

Study on a chelating removal system for dissolving barite mud cake

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To reduce drilling fluid damage, an oligomeric chelating agent DTPA-J was obtained by modifying DTPA with 1,3-dichloropropane to improve the solubility of barite, which was a non-toxic reagent and met environmental requirements. DTPA-J was chosen for the system of removal of chelating substances from the point of view of its corrosion, biotoxicity and dissolution of barite. The system consisted of a catalyst, a transforming agent and a glue breaker, which had a high efficiency of dissolving barite mud cake in a water-based drilling fluid, and the rate of weight loss of the mud cake exceeded 90%. In addition, the chelation removal system dissolved the additives pressed into the pores of the core, which can significantly improve the efficiency of restoring the permeability of the contaminated core. The application of the chelation removal system is of great importance for reservoir protection and subsequent oil and gas production, which can reduce requirements for the effectiveness of reservoir protection with water-based drilling fluids.

Keyword: Mud cake; Barite; Blocking remover; Reservoir protection; Glue breaker; Water-soluble polymer; Water-based drilling fluids.

Дослідження системи видалення хелатуючих агентів для розчинення бариту.
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Для зменшення пошкодження бурового розчину, був отриманий різновид олігомерного хелатного агента ДТРА-Ж шляхом модифікації ДТРА 1,3-дихлорпропаном для покращення розчинності бариту, який був нетоксичним реагентом і відповідав екологічним вимогам. ДТРА-Ж був обраний для системи видалення хелатуючих речовин з точки зору його корозії, біотоксичності та розчинення бариту. Система складалася з каталізатора, перетворювального агента та розривника клею, які мали високу ефективність розчинення баритової глинястої кірки в буровому розчині на водній основі, а швидкість втрати ваги глинястої кірки перевищувала 90%. Крім того, система видалення хелатів розчинила добавки, які були втиснуті в пори ядра, що може значно покращити ефективність відновлення проникності забрудненого ядра. Застосування системи видалення хелатів має велике значення для захисту пласта та подальшого видобутку нафти та газу, що може знизити вимоги до ефективності захисту пласта буровими розчинами на водній основі.

1 Introduction

With the development of drilling technique and the consumption of shallow oil resources, oil and gas exploration has been gradually forced to transit from shallow reservoirs to deep strata, which requires the fluid column pressure created by the high-density drilling fluid to balance the formation pore pressure

[1-4]. The high content of barite in high-density drilling fluid will increase the possibility of blocking reservoir. For horizontal wells, the mud cake formed in the lower parts of the horizontal section is denser due to the grinding and pressing of the drilling tool, resulting in more serious damage to the reservoir caused by barite mud cake [5-7]. Especially for low perme-

ability reservoir, barite mud cake will cause more damage to the reservoir [8]. It is difficult to remove the blockage with barite mud cake by subsequent acid treatment, since the solubility of barite in various inorganic acids and organic acids are very small [9-11]. In addition, due to the lack of high-quality barite resources, the density of barite currently used in drilling fluids is generally low, resulting in an increase in the amount of associated minerals in the barite, which may negatively affect the removal effect of blockage with barite mud cake [12-13]. Obviously, eliminating the potential reservoir damage from barite mud cake is essential for improving the production of oil and gas wells [14]. Unfortunately, the mechanism of barite blocking and removal, the development of removal strategies, and the main components of a chelating barite remover are not well understood; this may seriously restrict the development of chelating blocking removal technology for reservoir protection [15-16]. At present, the core treatment agent is chelating barite blockage remover, which can efficiently dissolve barite and the barite mud cake [17]. The chelating barite blocking remover consists of multiple nitrogen groups and carboxylic acid groups. The nitrogen groups are located in the center of the molecule, and the carboxylic acid groups are distributed externally, chelating cations to form a stable complex compound [18-19]. There are a variety of chelating agents commonly used in the completion process of dissolving barite and barite mud cake, such as nitrilotriacetic acid (NTA), ethylene diamine tetraacetic acid (EDTA), ethanolamine-N, N-diacetic acid (HEIDA), N-(hydroxyethyl) ethylenediamine triacetic acid (HEDTA), diethylene triamine penta acetic acid (DTPA), cyclohexanediamine tetraacetic acid (CDTA) and L-glutamic acid N, N-diacetic acid (GLDA). NTA is commonly used for well acidification, iron ion control and descaling, but the stability constant of its chelates with most metal cations is too low. In addition, NTA is carcinogenic to animals, making it a restricted chemical. EDTA can coordinate with many metal ions to form a complex compound, which can be used for stable control of iron ions or descaling during acidification and fracturing of oil and gas fields. However, EDTA is poorly biodegradable and almost insoluble in water, which has prompted engineer to search for viable alternatives. The molecular structure of HEIDA is similar to that of NTA, which is often used for descaling and acidification, and has

