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Effect of Cycloaliphatic Amine on the Shale Inhibitive Properties of Water-Based Drilling Fluid

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Abstract: In order to improve the inhibitive properties and high temperature resistance of shale inhibitor, cycloaliphatic amines were introduced as shale hydration inhibitors in water-based drilling fluids. Bulk hardness test, shale cuttings dispersion test, bentonite inhibition test and water adsorption test were carried out to characterize the inhibitive properties of the novel amines. Surface tension measurement, zeta potential measurement, XRD, contact angle test, SEM and TGA were performed to investigate the interaction between the cycloaliphatic amines and clay particles. The results indicated that cycloaliphatic amines exhibited superior inhibitive properties to the state of the art inhibitors. Moreover, the amines were high temperature resistant. The hydrophobic amine could intercalate into the clay gallery with monolayer orientation. The protonated ammonium ions neutralized the negatively charged surface. After adsorption, the hydrophobic segment covered the clay surface and provided a shell preventing the ingress of water.

Keywords: Clay hydration and swelling, cycloaliphatic amine, high temperature, inhibitive property, mechanism.

1. INTRODUCTION

Drilling in shale formations has been a challenge for a long time due to its strong potential for wellbore instability [1]. While drilling a shale formation, water in a water-based drilling fluid can filtrate out of the mud and invade the shale. The invaded water can interact with the smectite and other clay minerals in the shale and cause shale instability issues, such as swelling, dispersing, delaminating and fracturing [2].

Oil-based drilling fluids have been the fluid of choice for challenging hole sections like active shale formation for many years because of many advantages, including optimal shale stability, high temperature stability, good resistance to contamination, and excellent lubricity and so on. However, the concern about the increasing level of environmental regulation limits their wide use. Then people render to seek a high performance water-based drilling fluid that performs like oil-based drilling fluid especially with regard to shale stability, and also is environmentally acceptable [3-5].

In order to replace oil-based drilling fluid, the inhibitive property of water-based drilling fluid is of vital importance. During the past decades, extensive work has been carried out to evaluate and find solutions to mitigate and control the interaction between shale formations and drilling fluid [6], and many of the chemical inhibitor additives including potassium, sodium and calcium salts, PHPA, polyglycols, silicates, amine compounds and formates have been developed to improve the inhibition comparable to the oil-based drilling fluids [7, 8].

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Conventionally, various kinds of salts are added to the drilling fluids to control reactive clays. Among these inhibitors, potassium chloride has been the most widely applied type of salt for many years. However, the salt affects the biological ecosystems when a large amount is used. Even at certain circumstance, potassium chloride will promote the swelling of kaolinite. To combat the performance related issues and environmental impact, most recent research focused on the development of organic compounds. Amine compounds are introduced as alternative of potassium to suppress shale hydration. Polyether amines were designed and applied as shale inhibitors in the early 2000s [9].

The diamines, with uniquely designed molecular structure, have been evaluated to be excellent clay swelling inhibitors. Based on the inhibitors, a high performance water-based drilling fluid was established and obtained successful application around the world [10-13]. However, the recorded application temperature of polyether amine was less than 150°C. With the increase of well depth, high temperature resistant shale inhibitor is required.

With the assistance of molecular modeling technology, the underlying mechanism at the molecular level can be addressed, and some "rule-based" criteria for designing efficient clay swelling inhibitors have been proposed [14]. According to these rules, novel cycloaliphatic amine compounds were introduced as shale hydration inhibitive agent to stabilize shale and improve high temperature resistance [15].

In this paper, several experimental evaluation methods including bulk hardness test, shale cuttings hot-rolling dispersion test, bentonite inhibition test and water adsorption test were adopted to evaluate the inhibitive properties of the novel cycloaliphatic amine compounds. To understand the mechanism of the compounds, surface tension measurement,

zeta potential measurement, XRD, contact angle test, SEM and TGA were carried out to investigate the interaction between cycloaliphatic amine and clay.

2. EXPERIMENTAL PART

2.1. Materials

The sodium montmorillonite (Na-MMT) was obtained from Xia Zijie Bentonite Technology Co., LTD. The chemical composition of the sample was: SiO_2, 64.07%; Al_2O_3, 19.11%; CaO, 4.48%; MgO, 3.61%; Na_2O, 3.07%; Fe_2O_3, 2.64%, P_2O_5, 1.71%; K_2O, 0.72%. The cationic exchange capacity was measured to be 95 mmol/100g by ammonium acetate method. Drilling fluid bentontie was provided by Weifang Huawei Bentonite Group Co., Ltd, China, following the API standard. Cyclohexylamine (CHA), methylcyclohexanamine (MCHA) and N, N-Dimethylcyclohexylamine (DMCHA) were purchased from Sigma-Aldrich Co., LTD. Ultrahib, as an excellent shale inhibitor, was obtained from M-I SWACO, a Schlumberger company of USA. KCl was provided by Sinopharm Chemical Reagent Co., Ltd, China. All the reagents were used as received without further purification.

