Petroleum Science 22 (2025) 1380–1390

Contents lists available at ScienceDirect

Petroleum Science

journal homepage: www.keaipublishing.com/en/journals/petroleum-science

Original Paper

Experimental investigation on the effects of deep eutectic solvents (DES) on the wettability of sandstone samples

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A R T I C L E I N F O

Article history:
Received 19 January 2024
Received in revised form
29 November 2024
Accepted 3 December 2024
Available online 7 December 2024

Edited by Min Li

Keywords:
Deep eutectic solvents
Surfactant
Wettability alteration
Sandstone rock

A B S T R A C T

Recently, deep eutectic solvents (DES) have received great attention in assisting water flooding and surfactant flooding to improve oil recovery because they can reduce the interfacial tension (IFT) between oil and water, inhibit surfactant adsorption, and change the wettability of rock. However, the effects of DES on the wettability of rock surface have not been thoroughly investigated in the reported studies. In this study, the effects of various DES samples on the wettability of sandstone samples are investigated using the Amott wettability measurement method. Three DES samples and several DES solutions and DES-surfactant solutions are firstly synthesized. Then, the wettability of the sandstone samples is measured using pure saline water, DES solutions, and DES-surfactant solutions, respectively. The effects of the DES samples on the wettability of the sandstone samples are investigated by comparing the measured wettability parameters, including oil displacement ratio (I_o), water displacement ratio (I_w), and wettability index (I_A). The Berea rock sample used in this study is weakly hydrophilic with I_o, I_w, and I_A of 0.318, 0.032, and 0.286, respectively. Being processed by the prepared DES samples, the wettability of the Berea sandstone samples is altered to hydrophilic ($0.7 > I_A > 0.3$) by increasing I_w but lowering I_o. Similarly, DES-surfactant solutions can also modify the wettability of the Berea sandstone samples from weakly hydrophilic to hydrophilic. However, some DES-surfactant solutions can not only increase I_w but also increase I_o, suggesting that the lipophilicity of those sandstone samples will be improved by the DES-surfactant solutions. In addition, micromodel flooding tests confirm the promising performance of a DES-surfactant solution in improving oil recovery and altering wettability. Moreover, the possible mechanisms of DES and DES-surfactant solutions in altering the wettability of the Berea sandstone samples are proposed. DES samples may improve the hydrophilicity by forming hydrogen bonds between rock surface and water molecules. For DES-surfactant solutions, surfactant micelles can capture oil molecules to improve the lipophilicity of those sandstone samples.

1. Introduction

Wettability is used to describe the tendency of rock surface to preferentially spread or attract a specific fluid in the presence of other immiscible fluids (Ahmed, 2010). In petroleum engineering, wettability of rock surface is one of the important factors affecting oil recovery factor because it determines how difficult it is to sweep the residual oil from porous media (Agbalaka et al., 2008). For the water-wet rock surface, oil occupies the central position of larger pores while water is attached to rock surface (Alhosani et al., 2021). As a result, oil is readily to be swept by the injected injectant. As for the oil-wet rock surface, oil is coated to rock surface while water is located in the center of larger pores (Branco and Natália, 2017). Thus, less oil can be flooded by the injected injectant because of the capillary forces. To help produce more residual oil, some methods

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https://doi.org/10.1016/j.petsci.2024.12.002

are proposed to alter the wettability of rock surface from oil-wet to water-wet or from weakly water-wet to strongly water-wet. Surfactants are widely used to alter the wettability of reservoir rocks (Wang and Kishore, 2015; Yang et al., 2021; Shi et al., 2022). For example, Souraki et al. (2019) experimentally investigated the feasibility of altering the wettability of carbonate reservoir rock using two surfactants (i.e., hexadecyl amino benzene sulfonic acid (HABSA) and cationic hexa decyl trimethyl ammonum bromide (CTAB)). They found that the two surfactants can alter the wettability of the rock surface from oil-wet to water-wet. Moreover, CTAB has a better performance in altering the wettability. Some polymers can also be used to alter the wettability of rock surface (Tang and Abbas, 2002; El-Hoshoudy, 2018). Al-Busaidi et al. (2023) evaluated the performances of three partially hydrolyzed polyacrylamide (HPAM)-based polymers in altering the wettability of rock. Their study indicates that the HPAM-based polymers are also effective in altering the wettability of dolomite. Besides, silica nanoparticles can also alter the wettability of rock surface by being adsorbed by the rock surface to form a hydrophilic or hydrophobic layer (Li et al., 2019).

DES, being synthesized by mixing a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA) at a certain temperature (García et al., 2015), are now widely used in different industrial applications because of their good availability, non-toxicity, good biodegradability, good recyclability, and environmental friendliness (Smith et al., 2014). Nowadays, the feasibility of improving oil recovery using DES samples is examined through numerical simulations and experimental investigations. Raut et al. (2023) used a conductor-like screening model for real solvent (COSMO-RS) model to predict the IFT reduction effect caused by DES samples. The simulation results indicate that 26 out of 217 DES samples can reduce the IFT between water and heavy oil by more than 50%. Kesarwani et al. (2022) found that DES samples can reduce the surfactant adsorption, lower critical micelle concentrations (CMC) of surfactants, and decrease the IFT between water and oil when they are used as additives in surfactant polymer (SP) flooding. Nowadays, researchers have devoted great efforts to examining the effects of various DES samples on improving oil recovery (Huang et al., 2019, 2020; Sanati et al., 2021; Atilhan and Santiago, 2022). From these reported studies, it can be concluded that the functions of DES samples in assisting water flooding and surfactant flooding include reducing IFT and surface tension, inducing emulsification, and inhibiting surfactant adsorption. Moreover, some researchers proposed that DES samples can also change the wettability of rock surface to improve oil recovery. Mohsenzadeh et al. (2015a) found that two DES samples prepared by mixing 1 mol of choline chloride with 2 mol of urea (or 2 mol of glycerol) can reduce the contact angle of sandstone samples. Shuwa et al. (2015) also found that the DES sample prepared by choline chloride and ethylene glycol can change the wettability of sandstone from oil-wet to less oil-wet. Moreover, Atilhan and Aparicio (2021) also proved that DES samples can alter the wettability of calcite rocks from oil-wet to water-wet using molecular dynamics simulation. Sanati et al. (2021) also obtained the conclusion that the wettability of dolomite rock can be changed to more water-wet using DES samples. Compared to the aforementioned agents for altering the wettability of rock surface, DES is more affordable and easier for production. Therefore, DES samples can act as a new potential media in enhancing oil recovery. However, the effects of various DES samples on the wettability of rock have not been thoroughly investigated because only scarce experimental data are available in the literature (Atilhan and Aparicio, 2021). Therefore, it is necessary to clarify the effects of DES samples on the wettability alterations of reservoir rocks to better understand the role DES samples play in improving oil recovery. In this study, the effects of various DES samples on the altering wettability of sandstone samples are investigated using the Amott wettability measurement method. First, three DES samples and various DES and DES-surfactant solutions are prepared with varied DES concentrations and different surfactants. Herein, these DES samples have hydrogen donors, resulting in different accommodation patterns of the polar sites. Thus, these DES samples will generate different molecular interactions when they are mixed with other substances (Cotroneo-Figueroa et al., 2022). Then, the wettability of the sandstone samples is determined using the prepared saline water and crude oil. The wettability of the sandstone samples is measured after those samples are treated by the prepared DES samples. The comparisons of the measured wettability parameters indicate that the prepared DES samples can alter the wettability of the sandstone samples by improving the hydrophilicity from weakly hydrophilic to hydrophilic and weakening the lipophilicity. Next, the wettability measurements are conducted using the prepared DES-surfactant solutions to observe the changes in the wettability parameters of the sandstone samples. Based on the experimental investigations, the DES samples can alter the wettability of the Berea sandstone samples from weakly hydrophilic to hydrophilic. After being processed by the DES samples, the parameter of I_w of those samples increases but the parameter of I_o decreases, suggesting that their hydrophilicity increases but lipophilicity decreases. In contrast, some DES-surfactant samples can also turn the wettability of the Berea sandstone samples from weakly hydrophilic to hydrophilic. However, those DES-surfactant samples will not only increase their hydrophilicity but also increase their lipophilicity.

