



## Original Paper

# A micro-crosslinked amphoteric hydrophobic association copolymer as high temperature- and salt-resistance fluid loss reducer for water-based drilling fluids



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## ABSTRACT

During ultradeep oil and gas drilling, fluid loss reducers are highly important for water-based drilling fluids, while preparing high temperature- and salt-resistance fluid loss reducers with excellent rheology and filtration performance remains a challenge. Herein, a micro-crosslinked amphoteric hydrophobic association copolymer (i.e., DADC) was synthesized using N,N-dimethyl acrylamide, diallyl dimethyl ammonium chloride, 2-acrylamido-2-methylpropane sulfonic acid, hydrophobic monomer, and pentaerythritol triallyl ether crosslinker. Due to the synergistic effects of hydrogen bonds, electrostatic interaction, hydrophobic association, and micro-crosslinking, the DADC copolymer exhibited outstanding temperature- and salt-resistance. The rheological experiments have shown that the DADC copolymer had excellent shear dilution performance and a certain degree of salt-responsive viscosity-increasing performance. The DADC copolymer could effectively adsorb on the surface of bentonite particles through electrostatic interaction and hydrogen bonds, which bring more negative charge to the bentonite, thus improving the hydration and dispersion of bentonite particles as well as the colloidal stability of the drilling fluids. Moreover, the drilling fluids constructed based on the DADC copolymer exhibited satisfactory rheological and filtration properties ( $FL_{HTHP} = 12$  mL) after aging at high temperatures (up to 200 °C) and high salinity (saturated salt) environments. Therefore, this work provided new insights into designing and fabricating high-performance drilling fluid treatment agents, demonstrating good potential applications in deep and ultradeep drilling engineering.

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## 1. Introduction

Oil and gas are the material basis for human production and life, which will still be the primary energy consumption in the world by 2050 (Liu et al., 2022a). With the development of the global oil and gas industry, the oil and gas exploration has rapidly extended from shallow to ultra-deep layers (Zhang et al., 2023). Drilling is the only way to establish the connection channel between the ground and underground reservoirs, which is the core project of petroleum

engineering (Li et al., 2022a). As the blood of drilling engineering, drilling fluids can carry cuttings, cool and lubricate bits and drill strings, suspend and release cuttings, balance wellbore pressure, and protect oil and gas reservoirs (Li et al., 2022b, 2022e; Sun et al., 2021b). Water-based drilling fluids (WDFs) composed of water, bentonite, weighting agents, and various chemical treatment agents have been playing a leading role in drilling engineering due to their low cost, convenient construction, and environmental non-pollution (Balaga and Kulkarni, 2022; Wang and Ding, 2021; Yang et al., 2022). However, with the increase of drilling depth, the geological conditions become increasingly complex: ultra-high temperature, ultra-high pressure, ultra-high salt, etc., leading to a deterioration in the performance of WDFs (Bai et al., 2022; Balaga

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and Kulkarni, 2022; Gautam et al., 2022; Zhu et al., 2021).

Fluid loss reducers, as the essential chemical additive for WDFs, can form an adsorption layer on the surface of bentonite through hydrogen bonds and electrostatic interaction to promote the dispersion of bentonite particles. In addition, the filtrate reducer and the particles in the drilling fluids are deposited on the wellbore under pressure, forming a thin and dense mud cake, thereby reducing the invasion of the drilling fluids into the formation (Aghdam et al., 2020; Liu et al., 2020a; Ricky et al., 2022). Generally, the existing fluid loss reducers are divided into natural/natural modified polymer and synthetic polymer (Gautam et al., 2022). Various environmentally friendly natural polymers, such as starch, cellulose, humic acid, and tannin were prone to oxidation and hydrolysis, which could not meet the complex drilling conditions of high temperature (Al-Hameedi et al., 2019). Chemical modification methods such as esterification, etherification, and grafting have been applied to natural polymers to improve their temperature resistance and fluid loss reduction (Al-Hameedi et al., 2019; Li et al., 2015, 2016, 2021; Liu et al., 2020c; Murtaza et al., 2021; Rana et al., 2021; Zhu and Zheng, 2021). However, due to many irregular glycoside bonds (low bond energy) in natural polymers-based materials, their applicable temperature is usually lower than 180 °C, limiting their application in high-temperature environments (Sun et al., 2021b). Synthetic polymer fluid loss reducer can significantly improve the temperature resistance (up to 200 °C) by introducing temperature-resistant monomers into the polymer, which has become a research hotspot in petroleum engineering in recent years (Gautam et al., 2022). Nonetheless, in deep salt gypsum layers, temperatures up to 200 °C and salt concentrations reaching saturation, the conformational changes of polymer chains, such as degradation, curling, cross-linking, etc., which has become a significant challenge for synthetic polymer fluid loss reducer (Sun et al., 2020a, 2021a).

To improve the temperature and salt resistance of synthetic polymer fluid loss reducers, researchers have done much research on the following two aspects. Firstly, by introducing rigid groups (imidazole ring, benzene ring, pyridine ring, etc.) and salt-resistant groups (amphoteric ion group, etc.) into the polymer's side chain, the polymer's temperature and salt resistance could be enhanced (Chang et al., 2019; Ghaderi et al., 2019; Li et al., 2023; Liu et al., 2020b; Sun et al., 2020a). For example, Jiang et al. synthesized a polymer fluid loss reducer containing multiple bulky cyclic structures through free radical polymerization with a temperature resistance of up to 240 °C (Wang et al., 2021). Li et al. reported a novel tetra-copolymer with multi-ring side groups, showing excellent filtration loss reduction in high-temperature and salt environments (Li et al., 2023). Secondly, hydrogen bonds, hydrophobic association, and micro-crosslinking were introduced to control the polymer's condensed structure and improve the polymer's high temperature and salt resistance (Davoodi et al., 2019; Shen et al., 2020; Wang and Ding, 2021; Zhang et al., 2023). Recently, Sun et al. synthesized a novel fluid loss reducer through free radical polymerization of lauryl methacrylate and other monomers, which exhibited significant hydrophobic association characteristics as well as outstanding temperature- and salt-resistance (Sun et al., 2021b). However, most hydrophobic association polymers require the addition of surfactants in the preparation process, which would lead to severe foaming during the drilling process, thus increasing the viscosity of the drilling fluids. Therefore, it remains a challenge to precisely design molecular architectures that integrate multiple temperature- and salt-resistant mechanisms into one synthetic polymer, thereby endowing it with excellent performance.

Herein, a novel polymer fluid loss reducer (i.e., DADC) with perfect temperature and salt resistance was prepared by

copolymerization of N,N-dimethyl acrylamide (DDAM), diallyldimethyl ammonium chloride solution (DMDAAC), 2-acrylamido-2-methylpropanesulfonic acid (AMPS), hydrophobic monomer (C12VIMCl) and pentaerythritol triallyl ether crosslinker (APE), as shown in Fig. 1. The key to this copolymer was the integration hydrogen bonds, electrostatic interactions, hydrophobic associations, and micro-crosslinks into one molecule. First, the rigid ring units (imidazole, quaternary ammonium) and micro-crosslinking limited the internal rotation of the polymer chain and increased the rigidity of the polymer molecular chain, thus endowing the polymer with excellent temperature resistance performance (Li et al., 2023). Second, the electrostatic interaction between positive (quaternary ammonium and imidazole cations) and negative (sulfonic acid) groups in polymer molecules formed “internal salt bonds” in freshwater solutions. Nevertheless, in high-concentration saline solutions, the metal counter ions disrupted the “internal salt bonds” in polymer molecules, which resulted in the formation of electrostatic interaction between the positive and the negative charge on the surface of the bentonite particles, thus increasing the adsorption of the polymer on the surface of the bentonite particles (Cheng et al., 2023; Sun et al., 2020a; Zhang et al., 2022). Moreover, imidazole ionic liquids increased the solubility of hydrophobic long-chain alkyl groups, enabling molecules to form strong association network structures, thus increasing the hydraulic radius of polymer chain rotation and improving the temperature and salt resistance of the polymer (Chen et al., 2022; Sun et al., 2021b). Owing to its delicate design, the resultant DADC copolymer exhibited excellent rheological and filtration properties under high-temperature (up to 200 °C) and high salinity (saturated salt) environments, which was expected to be applied in deep and ultradeep well drilling.