2.2. Inhibitive Evaluation Methods

2.2.1. Bulk Hardness Test

The bulk hardness test was used to determine the hardness of the shale sample after exposure to a test fluid. The continued increasing hardness of the shale under test conditions indicated the effect of the shale inhibitor on reducing the clay's tendency to imbibe water from the aqueous environment of the drilling fluid [16]. In this test, the shale cuttings were hot rolled in the testing fluids at 77°C for 16 hours. After that the cuttings were washed with water and then placed into the bulk hardness tester (Fig. **1**). The cuttings were extruded through a perforated steel plate utilizing the bulk hardness tester. The force required for extrusion was recorded as bulk hardness. All tests began with the same amount of shale [17]. Depending upon the hydration of the cuttings, the torque may reach a plateau region or may continue to rise during the extrusion. The harder the cuttings was, the higher torque reading would be [18, 19]. The cuttings were obtained from Sha Hejie formation of Shengli oilfield, which were mainly composed of clay minerals.

2.2.2. Cuttings Hot-Rolling Dispersion Test

This test involves exposing a weighed quantity of sized shale pieces to a formulated fluid in a conventional rolling oven cell. Volume of 350 mL of solution with inhibitors of various concentrations and 50g of shale cuttings (2-5 mm) obtained from the upper layer of Shahejie formation in Dagang oil field were added into sealed cells. After hot rolling at 77°C for 16h, the cuttings were washed with 10% KCl solution and screened through 40-mesh sieve. The recovered cuttings were dried at 105°C for 4 hours. The moisture content of the shale and the percentage recovery of the shale were determined [20].

Fig. (1). Bulk hardness tester.

2.2.3. Bentonite Inhibition Test

Volume of 400 mL of water containing 12 g inhibitor was treated by 5w/v% drilling fluid bentonite. After stirring for 30min, the dispersion was hot rolled at 70°C for 16h. Then the rheological properties were measured as the samples were cooled to room temperature. After that equivalent amount of drilling fluid bentonite was added and the procedure was repeated until the dispersion became too viscous to be measured.

2.2.4. Water Adsorption Test

The procedure of water adsorption test was illustrated in the following section.

2.3. Interaction Between MCHA and Montmorillonite

MCHA with a range of concentrations was added into 2w/v% prehydrated Na-MMT dispersions and shaken for 24 h to reach adsorption equilibrium. The dispersion was centrifuged and washed with deionized water several times. One part of the sediment was prepared for XRD measurements directly. The other part was dried at 105°C and ground to powder for XRD measurements, water adsorption test, SEM and TGA. The test procedure of zeta potential was reported previously and measured at room temperature using the Zetasizer 3000 electric potential and granularity meter (Malvern instrument, United Kingdom) [21]. The water adsorption test was as follows, modified MMT powders (1 g) was placed in a sealed glass desiccator with water in the bottom. The amount of adsorbed water was calculated from the mass gain. XRD analysis was performed by using an X'pert PRO MPD diffractometer with Cu target at a generator voltage of 45 kV, current of 50 mA. Samples are measured scanning 2θ angle from 2° to 15°. The basal spacing is analyzed by using Bragg's equation. The value for n=1 is calculated from $2d\sin\theta=n\lambda$. Contact angle test was carried out with a contact angle measurement HARKE-SPCA (Beijing Harke Testing Equipment Factory). MCHA with a sequence of concentration was incorporated into 2 wt% Na-MMT dispersion. After reaching adsorption equilibrium, the dispersions were coated on the surface to

glass slides following the procedures described by Wu [22]. The glass slides were left air dried. Contact angles were determined with the static sessile drop method to characterize the hydrophilic or hydrophobic property of testing materials using digital camera equipped on contact angle tester. The measurement was performed with distilled water [23]. SEM was observed using a Hitachi S-4800 scanning electron microscope to study the morphological features of the powdered samples. Thermal gravimetric analysis (TGA) was performed on a WCT-2D (Beijing Optical Instrument Factory) instruments thermal analyzer at a scan rate of 20°C/min under nitrogen flow. The solution surface tension was measured at 25°C with DCAT21 surface/interface tensiometer (Beijing Eastern-Dataphy Instruments Co., Ltd).

3. RESULTS AND DISCUSSION

3.1. Inhibitive Properties Evaluation

3.1.1. Bulk Hardness Test

Fig. (**2**) presents the inhibition variance of different shale inhibitors. The cuttings, mainly composed by clay minerals, would easily disperse into small particles and became soft after adsorbing water. An effective shale inhibitor can suppress the hydration and dispersion of cuttings, contributing to a high shale cuttings recovery and high hardness. As shown in Fig. (**1**), the torque increased gradually after a few turns for inhibitor systems, while more turns need for fresh water system. The higher slope of the curve showed, the harder the recovered cuttings were. The slope sequence of the curves were as follows, MCHA> Ultrahib > CHA> DMCHA > KCl. In this case the results showed that MCHA performed better than other inhibitors, while CHA and DMCHA also exhibited excellent results.