2. Experimental section

2.1. Materials

Cetyltrimethylammonium bromide (CTAB, 99%), sodium dodecyl sulfate (SDS, 99%), alkyl polyglucoside (APG, ~50% in H_2O, paste-like), choline chloride (ChCl, 98%), urea (U, 99.5%), glycerol (GC, 99%), glycolic acid (GA, 99%), sodium chloride (NaCl, 99%), calcium chloride ($CaCl_2$, 97%), magnesium chloride ($MgCl_2$, 99%), sodium sulfate (Na_2SO_4, 99%), sodium bicarbonate ($NaHCO_3$, 99.7%), sodium carbonate (Na_2CO_3, 99.5%), and potassium chloride (KCl, 99%) are purchased from Sigma-Aldrich and used as received. Distilled water is home-made. Saline water is made by adding various salts into distilled water, as shown in Table 1. Crude oil is provided by Daqing Oilfield Company (China). The fractions of the saturates, aromatics, resins, and asphaltenes in the crude oil are 56.56, 25.41, 17.21, and 0.82 wt%, respectively. Cylindrical Berea sandstone samples (100–140 mesh) with a permeability of 400–500 μm^2 are used in the experiments.

2.2. Preparation of DES samples

A DES sample is prepared by mixing and heating HBA and HBD until they form a homogeneous liquid. Hydrogen bonds will be formed to connect the HBA and HBD in this process (Chen et al., 2019; Marcus, 2019). In this work, 3 DES samples are synthesized to prepare the DES solutions and the DES-surfactant solutions. These solutions are used to thoroughly investigate the effects of various DES samples and DES-surfactant mixtures on the

Table 1
The amounts of various salts used in preparing the saline water.

Component	H_2O	NaCl	$CaCl_2$	$MgCl_2$	Na_2SO_4	$NaHCO_3$	Na_2CO_3	KCl
Mass, g	3000	4.273	0.384	0.150	0.045	14.255	0.803	0.032

wettability of rock samples. Meanwhile, the performance of those samples in altering the wettability of sandstone samples are also compared. Herein, the detailed description of preparing the DES sample of ChCl/U is used as an example to show the preparation process of those DES samples. Typically, 13.692 g (0.1 mol) of ChCl is added to a beaker. Then, 12.012 g (0.2 mol) of U is added to the beaker. The beaker containing two agents is then transferred into an oven and heated for several hours at a temperature of 80 °C. After the two agents are completely dissolved into a homogeneous liquid, the DES sample of ChCl/U is successfully prepared. Using the same methods, other two DES samples are prepared using different agents with different molar ratios. The molar ratios between ChCl and various HBDs and the corresponding temperatures for preparing these DES samples are listed in Table 2.

2.3. Preparation of the DES and DES-surfactant solutions

Several DES solutions and DES-surfactant solutions are prepared using the saline water to determine their effect on the wettability of the sandstone samples. Six DES solutions with the concentrations of 0.5% and 1.5% are prepared by adding certain amounts of the three synthesized DES samples to saline water. Then, some surfactant solutions are prepared using three surfactants at a fixed concentration of 0.02 wt% which is much smaller than the critical micelle concentrations (CMCs) of these surfactants. Afterwards, different amounts of the three DES samples are added into the surfactant solutions to prepare the DES-surfactant solutions with DES concentrations of 0.5%, 1%, and 1.5%.

2.4. Wettability measurements

In this section, the procedure of measuring the wettability of sandstone samples using the Amott wettability measurement method (Anderson, 1986; Li et al., 2021) will be shown. The lengths (L) and diameters (D) of the Berea sandstone samples used in these experiments are in the ranges of 4.7–5.2 cm and 2.5–2.9 cm, respectively. The average porosity (Φ), permeability (K), and irreducible water saturation (S_{wi}) of these cores are around 20.3%, 420×10^{-3} μm^2 (420 mD), and 6.2%, respectively. The wettability tests are conducted with the following steps.

(1). Sample processing: The sandstone samples are processed by sequentially being immersed in saline water for 12 h, being flooded by crude oil to reach irreducible water saturation, and being aged in crude oil for 7 d at 45 °C.
(2). Water imbibition: Water is spontaneously imbibed into the aged Berea sandstone samples by immersing them into the saline water using a water imbibition meter (Fig. 1(a)) at 45 °C. The volume of the displaced oil by the spontaneous imbibition of the water is recorded as V_{os}.
(3). Water flooding: The Berea sandstone samples are flooded by water using a core flooding apparatus (Fig. 1(c)) at 45 °C until no oil is produced by the forced displacement of water. The volume of the displaced oil by the forced displacement is recorded as V_{of}.
(4). Oil imbibition: Using the same method as the water imbibition, oil is spontaneously imbibed into the Berea sandstone samples. The volume of the displaced water by the spontaneous imbibition of crude oil is recorded as V_{ws}.
(5). Oil flooding: Using the same method as the water flooding, oil is flooded into the Berea sandstone samples to displace water. The volume of the displaced water by the forced displacement of crude oil is recorded as V_{wf}.

More wettability measurements are then conducted by changing the prepared DES solutions or the DES-surfactant solutions. Based on the recorded four volumes (V_{os}, V_{of}, V_{ws}, and V_{wf}), the parameters for characterizing the wettability of the sandstone samples can be obtained by the following formula (Isah et al., 2023):

$$I_w = \frac{V_{os}}{V_{os} + V_{of}} \tag{1}$$

$$I_o = \frac{V_{ws}}{V_{ws} + V_{wf}} \tag{2}$$

$$I_A = I_w - I_o \tag{3}$$

where I_w is the displacement-by-water index, I_o is the displacement-by-oil index, and I_A is the wettability index. I_w and I_o can tell us the hydrophilicity and lipophilicity of the sandstone samples. A rock sample has a stronger hydrophilicity or lipophilicity if the I_w or I_o has a larger value. I_A indicates the overall wettability of a rock sample. A rock sample is strongly lipophilic, lipophilic, weakly lipophilic, neutral, weakly hydrophilic, hydrophilic, and strongly hydrophilic if I_A lies in the range of $-1.0 < I_A < -0.7$, $-0.7 < I_A < -0.3$, $-0.3 < I_A < -0.1$, $-0.1 < I_A < 0.1$, $0.1 < I_A < 0.3$, $0.3 < I_A < 0.7$, and $0.7 < I_A < 1.0$, respectively (Isah et al., 2023).

Table 2
The HBA-HBD molar ratios and heating temperatures used for preparing the DES samples.

DES sample (HBA/HBD)	Molar ratio, mol:mol	Preparing temperature, °C
ChCl/U	1:2	80
ChCl/GC	1:3	80
ChCl/GA	1:1	120

2.5. Micromodel flooding test

Micromodel flooding tests are carried out to investigate the effects of the SDS solution and the 1.5% ChCl/GC-SDS solution on the oil recovery and residual oil distribution. The micromodel is prepared by etching the surface of a selected area of a glass plate using hydrofluoric acid. The images in the dynamic flooding process are captured by an image capture system named eBUS Player for JAI. The experimental apparatus used in the micromodel flooding tests is shown in Fig. 2. The micromodel flooding tests are conducted in the order of oil injection, water flooding, SDS solution flooding, and ChCl/GC-SDS flooding. More specifically, the crude oil sample is consistently injected into the micromodel by a single syringe pump with a flow rate of 200 μL/min until the micromodel is saturated by oil. Subsequently, the saline water is injected into the micromodel with a flow rate of 30 μL/min to displace the saturated oil until no more oil is produced from the outlet. Next, the SDS solution is injected into the micromodel with a flow rate of 30 μL/min to displace the residual oil. Finally, the ChCl/GC-SDS solution is injected with the same flow rate to further sweep the residual oil. In those processes, the change in the distribution of oil in the micromodel is monitored and captured by the image capture system.

