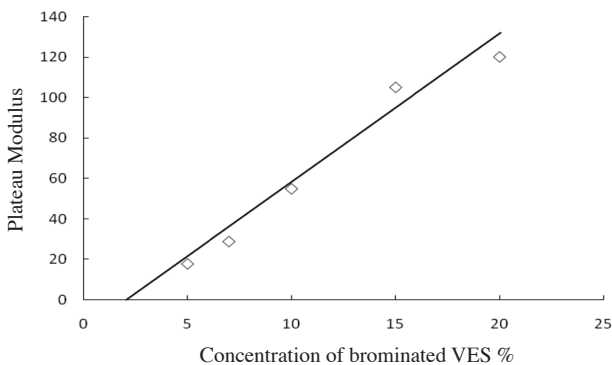


FIGURE 7. Plateau modulus G_0 vs concentration of potassium 9-bromo stearate in 8% KCl

expression gives the storage modulus G' and loss modulus G'' .

$$G^*(\omega) = G'(\omega) + iG''(\omega). \tag{3}$$

$$G'(\omega) = G \frac{(\omega m)^2}{1 + (\omega m)^2}. \tag{4}$$

$$G''(\omega) = G \frac{\omega m}{1 + (\omega m)^2}. \tag{5}$$

These expressions represent the frequency dependence of the stress with respect with strain. It is normal to represent these as two moduli which determine the component of stress in phase with the applied strain and the component out of phase by 90° . Plotting the loss modulus G'' against frequency, G'' will first increase from zero to $G/2$, and then reduces to zero at high frequency giving a bell shaped curve. The storage modulus G' increases from zero to a maximum value and as the frequency tends to infinity the complex modulus and the storage modulus equate (Goodwin & Hughes 2000). The maximum in the curve for G'' and the crossover point between the curves G' and G'' occurs at τ_m . For viscoelastic fluids this time constant is often called relaxation time.

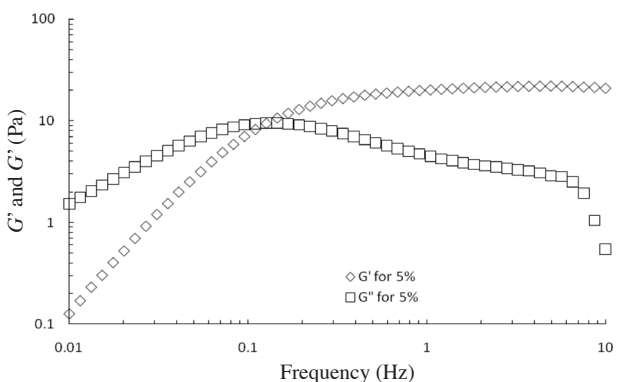


FIGURE 8. G' and G'' versus frequency for potassium 9-bromo stearate in 8% KCl

Figure 9 shows the variation of the elastic modulus and the viscous modulus in a single graph for a 10% solution of potassium 9-bromo stearate in 8% KCl. There is a clear viscoelastic behavior, as at low frequencies the viscous or loss modulus, G'' , is higher than the elastic or storage modulus, G' , indicating that the sample behaves as a liquid, whereas at high frequencies ($G' > G''$), the sample behaves as a solid. Figure 9 also shows that in the low ω region, the data points can be fitted to the Maxwell equations, but in the high ω region, experimental data deviate from the model. The G'' to G' crossover frequency shifts to lower values suggesting a slower relaxation process. This is a further indication of the formation of worm-like micelles (Varade et al. 2007) where the longer length micelles form an entangled network and therefore undergo stress relaxation slowly.

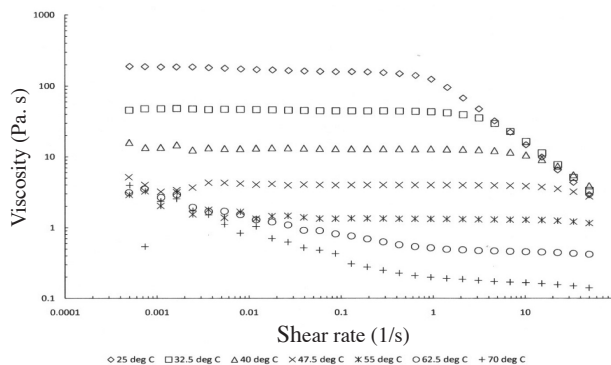


FIGURE 9. Viscosity against shear rate for a 20% solution of potassium 9-bromo stearate in 8% KCl at various temperatures

Another parameter that affects the micellar structure and rheology of wormlike micelles is temperature. Figure 10 presents the effect of temperature on the viscosity for a solution of potassium 9-bromo stearate in 8% KCl. The zero shear viscosity η_0 decreases as the temperature increased till about 60°C where the Newtonian plateau is no longer evident. The living polymer model of worm-like micelles considers two process; reptation or reptile like motion of worm-like micelles along their own contours and reversible scission (Yesulata et al. 2006). The kinetics of the scission is very sensitive to temperature. The living polymer model predicts an exponential decay of the average micellar length and hence the viscosity of wormlike micellar solutions with increasing temperature. The relationship between viscosity and temperature is given by the Arrhenius equation (Dantas et al. 2003).

$$\eta = A \exp \left[\frac{E_a}{kT} \right], \tag{6}$$

where A is a constant characteristic of the material, k the Boltzman constant, T the absolute temperature and E_a the activation energy.

