

Study of a dual-functional degradable additive for fluid loss control and acid generation in hydraulic fracturing

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ABSTRACT

This paper presents a degradable acid-generating filtrate reducer (DA-FR) synthesized via the dissolution-evaporation-precipitation (DEP) method. When incorporated into a fracturing fluid with a viscosity of 30 mPa·s, DA-FR at mass concentrations ranging from 1% to 3% enables the static fluid loss performance to meet industry standards. The material exhibits a degradation rate of 8.3% after 2 hours at 160 °C, and achieves complete degradation within 24 hours. The suspended particles in the degraded fluid have an average size of approximately 80 nm, significantly smaller than those observed in conventionally broken fracturing fluids. Furthermore, DA-FR demonstrates favorable acid-generating capability: a 5% solution of its degradation products dissolves marble powder at a rate of 28.4%, and the rock surface exhibits abundant loose pores following dissolution. Consequently, DA-FR serves a dual function, it acts as a fluid loss control additive to mitigate filtrate invasion during fracturing operations, thereby enhancing the fracture propagating efficiency of the fracturing fluid, while simultaneously providing acid-etching functionality that improves near-fracture matrix permeability and promotes fracture conductivity.

Keywords: hydraulic fracturing, filtrate reducer, degradable acid generation, dissolution, fracture conductivity.

INTRODUCTION

Slickwater volumetric fracturing has become a key technology for stimulating unconventional oil and gas reservoirs [1, 2]. Unlike conventional crosslinked gel fracturing fluids, slickwater is characterized by low viscosity, typically ranging from 5 to 10 mPa·s. In naturally fractured or high-permeability reservoirs, this low viscosity can exacerbate fluid leak-off, leading to several adverse effects on reservoir stimulation. First, it compromises fracture propagation efficiency: excessive filtrate loss in the near-wellbore region results in insufficient fluid at the fracture tip and a reduction in net pressure within the fracture, potentially causing the actual fracture width and

length to fall significantly below design values [3]. Second, in fractured reservoirs with well-developed natural fractures, rapid fluid loss reduces the effective width of induced fractures, which may impede proppant transport. This may lead to premature proppant settling and accumulation near the fracture entrance or in the near-wellbore zone, substantially increasing the risk of sand plugging [4]. Third, in reservoirs rich in water-sensitive clay minerals, large volumes of fracturing fluid filtrate invading the reservoir matrix may trigger clay swelling and pore blockage, causing a sharp decrease in permeability and resulting in formation damage [5–7]. To address these challenges, a range of countermeasures have been adopted in field practice, including: (1) adopting

variable-viscosity slickwater technology, using low-viscosity fluid during fracture initiation and higher-viscosity fluid during proppant transport; (2) optimizing proppant size and pumping schedules; (3) incorporating additives such as clay stabilizers, flowback aids, water-block preventives, and fluid-loss control agents to reduce filtrate invasion and formation damage; and (4) designing a rational flowback schedule to enhance fracturing fluid recovery efficiency and improve the flowback capacity of the liquid phase [8–10].

In response to the above challenges, extensive research efforts have been undertaken to clarify the relationship between fracturing-fluid leak-off behavior and fracture initiation, propagation, and overall stimulation effectiveness. These investigations have been carried out via approaches including the development of fluid-loss control additives, laboratory physical modeling experiments, and numerical simulations [11–15]. Currently, fluid loss control additives can be broadly classified into synthetic polymers, natural macromolecular materials and their derivatives, and organic–inorganic composite materials. Among these, polymer-based agents are the most widely used, encompassing multi-component copolymerized modified polyacrylamides, hyperbranched polymers with dendritic structures, gel-type polymers with three-dimensional network architectures, and cross-linked polymers [16–19]. Natural macromolecular materials, including starch, cellulose and lignin, offer advantages such as wide availability, low cost, and environmental biodegradability [20–22]. Organic–inorganic composite fluid loss control agents are typically formed via physical blending, surface covalent grafting, or polymer intercalation, resulting in composites of polymers and micro/nano-particulate materials [23–26]. To date, the working mechanisms of fluid-loss control additives can be grouped into three primary categories: (1) Water absorption swelling and physical plugging: Crosslinked polymers, for example, possess complex spatial network structures that can swell upon water absorption to block pores. Their long-chain molecules may also act as colloidal particles to physically plug the filter cake [27–30]. (2) Adsorption: Adsorption groups attach to the surface of bentonite particles, forming a hydrated film that prevents clay particle aggregation and ensures uniform particle distribution. Meanwhile, molecular chains are closely aligned and stacked via electrostatic forces or hydrogen bonding, creating a layered structure