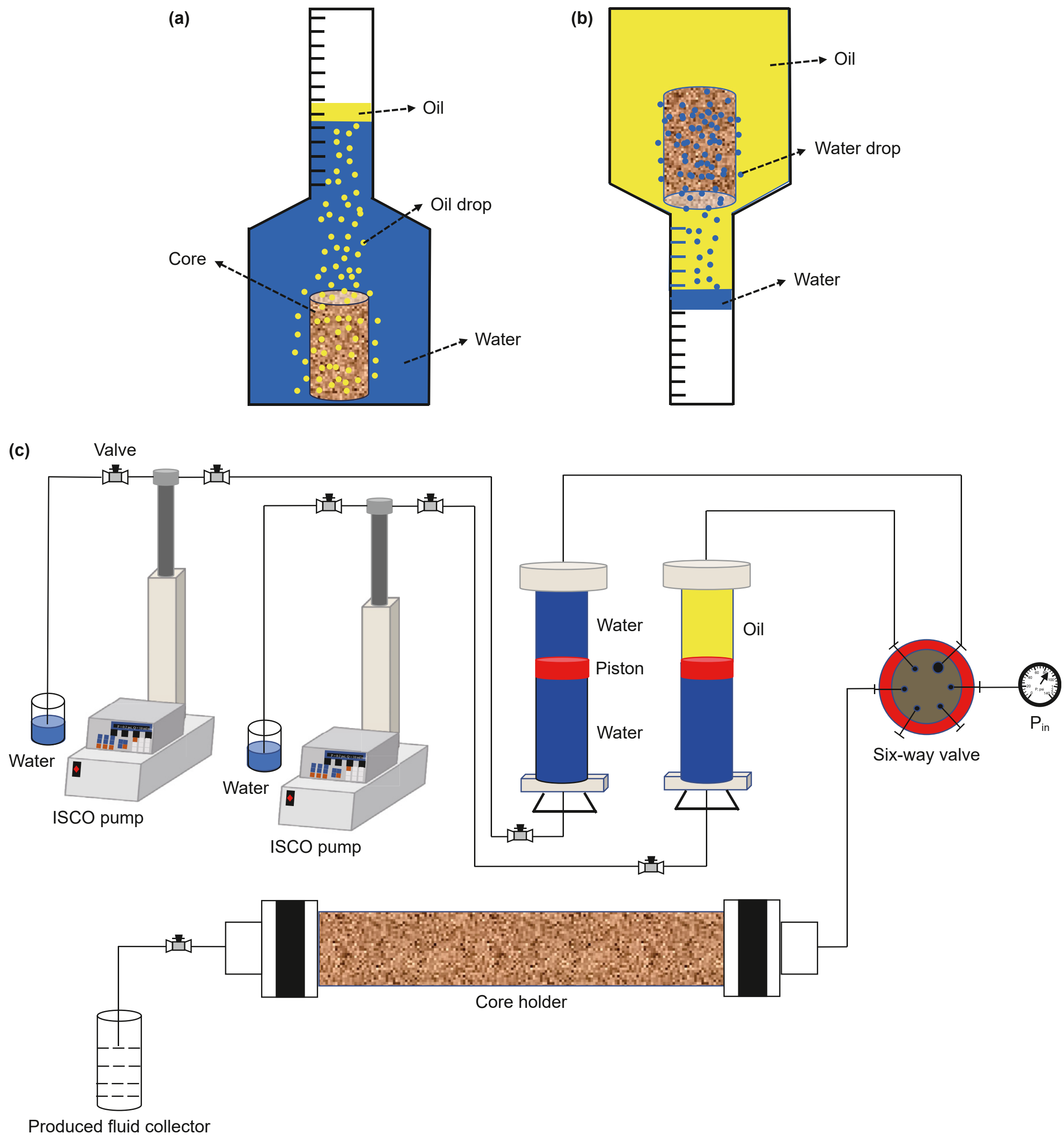

Fig. 1. Schematic showing the devices used in the imbibition experiments and flooding experiments: **(a)** water imbibition meter; **(b)** oil imbibition meter; and **(c)** the core flooding apparatus.

3. Results and discussion

3.1. The effects of DES on the wettability of sandstone samples

This section discusses the effects of various DES samples on the wettability of the sandstone samples. The results of the wettability measurements using the pure saline water and DES solutions are firstly shown. The properties of the sandstone samples and the corresponding solutions used in those wettability measurements are shown in Table 3. The recorded V_{os}, V_{of}, V_{ws}, and V_{wf} in the water imbibition, water flooding, oil imbibition, and oil flooding processes are shown in Table A1. The values of I_w, I_o, and I_A can be calculated using Eqs. (1)–(3) (See Fig. 3). From Fig. 3, it can be concluded that the Berea sandstone samples are weakly hydrophilic because the measured I_A equals 0.286 when the wettability is measured using the saline water. Compared to the saline water, I_w significantly increases while I_o remarkably decreases when the DES solutions are used during the measurements. Those variations suggest that the prepared DES samples can improve the hydrophilicity and weaken the lipophilicity of the sandstone samples. Meanwhile, the I_A of these sandstone samples also changes to the range of $0.3 < I_A < 0.7$, demonstrating that the DES samples alter the wettability of those sandstone samples from weakly hydrophilic to hydrophilic. Moreover, it can be found that the DES solution of 1.5% ChCl/GA has the most pronounced effect on altering the wettability of the sandstone sample by improving I_A from 0.286 to 0.580. Based on these findings, it can be concluded that the prepared DES samples can alter the wettability of the

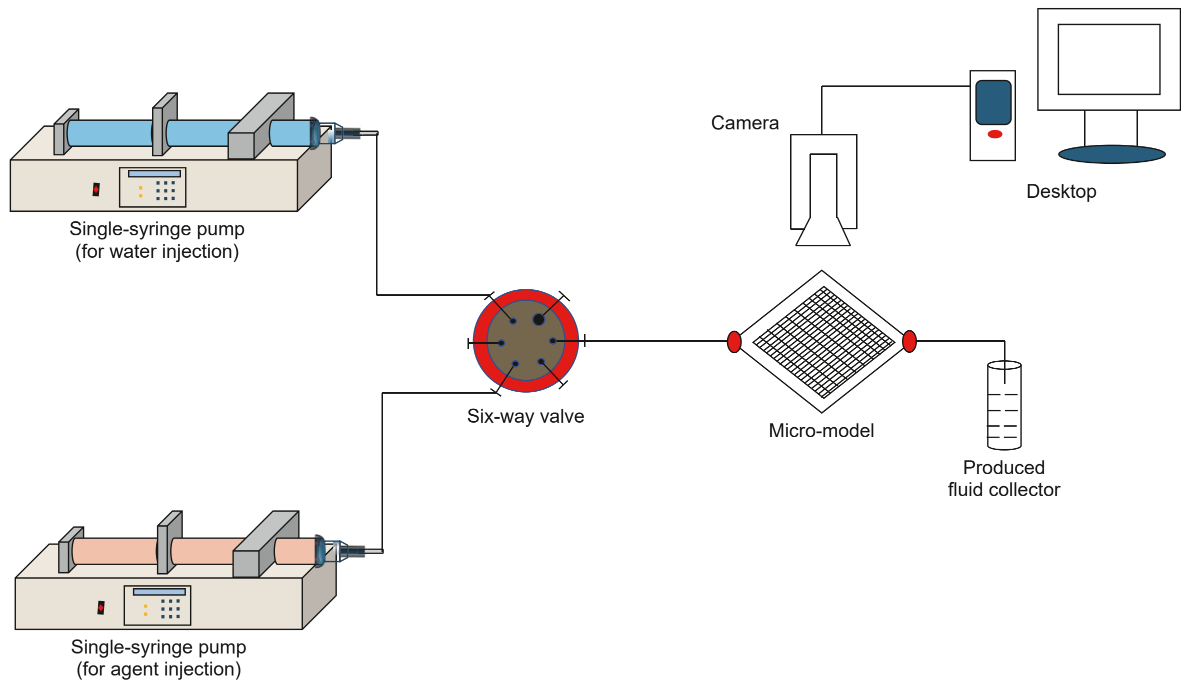

Fig. 2. Experimental apparatus used in the micromodel flooding tests.

