Experimental study on CO₂ capture by using n-butylamine to plug the gas channeling to enhanced oil recovery

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Abstract
The national policy of peak carbon dioxide emission and carbon neutrality has pointed out the technological direction for the development of the petroleum industry in China. In order to efficiently utilize CO₂ gas source to enhance oil recovery, n-butylamine is taken as the plugging channeling agent for experiment study to plug the produced channeling-path during the process of CO₂ flooding in ultra-low permeability reservoir. The contents of the experiment included three parts: reaction mechanism of n-butylamine with CO₂, evaluation of the injection performance of n-butylamine, and the extent of enhanced oil recovery after plugging the gas channeling by using n-butylamine. Reaction product of n-butylamine and CO₂ is white solid, which is a type of organic urea so that it can be used to plug the gas channeling. N-butylamine has a good injection performance after adding protecting slug on the condition of high temperature. 80% of the whole volume of core can be spread after injecting 0.3 PV of n-butylamine. During plugging and displacement experiment of heterogeneous cores, oil recovery can be greatly enhanced by 25–30% after injecting n-butylamine. Experimental results show that it can provide a new train of thought for the gas injection development of fractured, heterogeneous and ultra-low permeability reservoirs by using n-butylamine to plug the high permeability area.

Keywords Carbon capture · Plugging gas channeling · n-butylamine · Enhanced oil recovery · Ultra-low permeability reservoirs

Introduction
Carbon dioxide emissions have become a major problem due to the adverse effects of greenhouse gases on the climate. To date, global carbon dioxide emissions have exceeded 30 Gt, most of which come from the burning of fossil fuels (Chen et al. 2014). Emission levels will continue to increase in the coming decades as economies and populations grow.

The continued reliance on fossil fuel combustion for many energy applications has led to the need to develop different low-carbon technologies to reduce emissions (Wang and Song 2020; Wang et al. 2020b). Therefore, many countries have proposed corresponding low-carbon or carbon-reduction policies. For example, in September 2020, China clearly proposed the goals of “carbon peaking” in 2030 and “carbon neutrality” in 2060.

A potentially scalable technology to reduce CO₂ emissions is CO₂ capture and storage (CCS). CCS technology involves capturing CO₂ from industrial exhaust and transporting it for injection into safe geological reservoirs, which will contribute about 19% of emissions reductions in 2050. However, the cost of CCS technology is high. To reduce the cost, CO₂ capture and utilization is combined with CCS to form the unified CO₂ capture, transport and storage (CCUS) systems. CO₂ utilization can be treated as a resource conservation strategy, which can be utilized for enhanced oil recovery (EOR) by injection into depleted oil reservoirs (Budzianowski 2017; Zhang et al. 2021, 2020).
For petroleum company in China, it is a practical method to enhance oil recovery from low permeability reservoirs or enhance multi-component shale gas recovery and its sequestration in realistic kerogen by using CO₂ (Wang et al., 2020a, 2021a, b, 2022). The utilization of CO₂ to enhance oil and gas recovery can implement the national policy of peak carbon dioxide emission and carbon neutrality, which can not only achieve the transfer of carbon, but also achieve the efficient development of low permeability oil reservoirs (Zou et al. 2021; Vikara et al. 2017; Curtis 2014).

Due to the heterogeneity of the low permeability oil reservoir, such as fractures and dominant pore channels, as well as the high fluidity ratio between CO₂ and crude oil, the phenomenon of gas channeling of CO₂ flooding is serious (Xiong et al. 2017; Pu et al. 2020; Darabi et al. 2016). How to effectively plug the gas channeling is the key to improve the development effect of CO₂ gas flooding.