good biodegradability and solubility. HEDTA is similar in structure to EDTA, and it is formed by replacing one of the carboxyl groups in the EDTA molecule with a hydroxyl group. The addition of a hydroxyl group increased the solubility of HEDTA, but decreased its stability constant. DTPA has the highest stability constant among the chelating agents commonly used in the petroleum industry. Moreover, DTPA has little corrosion to metals, and does not produce poisonous gas during the chelation reaction. CDTA has good water solubility and chelation, which can be used for acidizing modification of carbonate reservoir in the petroleum industry. GLDA with biodegradation and low corrosion is obtained from L-glutamic acid, and it is a relatively new chelating agent [20].

The previous studies have shown that DTPA is the best for the dissolution of barite. However, it forms a complex with barium ions with poor structural stability [21]. Therefore, a kind of oligomer chelating agent DTPA-J was prepared by modifying DTPA with 1, 3-dichloropropane to improve the solubility of barite. Compared with common chelating agents, DTPA-J was evaluated in this research in terms of its corrosion activity, biotoxicity and the dissolution of barite. The effect of catalyst, converting agent and glue breaker on the performance of the oligomer chelating agent in dissolving barite was then discussed. Finally, a chelating removal system was developed to reduce the damage of the reservoir from mud cake and barite.

2. Experimental

2.1 Materials

As a weighting agent for drilling fluid, barite was purchased from Hubei Hanke Tech. Co., Ltd, and its particle size was in the range of 5-15 μ m. Analytical grade potassium carbonate (K_2CO_3), potassium chloride (KCl), potassium formate (HCOOK), potassium nitrate (KNO_3), sodium persulfate ($Na_2S_2O_8$), 1,3-dichloropropane, anhydrous ethanol, potassium iodide (KI), dichloromethane and potassium hydroxide (NaOH) were purchased from Wuxi Jingke Fine Chemical Co. Analytical grade EDTA (≥ 98.5 wt%), HEIDA (≥ 98.0 wt%), HEDTA (≥ 99.0 wt%), DTPA (≥ 98.5 wt%), CDTA (≥ 97.5 wt%), GLDA (≥ 95.0 wt%) and oxalic acid (≥ 98.0 wt%) were provided by Sinopharm Chemical Reagent Co., Ltd. Inhibitor (code LTMA-10), lubricant (LUBE), viscosifier (code VIS-B), filtrate reducer (code STARFLO), plugging agent (code

HISEAL), and gel-breaking stabilizer (code KM19) as the main components of constructing drilling fluid were obtained from Jingzhou Jiahua Tech. Co., Ltd. N80 steel was provided by CNPC Petroleum Pipe Research Institute. The sandstone cores ($\Phi 25$ mm \times 50 mm) were provided by Shanghai Branch of CNOOC Ltd., which was acquired from low permeability well at 3500 m in the East China Sea.

2.2 Preparation and structural characterization of oligomer chelating agent

After mixing 100 mL deionized water and 100 mL anhydrous ethanol, the mixture was added to a three-port round-bottom flask equipped with a stirrer, thermometer and condenser tube, followed by adding 50 g DTPA, 30 g 1, 3-dichloropropane and 2.0 g KI. NaOH was used to adjust the pH of the reaction system to 9. The reaction temperature was kept at 90 °C, and the reaction was carried out under stirring conditions for 6 h. At the end of the reaction, the reaction system was steamed to remove the solvent, washed with methylene chloride, and then the product was freeze-dried. The structure of oligomer chelating agent DTPA-J was analyzed by a Nicolet710 Fourier transform infrared spectrometer.

2.3 Barite Dissolution Measurement

Chelating agents, catalyst and alkaline converter were firstly added to a beaker containing 100mL tap water. Then, potassium hydroxide was used to adjust the pH value of the above system to about 13, and a chelating removal system was obtained. 1 g barite was added into the system, which was sealed and placed in a water bath at 90 °C for 10 hours. After the completion of the complex reaction, barite was filtered and dried at 105 °C for 4 hours, and the dissolution rate was finally calculated.

