

# Stability and Rheological Behavior of Sulfonated Polyacrylamide/ Laponite Nanoparticles Dispersions in Electrolyte Media

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## ABSTRACT

Due to the importance of nanoparticles stability in industrial applications, in this research, stability of laponite nanoparticles dispersions, containing different concentrations of sodium sulfonated polyacrylamide (SPA), was investigated in electrolyte media for oil reservoirs applications. In this regard, effects of parameters, such as polymer concentration, temperature, and ionic strength, were studied via different methods such as Fourier transform infrared (FTIR) spectroscopy, dynamic light scattering (DLS), and zeta potential. In FTIR spectra of SPA-laponite dispersion, in addition to typical peaks of laponite, there was a weak peak at  $1040\text{ cm}^{-1}$  characterizing SPA polymer. The z-average particle sizes of laponite particles increased after 168 h of aging in the presence of SPA polymer. Zeta potential measurements showed that adsorption of anionic groups of polymer on particle surface during the aging process has led to a decrease in zeta potential value (toward more negative values). It was observed that dispersion stability depended on polymer concentration, ionic strength of aqueous media, and temperature. Visual observations showed that the stability of laponite nanoparticles in electrolyte media was improved by increasing the SPA polymer concentration. However, dispersion stability decreased with increasing temperature and ionic strength. The rheological studies showed that the viscosity curves of SPA-laponite dispersions were located below those of the corresponding pure SPA polymer solutions. In addition, the samples had their shear viscosities increased by increasing the SPA polymer concentration. Consequently, particle settling was hindered by increasing the polymeric matrix viscosity. Furthermore, it was shown that laponite nanoparticles' stability in electrolyte media could be improved by decreasing the power-law coefficient using a power-law equation fitted to the polymer solution of viscosity-shear rate data.

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## 1. Introduction

Laponite is a synthetic disc-shaped

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nanoparticle with a diameter and layer thicknesses of about 25 nm and 1 nm, respectively [1, 2]. The chemical formula for laponite is  $\text{Na}^{+}_{0.7}[(\text{Si}_8 \text{Mg}_{5.5}\text{Li}_{0.3})\text{O}_{20}(\text{OH})_4]_{0.7}$ . In an aqueous medium, the sodium ions dissociate, thereby leading to a net negative charge on the nanoparticles surface [3, 4]. Laponite dispersions thus consist of a collection of negatively charged disks and counterions, which are mainly positively charged sodium ions and solvent molecules [5]. In salt-free water, the laponite particles are stabilized by electrostatic repulsion between the negatively charged faces and form transparent colorless aqueous dispersions. However, aqueous laponite dispersions can exhibit different physical states, such as stable colloidal dispersion or viscoelastic sol, viscoelastic gel, elastic solid, etc. [6-7]. If the interactions were partially screened by the presence of salt, the particles aggregate by forming electrostatic bonds between the positively charged rims and the negatively charged faces. The aggregates grow until they either form a space-filling structure (gel) at higher concentrations or precipitate under the influence of gravity at lower concentrations [8].

Stabilization of nanoparticles is important in several industrial applications including enhanced oil recovery and drilling, paper production, and water treatment [9-12]. The degree to which the nanoparticles are stabilized can be dependent on the balance between attractive and repulsive forces acting between the colloidal particles. Two approaches have been commonly used to improve stability of particles dispersions: electrostatic repulsion and steric stabilization. Electrostatic repulsion is achieved by changing the surface charge, while steric stabilization is typically attained by the