CO₂ Capture is considered to be an important and feasible method to control CO₂ content in the near future. Although there are many methods to capture CO₂, the chemical method is relatively economical and reliable (Gómez-Díaz et al. 2021). Among the chemical methods, CO₂ capture by using organic amines is the most common. The literature on the reaction of organic amines with CO₂ was first reported in 1920. At present, large-scale separation of CO₂ by the absorption process of aqueous amines has been commercialized in the worldwide. The method of wet scrubbing by using organic amine (such as ethanolamine) has been used industrially to capture CO₂ for the past 50 years. During the absorption process, amine molecules react with CO₂ to form compounds. As shown in Fig. 1, amines react with CO₂ to form the different product. The selectivity of CO₂ capture of the amine solution is higher than that of N₂, and the CO₂ capture capacity is not strongly affected by CO₂ partial pressure. Therefore, amine-based systems can efficiently remove carbon dioxide. After decades of development, the technology of CO₂ capture by using organic amines is mature and reliable for petroleum and petrochemical industry (Ume et al. 2012; Wang 2008). Therefore, it is a potential enhanced oil recovery (EOR) technology to plug the gas channeling channel of CO₂ flooding by using the CO₂ capture technology of organic amine.

In the gas dominant transportation pathway of the reservoir, the concentration of CO₂ is high, so that the injected organic amine can react with CO₂ and the formed product can plug the gas dominant transportation pathway in the form of physical plugging. This method can not only capture CO₂, but also can greatly improve oil recovery. However, there are few research reports in this academic field, and no effective technical methods have been formed. Therefore, in this paper, an organic amine is studied to capture CO₂ and then to be used to EOR based on reservoir conditions. The first is to carry out the study of the chemical reaction mechanism between organic amine and CO₂. The second is to carry out the study of plugging to EOR by using the reaction products. The aim is to develop a new EOR method.

**Experiment**

**Materials and equipment**

The used oil sample and water sample are produced from Wuqi oilfield of Shaanxi Yanchang Petroleum (Group) Co., Ltd. The ground surface viscosity of oil sample is 12.86 mPa.s, and the subsurface viscosity of oil sample is 4.64 mPa.s at 75 °C. The water sample is NaCl water type with the salinity of 43,000 mg/L. N-butylamine (CH₃CH₂CH₂CH₂NH₂) is chemical pure with the boiling point of 77.8 °C, which is obtained from Sinopharm Group in China. Ethanol is analytical reagent, which is also obtained from Sinopharm Group in China.

All the cores used in the experiment are natural outcrop square cores with the three dimensional size of 4.5 × 4.5 × 30 cm³. After the cores are saturated with the water sample, the oil sample is continuously injected into the cores at the injection rate of 1 mL/min until only oil is produced from the outlet. Thus, the oil saturations of the cores can be calculated according to the amount of the produced water and the porosity of the cores. After the cores are saturated with the oil for 24 h of the aging period in the oven, the fractured artificial cores can be obtained by fracturing a homogeneous core along the injection direction from the middle part, and then, the two pieces of the core are directly closed without any filling.

The equipment include a Nicolet 6700 FTIR Fourier infrared spectrometer (Thermo Fisher, USA), a CO₂ gas cylinders with the CO₂ purity of 99.9%, a gas flow meter. The physical simulation experiment device for displacement

![Fig. 1 General reaction schemes for the chemical absorption of CO₂ by secondary and tertiary amines](image-url)
is made by Jiangsu Lianyou Scientific Research Instrument Co., Ltd., in China, which is shown in Fig. 2. From Fig. 2, it can be observed that the outlet is directly connected to the atmosphere, and the injection pressure of a fluid is equal to its flow resistance across the core or the capillary.

**Reaction experiment of n-butylamine with CO₂**

First, two different volumes of n-butylamine are measured and then are placed into the two beakers. (One volume is 25 mL and the other volume is 50 mL.) Thus, the two beakers are put into an intermediate container with a volume of 500 mL and then connected with another intermediate container with high-pressure CO₂ gas (1200 mL volume, pressure of 8 MPa). After the two intermediate vessels are connected, the change of CO₂ pressure is observed. In the experiment, four temperatures of 45 °C, 60 °C, 75 °C, 90 °C are set, respectively. After 24 h, the two beakers are taken out from the intermediate container, and the morphology of the reaction products in the beakers is observed, and the structure is analyzed by infrared spectrum. The experiment flowchart is shown in Fig. 3.