Table 3
The properties of the sandstone samples and the corresponding solutions used in the wettability measurements.

Core No. #	L, cm	D, cm	Φ, %	K, 10^{-3} μm^2	S_{wi}, %	Solution
1	4.906	2.528	18.74	482.373	6.55	Pure saline water
2	4.904	2.520	20.90	479.159	6.99	0.5% ChCl/U
3	4.884	2.520	20.72	383.725	6.76	1.5% ChCl/U
4	4.936	2.530	20.14	423.311	6.02	0.5% ChCl/GC
5	4.908	2.520	20.80	531.269	6.08	1.5% ChCl/GC
6	4.890	2.520	20.25	414.367	5.89	0.5% ChCl/GA
7	5.220	2.520	19.22	425.417	5.38	1.5% ChCl/GA

Note: 0.5% ChCl/U represents a ChCl/U solution with a weight concentration of 0.5%.

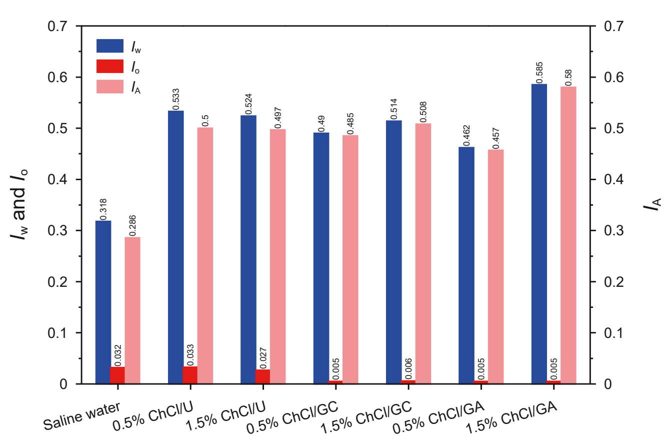

Fig. 3. The calculated I_w, I_o, and I_A of the Berea sandstone samples when measuring their wettability using different DES solutions.

sandstone samples by improving their hydrophilicity and simultaneously weakening their lipophilicity.

Table 4
The properties of the sandstone samples and the corresponding DES-CTAB solutions used in the wettability measurements.

Core No. #	L, cm	D, cm	Φ, %	K, 10^{-3} μm^2	S_{wi}, %	DES-CTAB solutions
8	5.036	2.532	20.33	431.204	4.940	0.5% ChCl/U-CTAB
9	4.994	2.534	20.60	423.254	5.510	1% ChCl/U-CTAB
10	4.944	2.526	20.52	436.725	5.980	1.5% ChCl/U-CTAB
11	4.902	2.588	19.53	407.370	6.720	0.5% ChCl/GC-CTAB
12	4.932	2.520	19.47	336.779	5.650	1% ChCl/GC-CTAB
13	4.790	2.526	20.73	395.816	6.980	1.5% ChCl/GC-CTAB
14	4.870	2.530	20.97	443.158	7.07	0.5% ChCl/GA-CTAB
15	5.000	2.528	20.54	384.087	7.380	1% ChCl/GA-CTAB
16	5.200	2.528	20.72	400.667	6.149	1.5% ChCl/GA-CTAB

Note: 0.5% represents a DES-CTAB solution containing a DES concentration of 0.5 wt% and a CTAB concentration of 0.02 wt%.

3.2. The effects of DES-surfactant on the wettability of sandstone samples

In this section, the effects of various DES-surfactant solutions on the wettability of the sandstone samples are investigated. The properties of the sandstone samples and the corresponding DES-CTAB solutions used in the wettability measurements are shown in Table 4. The volumes of the collected oil and water during the water imbibition, water flooding, oil imbibition, and oil flooding (i.e., V_{os}, V_{of}, V_{ws}, and V_{wf}) in the wettability measurements are shown in Table A2. Using these data, the values of I_w, I_o, and I_A of the sandstone samples can be determined when their wettability is measured using different DES-CTAB solutions, as shown in Fig. 4(a). Compared to using the saline water, various DES-CTAB solutions will lead to increases in the I_w and I_A of the Berea sandstone samples. Meanwhile, the I_A of these samples is in the range of $0.3< I_A < 0.7$, suggesting that the DES-CTAB solutions can also alter the wettability of the sandstone samples from weakly hydrophilic to hydrophilic. However, the solution of 1% ChCl/U-CTAB weakens the hydrophilicity of the sandstone sample by decreasing the I_A from 0.286 to 0.268. Fig. 4(b) shows the comparison of the I_A values

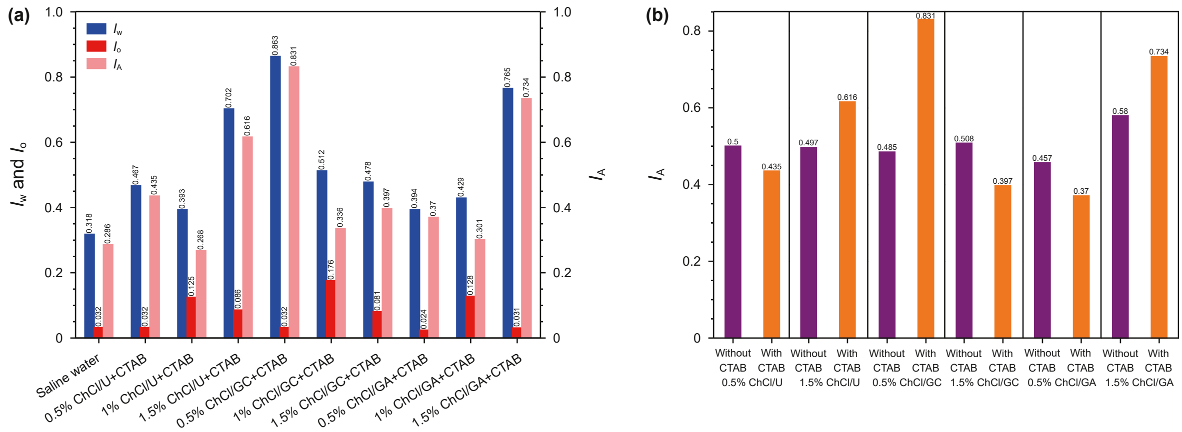

Fig. 4. The effects of DES-CTAB solutions on the wettability of the sandstone samples: **(a)** the calculated I_w, I_o, and I_A of the sandstone samples when measuring their wettability using different DES-CTAB solutions; **(b)** the comparison of the measured I_A of the sandstone samples between using pure DES solutions and DES-CTAB solutions.

obtained by using the DES solutions and the DES-CTAB solutions. It can be seen that the addition of CTAB to the DES samples of 1.5% ChCl/U, 0.5% ChCl/GC, and 1.5% ChCl/GA can further improve the hydrophilicity of the Berea sandstone samples. In contrast, adding CTAB to the DES samples of 0.5% ChCl/U, 1.5% ChCl/GC, and 0.5% ChCl/GA will weaken the hydrophilicity of the Berea sandstone samples. Therefore, it can be concluded that the concentration and type of the DES sample are crucial factors affecting the performance of the DES-CTAB solutions in altering the wettability of the sandstone samples.

