Application of environmentally friendly amphoteric polyacrylamide hydrophobically modified with plant oil as additive in water-based drilling fluid

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Abstract. Hydrophobic associating polymers show great potential in formulation of high-performance drilling fluids, due to their hydrophobic associative ability in high-temperature and high-salt conditions. Herein, a novel amphoteric polyacrylamide of poly(acrylamide/2-acrylamido-2-methyl-1-propanesulfonic acid/methylacrylethyl trimethylammonium chloride) hydrophobically modified with epoxidized soybean oil denoted as PAADE was prepared and applied in water-based drilling fluid, and one without hydrophobic modification (PAAD) was also used for comparison. Compared with PAAD, PAADE displayed characteristic association behavior with a critical association concentration (CAC) of 0.3 w/v%, and a salt thickening phenomenon over 3 w/v% NaCl concentration. The impacts of PAADE and PAAD on rheological and filtration properties of bentonite-based drilling fluid (BTDF) were evaluated under different salinity. The fitted Herschel-Bulkley rheological parameters indicated that BTDF containing PAADE showed stronger yield stress, easier flowability and better salt resistance at a reasonable concentration below CAC. Meanwhile, PAADE largely reduced the fluid loss of BTDF before and after thermal aging at 150°C, showing high temperature tolerance. The addition of NaCl promoted larger reduction of filtration. The superior properties of PAADE in BTDF were revealed to be the competitive results of salt-induced hydrophobic association and inter-particles configuration transition. Moreover, the incorporation of epoxidized soybean oil improved the biodegradability of PAADE.

1. Introduction
Drilling fluid plays an essential role in drilling operations, and the water-based drilling fluids (WBDFs) are most widely used worldwide accounting for near 80% of drilling operations [1]. However, the performance deterioration of WBDFs at harsh conditions such as high temperature and high salinity...
environments remains a big challenge [2]. Therefore, it is crucial to develop high-performance drilling fluid materials to maintain the properties of WBDFs at high temperature and high salinity conditions.

Hydrophobically associating polyelectrolytes (HAPs) are an interesting class of water-soluble polymers containing a small proportion of hydrophobic groups (1-3 mol%) [3, 4]. Due to the hydrophobic interactions, HAPs have the tendency to form molecular aggregates and physical cross-linked network depending on the polymer concentration where intra-molecular associations transform to inter-molecular association at a critical value [5]. The hydrophobic associations can be enhanced with the increase of temperature and salinity [6], as an entropy-favored process. The self-assembly of hydrophobic groups brings HAPs with the characteristics of better viscosifying ability and greater temperature and salinity resistance than conventional polymers.

The properties of HAPs are dominated by the structures of polyelectrolytes, type of hydrophobic comonomers and the hydrophobic length. HAPs can be employed as high-performance additives to tailor WBDFs properties by tuning the polyelectrolyte backbone structures and hydrophobic comonomers, as well as the length of hydrophobic blocks formed in micellar copolymerization [3]. However, commonly used hydrophobic monomers are petroleum-based synthetic materials, which are less environmentally friendly to protect the environment [7]. Plant oils as highly hydrophobic, renewable and biodegradable biomass materials seem to be promising alternatives to conventional hydrophobic monomers.

Plant oils are triacylglycerols of fatty acids with different number of carbon atoms (8 to 24) and unsaturated double bonds (0 to 7), and possible natural functional groups (hydroxyl, ester and epoxy groups). The double bonds and functional groups offer reactive sites for direct chemical modification or converting to polymerizable monomers [8]. More recently, new strategies for the transformation of plant oils into polymerizable vinyl monomers were proposed [9,10], and the resulting monomers show high activity in free radical polymerization.

Therefore, inspired by above-mentioned works, herein a plant oil-based methacrylate monomer (ESO-MA) was prepared from epoxidized soybean oil (ESO) with long aliphatic chains and functional epoxy groups. The amphoteric polyelectrolytes of acrylamide (AM), 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS), methylacrylethyl trimethylammonium chloride (DMC) with and without hydrophobic modification with ESO-MA were prepared and applied in water-based drilling fluid. A comparative study of association behavior tests in aqueous solution, rheological and filtration performance tests in bentonite-based drilling fluid (BTDF) was carried out to reveal the structure-property relationships and the contribution of hydrophobic association in harsh conditions. Furthermore, the biodegradability of PAADE and PAAD was measured.