**Evaluation of injection performance and plugging capability of n-Butylamine**

The injection performance of n-butylamine is evaluated by displacement experiment. Four temperatures of 45 °C, 60 °C, 75 °C and 90 °C are set in the experiment. Besides, a comparison experiment is also set. One group is injected with n-butylamine directly, while the other group is added with protective slug before n-butylamine injection. As a liquid, the viscosity of ethanol is close to that of n-butylamine solution, so that ethanol is selected to be a protective slug between CO₂ and n-butylamine during injection, which can better separate CO₂ and N-butylamine that gas (such as N₂). The changes of the pressure during injection are observed to evaluate the influence of protective slug on injection performance. The permeability of the core used in the experiment is 6 × 10⁻³ μm².

As an important parameter to evaluate the properties of the plugging channeling agent, the breakthrough pressure can directly reflect the capacity of the plugging. Breakthrough pressure gradient is equal to breakthrough pressure/core length (MPa/m). The testing method of breakthrough pressure is as follows. First, the core with a certain permeability value is selected and then be placed into the core holder. Subsequently, high-pressure CO₂ gas is injected into the core, and the gas pressure in the core is controlled to be 7 MPa by using the backpressure valve. Thus, 0.1 PV alcohol, 0.3 PV n-butylamine and 0.1 PV alcohol are injected in sequence. When this is done, the core holder is closed. After the n-butylamine in the core is fully reacted with high-pressure CO₂ gas, the gas flooding is started. The outlet end of the core is directly connected with the atmosphere, and the injection pressure is equal to the difference of displacement pressure. Until the gas flow at the outlet end is suddenly increased and the inlet pressure of the core is decreased sharply, the inflection point value at the pressure curve at the inlet end of the core is the breakthrough pressure of the plugging channeling agent. To start the low permeability layer, the plugging channeling agent must have a certain breakthrough pressure. The experimental device for measuring the breakthrough pressure of the reaction product of n-butylamine and CO₂ is shown in Fig. 1, and the core parameters for the experiment are shown in Table 1.

**Enlarging the sweep volume evaluation of n-butylamine**

The enlarging sweep volume evaluation experiment is carried out by using the displacement experimental device. During the experiment, high-pressure CO₂ gas is first injected...
into the core, and the gas pressure in the core is controlled to be 7 MPa through the backpressure valve. Thus, a certain volume of protective slug is injected into the core, followed by separate injection of 0.1 PV and 0.3 PV of \( n \)-butylamine. After 48 h, the reaction between \( CO_2 \) gas and \( n \)-butylamine is completed, and then, the core is fractured and exposed to air. Because the organic amines can absorb moisture from the air, there have different colors in the surface of the core. By observing the color change of the core section, the range of the enlarging sweep volume of \( n \)-butylamine can be judged. The migration of \( n \)-butylamine under formation conditions can be investigated by contrast experiments, and the capacity of \( n \)-butylamine to expand sweep volume can be analyzed. The permeability of the core used is \( 6 \times 10^{-3} \mu m^2 \).

**Experiment of plugging the gas channeling and oil displacement**

The experiment of plugging the gas channeling and oil displacement is carried out by using the displacement experimental device, which can be judged by the extent of EOR by injecting \( n \)-butylamine to plug the \( CO_2 \) gas channeling.