2.4 Corrosion Property Measurement

A certain amount of chelating agent was mixed in a glass containing 100 ml of tap water. Potassium hydroxide was used to adjust the pH value of the above solution to about 13. After mixing well, the N80 steel was placed in the solution at a temperature of 90 °C for 72 hours. Corrosion of N80 steel from chelating agents was calculated using the static mass loss method.

2.5 Biototoxicity Evaluation

The toxicity level was measured at a concentration of 50% of the maximum effect (EC_{50}) for luminescent bacteria using the method rec-

ommended by the National Standards of the People's Republic of China GB/T 15441-1995 (Water quality determination of the acute toxicity-luminescent bacteria test).

2.6 Mud Cake Dissolution Measurement

The formula of the water-based drilling fluid was: tap water + 0.3 wt% NaOH + 0.20 wt% Na_2CO_3 + 1.5 wt% inhibitor (LTMA-10) + 2.0 wt% lubricant (LUBE) + 0.7 wt% viscosifier (VIS-B) + 4.0 wt% filtrate reducer (STARFLO) + 3.0 wt% plugging agent (HISEAL) + 2.0 wt% gel-breaking stabilizer (KM19) + barite ($\rho = 1.5$ g/cm³). The drilling fluid was firstly aged at 90 °C for 16 h and then tested for API fluid loss using a ZNZ-D6 medium pressure filter loss instrument (Qingdao Hengtaida Electromechanical Equipment Co., Ltd., Qingdao, China). The API filter paper was used as the fluid loss medium, and its mass was described as m_0 . Barite mud cake was obtained after 30 min filtration under the condition of a pressure difference of 0.7 MPa; then it was dried and weighed (m_1), and placed in the chelating removal system for 10 hours at 90 °C. Finally, the dissolved mud cake was dried and weighed (m_2). The weight loss rate K of mud cake before and after dissolution reaction was calculated according to the formula below:

$$K = (m_1 - m_2) \times 100\% / (m_1 - m_0).$$

2.7 Reservoir protection measurement

According to the industrial standard SY/T 6540-2002, permeability recovery experiment of the water-based drilling fluid was carried out, and the effect of the chelating removal system on the permeability of the contaminated core was analyzed. Sandstone cores were used to measure the permeability (K_g) in air, and the permeability (K_0) of the cores treated with saturated brine was tested. After that, the cores were contaminated with the water-based drilling fluid at 90 °C for 2 h. The permeability (K_{od}) of cores treated with the water-based drilling fluid was tested. The rate of permeability recovery K_{od} / K_0 was calculated to analyze reservoir protection performance of the water-based drilling fluid. In addition, after the cores were contaminated with the water-based drilling fluid, the mud cake at the end of the cores was dissolved by the chelating removal system for 10 h. After the dissolution reaction, the rate of permeability recovery K_{od} / K_0 was tested.

3 Results and Discussion

3.1 Analysis on Infrared Spectrum of DTPA-J

The FTIR spectrum of the oligomer chelating agent DTPA-J is shown in Fig. 1. As indicated, the spectrum absorption peak at 2956.2 cm^{-1} was the stretching vibration of C-H, and the spectrum absorption peak at 1472.3 cm^{-1} was the bending vibration of CH_2 . The peak at 712.3 cm^{-1} is characteristic of $-(\text{CH}_2)_n-$. The peak at 3312.0 cm^{-1} is the stretching vibration peak of the hydroxyl group in the carboxyl group. The spectrum absorption peak at 1183.7 cm^{-1} was the stretching vibration of the C-N bond. The absorption peak at 1043.6 cm^{-1} is the characteristic peak of the quaternary ammonium salt groups. The peaks at 1741.0 cm^{-1} and 1420.1 cm^{-1} were the asymmetric and symmetrical stretching vibration peaks of the carbonyl group in carboxylic acid, respectively. The above analysis indicated that the designed molecule was successfully synthesized.