## 2. Materials and methods

### 2.1. Materials

N,N-dimethylacrylamide (DDAM), diallyl dimethyl ammonium chloride solution (DMDAAC, 60 wt%), 2-acrylamido-2-methylpropanesulfonic acid (AMPS), 1-vinyl imidazole, 1-Chlorododecane and pentaerythritol triallyl ether (APE) were supplied by Aladdin Reagent Company (Shanghai, China) and used as received. NaOH, NaCl, KPS, and Na<sub>2</sub>CO<sub>3</sub> were bought from Sinopharm Group Chemical Reagent Company Limited. Bentonite was purchased from CNPC Bohai Drilling Engineering Co., Ltd (China). The high-temperature resistant fluid loss reducer Driscal-D was purchased from Chevron Phillips Chemical Co., Ltd. The other drilling fluid additives, including ultra-fine calcium carbonate, polyalcohol, white oil, emulsifier, polyester lotion, and barite weighting agent, were purchased from Deshunyuan Petroleum Technology Co., Ltd (Shandong, China). Other reagents do not require purification and can be directly used.

### 2.2. Preparation of micro-crosslinked amphoteric hydrophobic association copolymer

The hydrophobic ionic liquid monomer C12VIMCl was prepared according to the literature (Sun et al., 2018). The micro-crosslinked amphiphilic hydrophobic association polymer DADC (DDAM-AMPS-DMDAAC-C12VIMCl) was synthesized through free radical aqueous solution copolymerization using KPS as the initiator referring to previous methods (Cao et al., 2017; Liu et al., 2016). Briefly, a certain mass of DDAM (6.93 g, 0.07 mol), AMPS (10.35 g, 0.05 mol), DMDAAC (3.22 g, 0.02 mol), C12VIMCl (2.98 g, 0.01 mol) and crosslinking agent APE (0.68 g, 3 % of the total mass of the monomer) was dissolved in distilled water (solid content: 20 wt%)

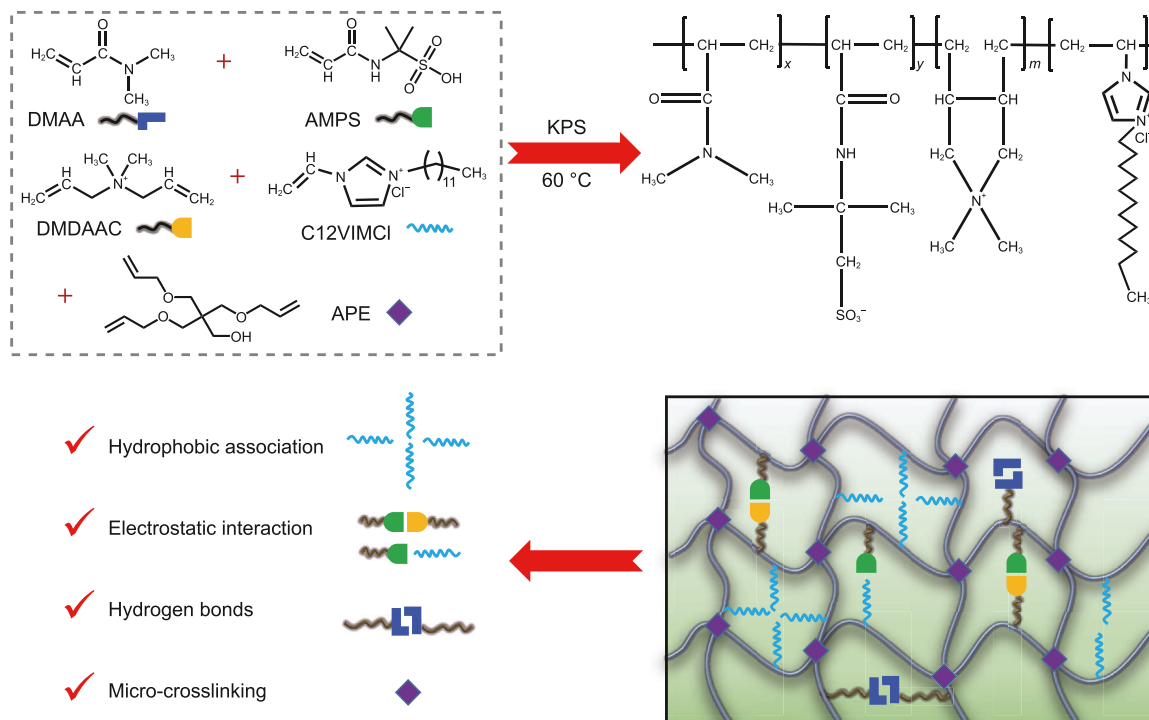


Fig. 1. Synthesis and schematic diagram of micro-crosslinked amphoteric hydrophobic association copolymers.

to obtain the reaction solution. Then, the pH of the reaction solution was adjusted to 8 using 1 mol/L NaOH solution to achieve a slightly alkaline environment. Transfer the reaction solution to a three-necked flask and stir for 30 min in a nitrogen-bubbling atmosphere to remove oxygen. Afterward, the KPS initiator (0.25 g, 1 wt% of solid mass) was added to initiate the polymerization reaction at 60 °C for 4 h. Finally, the obtained viscous reactants were precipitated in ethanol to remove unreacted monomers and dried at 80 °C in an oven for 5 h to get the target copolymer.

### 2.3. Characterization

The nuclear magnetic resonance (NMR) spectrum of the sample was performed on an Avance NEO 400 MHz NMR spectrometer (Bruker, Germany) with deuterated water ( $D_2O$ ) as the solvent. Fourier transform infrared spectroscopy (FT-IR) of samples was recorded on a Nicolet 6700 FT-IR spectrometer (Nexus, USA) using a standard KBr pellet technique in the 500–4000  $cm^{-1}$ . The TGA measurements were carried out on a TG 209 thermal gravimetric analyzer with temperatures ranging from 40 to 600 °C at a heating rate of 10 °C/min. The micromorphology of samples was observed using Scanning electron microscopy (SEM, ZEISS, EVO-15, DE). Rheological properties of polymer solutions in deionized and salt water were measured using TA, DHR-2 rheometer with a parallel-plate geometry (25 mm in diameter, 1 mm between gap distances). The viscosity of the polymer solution was measured as a function of shear rate over a shear rate range of 1–200  $s^{-1}$  at 25 °C in deionized and salt water. The storage modulus ( $G'$ ) and the loss modulus ( $G''$ ) were measured as a function of the oscillation mode with the shear strain maintained at 1%, and the frequency varied from 0.1 to 10 Hz.

### 2.4. Drilling fluids preparation and performance evaluation

Add 40 g bentonite and 2.4 g anhydrous sodium carbonate to 1000 mL fresh water under high-speed stirring, then seal for 24 h to obtain the base slurry. A certain amount of DADC copolymer was added to the high-speed stirred base slurry and sealed for 24 h to obtain the DADC copolymer-based drilling fluids. In addition, a certain amount of NaCl was added to the DADC copolymer-based drilling fluids and stirred for 20 min to get salt-contaminated drilling fluids. Temperature- and salt-resistance drilling fluids system: add DADC copolymer to 300 mL of base slurry and stir for 20 min at 5000 r/min. Afterward, sodium chloride, superfine calcium carbonate plugging agent, polyalcohol plugging agent, white oil lubricant, emulsifier, polyester lotion and barite weighting agent were added in turn. It was worth noting that after adding each additive to the drilling fluids system, it needed high-speed stirring for 20 min.

The drilling fluids were placed in a rolling heating furnace with different temperatures for 16 h. Pour the maintained drilling fluids into a cup, measure the values displayed on the dial at 600 r/min and 300 r/min using a six-speed rotary viscometer, and calculate the apparent viscosity (AV), plastic viscosity (PV), and yield point (YP) of the drilling fluids according to the formula:

$$AV = \frac{1}{2}\theta_{600} \text{ (mPa} \cdot \text{s)} \quad (1)$$

$$PV = \theta_{600} - \theta_{300} \text{ (mPa} \cdot \text{s)} \quad (2)$$

$$YP = 0.511(\theta_{300} - PV) \text{ (Pa)} \quad (3)$$

where  $\theta_{600}$  and  $\theta_{300}$  are the values of the rotary viscometer at the corresponding speed.

The API filtration of drilling fluids was measured by a low-

pressure-low-temperature (LPLT) filter press API (Fann Instrument Company) at a fixed pressure of 0.7 MPa for 30 min. The high-temperature and high-pressure (HTHP) filtration was tested by a high-temperature and high-pressure filtration apparatus (GGS42-2, Qingdao Tongchun Petroleum Instrument Co., Ltd.) at a fixed pressure of 3.5 MPa for 30 min.

## 2.5. Mechanism analysis

**Microscopic morphological analysis of mud cake.** Observe the surface morphology of dried mud cakes using scanning electron microscopy.

**Zeta potential and particle size analysis.** The Zeta potential of the drilling fluids was measured at room temperature using a Malvern Zetasizer Nano Analyzer (Malvern, UK). The particle size distribution of the drilling fluids was analyzed using an ultra-high-speed intelligent particle size analyzer (Mastersizer 3000, Malvern Instruments Co., Ltd.).