of “bentonite–polymer chain–water molecules,” thereby improving filter cake quality and fluid loss control performance [31–33]. (3) Generation or release of nano-microspheres via high-temperature degradation: Under high-temperature and high-pressure conditions, natural macromolecules undergo hydrothermal carbonization, forming carbon nano-microspheres or nanocomposite particles. These particles can fill the micro-pores within the filter cake, thereby enhancing its compactness and reducing permeability [34, 35].

As the exploration and development of oil and gas resources increasingly target tight and unconventional reservoirs, enhancing the efficiency of fracture generation during volumetric fracturing and optimizing the conductivity of induced fractures have become essential for the cost-effective exploitation of these reservoirs [36, 37]. In response to the challenges associated with volumetric fracturing stimulation as previously described, this study introduces a functional agent, termed DA-FR, developed utilizing the DEP method. This method synergistically combines fluid loss control with slow-release acid-generating properties. The primary function of DA-FR is to markedly decrease fluid loss of slickwater during volumetric fracturing in unconventional reservoirs, thereby augmenting the net pressure within fractures, enhancing fracture propagation efficiency, and mitigating the risk of sand blockage. Simultaneously, during its slow degradation, DA-FR generates acid in situ, facilitating mild acid etching on the surfaces of induced fractures, which further improves fracture conductivity. The DA-FR agent developed in this work provides a new methodology and technical approach centered on “controlled fluid loss, enhanced fracture propagation, and mild acid etching” for the efficient development of unconventional gas reservoirs.

EXPERIMENTAL

Reagents and materials

The fluid loss control agent (DA-FR) was self-synthesized in the laboratory. Sodium hydroxide, phenolphthalein, angelic anhydride, polylactic acid, and dichloromethane were purchased from Chengdu Kelong Chemical Reagent Factory, all of analytical grade. Natural marble blocks were obtained from Chengdu Keping Technology Co., Ltd.

Equipment

A model MK-SD6A medium-pressure filter press was used, with a filtration area of 45.6 cm², an operating pressure of 0.69 MPa, and a fluid injection volume of 350 mL. Scanning electron microscopy (SEM) was performed using a ZEISS EVO MA15 instrument, capable of image magnifications ranging from 5× to 1,000,000×. A constant-temperature oven, an electronic balance, and a high-temperature high-pressure reactor were all standard laboratory equipment. The constant-temperature oven had a maximum operating temperature of 180 °C. The analytical balance had a precision of 0.0001 g. The high-temperature high-pressure reactor was lined with polytetrafluoroethylene (PTFE), rated for temperatures up to 260 °C and pressures up to 8 MPa.

Preparation of DA-FR

Two primary functions were considered in the design of DA-FR. First, it can generate H⁺ ions through dissolution, degradation, and hydrolysis, thereby exhibiting the characteristics of a retarded acid. Second, its relatively long degradation time – with negligible degradation within 2 hours – enables it to function as a fluid loss control agent. Based on this concept, the structure of DA-FR consists of two main components: one is the organic acid anhydride material, angelic anhydride, which can produce a substantial amount of H⁺ upon hydrolysis; the other is degradable polylactic acid (PLA), whose molecular structure features poor solubility and slow degradation, ultimately forming small-molecule organic acids after degradation. These two materials are dissolved in dichloromethane, mixed in a certain proportion to react, and then dried and crushed after solvent removal to obtain DA-FR, which has a density of 1.58 g/cm³. When used as a fluid loss control agent, DA-FR should be crushed to a particle size of 200–300 mesh.