2. Materials and methods

2.1 Materials

PAADE and PAAD used in this work were prepared in our laboratory according to previous work [11]. The overall synthetic strategy of PAADE and PAAD are shown in Fig.1. In the micellar copolymerization process, the plant oil-based monomer (ESO-MA) was solubilized in micelles formed by cetyltrimethyl ammonium bromide methanol (CTAB), which showed a larger solubilization ability for the large hydrophobes than sodium dodecyl sulfate (SDS). As the turbidity of surfactant solutions containing different concentrations of ESO-MA in Fig.2, the solubilization limit for SDS was estimated to be around 0.4 w/v%, while that for CTAB reached above 1.0 w/v%. Additionally, the number of hydrophobic monomers per micelle ($N_h$) could be estimated to be 20.15 using Eq. (1):

$$N_h = \frac{[M]}{(S)\cdot CMC}/N_{agg}$$

in which $[M]$ is the concentration of ESO-MA, $N_{agg}$ is the aggregation number of CTAB, $[S]$ is the surfactant concentration, and $CMC$ is the critical micelle concentration of CTAB.
Sodium chloride (NaCl, AR) was supplied by Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Sodium bentonite (Na-Bent) was supported by CNPC Exploration and Development Research Institute. Deionized water was utilized in all experiments.

Fig. 1. Synthetic routes of PAAD and PAADE

2.2 Association behavior

2.2.1 Rheological test
The steady shear rheology of polymer aqueous solution was performed on a Brookfield rotation viscosimeter (Brookfield DV2T, USA) in the shear rate range of 0-200 s⁻¹.

2.2.2 Hydrodynamic diameter
Hydrodynamic diameters of polymer aqueous solution were measured through dynamic light scattering (DLS) on a nanoparticle size analyzer (Brookhaven, USA).

2.2.3 SEM
Crosslinking network and morphological characteristics of PAAD and PAADE were observed on an S-4800 field emission scanning electron microscope (Hitachi, Japan).

2.3 Drilling fluid performance test
Bentonite-based drilling fluid (BTDF) was prepared according to the reported method [11]. PAADE and PAAD were added into the cured BTDF at a certain concentration under vigorous stirring for 30 min, to obtain BTDF/PAADE and BTDF/PAAD dispersions. The impact of salinity was also evaluated by adding various amount of NaCl.

The rheological properties of prepared dispersions were carried out on a Fann35A six-speed rotary viscometer at a temperature of 25°C and shear rates of 1022 s⁻¹, 511 s⁻¹, 340.6 s⁻¹, 170.3 s⁻¹, 10.22 s⁻¹ and...
5.11 s\(^{-1}\). The relationship between shear stress and shear rate was fitted by Herschel–Bulkley (H-B) model [12]. The constitutive equation is as follows:

\[
\tau = \tau_\gamma + K\gamma^n
\]

(2)

where \(\tau\) is the shear stress (Pa), \(\gamma\) is the shear rate (s\(^{-1}\)), \(\tau_\gamma\) is the H-B yield stress, \(K\) is the consistency coefficient (Pa·s\(^n\)) and \(n\) is the flow behavior index (dimensionless).

The filtration properties of prepared dispersions were conducted under both normal temperature/medium pressure and high temperature/high pressure (HTHP) conditions according to the American Petroleum Institute (API) procedure.

To understand the underlying mechanism of clay-polymer interactions, particle size distribution (PSD) of different dispersions were tested.

2.4 Biodegradability test

The biodegradability of PAADE and PAAD was determined by biodegradability index defined as the ratio of five-day biochemical oxygen demand (BOD\(_5\)) and chemical oxygen demand (COD) [13]. The test procedures were in accordance with Chinese industry standard SY/T 3788.