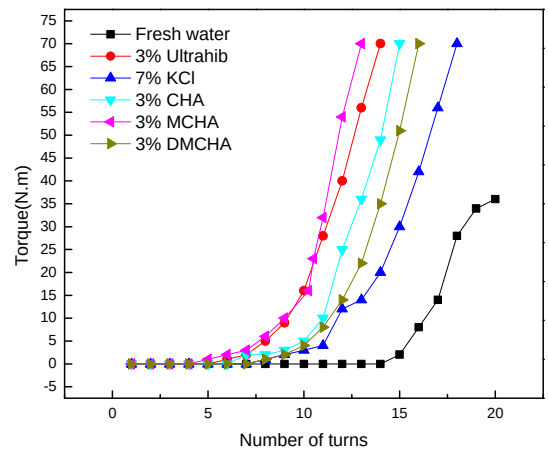


Fig. (2). Bulk hardness test comparing the torque readings on shale cuttings from various inhibitor systems.

3.1.2. Shale Cuttings Hot-Rolling Dispersion Test

As shown in Fig. (**3**), shale cuttings recovery was virtually low in fresh water. At low temperature of 77°C, the shale recovery of fresh water was 25.98%. After incorporating shale inhibitors, the shale recovery reached 79.5%, 85.2%, 82.53%, 87.6% and 84.27% for KCl, Ultrahib, CHA, MCHA and DMCHA respectively. CHA, MCHA and DMCHA exhibited higher level of inhibition than potassium chloride and Ultrahib. The shale cuttings recovery decreased with the increase of temperature. For one hand, the increase of temperature aggregates the shale hydration. For the other

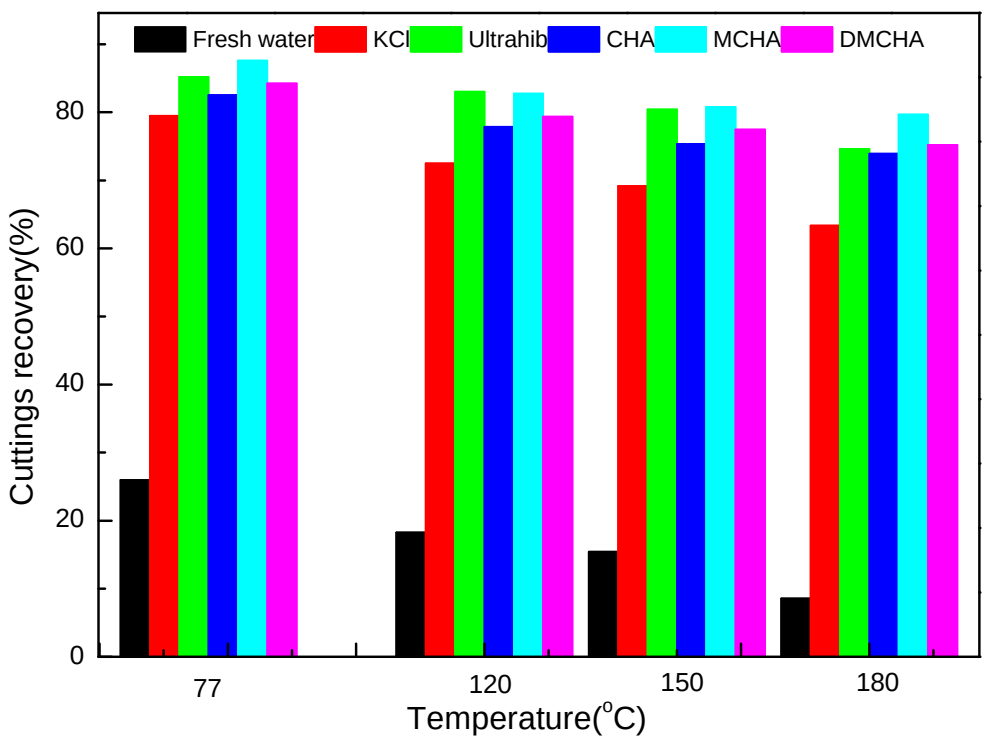


Fig. (3). Shale cuttings recoveries of different inhibitors at different temperatures.

hand, the adsorption of inhibitors, and especially several polymers onto the clay surface will become weak, which leads to the reduced inhibition effect. However, the shale cuttings recovery of CHA, MCHA and DMCHA was still high compared with that of low temperature, demonstrating that CHA, MCHA and DMCHA performed well at high temperatures.

3.1.3. Bentonite Inhibition Test

The bentonite inhibition test method was used routinely as a screening method to determine the ability of a product to prevent bentonite from swelling and maintain a low rheological profile. This test method was designed to simulate the incorporation of highly active drilled solids into a drilling fluid similar to what occurs while drilling water-sensitive shale in the real-time field application [24].