adsorption of long-chain organic molecules (e.g., polymers) on the colloids-particles surfaces via methods, such as chemical grafting or physical adsorption, to prevent them from flocculating [9, 14]. In this regard, several investigations have been carried out on the effect of different polymers on stability behavior of nanoparticle dispersions in different media. For example, Khoshniyat et al. [9] studied surface modification of bentonite nanoclay particles by chemical grafting of different copolymers. Based on the turbidimetry data provided, they showed that stabilities of unmodified and modified bentonite nanoclays in electrolyte solution decreased as the time passed since their preparation so that the nanoparticles can be settled completely in these solutions after 24 h. However, different settlement rates were observed for unmodified and modified bentonite nanoclays with the poly (1-vinyl pyrrolidone-*co*-styrene)/bentonite nano clay particles being settled at the lowest rate. They concluded that surface modification of nanoclays may not guarantee their long-term stability in electrolytes of high salinity. Deng et al. [15] studied the interaction of cationic, anionic, and nonionic polyacrylamides (PAM) with clay minerals. They indicated that anionic PAM had the lowest affinity for the clay surfaces; in other words, it served as a stabilizer for the clay dispersions. Sun et al. [16] demonstrated that grafting of polyvinyl alcohol-*co*- vinyl acetate-*co*-itaconic acid on the zero-valent iron nanoparticles (nZVI) surface can serve as an effective dispersant of nZVI. Mongondry et al. [17] studied the influence of polyethylene oxide (PEO) on the aggregation and gelation of laponite dispersions, and conclude that in the presence of PEO, the steric hindrance of chains adsorbed into the laponite particles inhibits

aggregation. Kurecic et al. [18] investigated the effects of non-ionic polysaccharide-based surfactants to stabilize organically modified clay particles in aqueous dispersions. Their study showed that by using these surfactants, the wettability of clay particles was improved, and consequently, enhancement of their dispersion ability in water-based systems was observed. Gareche et al. [19] studied the influence of low molecular weight PEO on the stability and rheology of bentonite suspensions. They concluded that the PEO presents an affinity for the bentonite particles and gradually coats the clay particles, slowing down their kinetic aggregation. The reduction of particle-particle interaction took place until steady values of the yield stress. The stability of the suspension was enhanced by increasing polymer adsorption. Indeed, the PEO can adopt diverse possible mechanisms: adsorption on the surface of particles, intercalation between the platelets of clay or favoring flocculation by forming bridges between particles. Sakthivelu [20] conducted a sedimentation study of nano kaolinite suspensions in water as well as in aqueous solutions of polyvinyl alcohol (PVA). Their results demonstrated that the adsorption of PVA lowers the viscosity of system resulting in an increase of solid loading for the same viscosity level. Pooja et al. [21] evaluated the effect of xanthan gum on biocompatibility and stability of gold nanoparticles. They found that xanthan gum can act as both size reducing and stabilizing agents in the synthesis of gold nanoparticles. Alkilany et al. [22] studied the synthesis of monodispersed gold nanoparticles with exceptional colloidal stability. Polyethylene glycol-*g*-polyvinyl alcohol (PEG-*g*-PVA) grafted copolymer was employed as a reducing, capping, and

stabilizing agent.

Logie [23] studied the effect of poly(ethylene glycol) (PEG) density on stability of self-assembled nanoparticle micelles based on amphiphilic block copolymers, and concluded that increased PEG density leads to micelles that resist aggregation during lyophilization, allowing for resuspension in aqueous media with narrow distribution. Aguilar-Castillo contrasted the stability behavior of nanoparticles in biologically relevant media [24]. Their results showed the absence of larger aggregates for most of the polymers examined. Soliman et al. [25] applied the phase transfer and polymer coating method to improve the stability of metallic nanoparticles for biological applications. Also, Kumar et al. [26] studied the phase behavior of nanoparticle (silica)-polymer (polyethylene glycol) system. It is observed that at low polymer concentrations, the stability of the nanoparticles in one phase is due to the dominance of electrostatic repulsion over the depletion attraction. On further addition of polymer, depletion attraction increases sufficiently to give rise to nanoparticles clustering in the two-phase system. The re-entrant phase arises due to the reduction in depletion attraction as a result of polymer-polymer repulsion at higher polymer concentrations. Recently, Maurya and Mandal [27] demonstrated that polyacrylamide gets adsorbed onto the surface of silica due to hydrogen bonding, and the silica particle acts as physical cross-linker between polymeric chains, resulting in improvement of viscosity.