3.2 Analysis on Barite Dissolution of Chelating Agents

The dissolution efficiency of barite was measured when the dosage of chelating agent was 10 wt%; the experimental results are shown in Fig. 2. As indicated, DTPA had a stronger ability to dissolve barite than the other four common chelating agents, which was consistent with the results of existing reported studies [9]. In addition, DTPA-J showed the strongest dissolution effect for barite with a dissolution rate of 55.9%. Due to the oligomer chelating agent DTPA-J with the largest molecular chain among the chelating agents, it can effectively form coordination bonds with barium ions to form inner orbital coordination compounds. Due to the effect of molecular thermal motion, the above coordination compounds went from the surface of the barite particle into the solution, which ultimately led to the dissolution of barite [22].

3.3 Analysis on Corrosion Property of Chelating Agents

As organic weak acid, chelating agents have a certain corrosive effect on metal. Therefore, the corrosion behavior of the six chelating agents on metal was evaluated. N80 steel as corrosion object soaked in 10 wt% chelating agents. The testing results are shown in Fig. 3. HEIDA had the highest corrosion effect on N80

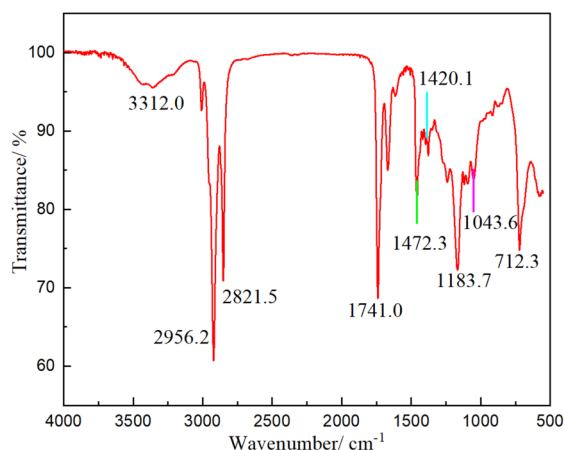


Fig.1 FTIR spectrum of DTPA-J

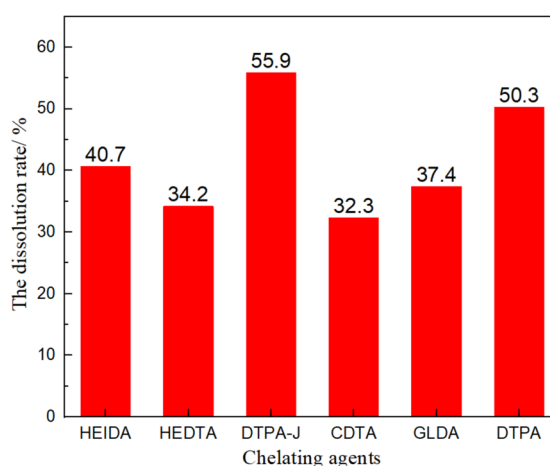


Fig. 2 Dissolution efficiency of barite with different chelating agents

steel, which can greatly shorten the life of the metal. In addition, the corrosion rates of DTPA, DTPA-J and GLDA on N80 steels were all less than 0.06 mm a^{-1} . Compared with DTPA, the corrosion rate of DTPA-J on metal was smaller, which indicated that DTPA-J with the quaternary ammonium salt structure can reduce the corrosion reaction of the metal surface due to the principle of electronuclear repulsion.

3.4 Biototoxicity Analysis of Chelating Agents

Table 1 shows the EC_{50} value of the six chelating agents. If the EC_{50} value of the chelating agent is greater than 30000 mg L^{-1} , it is considered as a non-toxic reagent and meets environmental requirements. The EC_{50} values of HEIDA, DTPA-J, GLDA and DTPA were more than $30,000\text{ mg L}^{-1}$, respectively, which can meet the national standard GB/T 15441-91 of the People's Republic of China. GLDA was extracted from L-glutamate or monosodium

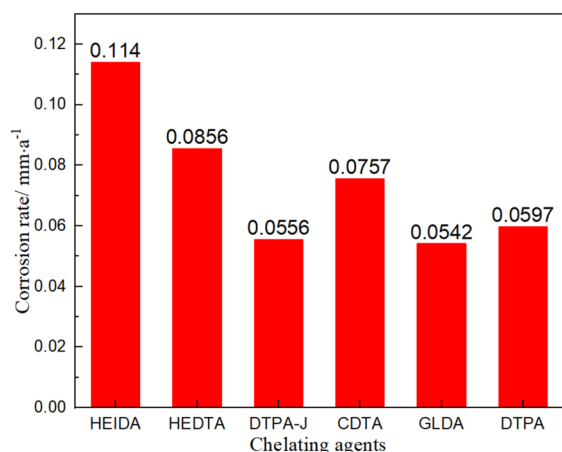


Fig. 3 Effect of chelating agents on corrosion behavior of N80 steel

glutamate, resulting in its best compatibility with the environment. However, the ability of GLDA to dissolve barite was lower than that of DTPA-J. Therefore, DTPA-J was selected as the chelating agent for the chelating removal system in terms of its corrosion, biotoxicity and the dissolution of barite.