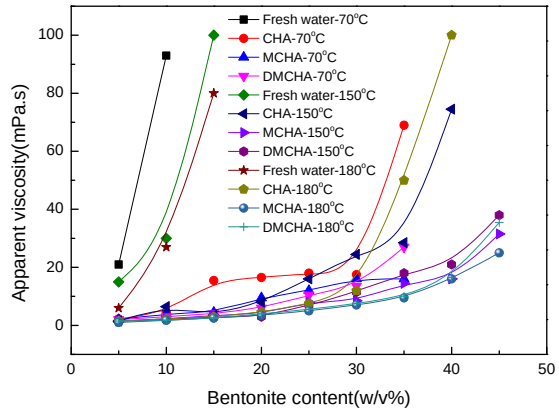
As depicted in Fig. (**4**), when the testing temperature was 70°C, the apparent viscosity and yield point value for the dispersion without inhibitor increased abruptly after addition of 10 w/v% bentonite, whereas for the dispersions with inhibitors, the apparent viscosity and yield point value increased abruptly merely at higher bentonite concentrations, corresponding to 30w/v%, 40w/v% and 35w/v% for CHA, MCHA and DMCHA respectively. The results demonstrated that cycloaliphatic amine exhibited high performance in inhibiting the hydration and swelling of bentonite. With the increase of temperature, the dehydration of clay dominated, therefore the bentonite inhibition capacity for the inhibitors improved. It also could be seen that, the cycloaliphatic amines were effective at high temperatures, thus it could be used for high temperature shale inhibitors.

3.1.4. Water Adsorption Test

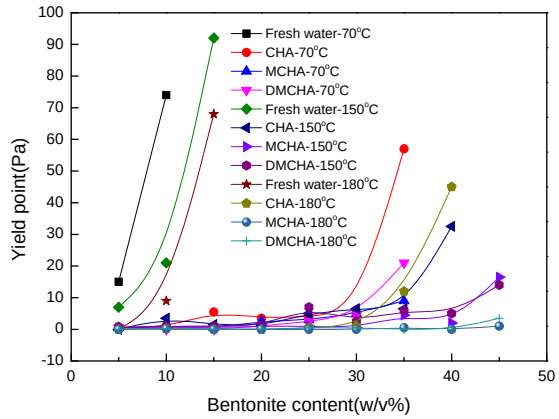
Water adsorption test was carried out to characterize the water affinity of clay surface after modification by MCHA. The water adsorption rate was recorded as a function of time. As depicted in Fig. (**5**), for the pure bentonite, the water adsorption rate increased significantly at the initial 48 hours, and then increased with a flatten rate. Compared with the pure bentonite, the water adsorption rate of modified bentonite was much lower, indicating the less hydrophilicity. The higher the concentration was, the lower water affinity of modified bentonite was obtained. However, the difference among the three testing samples was not noticeable, which was related to the adsorption of MCHA onto the clay. The results indicated that the interaction between MCHA and clay reduced the water affinity, which improved the stability of clay.

3.2. Properties in Water-Based Drilling Fluid

The properties of cycloaliphatic amine in water-based drilling fluid were evaluated. The fluid formulas were shown in Table **1**. The data in Table **2** clearly indicated the stability of the customized fluid before and after hot rolling. The apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) remained relatively stable after ageing at 120°C. Also of note, the API fluid loss changed little before and after hot rolling. Shale cuttings hot-rolling dispersion test was conducted to evaluate the inhibitive property of the two fluids. The shale recovery of the shale cuttings in fresh water was 5.05%, and increased to as high as 85.27% and 82.77% for the two fluids respectively, demonstrating highly inhibitive properties.



(a)



(b)

Fig. (4). Bentonite inhibition test comparing the rheological profile of different shale inhibitors (3%w/v) and the base fluid. Note: (**a**) Apparent viscosity; (**b**) Yield point.

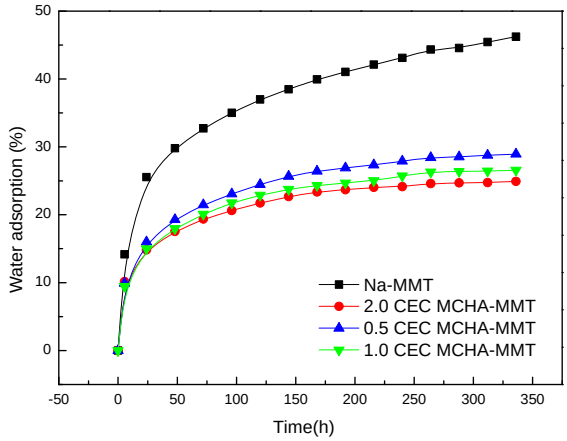


Fig. (5). Water adsorption rate as a function of time.

Table 1. Composition of the inhibitive fluids.

Inhibitive Fluid 1		Inhibitive Fluid 2	
Product	**Content**	**Product**	**Content**
Water	400mL	Water	400mL
Encapsulator	1.2g	Encapsulator	1.2g
Shale inhibitor (CHA)	12g	Shale inhibitor (MCHA)	12g
Polyanionic cellulose	4g	Polyanionic cellulose	4g
Xanthan gum	1.2g	Xanthan gum	1.2g
Superfine $CaCO_3$	20g	Superfine $CaCO_3$	20g
Lubricant	12g	Lubricant	12g

Table 2. Fluid properties of designed inhibitive fluids.