Static filtration test

A fracturing fluid system (1 L) was prepared according to the formulation. DA-FR was added at concentrations of 1%, 3%, and 5% (by weight), and the mixtures were uniformly stirred before being placed into a model MK-SD6A medium-pressure filter press. The tests were conducted at room temperature under a differential pressure of

3.5 MPa, and the filtrate volumes were measured at different time intervals. A plot of cumulative filtrate volume (V) against the square root of time ($\sqrt{t}$) yields a straight line, where the slope of the line is denoted as m and the intercept as h , the effective filtration cross-sectional area of the filtration medium denoted as A . The filter-cake-controlled filtration coefficient (C_3), filtration rate (v_c), and initial filtrate volume (Q_{sp}) can then be calculated from Equations 1 to 3.

$$C_3 = 0.005 \times \frac{m}{A} \quad (1)$$

$$v_c = \frac{C_3}{\sqrt{t}} \quad (2)$$

$$Q_{sp} = \frac{h}{A} \quad (3)$$

Degradation performance

Equal amounts of DA-FR were weighed and placed into four high-temperature high-pressure reactors. The reactors were then placed in a constant-temperature, the initial mass of the DA-FR was recorded as m_0 . Timing commenced once the temperature reached the preset value. One reactor was taken out at each of the following time intervals: 2 h, 8 h, 16 h, and 24 h. The contents were filtered through a quantitative filter paper (the filter paper was pre-weighed, with its mass recorded as m_2). The residue along with the filter paper was dried at 103–105 °C to constant weight, and the total mass (m_1) was recorded. The degradation rate (η) was then calculated according to Equation 4.

$$\eta = \frac{m_0 - m_1 + m_2}{m_0} \times 100 \quad (4)$$

Acid generating performance

A 100 mL aliquot of DA-FR solution at various concentrations was prepared in a clean beaker and heated in a constant-temperature water bath at different temperatures for 4 hours. Subsequently, 5 mL of the heated acid solution was transferred into a clean Erlenmeyer flask. Three to four drops of standard phenolphthalein indicator were added to the flask, and the acid solution was titrated with 20 wt% NaOH solution. Titration was stopped when the solution turned light pink and the color did not fade after shaking and standing for a period of time; back-titration with acid until the solution became colorless confirmed the

endpoint. The mass of NaOH solution consumed was recorded, and the H^+ concentration in the acid solution was calculated based on the amount of OH^- consumed, which was then converted to an equivalent HCl concentration. To minimize errors and the effect of time on acid generation, three titrations were performed on the acid solution after 4 hours of heating, and the average value was taken.

Dissolution capability

Two methods, namely dissolution rate measurement and microscopic morphology scanning, were employed to evaluate dissolution capability. The dissolution rate was assessed using actual rock cuttings obtained from an oilfield. The mineral composition of the cuttings consisted of an average quartz content of 52.27%, feldspar content of 23.62%, calcite content of 9.23%, and clay mineral content of 14.88%. The cuttings were crushed and sieved through a 100-mesh screen, then dried at 103–105 °C for 2 hours and stored in a desiccator for later use. A 20 g portion of the rock powder (initial mass recorded as G_0) was added to 200 mL of degradation solution and allowed to react at a predetermined temperature for 6 hours. After the reaction, the mixture was filtered through quantitative filter paper (pre-weighed filter paper mass recorded as G_2), and the residue along with the filter paper was dried at 103–105 °C to constant weight. The total mass (G_1) of the sample plus filter paper was recorded. The dissolution rate (γ) was then calculated according to Equation 5.

$$\gamma = \frac{G_0 - G_1 + G_2}{G_0} \times 100\% \quad (5)$$

For apparent morphology evaluation, rock samples measuring 1 cm³ were used. One surface of each rock sample was marked for reference. The initial microscopic morphology of the marked surface was observed using scanning electron microscopy (SEM). Subsequently, the rock samples were immersed in the degradation solution at a solid-to-liquid mass ratio of 1:10 and allowed to react at a predetermined temperature for 6 hours. After the reaction, the rock samples were retrieved, rinsed with tap water until neutral, and the same marked surface was re-examined under SEM at identical magnifications to observe the morphological changes.