As the examples above show, even though a considerable deal of research has been undertaken on the use of different polymers for improving stability behavior of

nanoparticles in various conditions, to the best of our knowledge, there is no report concerning the stability and rheological behavior of sulfonated polyacrylamide (SPA)/laponite nanoparticles dispersions, especially in concentrated electrolyte media. Accordingly, most of the information reported in the present paper (e.g., FTIR spectra, dynamic light scattering and zeta potential tests results conducted on SPA/laponite dispersions, and finally evaluation of their rheological and stability behavior in electrolytes of high ionic strengths) is new and has not been reported elsewhere. It was found that the lower the power-law coefficient of the suspending fluid, the more stable the SPA/nanoparticles dispersion. In this regard, a series of SPA-laponite dispersions were prepared and aged in distilled water for proceeding to adsorption process, and then, their stabilities in electrolyte media were studied considering the effect polymer concentration, temperature and ionic strength as well as viscosity of suspending media.

## 2. Experimental

### 2.1. Materials

Tetra-sodium phosphate modified laponite was provided by Rockwood Co. (United Kingdom) under the trade name of Laponite XLS. Sulfonated polyacrylamide (SPA) with

an average molecular weight of  $8 \times 10^6$ , sulfonation degree of 25 % and water content of less than 10 wt % was provided by SNF Co. SPA is a copolymer of acrylamide and sodium salt of acrylamide propyl sulfonic acid (Fig. 1).

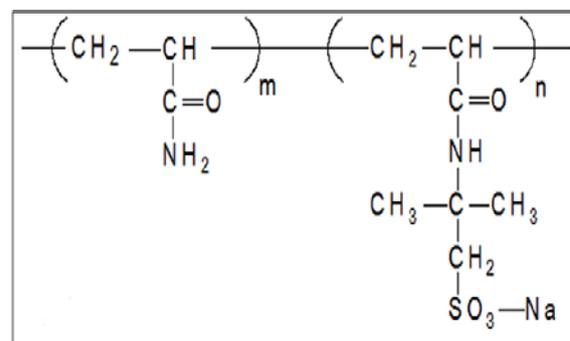


Figure 1. SPA chemical structure [28].

### 2.2. Sample preparation

For evaluating the effect of SPA polymer on laponite dispersions stability in electrolyte media, at the first step, separately-prepared SPA solution and laponite dispersion were mixed (using a magnetic stirrer for 24 h) and stored for 168 h at room temperature to proceed to the adsorption process of SPA polymer on laponite nanoparticles surfaces. Then, this dispersion was added to electrolyte solutions (with the salinity given in Table 1) containing different concentrations of SPA while stirring at room temperature for 24 h. The prepared samples were kept in the oven at desired temperature.

Table 1

Details of the used electrolytes.

Electrolyte type	Salt concentrations		Ionic strength (M)
	NaCl (M)	CaCl <sub>2</sub> (M)	
Electrolyte 1	2	0.2	2.6
Electrolyte 2	1	0.1	1.3
Electrolyte 3	0.5	0.05	0.65

Dispersions stability after 24 h thermal aging was recorded visually according to the definitions described in Table 2.

**Table 2**

Definitions used for characterization of dispersions stability by visual observations.

Statutes of stability	Description	Corresponding image
Stable	Homogeneous dispersion or Homogeneous viscous gel	
Semi-stable	Slightly phase separated or Slightly precipitated	
Unstable	Voluminous sedimentation or Coagulation	

The rheological behavior of the prepared samples was monitored using a concentric cylinder Physica MCR 501 rheometer (Anton Paar, Austria) equipped with a Peltier device for temperature control. To carry out the rheological tests, the system was first heated to desired temperature, and then the samples were introduced into the heated cylinder and subjected to shear strain. The steady flow measurements were carried out in order to determine the shear viscosity. Rheoplus software, associated with the instrument, was

utilized for recording and analyzing flow behavior data.