Fluid	Testing Condition	AV/mPa.s	PV/mPa.s	YP/Pa	FL/mL	6 Rev/Min	3 Rev/Min	Shale Cuttings Recovery/%
1	Before hot rolling	44.5	33	11.5	7.0	7	6	85.27
	After hot rolling	41.5	26	15.5	7.4	10	8	
2	Before hot rolling	44.5	34	10.5	8.0	8	7	82.77
	After hot rolling	42	27	15	8.0	9	7	

3.3. Inhibitive Properties Analysis

3.3.1 Surface Tension Measurement

MCHA generally consist of naphthenic group as the hydrophobic parts, as well as amine group as the hydrophilic portions. The balance between hydrophobic part and hydrophilic part rendered the compound amphiphilic in water. As shown in Fig. (**6**), the surface tension could be reduced to as low as 35 mN/m. The critical micelle concentration (CMC) was about 0.05 mol/L.

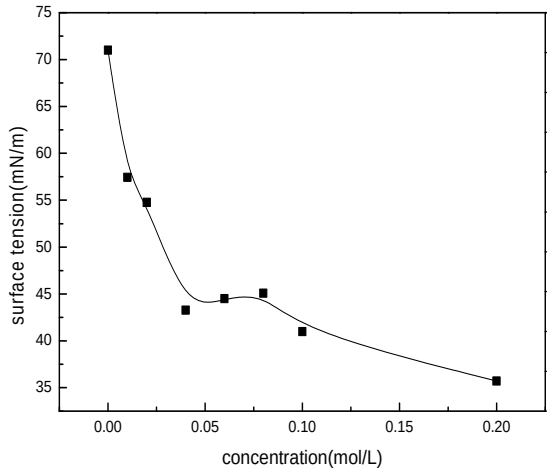


Fig. (6). Surface tension variation with concentration increase of MCHA.

For oil-based drilling fluids, pressure penetration into the shale pore space generally does not occur, mainly because of a high capillary entry pressure existing [25]. While for water-based drilling fluid, Zhang [26] *et al.* pointed out that, generally for a hydrophilic shale surface, a lower surface tension reduction would improve the shale stability because of the lower capillary suction pressure. Thus MCHA was capable of diminishing the suction of water into the shale pores.

3.3.2. Zeta Potential Measurement

A negative surface charge exists due to the isomorphous substitution in the octahedral layer for clay minerals, which is balanced by the exchangeable cations, typically sodium ions or calcium ions. The exchangeable cations can be replaced by other cationic ions, such as ammonium ions.

As shown in Fig. (**7**), when there was no inhibitor added into the bentonite dispersion, the zeta potential was -43mV, showing well dispersion. When the concentration of MCHA was 0.01mol/L, the zeta potential increased up to -28.9mV. Then there was slight change of zeta potential along with further increase of concentration, indicating that the adsorption of MCHA onto the clay surface reached saturation. It also demonstrated that MCHA adsorbed onto the clay surface with monolayer orientation, which would be verified in the following part of X-ray diffraction analyzing the interlayer space change of clay with the increase of MCHA concentration.

Amine group would be protonated when dissolved in aqueous solution [27]. The protonated ammonium ions neutralized the negatively charged clay surface and resulted in the increase of zeta potential. As the clay became less negatively charged, the trend of hydration and swelling decreased.

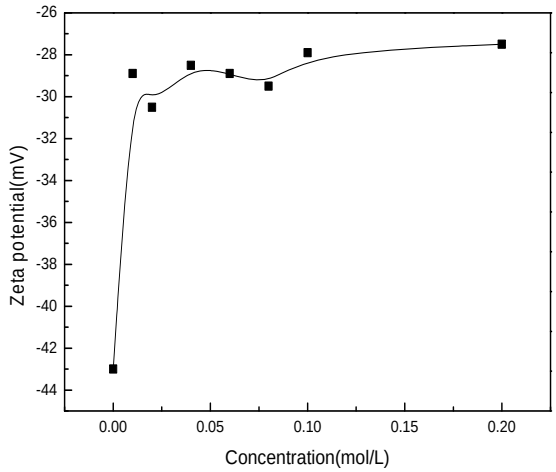


Fig. (7). Zeta potential variations as a function of MCHA concentrations.

3.3.3. XRD

The clay interlayer space after intercalation by MCHA was characterized by XRD, as shown in Fig. (**8**) and Fig. (**9**). When the concentration of MCHA was 0.01mol/L, the interlayer space increased from 1.19nm to 1.34nm. With the further increase of concentration, the interlayer space increased slightly, suggesting a monolayer arrangement at the clay surface, which was also observed in Fig. (**9**) for the wet samples.

As displayed in Fig. (**9**), the interlayer space of clay with wet sample was 1.95nm. The increase from 1.19 nm for dry sample to 1.95nm for wet sample was mainly affected by the ingress of water into the interlayer. However, the intercalation of MCHA reduced the interlayer space dramatically at low addition. In the process of intercalation, the protonated ammonium ions exchanged the hydrated sodium ions and their associated hydration shells, resulting in the collapse of the clay gallery. Thus the interlayer space decreased greatly and the hydration and swelling of the clay was reduced. It could also be seen that MCHA played an inhibitive role with very low concentration.