RESULTS

Static fluid loss control performance

In this study, the static fluid loss control performance was evaluated using a fracturing fluid with a viscosity of 30 mPa·s. The relationship between filtrate volume and time under different DA-FR addition concentrations is shown in Figure 1. It can be observed that as the DA-FR concentration increased from 1% to 3%, the filtrate volume gradually decreased, and the slope of the filtrate volume versus time curve became progressively smaller. Moreover, a distinct filter cake was observed on the filter paper for the fracturing fluid which containing 3% DA-FR. The filtration parameters calculated according to the static filtration test method and the corresponding formulas are presented in Table 1. It can be seen that at a DA-FR concentration of 1%, both the filtration rate and the filter-cake-controlled filtration coefficient already met the industry standard requirements. At a concentration of 3%, the filtration rate, filtration coefficient, and initial filtrate volume were all significantly lower than the industry standard. Therefore, the addition of only a small amount of DA-FR enables the fracturing fluid to exhibit excellent fluid loss control performance, demonstrating its potential as a fluid loss control agent in volumetric fracturing operations.

High-temperature degradation performance

The variation curve of the high-temperature degradation rate of DA-FR as a function of time is presented in Figure 2. It can be observed that DA-FR exhibits a relatively slow degradation rate, with less than 10% degradation within the first 2 hours regardless of temperature. Specifically, the degradation rate at 120 °C was only 3.6%, and at 160 °C it reached just 8.3%. This slow degradation characteristic ensures that the fluid loss control function of DA-FR is maintained during hydraulic fracturing operations. After 8 hours at high temperature, the degradation rate exceeded 95%, and after 10 hours it reached above 99.8%, indicating nearly complete degradation. This feature guarantees that DA-FR can be fully degraded during the shut-in stage following fracturing treatment and then flow back to the surface with the fracturing fluid. Consequently, it facilitates filter cake removal, cleans the fracture surfaces,

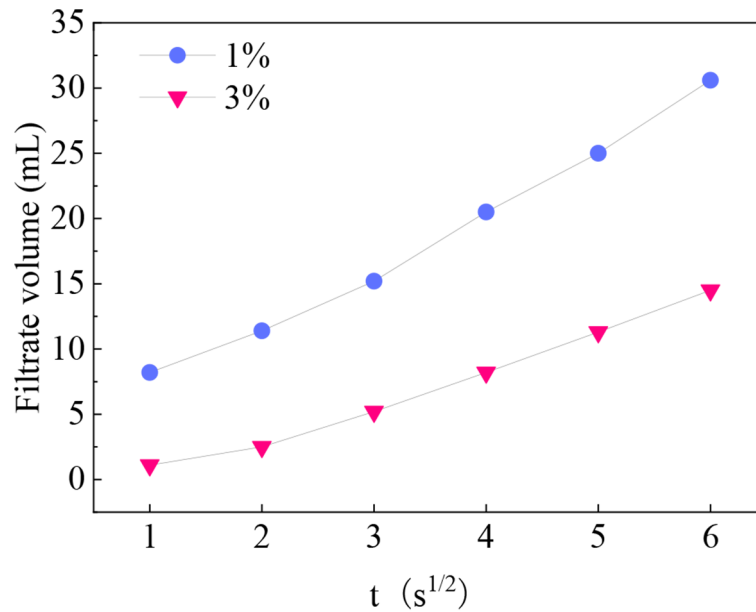


Figure 1. The variation curve of the leakage volume over time

Table 1. Static fluid loss parameters

DA-FR content	Fluid loss coefficient C_3	Filtration rate v_e	Initial filtrate volume Q_{sp}
Occupation standard	1.0×10^{-3}	1.5×10^{-4}	5.0×10^{-2}
1%	6.2×10^{-4}	1.0×10^{-4}	1.2×10^{-1}
3%	1.3×10^{-6}	2.1×10^{-7}	1.8×10^{-2}