The z-average particle size of laponite mineral in the prepared dispersions was determined using dynamic light scattering (DLS) method at wavelength of 532 nm by a Malvern NANO-ZS Zetasizer (Malvern Instruments, England). The scattering angle was fixed to be 173°. The zeta potential of the laponite particles was measured by NANO-ZS Zetasizer (Malvern-England). Required samples were prepared by dispersing of aged

nanoparticles-SPA specimens in distilled water at a concentration of 0.1 wt %.

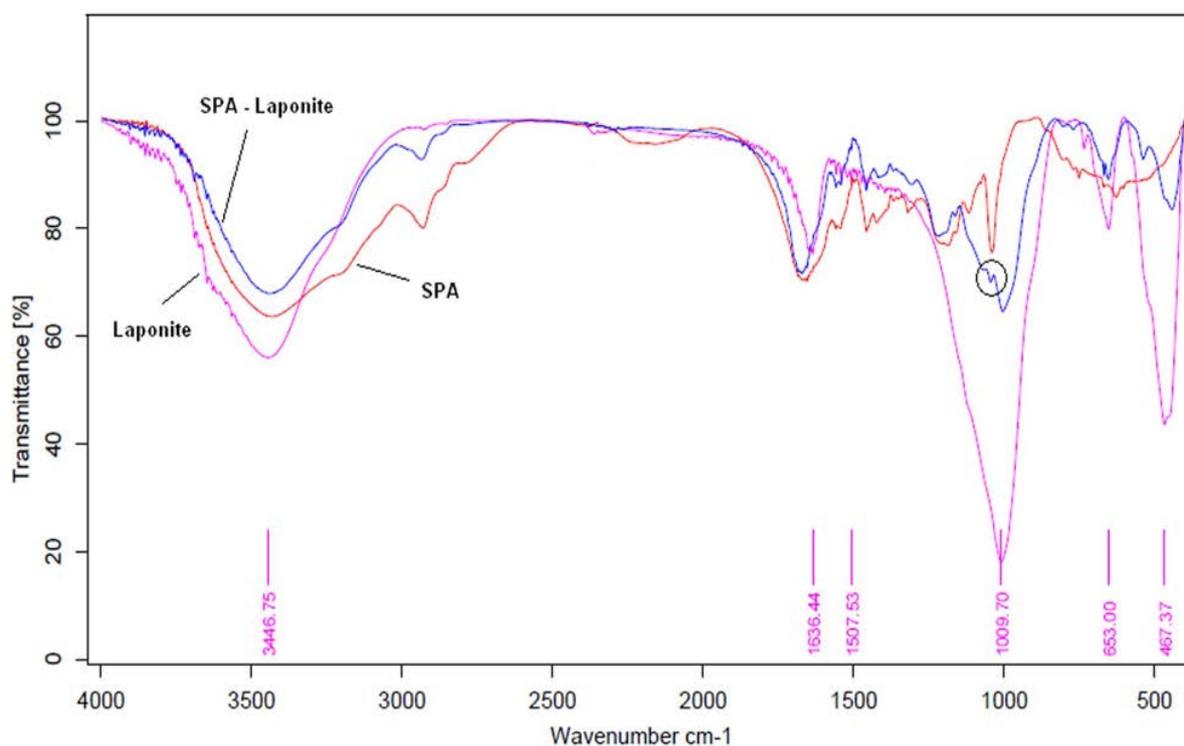
Fourier transform infrared spectra (FTIR) were taken with a Bruker Vertex V70 instrument (Germany), taking samples in KBr pellets.

### 3. Results and discussion

#### 3.1. Characterizations

Laponite dispersions are stable in a wide range of concentrations in distilled water; however, their stability decreases with increasing salt concentration. The main aim of this research is to evaluate the effect of SPA polymer on stabilization of these nanoparticles in electrolyte media. FTIR spectra of SPA polymer, laponite, and SPA-