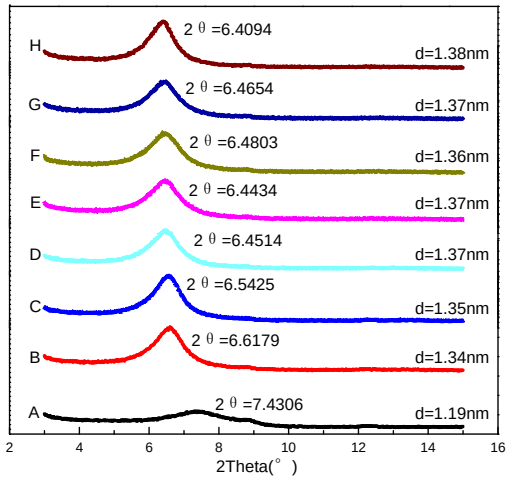


Fig. (8). X-ray diffraction patterns of MMT-MCHA samples at various concentrations (mol/L) with dry sample. Note: (A):0; (B):0.01; (C):0.02; (D): 0.04; (E):0.06; (F):0.08; (G): 0.1; (H):0.2.

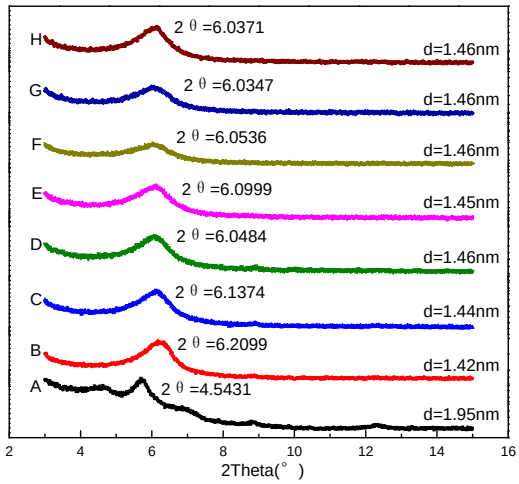


Fig. (9). X-ray diffraction patterns of MMT-MCHA samples at various concentrations (mol/L) with wet sample. Note: (A):0; (B):0.01; (C):0.02; (D): 0.04; (E):0.06; (F):0.08; (G): 0.1; (H):0.2.

3.3.4. Contact Angle Test

As shown in Figs. (**10**, **11**), the contact angle for water was 21.33^{o} measured on smectite film. The hydrophilic properties of MMT decreased after modification by MCHA. When the concentration of MCHA was 0.01mol/L, the contact angle increased to 35.35^{o}. Then the contact angle increased flatly with the further increase of content. It could be seen that, the contact angle as a function of concentration corresponded to the Langmuir type adsorption isotherms. When the concentration of MCHA reached adsorption saturation, the clay particles surface were covered by MCHA molecules, forming a hydrophobic shell which prevented the adsorption of water on the clay surface.

3.3.5. SEM

SEM was used to observe the microstructure and morphology of the Na-MMT and MMT modified with MCHA (Fig. **12**). The SEM photograph of Na-MMT illustrated the distinct laminate layer and non-aggregated structure. After intercalation with MCHA, the plate-like particles were stacked together in a disorder pattern to form loose and irregular agglomerates in some degree. The particle size became larger with slightly curled or crumpled edges. Also it could be seen, there were well-marked pores in the composite. The changes in the morphologies and particle size indicated that the intercalation was accompanied with adsorption [28]. The larger particle size and smaller surface of clay reduced the chance of contact with water, which was favorable to clay stabilization.

3.3.6. TGA

Thermogravimetric analysis was used to probe the thermal stability of organic clay and the microenvironment of intercalated organic molecules. For organic modified montmorillonite, several decomposition steps could be seen from the derivative weight loss curves. Generally, montmorillonite contained water to some extent, typically including adsorbed water and crystalline water. The total weight loss ranging from ambient temperature to 200°C could be attributed to the loss of adsorbed water [29].