**Adsorption capacity of copolymers on bentonite particles.** The adsorbed amount of the copolymer on the bentonite particles was determined using a total organic carbon (TOC) analysis apparatus (multi N/C 3100, Analytik Jena AG, Jena, Germany). The following equation calculated the adsorption amount:

$$K = \frac{m_1 - m_2 \times \frac{P}{L}}{m_0} \quad (\text{mg/g}) \quad (4)$$

where  $K$  is the adsorption quantity of the copolymer on bentonite particle (mg/g);  $m_0$  is the initial mass of bentonite particle in the drilling fluids (g);  $m_1$  is the initial mass of the copolymer in the drilling fluids (g);  $m_2$  is mass of high-temperature and high-pressure filtration loss (g);  $L$  is the mass proportion of elemental carbon in the copolymer (%);  $P$  is the amount of carbon element in the filtrate tested by TOC (mg/L).

## 3. Results and discussion

### 3.1. Characterization of DADC copolymer

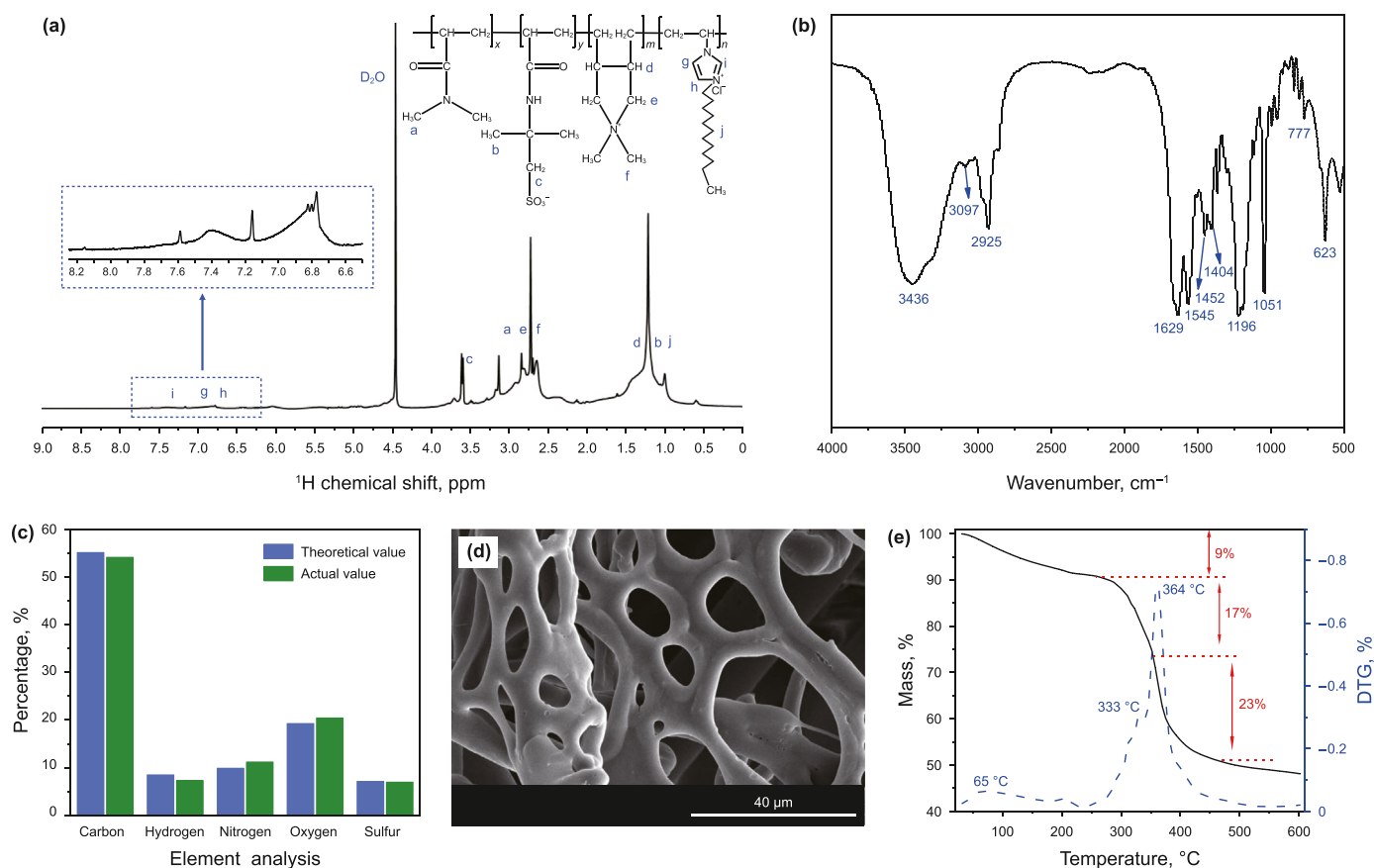
To prove the successful synthesis of the DADC copolymer, the product's chemical structure was confirmed by  $^1\text{H-NMR}$ , FT-IR, and elemental analysis. As shown in Fig. 2(a), the chemical shifts of the two methyl ( $-\text{CH}_3$ ) protons in the DDAM unit may be about 2.8 ppm. The chemical shifts of methyl ( $-\text{CH}_3$ ) and methylene ( $-\text{CH}_2-$ ) groups in the AMPS unit may be observed at 1.0 and 3.5 ppm, respectively. The chemical shift of methyl and methylene groups on quaternary ammonium in DMDAAC could be observed at 3.0–3.3 ppm. Significantly, the signal about 6.5–8.0 ppm should belong to the hydrogen atom on the imidazole ring, and the chemical shift of the extended chain alkyl group could be observed at 1.0 ppm, thus confirming the successful polymerization of hydrophobic monomer C12VIMCl in DADC copolymer (Sun et al., 2018). In addition, Fig. 2(b) showed the FT-IR spectra of the DADC copolymer, where the characteristic peak near  $2920\text{ cm}^{-1}$  should belong to the stretching vibration absorption peak of the  $-\text{CH}_2-$  group in the main and side chains of the copolymer. The vibration absorption peaks at  $3097$  and  $1552\text{ cm}^{-1}$  were attributed to  $-\text{CH}-$  on the imidazole ring and the stretching vibration of imidazolium cation from the hydrophobic monomer, respectively (Chen et al., 2022). The absorption peaks at  $1629$  and  $1452\text{ cm}^{-1}$  were due to the stretching vibration peak of the  $\text{C}=\text{O}$  group in amide from DDAM units. The cliffs at  $1404\text{ cm}^{-1}$  were attributed to the vibrational absorption peak of quaternary ammonium in DMDAAC. The peak at  $610\text{ cm}^{-1}$  corresponded to the bending vibration of  $\text{S}-\text{O}-\text{S}$ , while the peaks at  $1196$  and  $1051\text{ cm}^{-1}$  were attributed to the

stretching vibration absorption peaks of  $\text{S}-\text{O}$  and  $\text{S}=\text{O}$  in AMPS, respectively (Li et al., 2022a). Therefore, the FT-IR characterization results further suggested that the DADC copolymer was synthesized successfully. Moreover, as shown in Fig. 2(c), the elemental (C, H, N, O, S) analysis showed that the theoretical values of element content in the DADC copolymer were consistent with the actual measured values, thereby proving that all monomers successfully participated in the reaction.

Generally, the main chain and side chain of the polymer would undergo thermal degradation in high-temperature environments, which destroyed the molecular structure and performance of the polymer (Cao et al., 2017; Yang et al., 2019). Therefore, the temperature resistance of DADC copolymer had important significance for its application in deep and ultradeep oil and gas. Fig. 2(d) showed the self-assembly structure of the DADC copolymer in aqueous solutions obtained by SEM. The as-synthesized polymer had a three-dimensional network cross structure with a robust backbone connection due to the synergistic effects of hydrogen bonds, electrostatic interaction, hydrophobic association, and micro-crosslinking (Fig. 1), thus endowing the polymer with excellent thermal stability in high-temperature environments (Cheng et al., 2023; Wang and Ding, 2021). In addition, the thermogravimetric (TG)-differential thermogravimetric (DTG) curve of the DADC copolymer was shown in Fig. 2(e), with the mass loss divided into four stages. During heating from 30 to  $260\text{ }^\circ\text{C}$ , the mass loss of the sample was 9%, mainly due to the decomposition of residual water and a small amount of functional group in the sample. In the second stage, the thermogravimetric curve sharply decreased, possibly due to the rapid decomposition of hydrophilic amide groups in the copolymer chain. The third stage of mass loss of the copolymer started at  $360\text{ }^\circ\text{C}$ , where the sulfonic acid groups within the copolymer began to decompose rapidly, and the side chains of the copolymer also began to break, resulting in a weight loss of 23%. When the temperature exceeded  $500\text{ }^\circ\text{C}$ , the main chain of the copolymer gradually lessened, and the polymer began to carbonize, with a residual mass retention rate of 48%. The above thermogravimetric experiment could infer that DADC copolymer had good thermal stability and application in high-temperature environments.