| No | The type of core | length (cm) | width (cm) | height (cm) | porosity (%) | The permeability of matrix (10\(^{-3}\) μm\(^2\)) | The aperture of fracture (μm) |
|----|-----------------|-------------|------------|-------------|--------------|-----------------------------------|-----------------------------|
| 1# | Unfractured core | 30          | 4.5        | 4.5         | 15.8         | 50                                  | No                          |
| 2# | Unfractured core | 30          | 4.5        | 4.5         | 15.9         | 200                                 | No                          |
| 3# | Unfractured core | 30          | 4.5        | 4.5         | 16.1         | 600                                 | No                          |
| 4# | Unfractured core | 30          | 4.5        | 4.5         | 16.3         | 1000                                | No                          |
| 5# | Fractured core   | 30          | 4.5        | 4.5         | 16.7         | 10                                   | 200                         |
| 6# | Fractured core   | 30          | 4.5        | 4.5         | 16.9         | 10                                   | 500                         |

**Results and discussion**

**Reaction mechanism of \( n \)-butylamine and \( CO_2 \)**

**Appearance of the reaction product**

In a high-pressure \( CO_2 \) environment, the reaction product of \( n \)-butylamine with \( CO_2 \) is shown in Fig. 4. From Fig. 4, it can be observed that the product is a loose, white solid particle. It is the solid particles that can provide the material basis for plugging the gas channeling channel. The volume

| No | Temperature (°C) | Permeability of the heterogeneous core (10\(^{-3}\) μm\(^2\)) | Level difference of heterogeneity (times) | Porosity (%) | Oil saturation (%) |
|----|------------------|---------------------------------------------------------|------------------------------------------|--------------|-------------------|
| 7# | 75               | 1.4                                                     | 7.14                                     | 14.5         | 62.4              |
|    |                  | 10                                                      |                                          | 14.8         | 66.3              |
| 8# | 75               | 5.6                                                     | 89.3                                     | 14.6         | 64.3              |
|    |                  | 500                                                     |                                          | 16.2         | 68.8              |

| No | Temperature (°C) | Matrix permeability/10\(^{-3}\) μm\(^2\) | Porosity (%) | Oil saturation (%) | Fracture aperture (μm) |
|----|------------------|------------------------------------------|--------------|-------------------|------------------------|
| 9# | 75               | 1.4                                      | 14.5         | 62.5              | closed fractured       |
| 10#| 75               | 1.4                                      | 14.5         | 62.2              | 100                    |
of formed solid expands 3–4 times compared with that of liquid before the reaction, which also provides technical support for greatly expanding sweep volume.

**Analysis of the chemical structure of the reaction product**

The analysis result of the infrared spectrum of the product is shown in Fig. 5. At 3300 cm\(^{-1}\), the characteristics of the stretching vibration absorption peak of N–H bond are consistent. At 3000 cm\(^{-1}\), it accords with the characteristics of stretching vibration absorption peak of C–H bond. At 1600 cm\(^{-1}\), it accords with the stretching vibration absorption peak characteristics of N–C=O (carbonyl group). At 1460 cm\(^{-1}\), it accords with the characteristics of the bending vibration absorption peak of N–H bond. At 1200 and 1300 cm\(^{-1}\), it accords with the characteristics of the stretching vibration absorption peak of C–N bond. At 800 cm\(^{-1}\), it accords with the characteristics of the bending vibration absorption peak of C–N bond. The structure of the reaction product seems to be similar to that of \(N, N'\)-dibutylurea. However, they are not exactly the same. This indicates that this reaction mainly belongs to the process of reaction to form organic urea by using \(CO_2\) and organic amine. Meanwhile, it is also mixed with other side reactions (Chao et al. 2014; Kortunov et al. 2015; Parrino et al. 2016).

**Reaction characteristics of \(n\)-butylamine with \(CO_2\)**

The reaction process of \(n\)-butylamine with high-pressure \(CO_2\) gas at different temperatures is shown in Fig. 6. When \(n\)-butylamine once starts to contact with \(CO_2\) gas at high temperature and pressure, the pressure of \(CO_2\) gas is rapidly decreased, indicating that the reaction of \(n\)-butylamine begins immediately after contacting with \(CO_2\) gas under formation conditions. With the increase in the temperature, the greater the decrease in \(CO_2\) gas pressure there is, indicating that the higher the temperature, the more intense the reaction there is. Since the product is a loose solid, it does not affect the continuation of the subsequent reaction until the end of the reaction. After the complete reaction, the pressure drop amplitude is basically the same at different temperatures, indicating that temperature mainly affects the reaction rate, but does not affect the reaction process.