reduces flow resistance of oil and gas from the matrix to the fractures, and ultimately enhances fracture conductivity.

In addition, the particle size of the degradation products of DA-FR was measured using a laser particle size analyzer, and the resulting particle size distribution is presented in Figure 3. As can be seen from the figure, the particles are relatively small, primarily distributed within the range of 20–130 nm, with an average particle size of approximately 80 nm. This particle size is smaller than that of conventional fracturing fluid breakers after gel breaking. Therefore, compared with the broken gel fluid of conventional fracturing fluids, the degradation products of DA-FR are unlikely to cause additional permeability damage to the induced fractures, which is beneficial for formation protection.

Degradation-induced acid generation capability

The H^+ concentration in DA-FR degradation solutions at different concentrations was determined by titration and then converted to

an equivalent HCl concentration. The resulting acid generation capability of DA-FR is shown in Figure 4. As can be seen from the figure, DA-FR exhibits a strong acid generation capability, approaching approximately 50% of that of HCl. Specifically, a 40 wt% DA-FR solution produced an acid concentration close to 20% HCl. The slow degradation and acid release characteristics of DA-FR ensure that the fracturing fluid remains neutral during pumping through the surface pipeline and wellbore. Moreover, the density of DA-FR is only 1.58 g/cm^3 , which is much lower than that of quartz sand, making it easier to be carried and transported by slickwater. As also shown in Figure 4, the acid concentration increases almost linearly with increasing DA-FR concentration, indicating a positive correlation between acid generation capability and DA-FR concentration, with negligible influence from solubility. Therefore, in field operations, the concentration of DA-FR can be flexibly adjusted according to actual reservoir conditions and treatment requirements. Consequently, DA-FR can serve not only as a fluid loss control agent to reduce fracturing fluid leak-off during unconventional

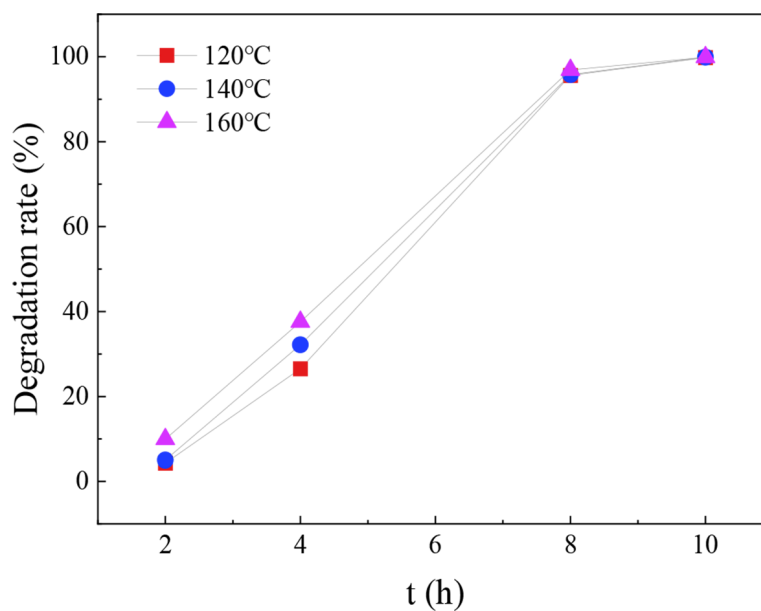


Figure 2. The variation curve of the high-temperature degradation rate of DA-FR as a function of time