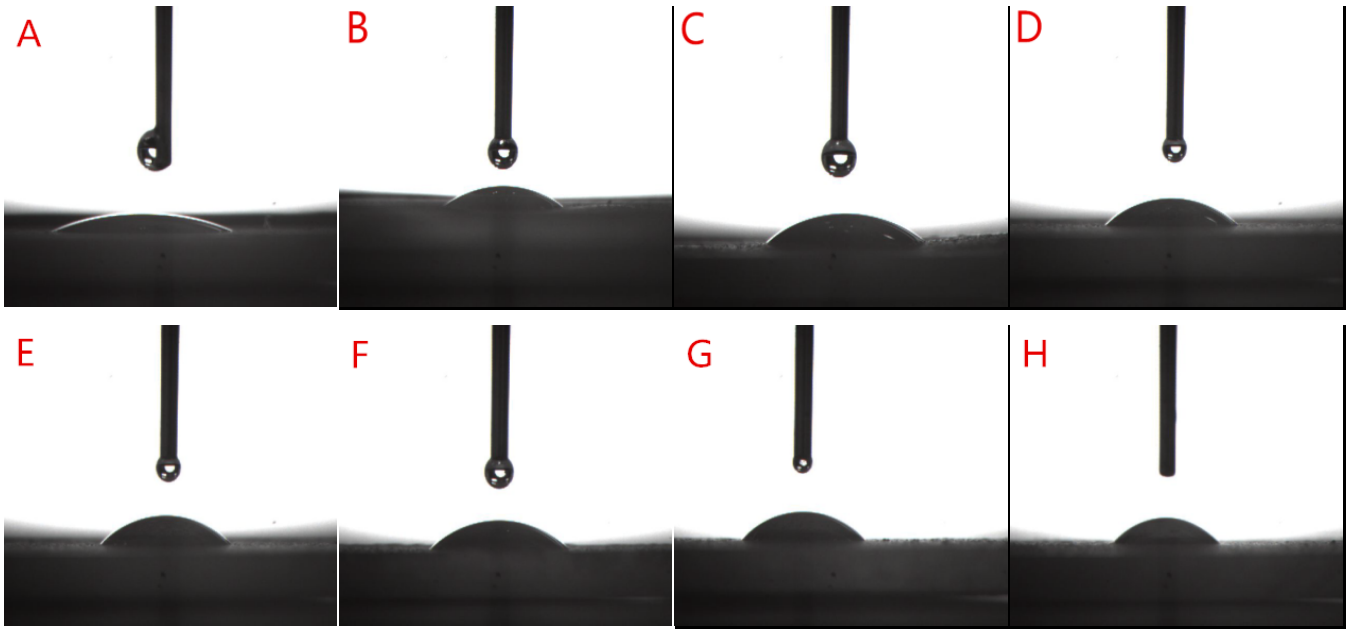


Fig. (10). Contact angle between water and modified bentontie with various concentrations (mol/L) of MCHA. Note: (**A**):0; (**B**):0.01; (**C**):0.02; (**D**): 0.04; (**E**):0.06; (**F**):0.08; (**G**): 0.1; (**H**):0.2.

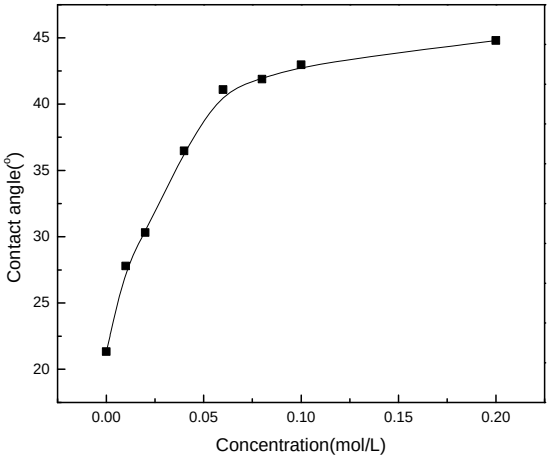


Fig. (11). Variation of contact angle with concentration of MCHA.

As depicted in Fig. (**13**), the decomposition between ambient temperature and 200°C for montmorillonite and MCHA-MMT varied greatly. For Na-MMT, the weight loss rate was 14.26%, while the weight loss of MMT modified by MCHA with concentration of 0.5CEC, 1.0CEC and 2.0CEC was 5.64%, 6.76% and 7% respectively. The result indicated that the intercalation of MCHA expelled the adsorbed water in the interlayer of clay, which was beneficial to the stability of clay.

3.3.7. Inhibitive Mechanism Analysis

There is a dynamic dissociation balance for amine groups in aqueous solution. When dissolved in water, the amine groups will be protonated into ammonium ions. In the process of interacting with clay, the protonated ammonium ions will neutralize the negatively charged clay surface, resulting in the reduction of hydration potential. Because the concentration of ammonium ions increases with the decrease of pH value, the inhibitive property of MCHA will be improved when the pH value was reduced to some degree. On the other hand, after adsorption, the hydrophobic segments, cyclohexane, which do not interact with the charged surface, staying at the surface of the clay, forming a hydrophobic film and preventing the entrance of water. The combined effect of the hydrophilic amine groups and the hydrophobic naphthenic groups weaken the hydration of clay. Because of the rigid structure of the cycloaliphatic amine, high temperature stability could be obtained.

CONCLUSION

Cycloaliphatic amines were chosen as potential shale hydration and swelling inhibitors based on the rules of molecular structure design. Based on the above tests and analyses, the following conclusions could be drawn.

The inhibitive evaluation experiments indicated that cycloaliphatic amines suppressed shale hydration and swelling effectively, superior to conventional potassium salt and commercial polyamine inhibitor. The cycloaliphatic amines were high temperature stable, which would be used in deep well drilling with high temperatures. The drilling fluid system evaluation test indicated that cycloaliphatic amine was compatible with common drilling fluid additives.