**Evaluation of injectivity and plugging capability of \(n\)-butylamine**

**Injection without protection of slug**

When \(n\)-butylamine is directly injected, the injection pressure is increased sharply and the normal injection is not possible to proceed. When only 0.1 PV is injected, the pressure is reached 5.5 MPa, and the injection experiment cannot be continued. When the core is taken out from the core holder, it is found that there are a lot of plugs at the entrance of the core. When the core is cut open, it is found
that the blockage almost only exists on the end face of the core, which is shown in Fig. 7. The black material in the red box is the reaction product. This further confirms that the enlarging sweep volume of \( n \)-butylamine is very small and \( \text{CO}_2 \) can rapidly react with \( n \)-butylamine in the core in the absence of a protective slug.

**Effect of protective slug on injection capacity of \( n \)-butylamine**

Ethanol is added to isolate \( n \)-butylamine from \( \text{CO}_2 \) and prevent the reaction between \( \text{CO}_2 \) and \( n \)-butylamine. After ethanol is injected, the corresponding relationship between injection pressure and injection amount of \( n \)-butylamine is shown in Fig. 8. It can be observed that the injection pressure of \( n \)-butylamine is in a reasonable range after the addition of protective slug. The injection pressure can be greatly reduced compared with that without protection. In addition, the injection pressure is decreased with the increase in the temperature. The higher the temperature, the more volatile \( n \)-butylamine and the stronger the injection capacity there is. In general, \( n \)-butylamine can be easily injected into the low permeability layer with the addition of protective slug.

**Evaluation of enlarging sweep volume of \( n \)-butylamine**

After high-pressure \( \text{CO}_2 \) gas is filled in the core with a permeability of \( 6 \times 10^{-3} \mu \text{m}^2 \), 0.1 PV protective slug is first injected, followed by separate injection of 0.1 and 0.3 PV \( n \)-butylamine, respectively. After the reaction, the core is taken out for fracturing and the profile of the core are observed, which is shown in Fig. 8. It can be observed that injection of 0.1 PV \( n \)-butylamine can affect 33% of the total core volume and injection of 0.3 PV \( n \)-butylamine can affect almost 80% of the total core volume. This is due to the volatile nature of small amines and their strong diffusion and migration ability in the core under high temperature. Figure 9 shows that injecting \( n \)-butylamine into the core can greatly improve the sweep volume of \( \text{CO}_2 \) flooding.

**Plugging capability of \( n \)-butylamine**

The experimental results are shown in Fig. 10. When the permeability of the core is less than \( 600 \times 10^{-3} \mu \text{m}^2 \), the breakthrough pressure of reaction product of \( n \)-butylamine and \( \text{CO}_2 \) can reach more than 4.8 MPa, and the pressure after breakthrough is maintained at about 2 MPa. This
shows that the reaction product has a certain plugging ability and a high residual resistance coefficient, which indicates that \( n \)-butylamine has the ability to plug the high permeability oil layer and displace the remaining oil in the low permeability oil layer. But when the core permeability is increased to 1000 × 10^{-3} \text{ μm}^2, the breakthrough pressure and residual resistance coefficient of the reaction product are greatly reduced, indicating that the plugging ability of the reaction product is limited under this condition. For the fractured cores, when the fracture opening is 200 μm, the breakthrough pressure of the reaction product can reach 5.8 MPa, and the pressure after breakthrough is maintained at about 3 MPa, indicating that the reaction product can effectively plug the gas channeling of the fracture. But when the fracture opening is increased to 500 μm, the reaction product has almost no blocking ability. Therefore, for reservoirs with too large permeability ratio or large-scale fracture, other supplementary measures need to be taken to cooperate with the channeling plugging of \( n \)-butylamine and \( \text{CO}_2 \).