Moreover, comparing the measured parameters using the DES solutions and the DES-CTAB solutions, the DES solutions can improve the hydrophilicity but weaken the lipophilicity of the sandstone samples. In contrast, the DES-CTAB solutions lead to increases in the I_w and I_o, suggesting that the hydrophilicity and lipophilicity of the sandstone samples are simultaneously improved.

Some results obtained from this study are consistent with the experimental results from the previous studies (Mohsenzadeh et al., 2015b; Atilhan and Aparicio, 2021; Sanati et al., 2022). For example, Sanati et al. (2021) investigated the effects of a DES-CTAB (ChCl/citric acid-CTAB) mixture and a pure DES (ChCl/citric acid) sample on changing the wettability of a carbonate sample through contact angle measurements. Their results suggested that both the pure DES sample and the DES-CTAB solution could alter the wettability from oil-wet to water-wet, and the DES-CTAB solution showed a better performance. This study also indicated that mixing CTAB with some DES sample could provide a better performance in altering the wettability to became more water-wet. However, some new findings are obtained in our study. First, for some types of DES samples, adding surfactants may not necessarily yield a better performance in altering the wettability of rock samples. Second, this study provides new insights into how mixing surfactants with DES samples affects the hydrophilicity and lipophilicity of sandstones.

SDS and APG are then used to examine the effects of the DES-SDS and DES-APG solutions on the wettability of the sandstone samples. The properties of the used sandstone samples and the corresponding DES-SDS and DES-APG solutions in the wettability measurements are shown in Tables 5 and 6. The volumes of the collected oil and water during the water imbibition, water flooding, oil imbibition, and oil flooding (i.e., V_{os}, V_{of}, V_{ws}, and V_{wf}) in the wettability measurements are shown in Table A3 and Table A4. The determined I_w, I_o, and I_A of those sandstone samples are shown in Figs. 5(a) and 6(a). From Figs. 5(a) and 6(a), similar conclusions as above can be obtained. The I_A of the sandstone samples significantly increases when using the DES-SDS and DES-APG solutions compared to using the saline water. Meanwhile, the I_A of those sandstone samples is in the range of $0.3 < I_A < 0.7$, demonstrating that the DES-SDS and DES-APG solutions alter the wettability of the sandstone samples from weakly hydrophilic to hydrophilic. Meanwhile, DES-SDS and DES-APG solutions can increase the I_w and I_o, indicating that they can simultaneously improve the hydrophilicity and lipophilicity of the sandstone samples. Similarly, Fig. 5(b) and 6(b) illustrate that most of the DES solutions have a stronger effect on improving the hydrophilicity of the sandstone samples than the corresponding DES-SDS and DES-APG solutions.

Furthermore, the measured I_w and I_o of those sandstone samples are summarized when their wettability is measured using different solutions, as shown in Fig. 7. As aforementioned, the I_w of all these sandstone samples increases when their wettability is measured using the prepared DES and DES-surfactant solutions. The increase

Table 5
The properties of the sandstone samples and the corresponding DES-SDS solutions used in the wettability measurements.

Core No. #	L, cm	D, cm	Φ, %	K, 10^{-3} μm^2	S_{wi}, %	DES-SDS solutions
17	4.872	2.528	20.11	367.183	6.20	0.5% ChCl/U-SDS
18	4.962	2.528	20.25	461.003	5.79	1% ChCl/U-SDS
19	4.958	2.526	20.58	481.829	6.08	1.5% ChCl/U-SDS
20	5.010	2.532	20.24	382.956	5.56	0.5% ChCl/GC-SDS
21	4.844	2.528	20.27	442.986	5.87	1% ChCl/GC-SDS
22	4.940	2.520	19.50	360.192	6.91	1.5% ChCl/GC-SDS
23	4.834	2.530	21.59	358.227	8.00	0.5% ChCl/GA-SDS
24	4.868	2.530	19.18	349.461	5.28	1% ChCl/GA-SDS
25	4.908	2.526	20.60	471.710	5.14	1.5% ChCl/GA-SDS

Table 6
The properties of the sandstone samples and the corresponding DES-APG solutions used in the wettability measurements.

Core No. #	L, cm	D, cm	Φ, %	K, 10^{-3} μm^2	S_{wi}, %	DES-APG solutions
26	4.978	2.528	20.21	367.148	8.20	0.5% ChCl/U-APG
27	4.972	2.540	20.25	365.837	5.16	1% ChCl/U-APG
28	4.878	2.526	20.35	453.231	6.43	1.5% ChCl/U-APG
29	4.942	2.522	20.44	377.044	5.855	0.5% ChCl/GC-APG
30	4.888	2.530	20.46	420.337	5.801	1% ChCl/GC-APG
31	4.888	2.532	20.16	352.863	7.15	1.5% ChCl/GC-APG
32	4.622	2.526	20.65	387.928	5.097	0.5% ChCl/GA-APG
33	4.822	2.534	20.57	417.984	5.76	1% ChCl/GA-APG
34	4.922	2.530	20.59	424.706	6.031	1.5% ChCl/GA-APG

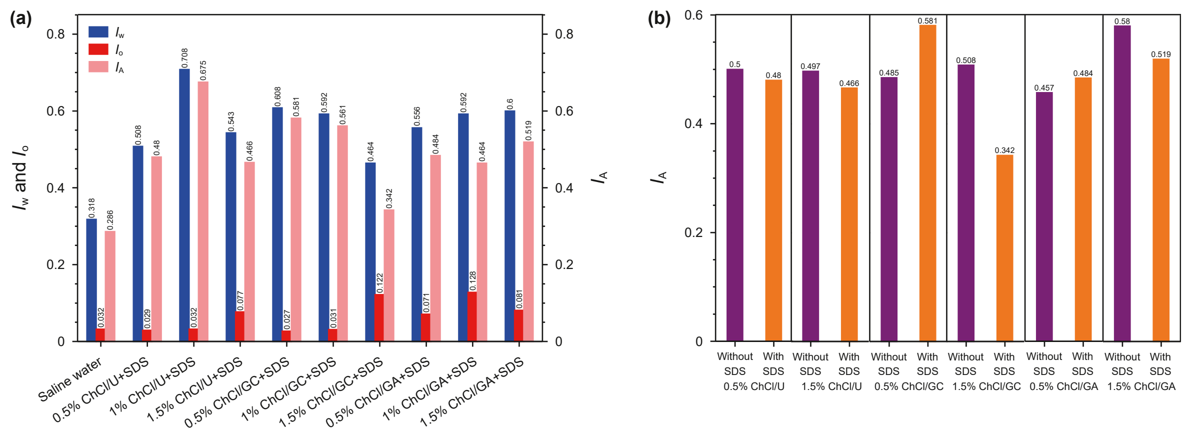

Fig. 5. The effects of DES-SDS solutions on the wettability of the sandstone samples: **(a)** the calculated I_w, I_o, and I_A of the Berea sandstone samples when measuring their wettability using different DES-SDS solutions; **(b)** the comparison of the measured I_A of the Berea sandstone samples between using pure DES solutions and DES-SDS solutions.