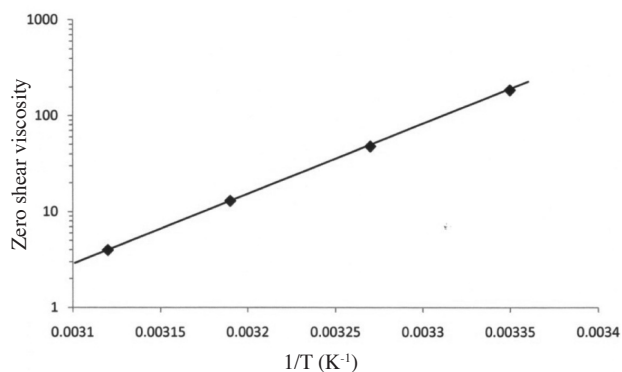


FIGURE 10. Zero shear viscosity (η_0) as a function of $1/T$ (K^{-1}) for 20% solution of potassium 9-bromo stearate in 8% KCl

The activation energy E_a was calculated from the slope of the graph of Figure 11 to be 7.12 kJ mol^{-1} . The decrease in viscosity with increasing temperature may be due to the shortening of the micelles or due to the formation of micellar joints in the networked structure ie micellar branching (Dantas et al. 2003).

Potassium oleate is a viscoelastic surfactant used widely as well bore fluids for fracturing formations. The shear viscosity of potassium oleate under varying shear rate is shown in Figure 11. The zero shear viscosity is varied from 528 Pa.s to 148 Pa s for concentrations of Potassium oleate from 20% to 5% in 8% KCl solutions. This is considerably higher than the brominated oleate. The viscosity at a shear rate of 100 s^{-1} usually referred to in oil field operations varied from 2.1 to 0.48 Pa s as compared to a variation from 1.5 Pa s to 0.16 Pa s for the brominated VES. As such the slope or degree of shear thinning is slightly greater for potassium oleate.

The partition coefficient and results of the gel break test of the potassium 9-bromo stearate and potassium oleate in 8% KCl is shown in Table 1. The partition coefficient for potassium 9-bromo stearate varied from 8% to 30%. This is a significant improvement when compared to potassium oleate which is virtually negligible. This improvement is due to the increased hydrophobicity of the oleate chain with the addition of bromine that increases its affinity for nonpolar solvents. The gel break test also shows an improvement of 2 to 4 hours again due to the increased hydrophobicity. These improvements to the partition coefficient and gel break time shows that the brominated

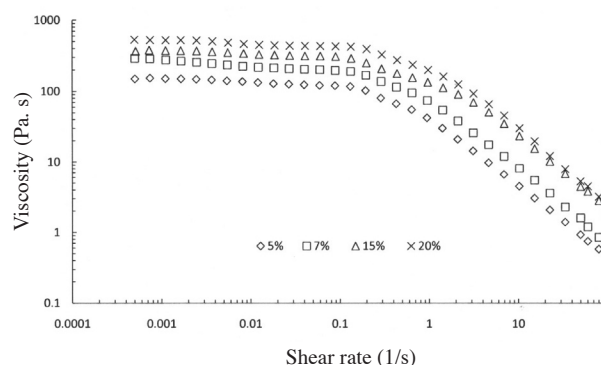


FIGURE 11. Viscosity as a function of shear rate for various concentration of potassium oleate

surfactant performs comparably with the surfactant of the said patent.

CONCLUSION

Bromination of oleic acid at its double bond can be prepared to yields of 98% using hydrogen bromide in acetic acid. The conversions were confirmed by the disappearance of the H-NMR peak at $\delta 5.35$ which represents the double bond at position C9 of the hydrocarbon chain of the oleic acid. The peak was replaced by another at $\delta 4.0$ which is the peak introduced by the bromine moiety. Bromination of the hydrocarbon chain did not cause the loss of viscoelasticity of the surfactant thus retaining the property which is essential for oilfield work where shear introduced by pumping lowers the viscosity for reduced power consumption. When shear is removed in the formation, the solution reverts to its viscous state. Bromination however reduced the zero shear viscosity from 528 Pa.s to 180 Pa.s. However the shear viscosity at 100 s^{-1} reduced from 0.48 Pa s to 0.16 Pa.s for a 5% non brominated and brominated VES solution in 8% KCl, respectively. This is still higher than the 0.1 Pa s deemed as suitable for proppant suspension. As such the brominated oleate is suitable for use as a fracturing fluid at a concentration as little as 5%. Bromination however reduced the gel break time and increased the partition coefficient significantly enabling easier oil field clean up operations which is gaining importance given that drilling will be spread to pristine regions like Alaska. The partition coefficient is comparable to the 11% claimed in the US7196041 patent.

TABLE 1. Partition coefficient and gel break time for various concentrations of bromo 9-stearate and potassium oleate in 8% KCl solution

Concentration %	Partition Coefficient %		Gel Break time hrs	
	9-bromo stearate	oleate	9-bromo stearate	oleate
1%	17.1	0	12	14
5%	13.28	0	82	84
10%	13.49	0	94	98

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