Usually, the viscoelasticity of polymer was crucial for the rheological properties of drilling fluids, which affect the rock-carrying capacity and stabilization of the wellbore during the drilling process (Sun et al., 2021b; Yang et al., 2022). Herein, the rheological properties, including storage modulus ( $G'$ ), loss modulus ( $G''$ ), and viscosity of DADC polymer solution, were characterized by a rotary rheometer. As shown in Fig. 3(a), with the continuous increase of frequency, the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) of the DADC copolymer solutions gradually increased. The loss modulus was higher than the storage modulus, suggesting that the polymer solution was mainly viscous. In addition, as the shear rate increased, the apparent viscosity of the DADC solution significantly decreased shown in Fig. 3(b), exhibiting the shear dilution characteristics of pseudoplastic fluids, which was beneficial for suspending rock cuttings at low shear rates and breaking rocks at high shear rates (Davoodi et al., 2019). Moreover, as the concentration increased, the hydrophobic association effect, hydrogen bonds effect, and electrostatic force of the copolymer greatly enhanced the intermolecular forces, thus resulting in a significant increase in storage modulus, loss modulus, and viscosity.

DADC was a micro-crosslinked copolymer that linked positively charged, negatively charged, and hydrophobic groups. To improve the temperature and salt resistance of copolymer and prevent the flocculation of bentonite particles, the molecular composition of DADC polymer was dominated by anions (sulfonate) (Gautam et al., 2022). In the freshwater environment, a portion of anionic



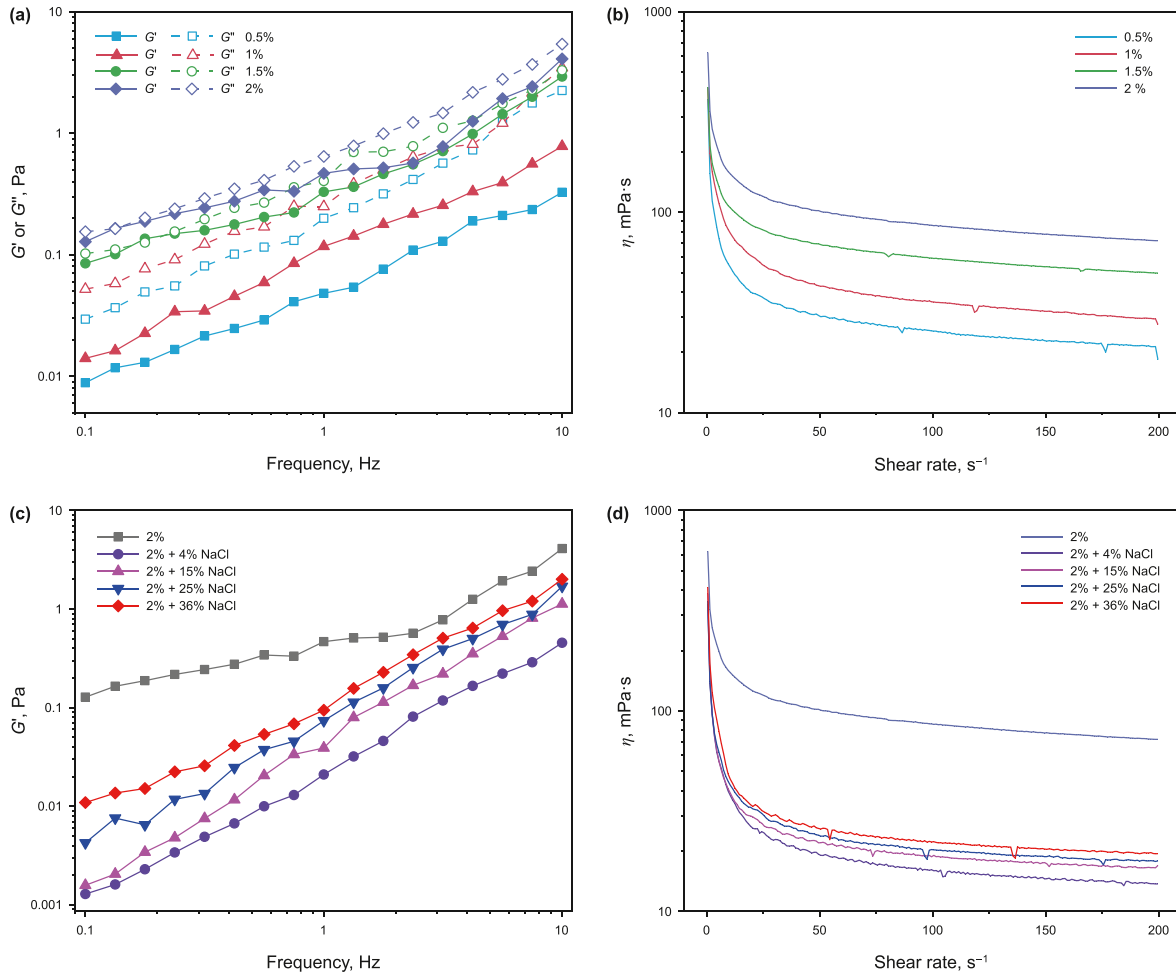
**Fig. 2.** (a)  $^1\text{H}$ -NMR curves of DADC copolymer; (b) FT-IR spectrum of DADC copolymer; (c) Elemental analysis of DADC copolymer; (d) Microstructure of DADC copolymer; (e) TGA and DTG curves of DADC copolymer.

(sulfonate) and cationic groups (quaternary ammonium and imidazolium cation) would form the “internal salt bonds”. Meanwhile, the remaining anions would form electrostatic repulsion, causing the chain segment to open (Tan et al., 2021). When a small amount of NaCl electrolyte was added, the metal sodium ions would shield the repulsion between anions groups (sulfonate), making the polymer chain curl and reducing fluid mechanics volume, thus decreasing the modulus and viscosity of the polymer solutions shown in Fig. 3(c) and (d) (Sun et al., 2020a). However, with the increase in salt concentration, on the one hand, sodium ions would disrupt the “internal salt bonds” in polymer chain, leading to a transition of the chain to a random conformation (Ghaderi et al., 2019; Sun et al., 2020b). What's more, salt increased the polarity of the solutions, significantly improved the hydrophobic association of long-chain alkyl in polymer chain, resulting in an enhanced spatial network structure of the polymer (Liu et al., 2020a; Zhang et al., 2023). When the salt concentration in the solution reached saturation, the volume of polymer fluid mechanics increased, and the viscosity of the solution no longer decreased or even increased. Therefore, DADC copolymer exhibited excellent comprehensive performance, with enormous application in high temperature and salt environments.

### 3.2. Performance of DADC copolymer in drilling fluids

During the drilling process, the drilling fluids play an important role in cooling, cleaning, and lubricating the drill bit, transporting rock cuttings out of the wellbore, avoiding repeated cutting, improving drilling efficiency, and balancing formation pressure, all

of which were closely related to the rheological properties of the drilling fluids (Aghdam et al., 2020). Therefore, the effects of DADC copolymer on rheological parameters (AV, PV, and YP) and fluid loss were measured based on API standards. As shown in Fig. 4, the AV, PV, and YP of the drilling fluids were positively correlated with the concentration of DADC copolymer in the solution. When 2 wt% DADC copolymer was added into the base slurry, the AV increased from 10 to 50 mPa·s, the PV increased from 6 to 40 mPa·s, the YP increased from 3 to 10 Pa, and the  $\text{FL}_{\text{API}}$  decreased from 15 to 3 mL compared with pure base slurry. This was because the DADC copolymer chain could be adsorbed on the surface of the bentonite particles through hydrogen bonds and electrostatic interaction, and the hydration groups suspended on the polymer chain could adsorb free water molecules, thus forming a layer structure of “bentonite-polymer chain-water molecules” and increasing the internal friction of drilling fluids (Li et al., 2022b). The intramolecular and intermolecular association aggregation of hydrophobic groups in DADC polymers also enhanced the spatial grid structure of drilling fluids, thereby increasing its structural viscosity (Shen et al., 2020; Sun et al., 2021b). As a result, the base slurry's viscosity increased with the DADC polymer's concentration. In addition, the DADC copolymer could prevent the bentonite particles from flocculating, thus promoting the formation of thin and dense mud cakes on the wellbore wall and reducing the filtration loss. It was worth noting that the viscosity-increasing effect of DADC copolymer was beneficial to reduce the filtration loss of drilling fluid, but the high viscosity of drilling fluids would reduce the mechanical penetration rate and even cause malignant accidents such as sticking (Sun et al., 2020b). Generally, the apparent viscosity of the drilling fluids