When interacting with clay, the cycloaliphatic amine was able to intercalate into the interlayer of clay with monolayer orientation with the driving force of electrostatic attraction between the protonated ammonium groups and the negative charge of clay. After intercalation, the clay surface was covered with the hydrophobic part of cyclohexane, which further prevented the adsorption of water. Therefore the combined effect contributed to the outstanding performance. The cycloaliphatic amine represented a new and significant improvement over state of the art shale inhibitors and

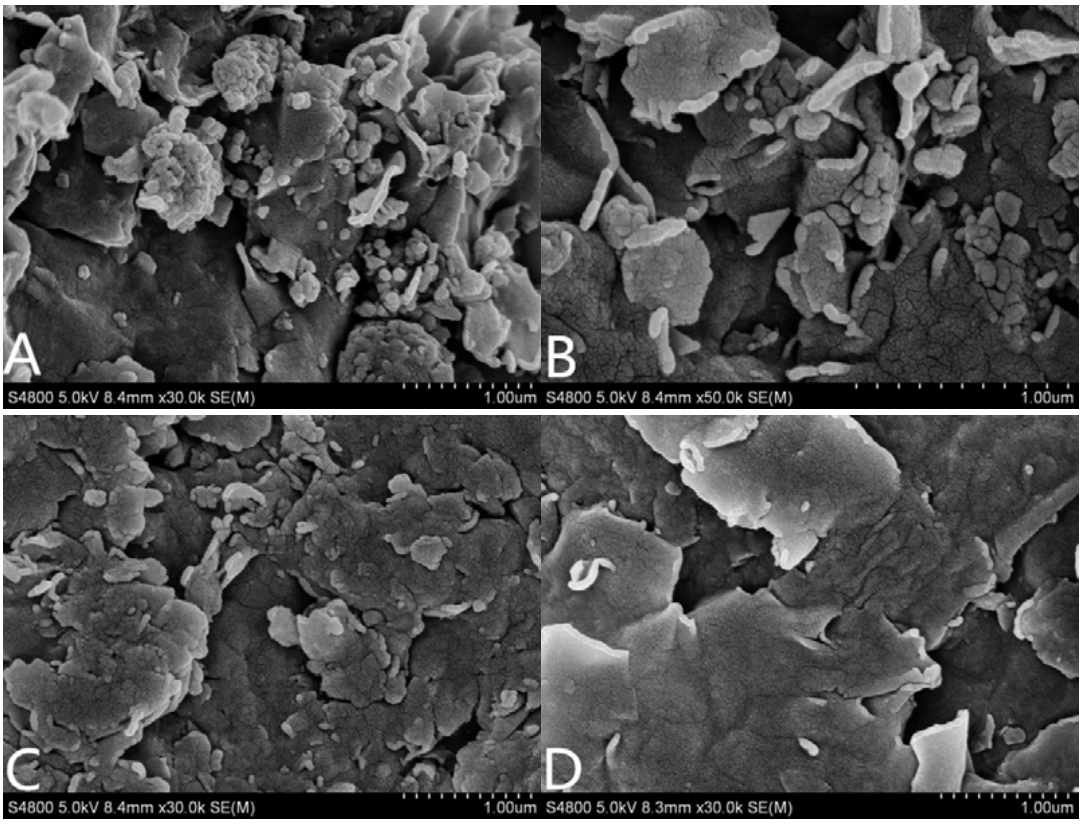


Fig. (12). SEM photographs of MCHA-MMT. Note: **A**: Na-MMT, **B**: 0.5CEC MCHA-MMT, **C**: 1.0 CEC MCHA-MMT, **D**: 1.5 CEC MCHA-MMT.

presented an opportunity to the drilling industry to meet the ever increasing demands for shale stability.

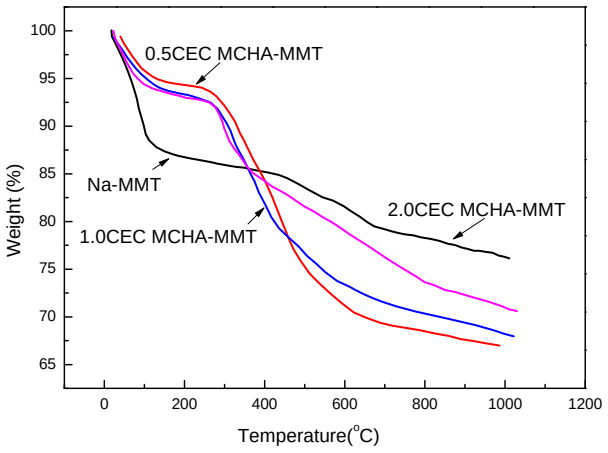


Fig. (13). TGA curves of Na-MMT and MMT modified with MCHA.

ABBREVIATIONS

API	=	American Petroleum Institute
AV	=	Apparent viscosity
CEC	=	Cation exchange capacity
CHA	=	Cyclohexylamine
CMC	=	Critical micelle concentration
DMCHA	=	N, N-Dimethylcyclohexylamine
FT-IR	=	Fourier transform infrared spectroscopy
MCHA	=	Methylcyclohexanamine
Na-MMT	=	Sodium montmorillonite
PHPA	=	Partially-hydrolyzed polyacrylamide
PV	=	Plastic viscosity
SEM	=	Scanning electron microscopy
TGA	=	Thermogravimetric analysis
XRD	=	X-ray diffraction
YP	=	Yield point

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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