laponite dispersions (aged in distilled water for 168 h) are presented in Figure 2, where there are three distinct peaks for laponite nanoparticle: Si-O band peak at  $1009\text{ cm}^{-1}$ , O-H band peak at  $1640\text{ cm}^{-1}$  and O-H stretching vibration peak at  $3444\text{ cm}^{-1}$ . There are also three distinct peaks for SPA polymer:  $\text{NH}_2$  group stretching vibration peak at  $3433\text{ cm}^{-1}$ , C=O stretching vibration peak at  $1659\text{ cm}^{-1}$  and C-N stretching vibration peak at  $1040\text{ cm}^{-1}$ . However, in FTIR spectra of SPA-laponite dispersion, in addition to the typical peaks of laponite, there is a weak peak at  $1040\text{ cm}^{-1}$  which is characteristic of SPA polymer and may consider physical adsorption of SPA polymer onto laponite surface after 168 h of aging.



**Figure 2.** FTIR spectra for SPA, Laponite and SPA-Laponite dispersions.

Dynamic light scattering (DLS) tests were conducted on laponite and SPA-laponite dispersions, so as to measure z-average particle sizes. Z-average size is the intensity-weighted mean size derived from cumulate analysis. Results are presented in Table 3. We

can see that the z-average size for pure laponite dispersion in distilled water has been about 47 nm. However, it has significantly increased to 519 nm following the 168 h-aging in the presence of SPA polymer. This behavior may be related to the adsorption of

polymeric chains on laponite surface which might contribute to the increased particle sizes. It is believed that the poly acryl amide chains are bound to the surface of the nano clay particles due to hydrogen bonding

between the oxygen atoms of nanoclay and the amide protons of the acryl amide and also due to complex formation between the metal ions on the nanoclay surface and the carbonyl oxygen on the acryl amide [29].

**Table 3**

DLS and zeta potential tests' results conducted on laponite and SPA-laponite dispersions aged in distilled water for 168 h.

Sample	Z-average particle size (nm)	Zeta potential (mV)
Laponite dispersion (0.2 wt %)	47.3	-1.02
SPA- Laponite dispersion (0.2 wt % - 0.2 wt %)	519	-119

Effect of SPA polymer adsorption on the surface charge of laponite particles was monitored through zeta potential measurements (Table 3). In recent years, zeta potential method has been successfully used to measure polymer-particle interactions in polymer-clay hybrids [28, 30]. Zeta potential measures the surface charge at solid/liquid interface in a solid/liquid system, by which one can explain the mechanism of components interaction at the interface. For these systems, the zeta potential value is strongly dependent on the composition of the aqueous phase. Surface active groups of the aqueous phase play an important role in adsorption processes at the interface and hence on the corresponding zeta potential value [28]. From Table 3, it can be seen that the aging process in the presence of SPA polymer leads to a decrease in zeta potential values from -1.02 mV (for pure laponite dispersion) to -119 mV (for 168 h-aged particles). It seems that the adsorption of anionic groups of polymer on the particles surfaces leads to such a decrease in zeta potential value (more negative). The more negative the zeta potential value is, the more electrostatic repulsion between particles

exists, enhancing their stability in electrolyte media.

### **3.2. Stability behavior**

FTIR, DLS and zeta potential results confirmed that the aging process in distilled water led to the adsorption of SPA polymer on laponite surface. Therefore, these aged particles were used in future steps for stability tests in electrolyte media. For this purpose, as mentioned earlier, aforementioned particles were mixed with electrolyte solutions containing different concentrations of SPA polymer, and then placed into an oven at a desired temperature. Samples' stabilities after 24 h were recorded visually according to the definitions described in Table 2, with the results presented in Table 4.

The dispersions' stabilities were seen to be dependent on polymer concentration, ionic strength of aqueous media, and temperature. As shown in Table 4, an increase in SPA polymer concentration improved the stability of dispersions in electrolyte media. As mentioned earlier, some of the SPA chains were adsorbed onto the laponite nanoparticles surfaces via hydrogen bonding between oxygen atoms of nanoclay and protons of amide group on polyacrylamide [29]. The

presence of sulfonated groups ( $-\text{SO}_3^{2-}$ ) of SPA polymer on laponite surfaces lowerd the screening effects of cations, such as  $\text{Ca}^{2+}$ , on anionic surfaces of laponite, thereby improving their stability. However, the electrostatic attraction between negatively charged laponite surfaces and electrolyte cations increased by increasing ionic strength of electrolytes, leading to lower stability of

dispersions in electrolytes of higher ionic strength (Electrolyte 1). In fact, by increasing the ionic strength of an electrolyte medium, screening effects of  $\text{Na}^+$  and  $\text{Ca}^{2+}$  cations on anionic groups of SPA polymer become more evident, resulting in less chain expansion, and consequently lower dispersion stability at higher ionic strengths.