3.5 Effect of DTPA-J of Different Dosage on the Barite Dissolution

Effect of DTPA-J with different dosage on the dissolution of barite was evaluated, and the experimental results are shown in Fig. 4. It was very interesting to note in Fig. 5 that the chelating removal system with 9 wt% DTPA-J was the most effective for dissolving barite, showing a dissolution rate of 58.4%. The traditional opinion that the higher the concentration of chelating agents, the faster is the dissolution of the barite was wrong. It was found that the higher concentration of chelating agent may even reduce the effect of barite dissolution. First of all, when the concentration of a chelating agent is very high, the competitive adsorption of the chelating agent on the surface of barite can lead to the reduction of chelating points, and its solubility to barite will naturally decrease. Secondly, the molecules of the chelating agent are relatively large and can form ste-

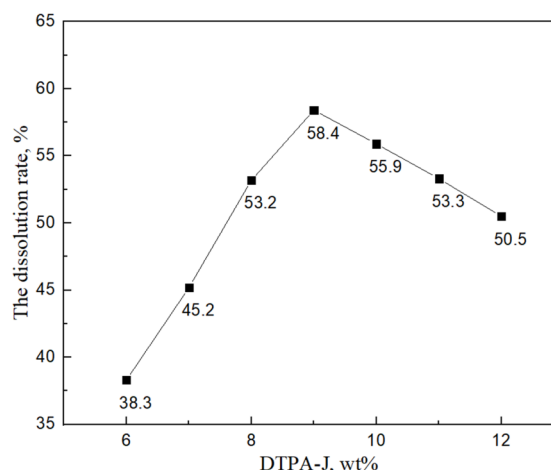


Fig. 4 Effect of DTPA-J with different dosage on barite dissolution

ric obstacles on the surface of the barite, which will weaken the process of chelating on the surface of the barite particles [23]. Therefore, the proper concentration of chelating agent can efficiently promote the dissolution of barite, and the optimum dosage of DTPA-J in a chelating removal system was 9 wt%.

3.6 Effect of Catalyst on Barite Dissolution

There are various chelating catalysts, such as oxalic acid, fluoride, disulfates, citrate, thio-sulfates, nitroacetate, mercaptoacetate, hydroxyacetate ammonium acetate and formate, wherein oxalic acid is the best chelating catalyst for barite dissolution. The formula of the chelating removal system was: 100 mL tap water + 9 wt% DTPA-J + oxalic acid + potassium hydroxide (pH=13). The effect of oxalic acid with different dosage in the chelating removal system on barite dissolution was evaluated, and the experimental results are shown in Fig. 5. As indicated, with an increase in the oxalic acid concentration, the dissolution rate of barite was effectively improved. With a concentration of 2.5 wt%, oxalic acid had the most obvious catalytic synergistic and solubilizing effect. Its mechanism consisted in the fact that

Table 1. EC₅₀ measurement of chelating agents

Chelating Agent	EC ₅₀ value (mg·L ⁻¹)	Standard (mg·L ⁻¹)	Result
HEIDA	56,000	30,000	Non-toxic
HEDTA	25,600		Low toxicity
DTPA-J	81,400		Non-toxic
CDTA	17,900		Low toxicity
GLDA	98,000		Non-toxic
DTPA	74,100		Non-toxic

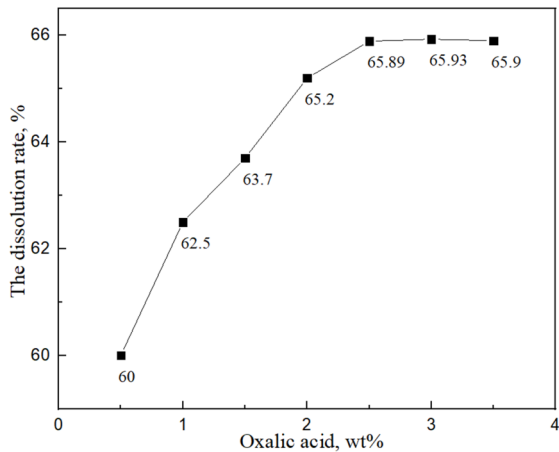


Fig. 5. Effect of oxalic acid with different dosage on barite dissolution

the exchange reaction between oxalate anion and sulfate can greatly improve the chemical properties of barite surface, thus increasing the reactivity of Ba^{2+} with DTPA-J and enhancing the DTPA-J solution activity for barite.