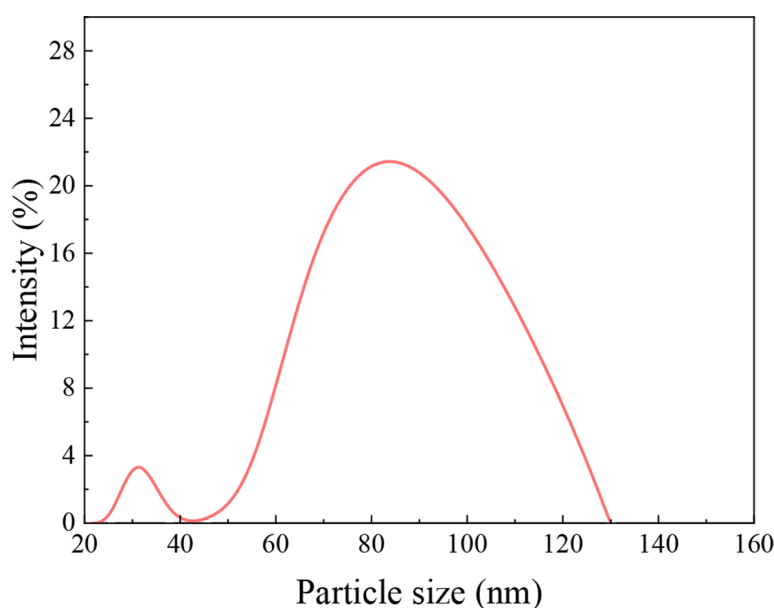


Figure 3. The particle size distribution of DA-FR after degradation

volumetric fracturing, thereby improving fracture propagation efficiency, but also as a slow-release acid for acid fracturing stimulation in carbonate reservoirs, enhancing fracture conductivity.

Fracture dissolution capacity

The dissolution rates of clastic rock cuttings were measured at 140 °C using DA-FR degradation solutions at mass concentrations of 2%, 4%, 6%, and 8%. The results are presented in Figure 5 (a). As shown in the figure,

the dissolution rate increases progressively with increasing DA-FR concentration, reaching 8.9% at a concentration of 8%. This indicates that the DA-FR degradation solution exhibits a strong dissolution effect on clastic rock, which is primarily attributed to the dissolution of calcite minerals. The dissolution rates of marble powder in DA-FR degradation solutions are shown in Figure 5 (b). It can be observed that the dissolution of marble powder by the DA-FR degradation solution is significantly greater than that of clastic rock. At a DA-FR concentration of 5%,