3. Results and discussion

3.1 Association behavior

3.1.1 Rheological properties of polymer solutions

The steady rheological curves of PAAD and PAADE aqueous solutions at different polymer concentrations are shown in Fig. 3. Both PAAD and PAADE solutions exhibited shear thinning behaviors with low viscosity at high shear rates while high viscosity at low shear rates, as a result of shear-induced destruction of polymer network. In comparison to PAAD, in the whole concentration range PAADE solution possessed much higher viscosity at low shear rate range (0.1-10 s\(^{-1}\)) while similar viscosity at high shear rate, displaying more marked shear thinning behavior. The particular hydrophobic groups along PAADE backbones contributed to the enhanced polymer network to increase viscosity, while the physical links were sensitive to the shear rate. By comparing the apparent viscosity at 0.1 s\(^{-1}\) as a function of polymer concentration, an inflection could be observed for PAADE, while the apparent viscosity of PAAD was almost linear with the concentration. The increase of hydrophobic groups along with increased concentration resulted in the change from intra-molecular to inter-molecular association [5], thus larger supra-molecular aggregates formed and the viscosity increased abruptly at a certain concentration defined as the critical association concentration (CAC). Accordingly, the CAC for PAADE was approximately 0.3 w/v%.

Fig. 3. Steady rheological curves of (a) PAAD and (b) PAADE aqueous solutions at different polymer concentration (\(T = 25^\circ\text{C}\))
To illustrate the impact of salt on the hydrophobic association behavior, the rheological properties of PAAD and PAADE solutions at concentration of 0.3 w/v% under different salinity were investigated. As shown in Fig.4, the viscosity of PAAD solution continuously decreased as NaCl concentration increased, with a large reduction of apparent viscosity at 0.1 s\(^{-1}\) from 88 mPa\(\cdot\)s to 33.6 mPa\(\cdot\)s and 10.5 mPa\(\cdot\)s at 3 w/v% and 5 w/v% NaCl concentration, respectively. The viscosity of PAAD mainly came from the spatial network formed by electrostatic interactions between amphoteric structure, which was vulnerable to the salt ions due to electrostatic screening effect. However, the viscosity of PAADE with both amphiphilic and amphoteric structures was the result of competition between salt-weakened electrostatic interaction and salt-enhanced hydrophobic association. At low salinity (0-3 w/v% NaCl), the electrostatic interactions contributed more to the viscosity which showed a large decrease as NaCl increased. Further addition of NaCl induced the formation of inter-molecular association, the viscosity conversely increased, showing a salt thickening behavior at the NaCl concentration higher than 3 w/v%.

3.1.2 Hydrodynamic diameter

The DLS describing hydrodynamic diameters of PAAD and PAADE solution under different salinity were performed and the results are shows in Fig.5. The \(D_h\) of PAADE solution was about 1167.25 nm, much larger than that of PAAD solution (359.22 nm). The hydrophobic association was contributed to the larger hydrodynamic volume which was responsible for the higher viscosity. After adding NaCl, the \(D_h\) of PAAD solution continuously decreased to about 189.08 nm at 10 w/v% NaCl. However, for PAADE solution, the \(D_h\) decreased to 796.1 nm first when NaCl concentration increased from 0 to 3 w/v%, while then increased to 909.69 nm at 10 w/v% NaCl, which corresponded well to the rheological results in which the salt thickening behavior appeared upon 3 w/v% NaCl content.
3.1.3 SEM
The morphological characteristics of PAAD and PAADE are shown in Fig.6. PAAD possessed overall silky-smooth surface while the lateral surface showed a crosslinked network structure. However, PAADE with hydrophobic fatty side chains showed a rough surface with many slender aggregations and strengthened network structure, and some spherical aggregates with a size centered at approximately 0.50 μm could be observed on the surface. Besides electrostatic and hydrogen bonding interactions, the extra hydrophobic association of pendant long carbon chains favored inter-molecular interactions, resulting in the improved crosslinking density and thicker aggregation network. Meanwhile, the polymer chains spontaneously formed coil-globule transition which triggered by the intrinsic molecular structures with hydrophobic fragments, thereby the nano-scale aggregates assembled.