**Table 4**

Stability behavior of SPA-Laponite dispersions in electrolyte media after 24 h aging at different temperatures.

SPA-Laponite concentration (wt % - wt %)	T= 25 °C			T= 75 °C		
	Electrolyte1 (I=2.6 M)	Electrolyte2 (I=1.3 M)	Electrolyte3 (I=0.65 M)	Electrolyte1 (I=2.6 M)	Electrolyte2 (I=1.3 M)	Electrolyte3 (I=0.65 M)
0 – 0.1	unstable	unstable	unstable	unstable	unstable	unstable
0.2 – 0.1	unstable	stable	stable	unstable	unstable	unstable
0.5 – 0.1	stable	stable	stable	unstable	semi-stable	stable
1 – 0.1	stable	stable	stable	semi-stable	semi-stable	stable

Effect of temperature on SPA-laponite dispersion' stability is shown in Table 4, where it can be seen that an increase in temperature lowers dispersion' stability in all electrolyte media. It is surmised that a rise of temperature increases the chain mobility and coiling probability, leading the polymeric chains to shrink on laponite surface [31-33]. In other words, temperature plays a kinetic role in nanoparticle dispersion' stability. Indeed, an increase in average kinetic energy with temperature gives rise to particle collisions, finally resulting in aggregation [35]. In addition, as adsorption process is an exothermic phenomenon [34] where lower polymeric chains will be adsorbed at higher temperatures, weakening the role of polymer in stabilization of laponite particles at higher temperatures. However, one can see high stability of two cases in Electrolyte 3 even at

temperature as high as 75 °C; this issue is discussed in the next section.

### 3.3. Rheological behavior

Evaluating the viscosity of polymeric systems is important for understanding their behaviors under different conditions. Viscosity of a polymer dispersion depends on many parameters such as polymer and particle concentrations, polymers' types and interactions, solvent quality, and temperature. According to Stoke's law, the terminal settling velocity ( $V_s$ ) for a particle of radius  $R$  is [36]:

$$V_s = (2gR^2(\rho_p - \rho_f))/9\eta \quad (1)$$

where  $\rho_p$  and  $\rho_f$  are densities of particles and solution, respectively,  $\eta$  is the solution viscosity, and  $g$  is the gravitational acceleration.

In order to attain a deeper insight into the role of SPA polymer in laponite nanoparticles stabilization in electrolyte media, the steady shear rheological behaviors of SPA solutions and SPA-laponite dispersions were evaluated, with results presented in Figures 3 and 4. One can see that at both temperatures, the samples had their viscosities continuously decreased with increasing shear rate; the results show the non-Newtonian behavior and the occurrence of shear thinning effect. Probably resulted from disentanglement process of polymeric chains due to shearing, this behavior is typical of polymeric systems. In addition, for SPA-laponite dispersions, this behavior may arise from the disintegration of

particles in the shear fields and orientation of particles along with flow direction [31]. Figures 3 and 4 further show that the corresponding viscosity curves of SPA-laponite dispersions are located lower than those of the corresponding pure SPA polymer solutions. One may surmise that this behavior is due to the adsorption of polymeric chains onto laponite surfaces due to hydrogen bonding and formation of complexes between them [37, 35]. In addition, shear viscosity of the samples is seen to increase with increasing SPA polymer concentration due to the increase in chain entanglement of the polymeric systems.