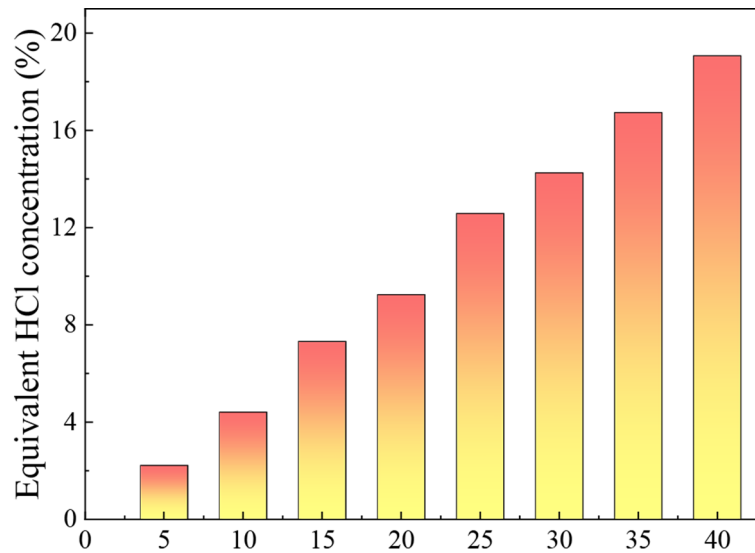


Figure 4. The degrade and acid generating ability of DA-FR

the dissolution rate of marble powder reached 28.4%, and at a concentration of 30%, it exceeded 90%. Therefore, during fracturing operations in tight sandstone or shale reservoirs, DA-FR can serve as a fluid loss control agent to enhance fracture propagation efficiency while simultaneously etching the fracture surfaces, improving near-fracture matrix permeability, and reducing the flow resistance of hydrocarbons from the matrix to the fractures. Furthermore, owing to its low density and slow degradation characteristics, DA-FR exhibits excellent transportability with the fracturing fluid. Consequently, in acid fracturing stimulation of carbonate reservoirs, DA-FR simplifies the fracturing fluid system; it can be directly dispersed in slickwater and injected during the fracturing operation without the need for additional acid preparation or

increase the viscosity of the acid solution to delay the acid-salt reaction, while effectively avoiding wellbore corrosion.

A comparison of the microscopic morphology of rock samples before and after dissolution by the DA-FR degradation solution is shown in Figure 6. Images (a) and (b) depict the surface morphology before dissolution, where the rock surface appears relatively smooth and flat. In contrast, images (c) and (d) show the surface morphology after dissolution, where a significant increase in surface roughness is observed, along with the formation of numerous loose pores and cavities. These observations further confirm that the DA-FR degradation solution exhibits a pronounced dissolution effect on carbonate rocks, which is expected to improve the permeability of the near-fracture matrix.

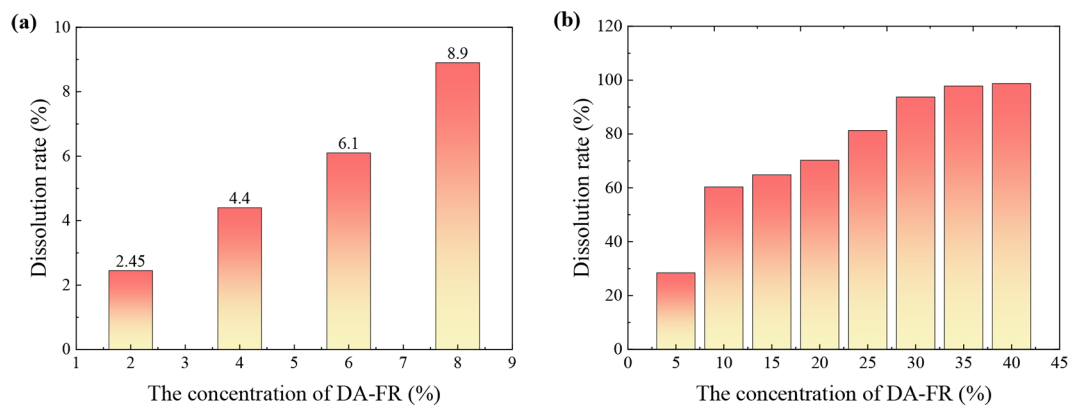


Figure 5. (a) DA-FR The dissolution ability of DA-FR degradation solution on shale cuttings, (b) The dissolution ability of DA-FR degradation solution on marble powder