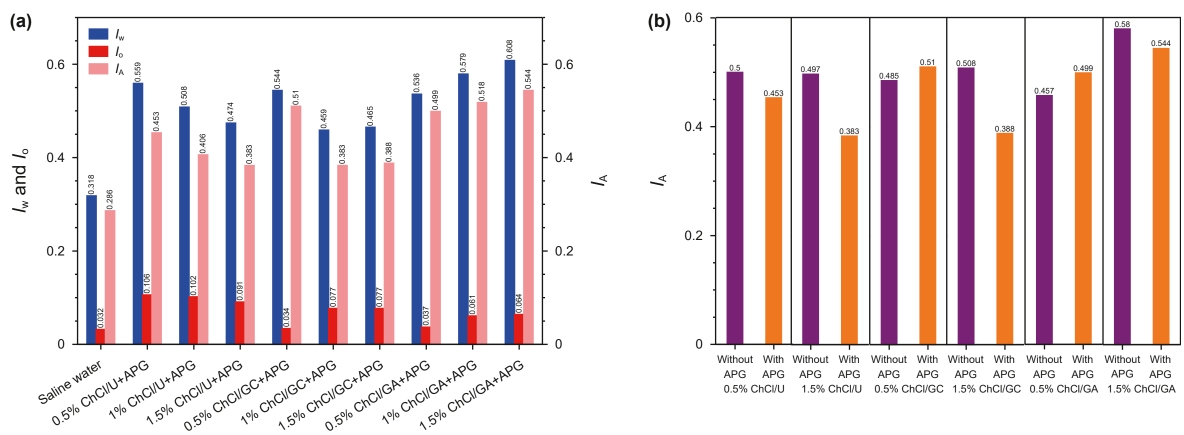

Fig. 6. The effects of DES-SDS solutions on the wettability of the sandstone samples: **(a)** the calculated I_w, I_o, and I_A of the Berea sandstone samples when measuring their wettability using different DES-APG solutions; **(b)** the comparison of the measured I_A of the Berea sandstone samples between using pure DES solutions and DES-APG solutions.

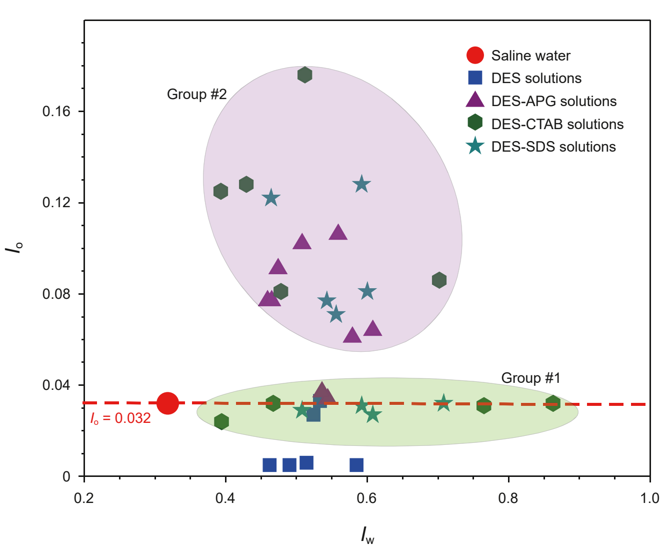

Fig. 7. The I_w and I_o of the sandstone samples when their wettability is measured using different solutions.

in the I_w suggests that the hydrophilicity of the sandstone samples is improved after being subjected to the DES and DES-surfactant solutions. In other words, the DES and DES-surfactant solutions can alter the wettability of the sandstone samples by improving the hydrophilicity. Meanwhile, the I_o of the sandstone samples becomes lower than 0.032 when their wettability is measured using the DES solutions (See the blue squares in Fig. 7). 0.032 is the I_o of the sandstone sample whose wettability is measured using the saline water. This result suggests that the DES samples can weaken the lipophilicity of the sandstone samples. However, the DES-surfactant solutions affect the wettability of the sandstone samples in different manners. These DES-surfactant solutions can be categorized into two groups based on the variations in the I_o of the sandstone samples, as shown in Fig. 7. The DES-surfactant solutions in group #1, as located in the green elliptic in Fig. 7, will not alter the lipophilicity of the sandstone samples because the I_o of those samples just slightly deviates from the red dash line. In contrast, the DES-surfactant solutions in group #2, as located in the magenta elliptic, will improve the lipophilicity of the sandstone samples because the I_o of those samples is much larger than 0.032. The DES-surfactant solutions belonging to the two groups are summarized in Table A5. Therefore, it can be concluded that the DES solutions prepared in this work can alter the wettability of the sandstone samples from weakly hydrophilic to hydrophilic by improving the hydrophilicity and weakening the lipophilicity of the sandstone samples. This will be beneficial for obtaining a higher oil recovery factor. As for the DES-surfactant solutions, they can alter the wettability of the sandstone samples in two different manners. First, some DES-surfactant solutions can alter the wettability from weakly hydrophilic to hydrophilic by greatly improving the hydrophilicity and simultaneously improving the lipophilicity of the sandstone samples. Second, other DES-surfactant solutions can alter the wettability from weakly hydrophilic to hydrophilic by just improving the hydrophilicity but having no effect on the lipophilicity of the sandstone samples.

Based on the wettability measurement using the saline water, the sandstone samples used in this work are found to be weakly hydrophilic. The chemical elements of silicon and oxygen on the surface of the sandstone samples can form hydrogen bonds with the saline water (Jia et al., 2022). Thus, water can spread along the surface of the sandstone samples, enabling these samples to be weakly water-wet, as shown in Fig. 8(a). When the wettability of the sandstone samples is measured using the DES solutions, the HBDs of the DES samples can also form hydrogen bonds with the elements of silicon and oxygen on the surface of the sandstone samples. Meanwhile, the HBAs of the DES samples can simultaneously form hydrogen bonds with the water molecules. As such, more water molecules can be bonded to the surface of the sandstone samples, leading to the alteration of the wettability of those samples from weakly hydrophilic to hydrophilic, as seen in Fig. 8(b). Moreover, when the wettability of the sandstone samples is measured using the DES-surfactant solutions, the surfactant molecules can be adsorbed by the surface of the sandstone samples through various ways, such as electrostatic attraction and hydrogen bonding (Kalam et al., 2021). The adsorbed surfactant molecules can form micelles to capture oil molecules, leading to an improvement in the lipophilicity of the sandstone samples. Meanwhile, the DES samples in the DES-surfactant solutions can form hydrogen bonds with water to improve the hydrophilicity. As a result, some DES-surfactant solutions can alter the wettability from weakly hydrophilic to hydrophilic by greatly improving the hydrophilicity and simultaneously improving the lipophilicity of the sandstone samples, as shown in Fig. 8(c). In addition, some researchers proposed some possible mechanisms of DES in altering the wettability of rock surface. For example, Sanati et al. (2021) proposed that DES samples altered wettability by forming hydrogen bonds with asphaltenic compounds of oil. In this way, the precipitation of asphaltenic compounds of oil on rock surface would be inhibited, leading to the rock surface being more hydrophilic. Moreover, the dissolution power of DES samples and the electrostatic attraction between the carboxylic groups of oil and choline cation may also attribute to the wettability alteration. In the presence of surfactants, the hydrophobic interaction between surfactants and oil could further enhance wettability alteration. However, more detailed experimental and theoretical studies are required to clarify the wettability alteration mechanisms caused by the combined use of DES and surfactant samples.