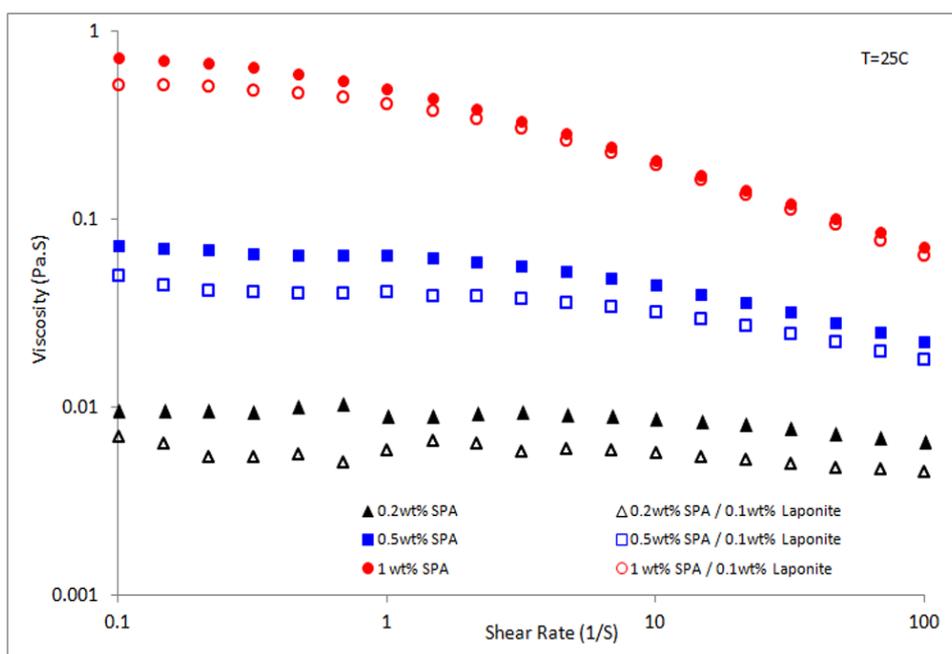


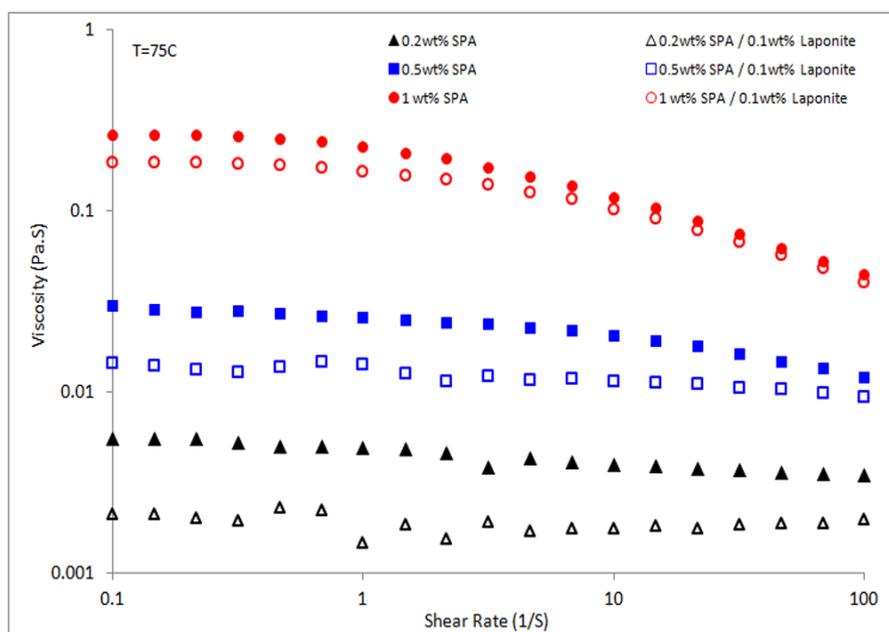
Figure 2. Viscosity-shear rate curves of SPA solutions and SPA-laponite dispersions at 25 °C in electrolyte media (Ionic strength=2.6M).