Fig. 6. SEM images of (a) PAAD and (b) PAADE

3.2 Drilling fluid performance

3.2.1 Rheological properties
The effects of PAAD and PAADE on the rheological property of bentonite-based drilling fluid (BTDF) are shown in Fig.7. The rheological curves were nonlinearly fitted by H-B model, which provided high correlation coefficients ($R^2$) between 0.97 and 0.99. All dispersions exhibited shear thinning behaviors ($n < 1$). The consistency coefficient ($K$) and yield stress ($τ_y$) of BTDF were improved by PAAD and PAADE. At the concentration of 0.2 w/v%, the $τ_y$ values for BTDF/PAAD and BTDF/PAADE were 5.84 Pa and 11.33 Pa, respectively; which further increased to 31.05 Pa and 35.47 Pa, respectively, at the concentration of 0.5 w/v%. Obviously, PAADE showed better yield stress-increasing effect on BTDF while similar impact on the consistency coefficient compared with PAAD, which indicated the stronger structural strength of spatial network formed between PAADE and clay, and similar flowability of BTDF/PAAD and BTDF/PAADE dispersions after applied shear stress exceeding the yield stress [14]. Noteworthily, the higher yield stress provided BTDF with stronger capacity for carrying drill cuttings under dynamic condition, while accompanied by larger shear stress (namely the larger pump pressure) required to restart fluid circulation at static condition [15]. Therefore, the yield stress of BTDF should be controlled in a reasonable range to ensure easy circulation of drilling fluid [16], and the appropriate concentration was 0.2 w/v% for PAADE, which was below the CAC of PAADE aqueous solution.

The invasion of electrolytes in BTDF can affect the thickness of electric double layer (EDL) of clay particles and inter-particle association types (edge-face, edge-edge or face-face) [17]. Fig.7b, c, d show the rheological curves of BTDF, BTDF/PAAD and BTDF/PAADE at different NaCl concentrations, respectively. The changes of rheological properties for BTDF could be divided into two regions dependent on the salt dosages, in which the $K$ and $τ_y$ values increased with added NaCl up to a maximum
at 3 w/v% NaCl, while further NaCl addition caused a decrease. This was attributed to the salt-induced inter-particle association transition. The addition of salt compressed the diffused EDL thickness and lowered the EDL repulsion force between clay particles, favoring the formation of flocculated structure, consequently the $K$ and $\tau_y$ increased. While excessive salt ions diminished EDL repulsion force and the attractive interaction dominated the face-face coagulation between particles leading to sedimentation at high salt content.

As for the bentonite/polymer dispersions, the rheological properties were improved at all tested salinity. As NaCl concentration increased from 0 to 3 w/v%, the $\tau_y$ of BTDF/PAADE showed a large increase to 17.86 Pa while that of BTDF/PAAD was almost unchanged. On the one hand, the NaCl promoted the flocculation of bentonite, leading to the increase of $\tau_y$; on the other hand, the salt ions weakened the electrostatic bonding between clay and polymers, leading to the de-flocculation of bentonite/polymer network. The two opposite effects caused the approximately unchanged $\tau_y$ in the NaCl concentration range between 0-3 w/v%, while the $K$ showed a large increase. When NaCl concentration increased over 3 w/v%, the salt caused the sedimentation of clay and the collapse of PAAD chains, resulting in the continuous decline of $K$ and $\tau_y$. However, PAADE showed better salt-resistance ability as the $\tau_y$ was higher at all salinity. A maximum $\tau_y$ was also observed at 3 w/v% NaCl addition, which was attributed to the flocculation of bentonite and the salt-enhanced hydrophobic interaction. The existence of hydrophobic groups outside of the clay surface favored the interaction with neighboring particles or flocs, providing steric stabilization effect on bentonite to withstand the sedimentation and maintained a certain $\tau_y$ at high NaCl concentration. Besides, BTDF/PAADE had a higher $\tau_y$ but a lower $K$ value compared with BTDF/PAAD, which meant that BTDF/PAADE dispersion would flow more easily due to the shear-sensitive particle aggregations bridged by hydrophobic interactions. However, an abrupt increase of $K$ occurred at 10 w/v% NaCl. It is reasonably proposed that the high salinity caused the desorption of PAADE, and the individual polymer chains with effective content below CAC were prone to form intra-molecular association, resulting in the generation of spherical polymer aggregates. The deformable polymer aggregates might resist certain shear stress, thereby increasing the consistency coefficient.
3.2.2 Filtration properties