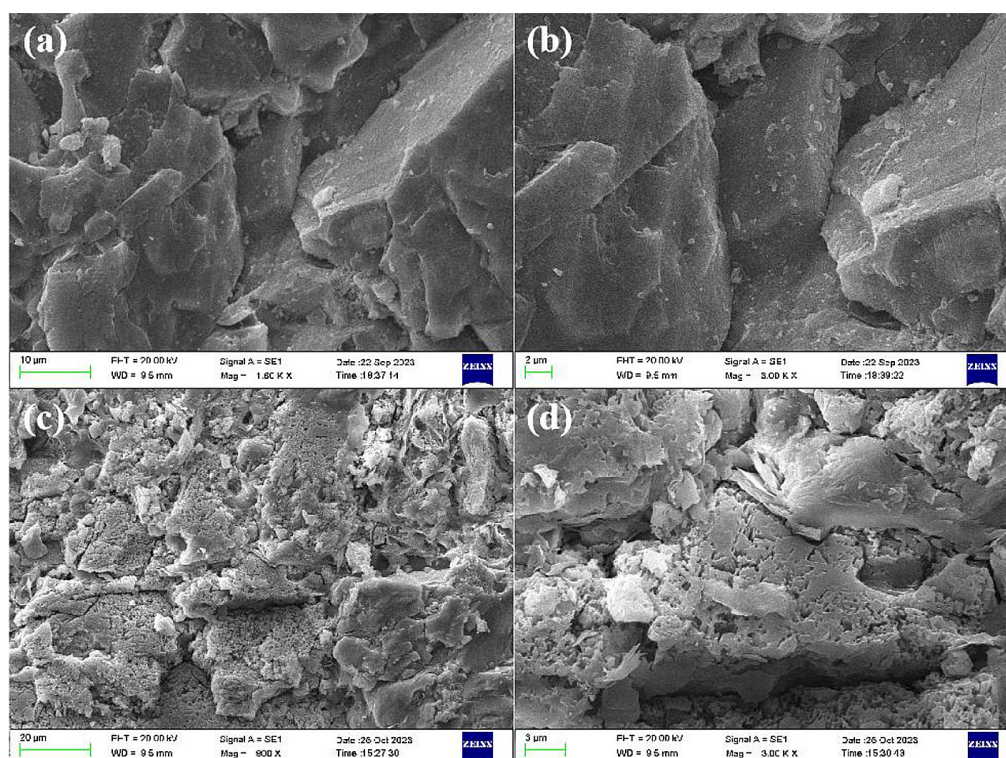


Figure 6. Comparison of microstructure before and after dissolution of rock samples by DA-FR degradation solution

CONCLUSIONS

This paper proposes a degradable acid- generating filtrate reducer DA-FR. DA-FR has a density of 1.58 g/cm³ and a particle size of 300 mesh, and it can significantly reduce the static filtration rate of fracturing fluids. At mass concentrations of 1–3 wt%, DA-FR enables the static filtration parameters of a fracturing fluid with a viscosity of 30 mPa·s to meet industry standards. This agent is nearly insoluble in water but undergoes slow degradation in aqueous solutions. After 2 h at 120 °C and 160 °C, the degradation rates are 3.6% and 8.3%, respectively; after 8 h, the degradation rates exceed 95% at both temperatures, and complete degradation is achieved after 10 h. The average particle size of the degradation products in the solution is approximately 80 nm, which is much smaller than that of conventional fracturing fluid breakers; thus, no additional damage to propped fracture conductivity is anticipated. DA-FR also exhibits a strong acid-generating capability. At a mass concentration of 40 wt%, the acid concentration produced by DA-FR is equivalent to that of 20 wt% hydrochloric acid. The degradation solution demonstrates

excellent dissolution performance, achieving a dissolution rate of 28.4% for marble powder at a DA-FR concentration of 5 wt%. After dissolution, extensive etching traces and loose pores appear on the rock sample surface, which helps improve near-fracture matrix permeability and fracture conductivity. Owing to its slow degradation and acid-releasing characteristics, DA-FR can be carried by slickwater to achieve deep-penetration volumetric acid fracturing. It can be primarily used as a fluid loss control agent in slickwater volumetric fracturing of tight sandstone and shale gas reservoirs to enhance stimulation efficiency. The accompanying acid etching effect also provides an auxiliary technical means for improving fracture conductivity in carbonate reservoirs.

Acknowledgements

This research was supported by the Zhongyuan Oilfield Postdoctoral Fund (No. 2026BY04), the Talent Project of Henan Province “Research on Key Technologies for Exploitation of Deep Tight/Shale Oil and Gas Reservoirs” (No. 264000510003), the Sinopec Ten Dragon Science and Technology Research Project (No. P24231).

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