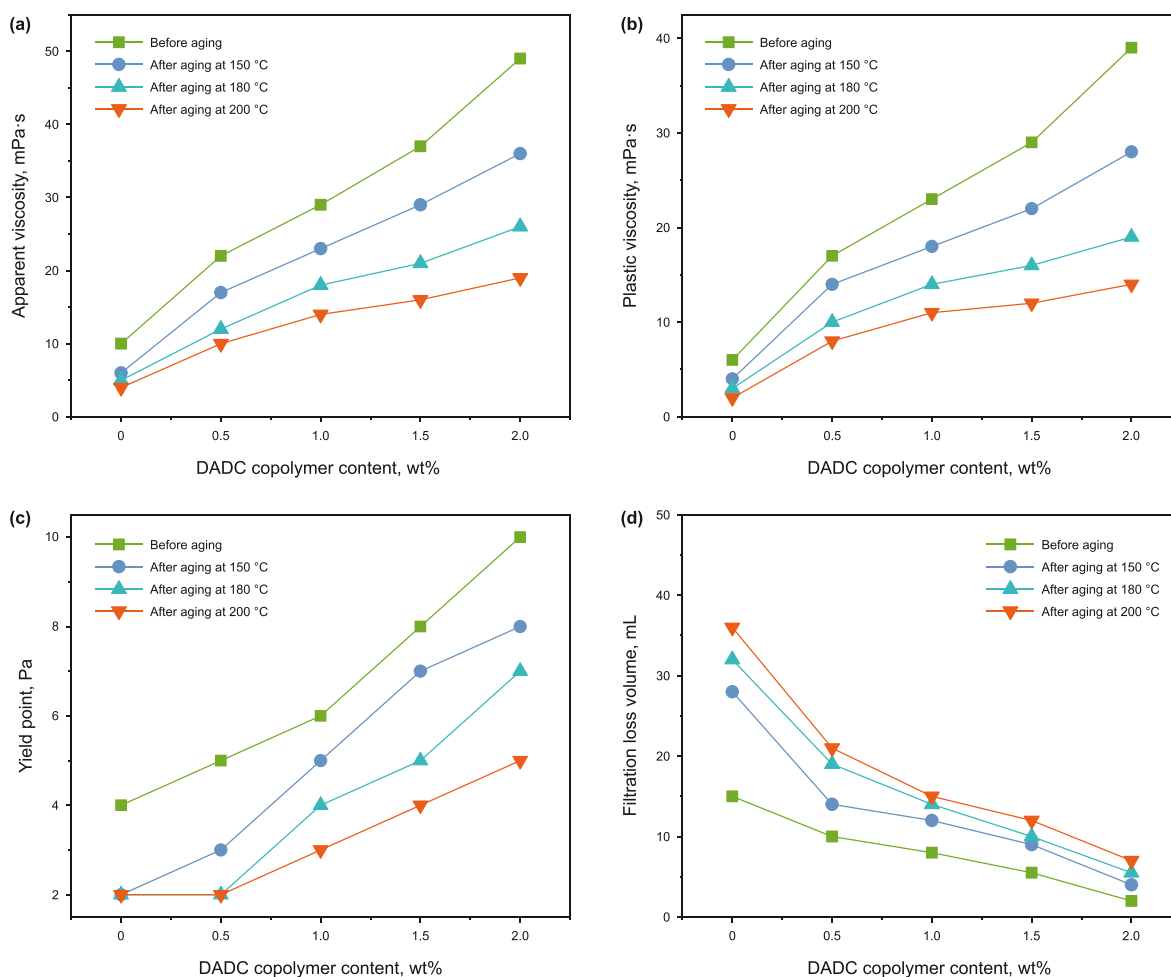
**Fig. 3.** (a) Viscoelastic properties of DADC copolymer solutions; (b) Viscosity curves of DADC copolymer solutions; (c) Viscoelastic properties of 2 wt% DADC copolymer solutions with different NaCl concentrations; (d) Viscosity curves of 2 wt% DADC copolymer solutions with different NaCl concentrations.

added with 2 wt% fluid loss reducers should be less than 80 mPa·s, while the apparent viscosity of the drilling fluids added with 2 wt% DADC copolymer was just 50 mPa·s, showing its low viscosity increasing effect.

In deep formations, the polymer fluid loss reducer, which could resist high-temperature oxidative degradation, was crucial for maintaining drilling fluids' rheological and filtration properties in harsh high-temperature conditions (Balaga and Kulkarni, 2022). As shown in Fig. 4, with the increase in aging temperature, the viscosity of the base slurry gradually decreased, and the API filtration loss gradually increased, confirming the high-temperature agglomeration of bentonite particles. However, for the base slurry (with 2 wt% DADC copolymer) after aging at 200 °C, the AV increased from 4 to 17 mPa·s, the PV increased from 2 to 13 mPa·s, the YP increased from 2 to 4 Pa. At the same time, the  $FL_{API}$  decreased from 36 to 7 mL compared with base slurry, demonstrating that the DADC copolymer could carry rock debris and reduce filtration in high-temperature environments.

Generally, when drilling into salt-gypsum formation, metal cations (sodium ions, calcium ions, magnesium ions, etc.) would enter the drilling fluids, destroying the electrostatic repulsion and leading to the aggregation or even flocculation of bentonite particles (Sun et al., 2021b). In addition, the proximity of the metal cation to the polymer chain would cause the polymer chain to curl, shrink in size, and even salt out, leading to the failure of the

performance of the fluid loss reducer (Zhang et al., 2023). Therefore, the drilling fluids with increased viscosity, shear resistance, and fluid loss reduction performance in saline environments were the key to drilling in salt gypsum formations. Herein, to study the temperature and salt resistance of DADC copolymer, the change of rheological and fluid loss of the drilling fluids accordant with different sodium chloride contents were performed. As shown in Fig. 5, when sodium chloride enters the drilling fluids, the viscosity of drilling fluids gradually decreased, and the API filtration increased due to the curling of polymer chains and reduction of hydrodynamic volume in brine. Remarkably, as the concentration of sodium chloride rose from 4% to 36%, the viscosity of the drilling fluids increased while the filtration decreased to a certain extent. This phenomenon was because as the salt concentration increased, the "internal salt bonds" formed by cations and anions in the polymer gradually opened, causing the polymer chain to expand and increasing the colloidal stability of drilling fluids. What's more, salt increased the hydrophobic association of copolymers, further enhancing the structural viscosity of drilling fluids (Cheng et al., 2023; Liu et al., 2020a; Sun et al., 2021b). As a result, the filtration volume of the drilling fluids (with 36 % NaCl) after aging at 200 °C was only 9.5 mL, demonstrating the DADC copolymer had excellent filtration-reducing abilities under high temperature and salinity conditions.



**Fig. 4.** Rheological properties of DADC copolymer-based drilling fluids before and after aging: (a) AV; (b) PV; (c) YP. (d) API filtration loss of DADC copolymer-based drilling fluids before and after aging at 150, 180 and 200 °C for different contents.

### 3.3. Analysis of filtration reduction mechanism of DADC copolymer

In this section, the role of DADC copolymer in reducing drilling fluids filtration was analyzed from the following aspects.

#### 3.3.1. Morphology of mud cake

During the drilling process, solid particles in the drilling fluids would deposit on the wellbore, forming a layer of mud cake, which was a crucial factor in determining the filtration loss. Usually, a good mud cake should have a dense and compact appearance with few leakage pores, thus effectively preventing the filtrate from infiltrating into the formation (Aghdam et al., 2020). Herein, we investigated the influence of DADC copolymer on mud cake morphology through intuitive and microscopic methods. As shown in Fig. 6(a), the mud cake formed by the base slurry aging at 200 °C was thicker (2.6 mm), thus resulting in an enormous filtration loss as described earlier. This was because high temperature intensified the molecular thermal motion and reduced the electrostatic repulsion between bentonite particles, which led to the aggregation of bentonite particles to form large particles on the surface of the mud cake shown in Fig. 7(a) (Liu et al., 2022b). However, as shown in Fig. 6(b)–(e), with the addition of DADC copolymer, the mud cake became thinner. In addition, the surface became smoother without noticeable pores and microcracks shown in Fig. 7(b)–(e), indicating the DADC copolymer effectively inhibited the aggregation of bentonite particles.