3.3. Micromodel flooding test results

Several micromodel flooding experiments are conducted to evaluate the effects of different agents on the residual oil distribution in a porous medium. The prepared micromodel for the flooding experiments is shown in Fig. 9(a). After oil saturation and initial water flooding, the oil distribution in the micromodel is presented in Fig. 9(b). From Fig. 9(b), we can see that a large amount of oil is trapped in the porous medium. Specifically, the trapped oil is distributed in small throats and pores connecting to those small throats. The sweep efficiency and displacement efficiency of water flooding are relatively low. Fig. 9(c)–(d) show the images of the micromodel captured in the process of SDS flooding and after SDS flooding. From these two figures, it can be found that SDS flooding can significantly improve oil recovery. SDS can decrease the interfacial tension between oil and the injected solution. As such, the residual oil can be dispersed in the injected solution, which helps the residual oil being washed out by the solution, e.g., regions A and B in Fig. 9(c). Meanwhile, SDS can alter the wettability of the micromodel, which enables the injected SDS solution to enter the throats. As a result, the originally residual oil being trapped in small throats can be swept by the injected SDS solution. Afterwards, the 1.5% ChCl/GC-SDS solution is injected into the micromodel after SDS flooding (See Fig. 9(e)). From Fig. 9(e), we can find that the oil recovery is greatly improved compared to the SDS flooding, and only little residual oil is trapped in the micromodel (See the red circle in Fig. 9(e)). Herein, several images in the DES-Surfactant solution flooding process are captured to illustrate the oil recovery improvement process. As shown in the yellow and red circles in Fig. 9(f), the residual oil is trapped within the small throats of the porous medium. After the injection of 1.5% ChCl/GC-SDS solution, the wettability of the micromodel becomes more water-wet. The change in the wettability enables the residual oil that is attached to the matrix surface to be displaced by the injected solution (See Fig. 9(g)–(h)). Comparison between Fig. 9(d) and (e) further indicates that most of the residual oil that is bonded to the matrix surface is produced after the 1.5% ChCl/GC-SDS solution flooding. Moreover, the 1.5% ChCl/GC-SDS solution has promising performance in reducing the interfacial tension between oil and the injected solution (Guo et al., 2024). Due to those factors, most of the residual oil in the post-surfactant flooding can be produced by the 1.5% ChCl/GC-SDS solution flooding.

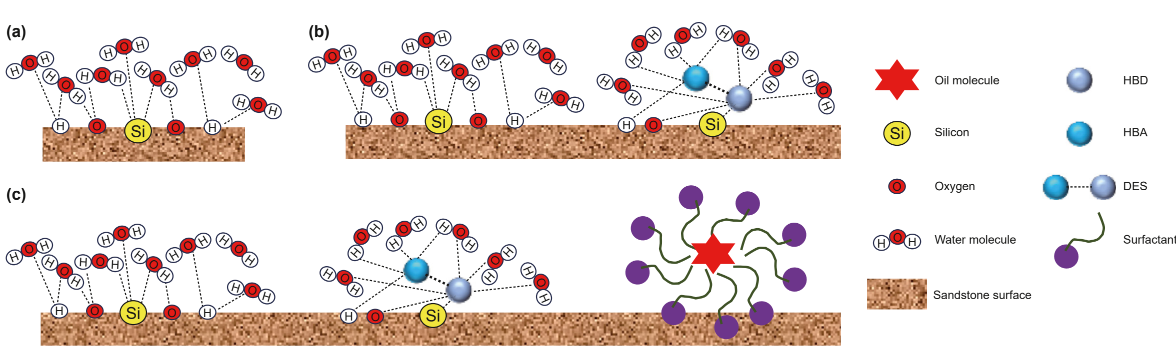

Fig. 8. The mechanisms of the DES solutions and the DES-surfactant solutions in altering the wettability of the sandstone samples: **(a)** a schematic showing the hydrogen bonds between water molecules and rock surface that help make the rock surface weakly hydrophilic; **(b)** a schematic showing the effect of the DES solutions on improving the hydrophilicity of the sandstone samples; **(c)** a schematic showing the effect of the DES-surfactant solutions on improving the hydrophilicity and the lipophilicity of the sandstone samples.

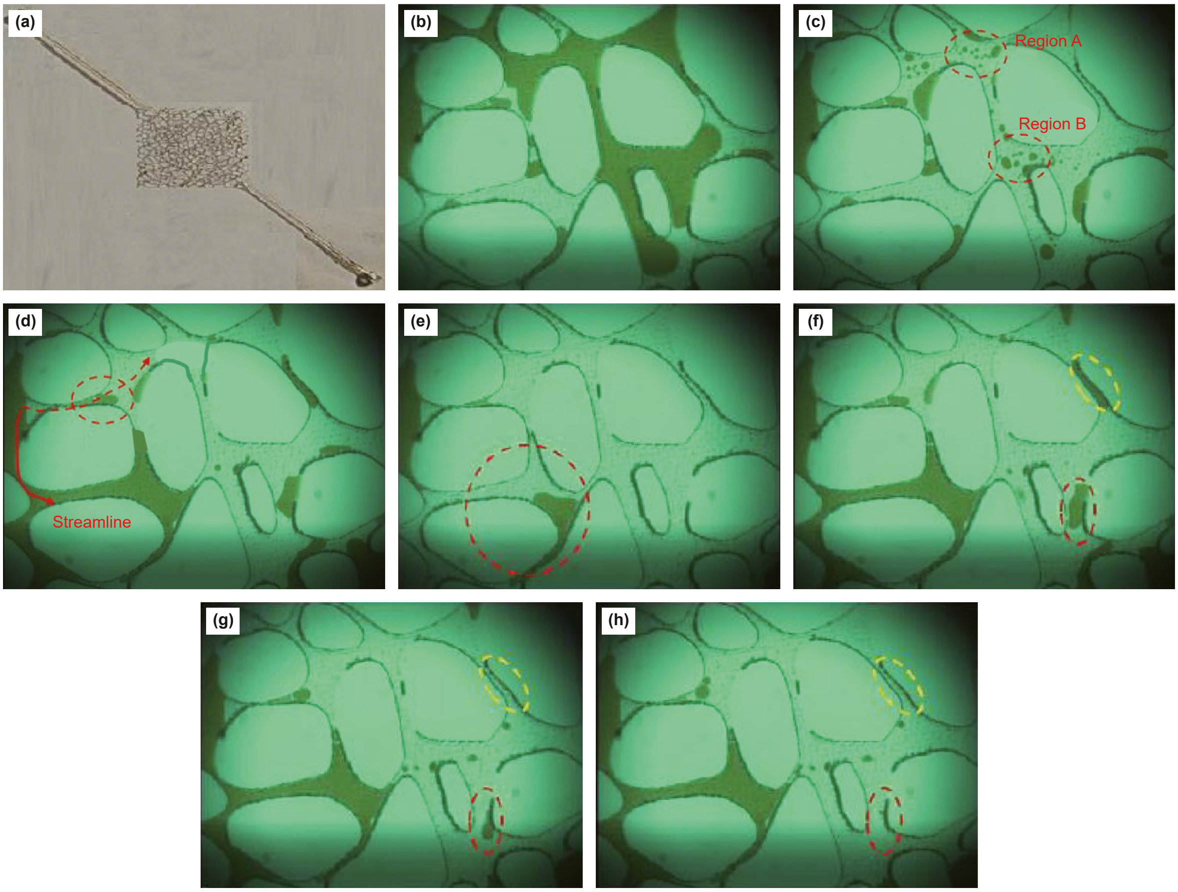

Fig. 9. Prepared micromodel for the flooding tests and captured micromodel images in different flooding stages: **(a)** micromodel; **(b)** the captured image after the water flooding; **(c)** the captured image during SDS solution flooding; **(d)** the captured image after SDS solution flooding; **(e)** the captured image after 1.5% ChCl/GC-SDS solution flooding; **(f)**–**(h)** the captured images during 1.5% ChCl/GC-SDS solution flooding.

4. Conclusions

This study investigates the effects of several DES samples and DES-surfactant solutions on the wettability of sandstone samples using the Amott wettability measurement method. The following conclusions can be obtained by comparing the measured wettability parameters, including the displacement-by-water index, the displacement-by-oil index, and the wettability index.