According to Stoke's law, viscosity of base-fluid plays an important role in precipitating stability of nanoparticles so that by increasing continuous phase viscosity, nanoparticles are less likely to accumulate, allowing for retarding the nanoparticles precipitation. In fact, viscosity represents stiffness of the

continuous phase and can be regarded as a macroscopic measure of the force opposing the settling. This is consistent with the stability results presented earlier (Table 4), indicating that the dispersion stability tends to be improved by increasing SPA polymer concentration. In addition, the decrease in

dispersion stability by increasing the temperature can be described by considering the effect of temperature on SPA polymer viscosity (Figure 4). As mentioned earlier, this behavior is a result of enhanced mobility

and coiling probability of polymeric chains at higher temperatures. Therefore, one may suggest that, at higher temperatures, the polymeric matrix does not have enough strength to oppose particles settling.



**Figure 3.** Viscosity-shear rate curves of SPA solutions and SPA-laponite dispersions at 75 °C in electrolyte media (Ionic strength=2.6M).

Furthermore, as mentioned earlier, at higher temperatures, the polymeric chains shrink on the laponite surface. Consequently, the prepared dispersions herein had better stability at lower temperatures.

SPA polymer solution of viscosity-shear rate data at different temperatures was fitted to a power-law equation defined as  $\eta = K(\dot{\gamma})^{n-1}$ , where  $\eta$ ,  $\dot{\gamma}$  and  $K$  are viscosity, shear rate, and consistency index, respectively. In this equation,  $n$  is power-law coefficient (or flow index) which is a measure of non-Newtonian-ness. The flow index is 1 for a Newtonian fluid, between 0 and 1 for a shear-thinning fluid, and greater than 1 for a shear thickening fluid.

As can be seen from Table 5, the value of power-law coefficient decreases with either increasing SPA polymer concentration or

decreasing the temperature from 75 °C to 25 °C. Regarding the stability results, one may surmise that the more shear thinning behavior (the lower the power-law coefficient) exhibited by of suspending fluid, the more stable would be SPA/nanoparticles dispersion.

**Table 5**

Power-law coefficient ( $n$ ) for SPA polymer at different temperatures in electrolyte media (Ionic strength=2.6M).

SPA concentration (wt %)	n	
	T= 25 °C	T= 75 °C
0.2	0.9881	0.9946
0.5	0.846	0.8818
1	0.7431	0.8259

#### 4. Conclusions

In the current study, effect of SPA polymer on stability of laponite nanoparticles in concentrated electrolyte media was investigated through evaluations such as FTIR spectrometry, DLS, zeta potential, and rheological tests. Accordingly, the following main conclusions can be drawn: FTIR, DLS, and zeta potential measurements confirmed the adsorption of SPA polymer onto laponite surface. The z-average size for laponite dispersion increased from 47 nm (in distilled water) to about 519 nm (after 168 h of aging in presence of SPA polymer) due to the adsorption of polymeric chains on laponite surface. Stability evaluations indicated the dependence of the dispersions stability on temperature, polymer concentration and ionic strength of aqueous media. By increasing SPA polymer concentration, the stability of dispersions in electrolyte media was improved. It is believed that the presence of sulfonated groups ( $-\text{SO}_3^{2-}$ ) on SPA polymer lowers the screening effect of cations, such as  $\text{Ca}^{2+}$ , on anionic surfaces of laponite, thereby improving their stability. However, dispersions' stability decreased with increasing temperature and ionic strength. Viscosity of suspending medium had a significant effect on stability of laponite nanoparticles in electrolyte media so that the particle settling was hindered with polymeric matrixes of higher viscosities. The shear viscosity of SPA-laponite dispersions increased with increasing SPA polymer concentration. By fitting the polymer solution of viscosity-shear rate data to a power-law equation, it was found that the lower the power-law coefficient of the suspending fluid, the more stable the SPA/nanoparticles dispersion. The findings of this research can

be used to promote long-term stability of nanoparticles in electrolyte media for applications in oil reservoirs. However, further investigations are required to address the remaining questions in this area.

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