3.7 Effect of Converting Agent on Barite Dissolution

There are various converting agents, such as potassium carbonate (K_2CO_3), potassium chloride (KCl), potassium formate (HCOOK), and potassium nitrate (KNO_3). The formula of the chelating removal system was: 100 mL tap water + 9 wt% DTPA-J + 2.5 wt% oxalic acid + converting agent + potassium hydroxide (pH = 13). The effect of 10 wt% converting agent used in the chelating removal system on barite dissolution was evaluated, and the experimental results are shown in Fig. 7. Also, the effect of K_2CO_3 with different dosage in the chelating removal system on barite dissolution was evaluated, and the experimental results are shown in Fig. 7. It was very fascinating to note in Fig. 5 that the converting agent K_2CO_3 in the chelating removal system was the most effective in promoting the chelating agent to dissolve barite. Its mechanism consisted in the fact that chelating agent promoted the conversion of low solubility $BaSO_4$ into high solubility $BaCO_3$ under the action of the alkaline converting agent. As a result, the chelating agent most likely forms chelates with Ba^{2+} ionized from $BaCO_3$, thereby quickly realizing the purpose of dissolving barite [24]. In Fig. 6, 12 wt% K_2CO_3 in a chelating removal system was the most effective in promoting the chelating agent to dissolve barite. Therefore, the recommended dosage of converting agent K_2CO_3 in the chelating removal system was 12 wt%.

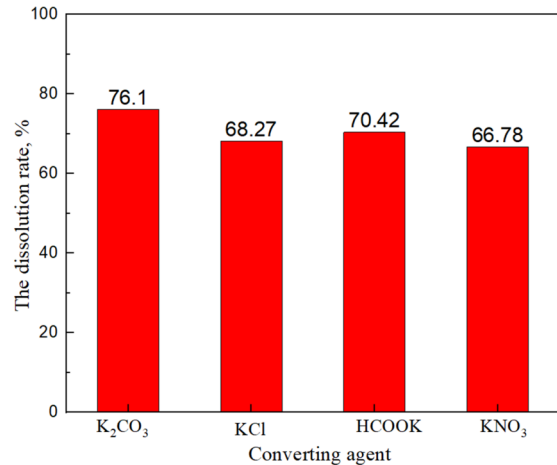


Fig. 6 Effect of converting agents on barite dissolution

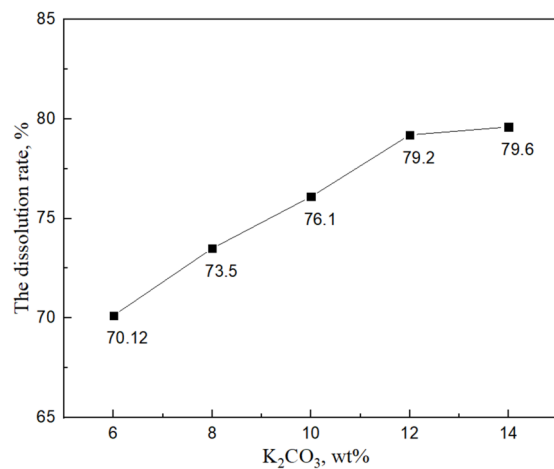


Fig. 7 Effect of converting agent with different dosage on barite dissolution

3.8 Analysis of the System for Dissolving Barite Mud Cake

The surface of barite particles is generally coated with water-soluble polymer, surfactants, lubricants, etc., forming an organic coating that does not contribute to the dissolution of the barite. In order to increase the contact surface between barite and chelating agents, the organic coating film on the surface of barite particles must be firstly dissolved. Glue breaker is used to dissolve the organic coating film on the surface of barite particles, and then chelating agents can quickly dissolve barite, thereby improving the dissolution rate of the mud cake. Therefore, the breaker was added to the system. The formula of the chelating removal system was: 100 mL tap water + 9 wt% DTPA-J + 2.5 wt% oxalic acid + 12 wt% K_2CO_3 + 2.0 wt% $Na_2S_2O_8$ + potassium hydroxide (pH=13). The above water-based drilling fluid was tested for API fluid loss using a ZNZ-D6 medium pressure