Fig. 6(f) showed that when the drilling fluids with 2 wt% DADC copolymer was contaminated with 4 wt% NaCl, the thickness of the mud cake increased to 1.4 mm and some large cracks between particles adhered to the surface of the mud cake shown in Fig. 7(f). This was because electrolytes would dehydrate bentonite particles, which could not protect them from aggregation in saline environments, thus resulting in a rougher surface of the mud cake (Sun et al., 2020b). However, as the NaCl content continued to increase, the thickness of the mud cake decreased to a certain extent, and there were no apparent pores or microcracks shown in Fig. 7(g)–(i). On the whole, through the analysis of the macroscopic and microscopic morphology of the mud cake, it was demonstrated that in high temperature and high salinity conditions, DADC copolymer still had good adsorption ability and enhancing the hydration effect of bentonite, thereby improving the quality of mud cake.

#### 3.3.2. Analysis of adsorption quantity, zeta potential and particle size distribution

Fluid loss reducer could adsorb onto the surface of bentonite particles through hydrogen bonds and electrostatic interactions, thus forming a colloidal dispersion system, which was crucial to the filtration reduction performance of the drilling fluids. Usually, the greater the adsorption capacity of polymers on the surface of bentonite, the smaller the particle size of the drilling fluids and the more stable the colloid dispersion system was (Dong et al., 2022).

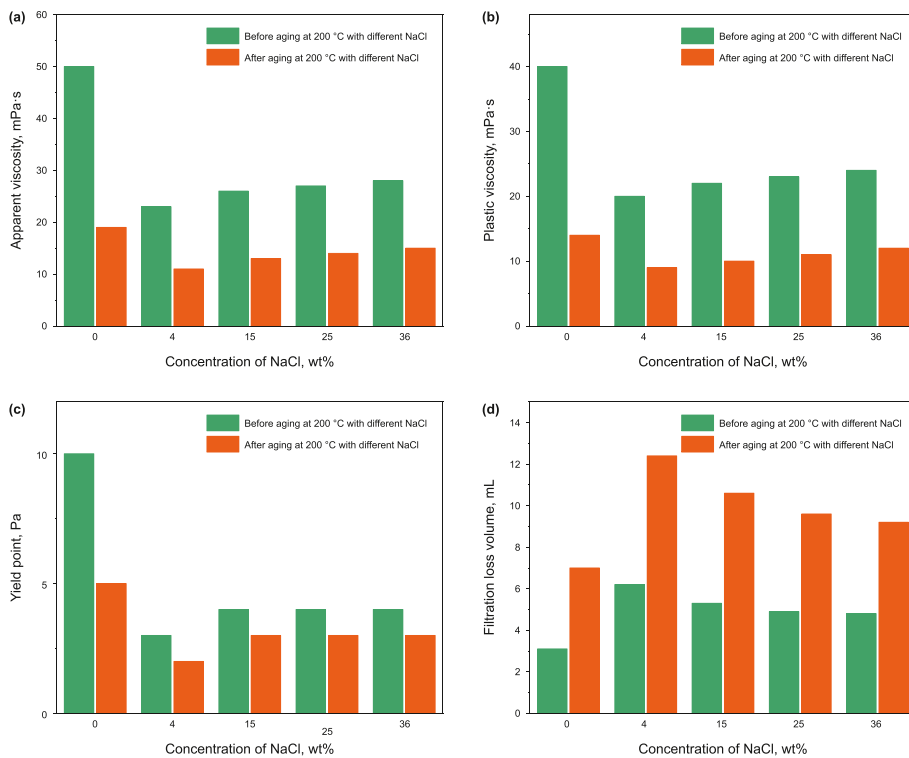


Fig. 5. Influence of salt (NaCl) contamination on the 2 wt% DADC copolymer-based drilling fluids: (a) AV; (b) PV; (c) YP; (d) filtration loss volume (FL<sub>API</sub>).

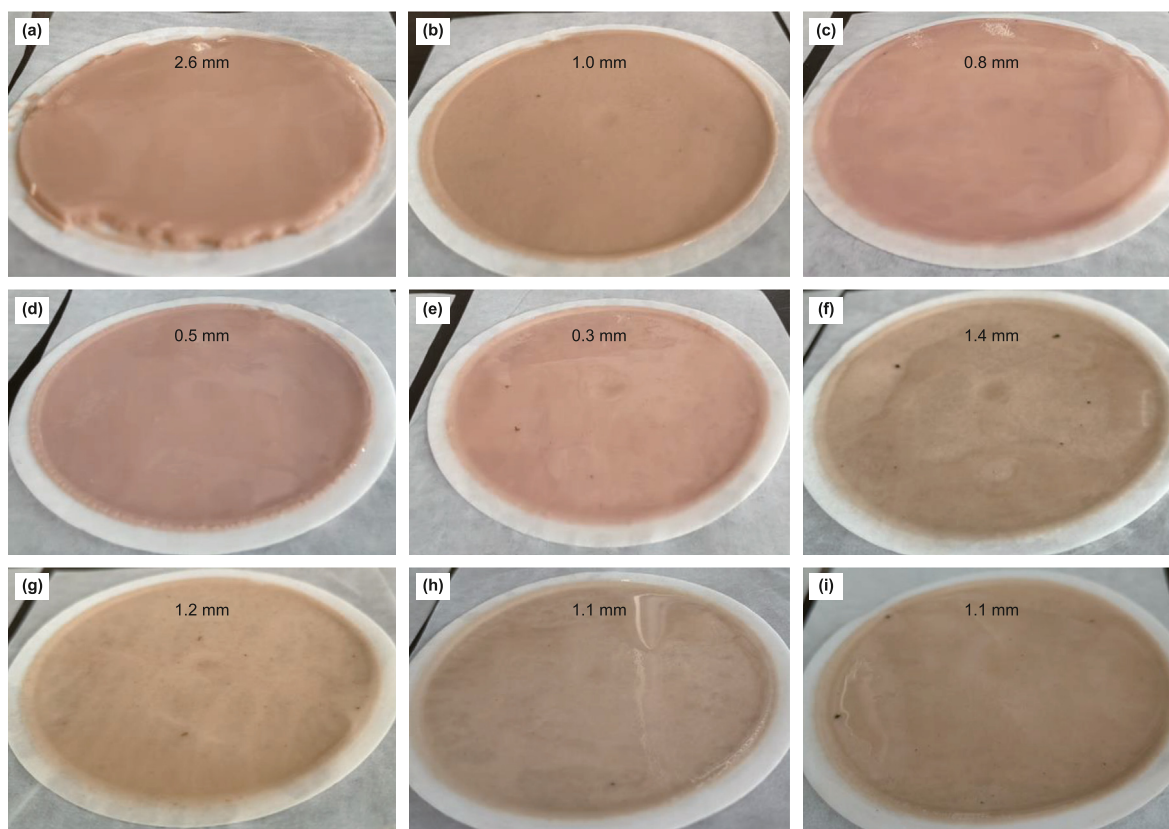
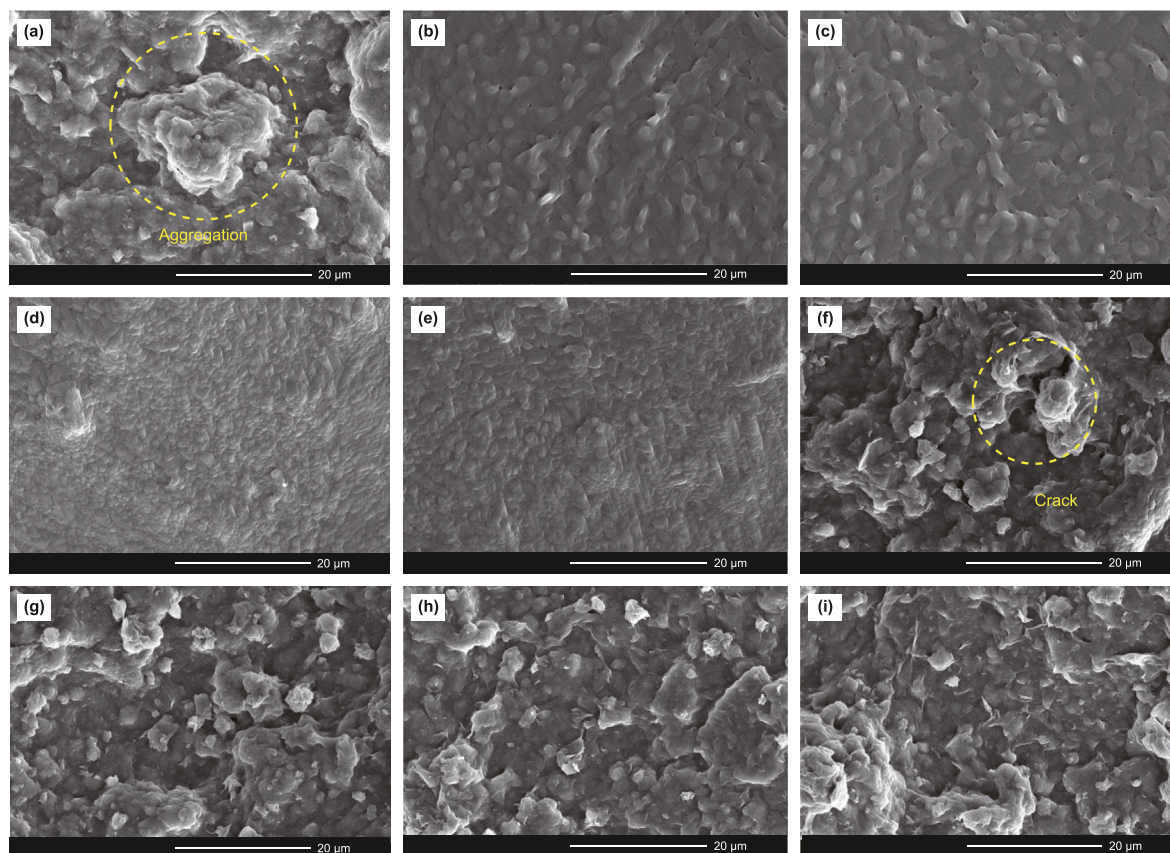