The effects of PAAD and PAADE at 0.2 w/v% concentration on the filtration properties of BTDF under different salinity before and after thermal aging are shown in Fig. 8. Before thermal aging, PAAD and PAADE both reduced the API filtration volume. The addition of NaCl at 10 w/v% caused the loss volume of BTDF and BTDF/PAAD increased to 81 mL and 83 mL, respectively. This uncontrolled fluid loss was attributed to the failure of colloid stability [18]. As expected, PAADE showed better capacity of reducing fluid loss especially at high NaCl content (10 w/v%) where PAAD almost lost the ability to reduce filtration, owing to the presence of hydrophobic blocks which could improve filter cake strength through crosslinked hydrophobic association. After thermal aging, the filtration volume of BTDF/PAAD showed little change, while that of BTDF and BTDF/PAADE exhibited contrary changes. The high temperature caused the flocculation and dehydration of bentonite particles, thus increasing the fluid loss of BTDF. On the contrary, the high temperature caused the desorption of PAADE and promoted the intra-molecular hydrophobic association to form nano-scale polymer aggregates, which could plug the pores and improve the quality of filter cake, thereby decreasing the fluid loss of BTDF/PAADE. Additionally, the higher the NaCl addition, the larger gaps of fluid loss between dispersions before and after aging.

To simulate the filtration behaviors under HTHP, the HTHP filtration loss of these three aged dispersions were tested under 150°C temperature and 3.5 MPa pressure difference. As shown in Fig. 8b, it is interesting to find that PAADE showed remarkable ability to reduce HTHP fluid loss, while PAAD could not. The real-time high temperature condition caused irreversible flocculation of clay particles and desorption and curl of PAAD chains, leading to the high HTHP fluid loss of both BTDF and BTDF/PAAD. The addition of NaCl deteriorated the HTHP filtration loss of BTDF as well. However, the BTDF/PAADE with NaCl showed larger HTHP fluid loss reduction compared with that without
NaCl. Specifically, the reduction rates were 44%, 51%, 52%, and 49% for BTDF/PAADE containing 0 w/v%, 3 w/v%, 5 w/v% and 10 w/v% NaCl, respectively. This phenomenon was also attributed to that the high temperature and high salt enhanced hydrophobic association and formation of polymer aggregates, which could act as deformable particles to fill small gaps and improve the seal of filter cake.

![Image](image.png)

Fig. 8. Effects of PAAD and PAADE at 0.2 w/v% concentration on the (a) API filtration performance and (b) HTHP filtration performance of BTDF under different salinity before and after aging at 150°C for 16 h

3.2.3 Particle size distribution

The particle size distributions (PSD) of BTDF, BTDF/PAAD and BTDF/PAADE are presented in Fig.9. The particle size of BTDF exhibited a unimodal distribution with a median particle diameter (D50) of 8.124 μm, and the incorporation of polymers widened the PSD and shifted the peaks to a larger value. The D10 values which represented the small particles in the dispersion system, were increased from 1.912 μm to 2.464 μm and 2.677 μm by PAAD and PAADE, respectively. Interestingly, a distinct bimodal distribution was observed for BTDF/PAADE, whose D90 value, an indicator for large particles or flocs, was significantly increased to 187.2 μm, proving the bridged flocs aggregations through hydrophobic interactions. While the D90 value of BTDF/PAAD slightly decreased.