1). The Berea rock sample used in this study is weakly hydrophilic with I_o, I_w, and I_A of 0.318, 0.032, and 0.286, respectively. Being processed by the prepared DES samples, the wettability of the Berea sandstone samples is altered to hydrophilic ($0.7 > I_A > 0.3$) with an increased I_w but a lowered I_o.
2). DES-surfactant solutions can also modify the wettability of the Berea sandstone samples from weakly hydrophilic to hydrophilic. However, some DES-surfactant solutions can not only increase I_w but also increase I_o, suggesting that the lipophilicity of those sandstone samples will be improved by the DES-surfactant solutions.
3). Micromodel flooding experiments confirm the promising performance of 1.5% ChCl/GC-SDS in improving oil recovery. Compared to pure SDS flooding, more residual oil can be displaced by the 1.5% ChCl/GC-SDS solution flooding. Meanwhile, the amount of oil being attached to the matrix surface is greatly reduced after 1.5% ChCl/GC-SDS solution flooding, suggesting that 1.5% ChCl/GC-SDS solution can alter the wettability towards more water-wet.
4). However, this study has the following shortcomings. First, this study cannot clarify the exact mechanism in altering the wettability of the sandstone samples of the DES samples and DES-surfactant samples. Second, the existing experiments cannot show the enhanced oil recovery (EOR) performance of the used DES samples and DES-surfactant samples.
5). This research can be further improved in the future. First, molecular simulations and other microscopic experiments can be used to study the distributions of DES samples and DES-surfactant samples on the surface of sandstone samples to clarify the exact mechanism in altering the wettability of those samples. Second, more dynamic core flooding experiments can be conducted to investigate the EOR performance of those samples. Those studies may provide some new insights into the applications of DES samples in reservoir engineering.

CRediT authorship contribution statement

Jun-Hui Guo: Methodology, Investigation, Funding acquisition, Formal analysis. **Yun-Fei Bai:** Methodology, Investigation, Data curation, Conceptualization. **Lin Du:** Writing – original draft, Methodology, Data curation, Conceptualization. **Li-Ying Wei:** Supervision, Methodology, Funding acquisition. **Yu Zhao:** Validation, Supervision, Funding acquisition. **Xian-Bao Zheng:** Funding acquisition, Formal analysis, Data curation, Conceptualization. **Er-Long Yang:** Supervision, Formal analysis, Data curation. **Zhi-Guo Wang:** Funding acquisition, Formal analysis, Data curation. **Hai**

Huang: Supervision, Methodology, Investigation, Data curation, Conceptualization. **Wen-Tong Zhang:** Supervision, Methodology, Investigation, Formal analysis. **Hua-Zhou Li:** Writing – review & editing, Supervision, Methodology, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work is financially supported by the Scientific Research and Technology Development Projects of PetroChina (2023ZZ22-02), the Local Efficient Reform and Development Funds for Personnel Training Projects, and the China Scholarship Council (CSC) via a Ph.D. Scholarship (No. 202008510128).

Appendix

Table A1
The measured V_{os}, V_{of}, V_{ws}, and V_{wf} when measuring the wettability of the sandstone samples using the saline water and DES solutions.

Water solutions	V_{os}, mL	V_{of}, mL	V_{ws}, mL	V_{wf}, mL
Pure saline water	0.70	1.50	0.05	1.52
0.5% ChCl/U	1.20	1.05	0.08	2.20
1.5% ChCl/U	1.10	1.00	0.05	1.80
0.5% ChCl/GC	1.25	1.30	0.01	2.00
1.5% ChCl/GC	0.90	0.85	0.01	1.70
0.5% ChCl/GA	1.20	1.40	0.01	2.00
1.5% ChCl/GA	1.20	0.85	0.01	1.90

Table A2
The measured V_{os}, V_{of}, V_{ws}, and V_{wf} when measuring the wettability of the sandstone samples using different DES-CTAB solutions.

DES-CTAB solutions	V_{os}, mL	V_{of}, mL	V_{ws}, mL	V_{wf}, mL
0.5% ChCl/U-CTAB	0.7	0.8	0.05	1.50
1% ChCl/U-CTAB	0.75	1.16	0.20	1.40
1.5% ChCl/U-CTAB	1.18	0.5	0.15	1.60
0.5% ChCl/GC-CTAB	1.3	5.03	0.05	1.50
1% ChCl/GC-CTAB	1.05	1.0	0.30	1.40
1.5% ChCl/GC-CTAB	1.1	1.2	0.15	1.70
0.5% ChCl/GA-CTAB	0.65	1.0	0.05	2.00
1% ChCl/GA-CTAB	1.05	1.4	0.25	1.70
1.5% ChCl/GA-CTAB	1.3	0.4	0.05	1.60

Table A3
The measured V_{os}, V_{of}, V_{ws}, and V_{wf} when measuring the wettability of the sandstone samples using different DES-SDS solutions.

DES-SDS solutions	V_{os}, mL	V_{of}, mL	V_{ws}, mL	V_{wf}, mL
0.5% ChCl/U-SDS	1.55	1.5	0.05	1.70
1% ChCl/U-SDS	1.5	0.62	0.05	1.50
1.5% ChCl/U-SDS	1.19	1.0	0.15	1.80
0.5% ChCl/GC-SDS	1.55	1.0	0.05	1.80
1% ChCl/GC-SDS	1.45	1.0	0.05	1.55
1.5% ChCl/GC-SDS	1.3	1.5	0.25	1.80
0.5% ChCl/GA-SDS	1.75	1.4	0.10	1.30
1% ChCl/GA-SDS	1.45	1.0	0.25	1.70
1.5% ChCl/GA-SDS	1.5	1.0	0.15	1.70

Table A4
The measured V_{os}, V_{of}, V_{ws}, and V_{wf} when measuring the wettability of the sandstone samples using different DES-APG solutions.

DES-APG solutions	V_{os}, mL	V_{of}, mL	V_{ws}, mL	V_{wf}, mL
0.5% ChCl/U-APG	1.65	1.3	0.25	2.10
1% ChCl/U-APG	1.55	1.5	0.25	2.20
1.5% ChCl/U-APG	1.35	1.5	0.20	2.00
0.5% ChCl/GC-APG	1.55	1.3	0.08	2.30
1% ChCl/GC-APG	1.7	2.0	0.20	2.40
1.5% ChCl/GC-APG	1.65	1.9	0.15	1.80
0.5% ChCl/GA-APG	1.5	1.3	0.08	2.10
1% ChCl/GA-APG	1.65	1.2	0.15	2.30
1.5% ChCl/GA-APG	1.55	1.0	0.15	2.20

Table A5
The DES-surfactant solutions belonging to the group #1 and the group #2.

Group #1	0.5% ChCl/GA + CTAB, 1.5% ChCl/GA + CTAB, 0.5% ChCl/U + CTAB, 0.5% ChCl/GC + CTAB 0.5% ChCl/U + SDS, 1% ChCl/U + SDS, 0.5% ChCl/GC + SDS, 1% ChCl/GC + SDS 0.5% ChCl/GA + APG, 0.5% ChCl/GC + APG
Group #2	1% ChCl/U + CTAB, 1.5% ChCl/U + CTAB, 1% ChCl/GC + CTAB, 1.5% ChCl/GC + CTAB, 1% ChCl/GA + CTAB, 0.5% ChCl/GA + SDS, 1% ChCl/GA + SDS, 1.5% ChCl/GA + SDS, 1.5% ChCl/GC + SDS, 1.5% ChCl/U + SDS 1% ChCl/GA + APG, 1.5% ChCl/GA + APG, 1% ChCl/GC + APG, 1.5% ChCl/GC + APG, 0.5% ChCl/U + APG, 1% ChCl/U + APG, 1.5% ChCl/U + APG

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