Fig. 6. Digital images of mud cakes deposited from base slurry (with different content DADC copolymer) after aging at 200 °C: (a) 0%; (b) 0.5 wt%; (c) 1.0 wt%; (d) 1.5 wt%; (e) 2.0 wt%; (f) 2 wt% + 4 wt% NaCl; (g) 2 wt% + 15 wt% NaCl; (h) 2 wt% + 25 wt% NaCl; (i) 2 wt% + 36 wt% NaCl.





**Fig. 7.** SEM images of mud cakes deposited from base slurry (with different content DADC copolymer) after aging at 200 °C: (a) 0%; (b) 0.5 wt%; (c) 1.0 wt%; (d) 1.5 wt%; (e) 2.0 wt%; (f) 2 wt% + 4 wt% NaCl; (g) 2 wt% + 15 wt% NaCl; (h) 2 wt% + 25 wt% NaCl; (i) 2 wt% + 36 wt% NaCl.

Herein, the adsorption capacity of DADC copolymer on the surface of bentonite was calculated by Eq. (4). In addition, zeta potential was used to characterize the colloidal stability, and the median particle size ( $D_{50}$ ) represented the particle size fraction of the drilling fluids.

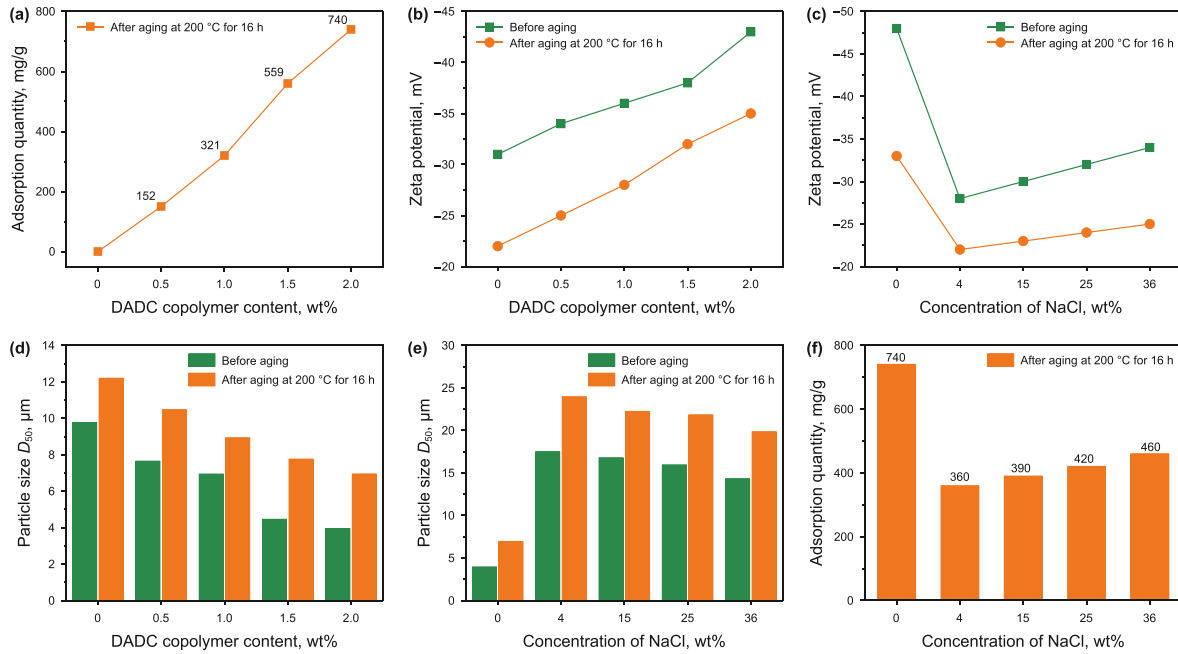
As shown in Fig. 8(a), the adsorption quantity increased with the DADC copolymer concentration. As a result, the anionic groups (sulfonate) of DADC copolymer endow bentonite particles with more negative charges, thus increasing the absolute value of the zeta potential of drilling fluids shown in Fig. 8(b). In addition, the drilling fluids' median particle size gradually decreases shown in Fig. 8(d). When the drilling fluids with 2 wt% DADC copolymer aging at 200 °C for 16 h, the adsorption quantity could reach 740 mg/g. The absolute value of zeta potential was 35 mV (higher than the 22 mV of the base slurry), demonstrating the DADC copolymer could improve repulsive force between the bentonite particles as well as the thermodynamic stability of drilling fluids in high-temperature environments.

Since the positively charged sodium ions could neutralize the anionic groups in the polymer as well as the negative charges on the surface of bentonite, the adsorption quantity of the polymer would reduce, thus resulting in the aggregation of bentonite particles. As demonstrated in Fig. 8(c), (e), and (f), when the drilling fluids with 2% DADC copolymer were contaminated with 4 wt% NaCl, the adsorption quantity and the absolute value of the zeta potential significantly decreased while the median particle size increased, confirming the electrolyte resulting in the aggregation of bentonite particles and damaging the colloidal stability of the drilling fluids. However, as the concentration of NaCl further increased, the gradual opening of the “inner salt bonds” enhanced

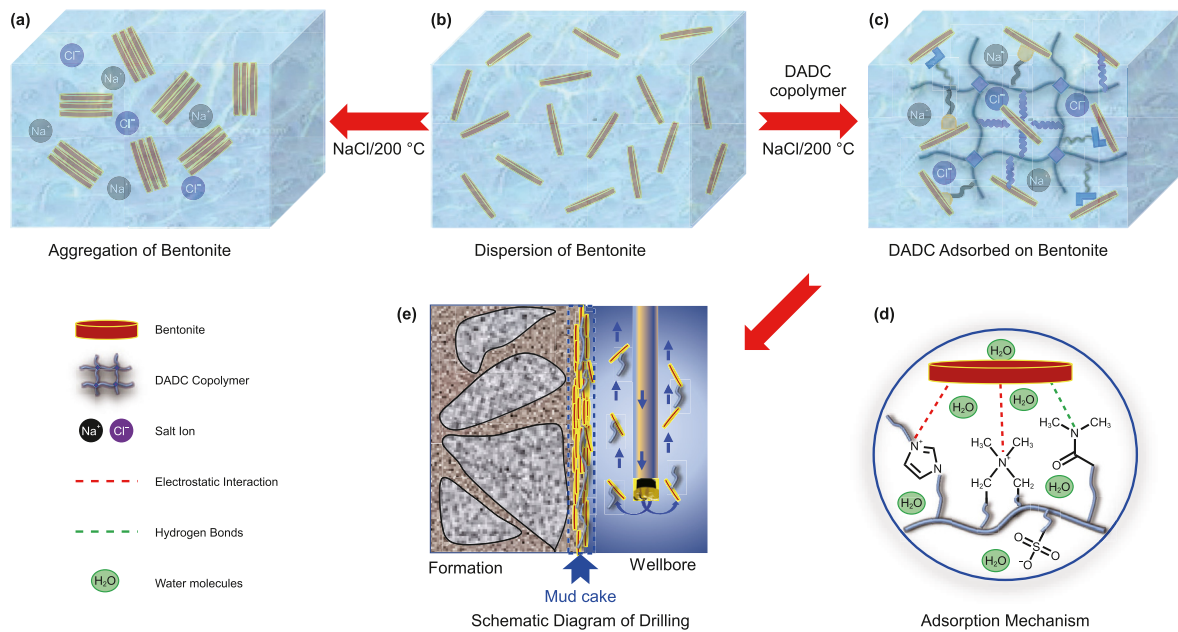
the ductility and the adsorption ability of DADC copolymers in high concentration salt environment, thus the absolute value of the zeta potential slightly increasing while the particle size decreasing (Li et al., 2022a; Tan et al., 2021). Even after aging in a saturated salt environment at 200 °C, the absolute zeta potential of the drilling fluid was 25 mV, and the median particle size of  $D_{50}$  was 20 μm, indicating that DADC copolymer could still effectively maintain the uniform dispersion of bentonite particles.