With the addition of NaCl, the D50 value of BTDF increased with increasing salt concentration from 0 to 3 w/v%, while then showing a slight decrease over the salt concentration range between 3-10 w/v%. This phenomenon exactly proved the changes of clay particle configuration from face-edge which had a larger equivalent particle size to face-face association with smaller equivalent particle size, consequently the dispersion-flocculation-sedimentation transition occurred at the NaCl concentration of 3 w/v%, where showed a maximum yield stress for BTDF. After introducing PAAD and PAADE, the D50 values both showed a slight increase trend in the salt concentration range between 0-5 w/v%, indicating a certain stabilization effects of PAAD and PAADE on the colloidal clay particles. However, the D90 values showed the opposite changes, namely a continuous growth for BTDF/PAADE while a decline trend for BTDF/PAAD, which verified that the undermined amphoteric interactions by salt ions caused de-flocculation of bentonite/polymer network while salt-enhanced hydrophobic interactions
inversely led to self-aggregation of flocs. Due to the fine particles contributed more to the flow resistance than the larger ones in suspensions [19], the BTDF/PAADE with larger particle aggregates at 0.5 w/v% NaCl addition could flow more easily than BTDF/PAAD, reflecting in the lower consistency coefficient in rheological results. When increasing NaCl to a high content of 10 w/v%, the particle sizes of BTDF/PAAD abruptly increased, while that of BTDF/PAADE showed a sudden decrease. The high salt ions favored the intra-molecular hydrophobic interactions of desorbed PAADE thus disaggregating large flocs, while the curled-up molecular chains of PAAD might wrap particles to form unstable agglomeration. This also confirmed the assumption that the formation of polymer aggregates resulted in abrupt increase of consistency coefficient for BTDF/PAADE at 10 w/v% NaCl concentration. Additionally, the D10 values of BTDF and BTDF/PAAD increased with the increase of salt concentration especially at a high salt content, while that of BTDF/PAADE was almost unchanged. This demonstrated that PAADE was capable of maintaining enough small particles, which was the reason that PAADE could lower fluid loss over the salinity range under study.

![Fig. 9. Particle size distributions of BTDF, BTDF/PAAD and BTDF/PAADE at different NaCl concentration](image)

3.3 Biodegradability
The biodegradability results are shown in Table 1. The ratio of BOD₅ and COD defined as biodegradability index (B.I.) was used as an indicator for biodegradation capacity. Generally, if B.I. > 0.3 the sample can be considered as biodegraded, in which if B.I. > 0.6 then the sample is quite biodegradable, and if B.I. is between 0.3 and 0.6 then seeding microorganisms is required. If B.I. < 0.3, the sample cannot be treated biologically [20]. PAAD was non-biodegradable with a B.I. of 0.16. However, it is interesting to find that after incorporating ESO-MA blocks, the biodegradability of the obtained new material greatly improved and the B.I. reached 0.34, indicating the PAADE was biodegradable with the aid of microorganisms.

| Sample | BOD₅ (mg/L) | COD (mg/L) | Biodegradability Index |
|--------|-------------|------------|------------------------|
| PAAD   | 709.00      | 4400       | 0.16                   |
| PAADE  | 806.78      | 2383       | 0.34                   |

4. Conclusions
(1) Compared with PAAD, PAADE with amphiphilic and amphoteric structures showed higher solution viscosity, and an association behavior at the critical concentration of 0.3 w/v%. The addition of NaCl first reduced the viscosity and then increased at 3 w/v% NaCl concentration where the hydrodynamic diameter started to increase due to salt-enhanced hydrophobic association.

(2) The PAADE showed more significant synergistic effect with bentonite in BTDF than PAAD, leading to the more prominent flocculated structure and stronger yield stress. Also, PAADE showed more significant stabilizing effect on the colloidal clay particles and prevent the face-face coagulation
to maintain enough small particles at all tested salinity. The hydrophobic shield, improved filter cake strength, as well as the formed polymer aggregates contributed to the reduction of fluid loss, especially at HTHP and high salinity conditions where hydrophobic association was enhanced.

(3) The ESO-MA blocks improved the biodegradability of PAADE, which could be considered as biodegradable additive with the aid of microorganisms.

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