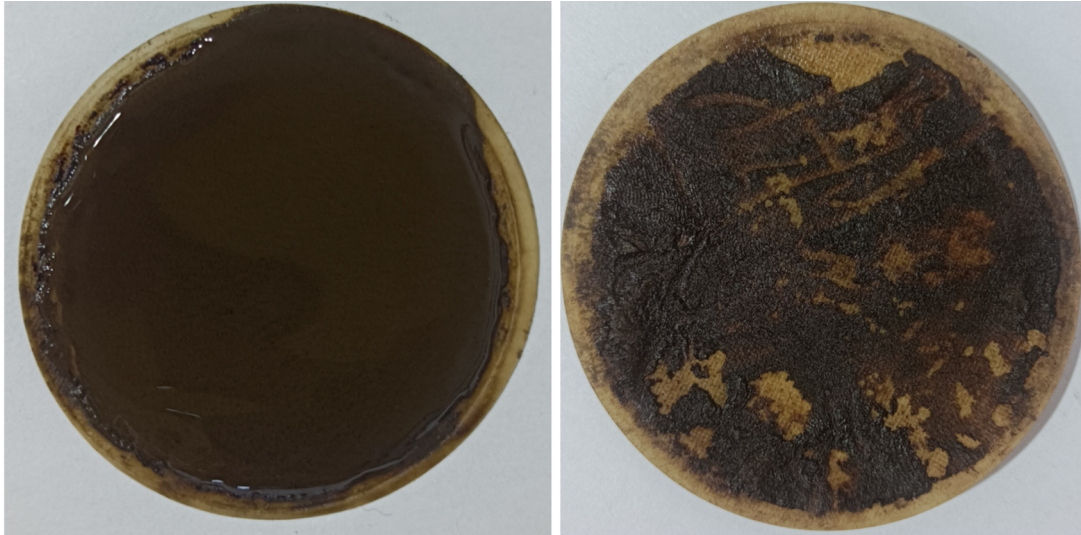


Fig. 8 The state of mud cakes (Lift: before dissolution reaction; and right: after dissolution reaction)

filter loss instrument, and the obtained barite mud cake was placed in the chelating removal system for 10 hours at 90 °C. The state of the mud cake before and after dissolution reaction is shown in Fig. 8, and the weight loss rate K of the mud cake in the chelating removal system was calculated. As indicated, the obtained barite mud cake before dissolution reaction was intact and unbroken, while after dissolution reaction, it was basically dissolved; this actually proves that the chelating removal system has effective dissolving performance for barite mud cake. In addition, the weight loss rate K of the mud cake was 91.5%.

3.9 Effect of the chelating removal system on cores

The effect of the chelating removal system on permeability recovery of the contaminated cores was analyzed, and the experimental results are shown in Table 2. It was clear from Table 2 that permeability recovery values of cores treated with the water-based drilling fluid were less than 75%, indicating that the additives of the drilling fluid blocked core pores. As expected, the chelating removal system will dissolve the additives squeezed into core pores, which can significantly improve permeability recovery values of the contaminated core.

3.10 Analysis of Mechanism for Mud Cake Dissolution

The diagram of the mechanism for mud cake dissolution is shown in Fig. 9. The mud cake dissolution process was divided into two stages. The first stage was the use of a glue breaker to dissolve an organic coating film on the surface

Table 2. The effect of the chelating removal system on the contaminated core

Core No.	K_g ($10^{-3} \mu\text{m}^2$)	K_o ($10^{-3} \mu\text{m}^2$)	K_{od} ($10^{-3} \mu\text{m}^2$)	K_{od}/K_o (%)
11	30.32	18.52	13.29	71.76
12	48.54	29.31	21.95	74.89
15	28.40	17.61	16.22	92.11
16	43.71	30.84	28.38	93.02

of barite particles, including water-soluble polymer, surfactants, lubricants, etc. The second stage was the chelating dissolution of barite. In the high pH environment, the central metal Ba^{2+} ions are separated from the crystal lattice and eventually enter the chelating agent solution. The chelating agent diffuses from the solution to the particle surface to form a low order adsorption complex. The low-order adsorption complex and the main component of barite are transformed into a high-order complex that can be detached from the particle surface. Therefore, barium ions in the barite mud cake can form chelates with the chelating agent, and the chelates move from the surface of barite particles into the solution, finally leading to the dissolution of barite particles.