### 3.3.3. Mechanism of DADC copolymer

Based on the above experimental results, the mechanism of DADC copolymer regulating rheology and filtration of drilling fluids under high temperature and high salinity environments was demonstrated as shown in Fig. 9. Usually, bentonite had a specific negative charge on its surface due to lattice substitution, which promoted the dispersion of bentonite particles in freshwater under electrostatic repulsion shown in Fig. 9(b) (Li et al., 2018). However, after the addition of electrolytes, metal cations with high-density positive charges diffused near the bentonite and adsorb on the surface of the bentonite particles, disrupting the electrostatic repulsion formed by negative charges between the bentonite particles, leading to the aggregation and flocculation of particles (Cao et al., 2017; Sun et al., 2021b). Meanwhile, the high temperature could simultaneously remove some hydroxyl groups and aluminum atoms in the aluminum octahedron, decreasing negative charges on the surface of bentonite particles, thereby causing aggregation of bentonite particles (Liu et al., 2022b). As a result, both of the above effects would cause the bentonite particles to aggregate shown in Fig. 9(a), the quality of mud cake to deteriorate, the rheological properties to deteriorate, and the filtration loss to increase sharply.



**Fig. 8.** (a) Adsorption quantity of DADC copolymer on bentonite particles; (b) Zeta potential values of drilling fluids with different contents of DADC copolymer; (c) Zeta potential values of 2 wt% DADC copolymer-based drilling fluids contaminated with different concentration of NaCl; (d) The particle size of drilling fluids with different contents of DADC copolymer; (e) The particle size of 2 wt% DADC copolymer-based drilling fluids contaminated with different concentration of NaCl; (f) Adsorption quantity of 2 wt% DADC copolymer-based drilling fluids contaminated with different concentration of NaCl.



**Fig. 9.** Schematic illustration of the mechanism of DADC copolymer in enhancing the rheology and filtration performance of the drilling fluids.

Nevertheless, as shown in Fig. 9(c) and (d), when the DADC copolymer was added to drilling fluids, the copolymer could effectively adsorb on the surface of bentonite particles through the strong electrostatic interaction and hydrogen bonds, thus forming a protective layer on the surface of bentonite particles. Based on the micro-crosslinking and rigid ring effect within the copolymer, it endowed the copolymer with excellent temperature resistance and improved the comprehensive performance in high-temperature environments (Li et al., 2023). In addition, the opening of the

“internal salt bonds” of copolymers in high-concentration saline environments increased the adsorption of copolymers on the surface of bentonite particles, thus hindering the aggregation of particles (Tan et al., 2021). Moreover, the hydrophobic association structure endowed the copolymer with excellent temperature and salt resistance and enhanced the spatial grid structure of drilling fluids (Sun et al., 2021b). During the drilling process, under the pressure difference between the static liquid column pressure of the drilling fluids and the formation pressure, the drilling fluids

**Table 1**  
Rheological and filtration performance of the drilling fluids.

Polymer	Aging time, h	AV, mPa·s	PV, mPa·s	YP, Pa	YP/PV, Pa/mPa·s	FL <sub>API</sub> , mL	FL <sub>HTHP</sub> , mL
DADC	0	96	72	24	0.33	0.5	–
	16	89	73	16	0.22	2	14
Driscal-D	0	113	88	25	0.28	0.5	–
	16	60.5	52	8.5	0.16	7.6	32

formed a thin and tough mud cake between the wellbore and the formation shown in Fig. 9(e), reducing the filtration loss. Overall, the multiple effects of DADC copolymer, including hydrogen bonds, electrostatic interaction, hydrophobic association, and micro-crosslinking, provided excellent rheology, filtration, and temperature- and salt-resistance to the drilling fluids.

### 3.3.4. Development of temperature- and salt-resistance drilling fluids

The complex geological conditions of ultra-high temperature and ultra-high salinity in deep formations posed a huge challenge to the performance of water-based drilling fluids. The saturated saltwater drilling fluids with ultra-high temperature resistance were constructed based on the DADC copolymer and other additives. The specific formulation was: 300 mL base slurry +5 wt% calcium carbonate +3 wt% DADC +4 wt% polymeric alcohol +2 wt% white oil +1 wt% emulsifier +3 wt% polyester lotion +36 wt% NaCl + barite, with the density being  $\rho = 2.0 \text{ g/cm}^3$ . In addition, the drilling fluids based on common high-temperature resistant Driscal-D polymer were used as control while keeping other treatment agents unchanged. Table 1 showed that the plastic viscosity of the drilling fluids based on DADC copolymer remained unchanged before and after aging at 200 °C, while the apparent viscosity slightly decreased. The dynamic shear force and the ratio of dynamic shear force to plastic viscosity were decreased, but all remained within a reasonable range. Specifically, the API of the saturated saline drilling fluids based on DADC copolymer aged at 200 °C was 2 mL, and the high-temperature and high-pressure filtration loss was 12 mL, demonstrating excellent filtration performance. In contrast, the saturated saline drilling fluids based on Driscal-D polymer exhibited a sharp decrease in viscosity/shear force and a sharp increase in filtration loss after aging at 200 °C, with a FL<sub>HTHP</sub> of up to 32 mL, which could not meet the requirements for safe drilling in ultradeep wells. Therefore, the above results indicated that DADC copolymer had excellent compatibility with other additives and could be used to construct high-temperature and high salt resistant drilling fluids, thus meeting the requirements of ultradeep well drilling operations.

## 4. Conclusions

In this work, a micro-crosslinked amphoteric hydrophobic association copolymer (DADC) as the temperature and salt-resistant fluid loss reducer was synthesized to enhance the performance of WBDFs. The basic physicochemical properties of DADC copolymer were characterized through NMR, FTIR, TGA, and rheological experiments. The rheological and filtration reducing performance of DADC copolymer controlled base slurry in high-temperature and high-salt environments have also been evaluated through API standards. The mechanism of action of the DADC copolymer was proposed based on experimental results such as scanning electron microscopy, potential, and particle size. Furthermore, the saturated saltwater drilling fluids with ultra-high temperature resistance were constructed using the DADC copolymer. Several specific conclusions from the results were as follows.

- (1) The TGA experiment showed that the DADC copolymer had good thermal stability with the initial decomposition temperature as high as 260 °C, demonstrating its potential for application in high-temperature-resistant drilling fluids.
- (2) Due to the anti-polyelectrolyte and hydrophobic association effects, the DADC copolymer solution exhibited good shear dilution and a certain degree of salt-responsive viscosity increasing performance.
- (3) DADC could significantly improve the rheological and filtration performance of the base slurry in high-temperature and high-salt environments. For the drilling fluids (with 2 wt% DADC copolymer), after aging at 200 °C, the AV increased from 4 to 17 mPa·s, the PV increased from 2 to 13 mPa·s, the YP increased from 2 to 4, while the FL<sub>API</sub> decreased from 36 to 7 mL compared with base slurry.
- (4) The adsorption capacity, zeta potential, and particle size distribution of drilling fluids further confirmed the DADC copolymer could effectively adsorb onto bentonite particles and inhibit the aggregation of particles in high-temperature and high-salt environments.
- (5) The drilling fluids constructed based on DADC copolymer had excellent rheological and filtration reducing properties. After aging at 200 °C, the FL<sub>API</sub> of the drilling fluids was 2 mL, and the FL<sub>HTHP</sub> filtration loss was 12 mL, demonstrating good potential applications in deep or ultradeep drilling engineering.

### CRedit authorship contribution statement

**Jian Li:** Conceptualization, Funding acquisition, Resources, Supervision. **Yu-Xi Ji:** Conceptualization. **Xiao-Xiao Ni:** Formal analysis. **Kai-He Lv:** Investigation. **Xian-Bin Huang:** Methodology. **Jin-Sheng Sun:** Investigation, Resources.

### Declaration of competing interest

We declare that we have no financial or personal relationships with others or organizations that can inappropriately influence our work. No professional or other personal interest in any product, service, or company could be construed as affecting the position presented in, or the review of, the manuscript entitled.

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