**Table 4** Plugging channeling experiment of heterogeneous core with \( n \)-butylamine

| No | Permeability of the heterogeneous core \((10^{-3} \text{ μm}^2)\) | Level difference of heterogeneity (times) | Oil recovery before plugging/% | Gas flow rate before plugging \((\text{mL/min})\) | Oil recovery after plugging (%) | Gas flow rate after plugging \((\text{mL/min})\) | Enhanced oil recovery after plugging (%) |
|----|-------------------------------------------------|-----------------------------------------|-------------------------------|-------------------------------------------|--------------------------------|--------------------------------|------------------------------------------|
| 7# | 1.4                                             | 7.14                                    | 12.5                          | 67                                        | 36.1                          | 105                           | 23.6                                     |
| 10 | 43.5                                            | 89.3                                    | 43.5                          | 1860                                      | 47.7                          | 180                           | 4.2                                     |
| 8# | 5.6                                             |                                         | 8.8                           | 77                                        | 35.3                          | 330                           | 26.5                                     |
| 500 |                                               |                                          | 53.5                          | 4600                                      | 60.3                          | 560                           | 6.8                                     |

**Experiment of EOR**

**Experiment of EOR of the heterogeneous core**

The experimental data of plugging gas channeling and oil displacement of the heterogeneous core are shown in Table 4. It can be observed that injection of \( n \)-butylamine can effectively plug the gas channeling in high-permeability zones to enhance oil recovery. On the one hand, the flow rate of \( \text{CO}_2 \) gas channeling is obviously decreased, and the flow rate of \( \text{CO}_2 \) at the outlet of the core in the high permeability layer is decreased to the same number order as that in the low permeability layer. On the other hand, in subsequent displacement after plugging, EOR of low permeability zones can be increased by 23.6% of No. 7# and 26.5% of No. 8#, and that of high permeability zones can be increased by 4.2% of No. 7# and 6.8% of No. 8#. This indicates that the \( \text{CO}_2 \) channeling channel formed by the gas flooding is plugged, and the flow is diverted to the low permeability core to initiate the remaining oil.

**Experiment of plugging \( \text{CO}_2 \) channeling and oil displacement of the fractured core**

The displacement data before and after plugging \( \text{CO}_2 \) channeling and oil displacement of fractured cores are shown in Table 5. It can be observed that \( n \)-butylamine can effectively plug the closed fracture and the opening fracture with the aperture of 100 μm. Before plugging, the gas injection is ineffective for EOR, and the recovery rate is only 5% of No. 9# and 3% of No. 10#. After plugging the \( \text{CO}_2 \) channeling channel, the recovery rate is significantly increased, reaching 33% of No. 9# and 27% of No. 10#. Moreover, the gas channeling rate of No. 9# is significantly reduced from 5,000 to 150 mL/min and the gas channeling rate of No. 10# is significantly reduced from 8,000 to 650 mL/min, indicating that the fractures are effectively plugged.
Conclusions

1. The reaction product of \( n \)-butylamine and \( \text{CO}_2 \) is an organic urea, which is white crystal and can be used to plug \( \text{CO}_2 \) channeling channel.

2. Protective slug should be added before \( n \)-butylamine injection. Under the action of protective slug, \( n \)-butylamine has a strong migration ability in the porous medium of high-temperature formation. The enlarging sweep volume is large. 0.3 PV \( n \)-butylamine can sweep 80% of the whole core.

3. \( N \)-butylamine has high plugging ability, which can plug the gas channeling channel of high permeability layer or fracture and activate the remaining oil in low permeability layer. The plugging experiment of \( n \)-butylamine can provide a technical idea for \( \text{CO}_2 \) injection development of fractured heterogeneous ultra-low permeability reservoirs.

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Declarations

Conflict of interest All authors declare that they have no conflict of interest.

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