4 Conclusions

It is shown that the glue solvent, which was used as an additive in the chelating agent removal system, can dissolve the organic coating film on the surface of barite particles; then the chelating agents can quickly dissolve the barite, which can improve the dissolution rate of the mud cake.

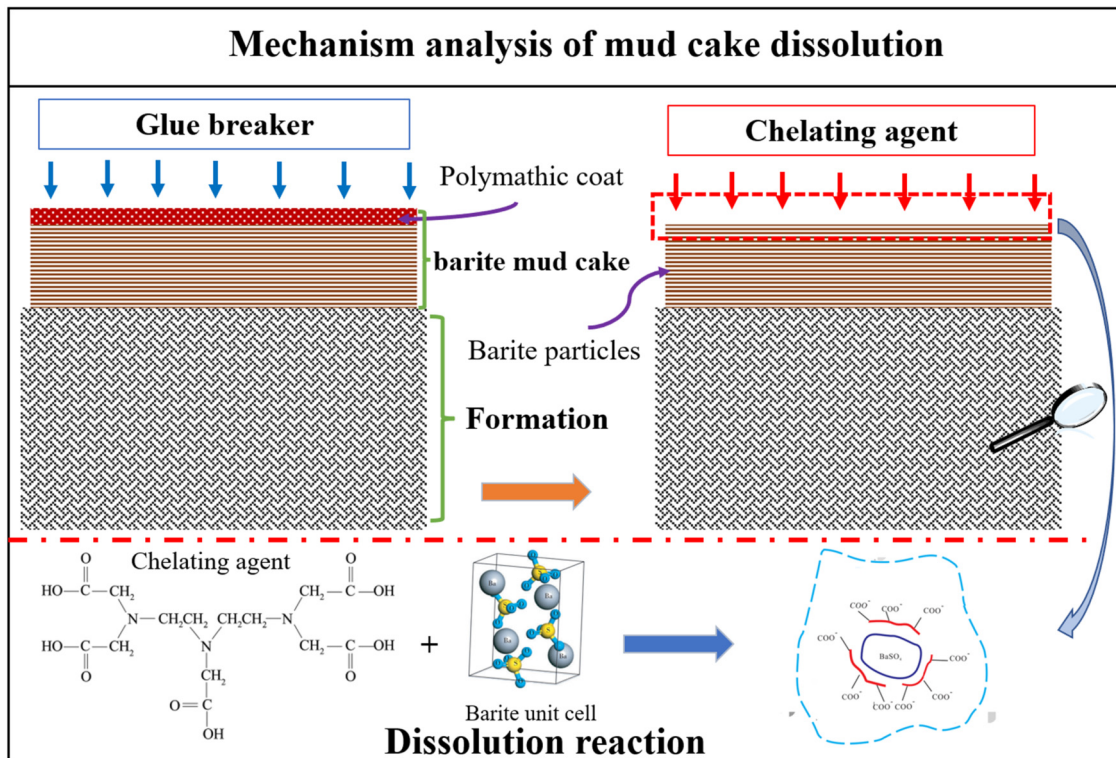


Fig. 9 Mechanism diagram of mud cake dissolution

The chelate removal system is effective for dissolving a barite mud cake. In addition, the chelating agent removal system dissolves additives that have been trapped in the core pores, which can significantly improve the permeability recovery performance of contaminated cores.

It was shown that the mud-cake dissolution process consists of two stages: the first stage is the use of an adhesive breaker to dissolve the organic coating film on the surface of the barite particles. The second stage is a chelating dissolution of barite. In a high pH environment, the central metal ion (Ba^{2+}) is separated from the crystal lattice and eventually enters the chelating agent solution, ultimately reaching the dissolution of the barite particles.

As a key additive of the chelating removal system, DTPA-J showed the strongest barite dissolution effect, which can effectively form coordination bonds with barium ion to form intra-orbital coordination compounds. DTPA-J is considered a non-toxic reagent and meets environmental requirements.

In addition, the influence of the catalyst, converter, and adhesive breaker on the effectiveness of the chelating agent in dissolving barite is discussed.

Finally, the chelating removal system was formulated.

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