



# Study on the desulfurization performance of iron/ethanolamine/deep eutectic solvent system

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## Abstract

Deep eutectic solvent (DES) was applied as the solvent of iron/alcohol amine system, and the prepared iron/ethanolamine/DES system was found to be a good desulfurizer for H<sub>2</sub>S removal. The absorbents were characterized by Fourier transform infrared spectroscopy. The iron/ethanolamine/DES system showed a significantly enhanced desulfurization performance compared with DES solution of iron or alcohol amine separately. Besides, the absorbents showed relatively stable desulfurization performance, which could keep a high H<sub>2</sub>S removal efficiency in a wide temperature range from 30–90°C. The iron/ethanolamine/DES system could be recycled for at least three times. The desulfurization product was analyzed by energy dispersive spectrum and X-ray diffraction, and the desulfurization product was identified as sulfur element.

**Keywords** Deep eutectic solvent · Hydrogen sulfide · Iron · Ethanolamine

## Introduction

The removal of hydrogen sulfide (H<sub>2</sub>S) has been considered an important work in the process of industrial production (Wang et al. 2018; Yuan et al. 2019). The common methods for low concentration H<sub>2</sub>S removal could be divided into dry method and wet method due to the phase of desulfurizers. Porous materials such as activated carbon, molecular sieve, clay, and MOFs are commonly used in the dry methods for the adsorption of H<sub>2</sub>S (Liu et al. 2020; Liu and Wang 2017a; Zhang et al. 2018a). Furthermore, alkaline or oxidizing substances (such as amine, metallic oxides) are often fixed on the surface of adsorbents to enhance the selective removal performance for H<sub>2</sub>S (Yang et al. 2019). Among the conventional wet methods, alcohol amine aqueous solution is one of the most common desulfurizers (Wang 2003). Besides, the chelate iron system is considered an effective desulfurizer which

could oxidize sulfide to element sulfur (Hua et al. 2006; Liu et al. 2019a; Wubs and Beenackers 1994). The conventional chelate iron system is obtained by complexation reaction of Fe<sup>3+</sup> and ligand in aqueous solution. The common ligand includes triethanolamine (TEA) and ethylenediamine tetraacetic acid (EDTA) (Iliuta and Larachi 2003; Lenninger et al. 2018). The sulfur load of chelate iron system is relatively large, and the sulfur element could be recovered in the form of sulfur. Therefore, the chelate iron system has been regarded as an effective desulfurization method. However, degradation of desulfurizer, regeneration and separation of sulfur limited the wider application of chelate iron system (Deshmukh et al. 2013; Eng et al. 2000). In addition, the current chelate iron systems are all based on water, and the application of new solvents in this field has been rarely reported.

Deep eutectic solvent (DES) is a kind of ionic liquids (ILs) which composed of hydrogen bond donor and receptor (Garcia et al. 2015). The DES possesses similar properties with conventional ILs and far lower cost. For gas separation, DES has been considered potential substitute of aqueous solution due to their high stability and low steam pressure. In the field of acid gas capture, the ILs (containing DES) were found to be good absorbents for CO<sub>2</sub> and SO<sub>2</sub> (Wang et al. 2019; Wang et al. 2020b; Yang et al. 2020; Zhang et al. 2018b). In present, a large amount of literature has reported the application of ILs for H<sub>2</sub>S removal. It was found that the ILs, especially that functionalized by alkaline or oxidizing substances,

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showed high H<sub>2</sub>S removal ability under suitable conditions (Liu et al. 2017b; Liu et al. 2021; Liu and Wang 2017b). However, the applications of DES for H<sub>2</sub>S removal are still rarely reported. In our previous works (Liu et al. 2019b; Wang et al. 2020a), the desulfurization performances of alkaline or oxidizing DES-based solutions have been investigated, and the results showed that the functionalized DES has excellent desulfurization performance. Therefore, after using non-aqueous solvent such as DES as the alternative solvent of water, the obtained desulfurizer will have its unique properties and show advantages in some aspects.

In this work, to study the desulfurization performance of non-aqueous chelating iron system, DES was applied as the solvent of iron/alcohol amine system, and the H<sub>2</sub>S removal performance of the prepared iron/alcohol amine/DES system was investigated using dynamic absorption experiment. It is found that the iron/alcohol amine/DES system should be a good desulfurizer for H<sub>2</sub>S removal. The samples were characterized by Fourier transform infrared (FTIR) spectroscopy, and the desulfurization product was analyzed by energy dispersive spectrum (EDS) and X-ray diffraction (XRD).

## Materials and methods

### Materials

Choline chloride (C<sub>5</sub>H<sub>14</sub>ClNO, ChCl) was purchased from Tianjin Zhiyuan Chemical Reagent Co., Ltd., China; ethylene glycol ((CH<sub>2</sub>OH)<sub>2</sub>, EG), ethanolamine (C<sub>2</sub>H<sub>7</sub>NO, MEA), diethanolamine (DEA), and triethanolamine (TEA) were

purchased from Tianjin Zhiyuan Chemical Reagent Co., Ltd., China; ferric chloride hexahydrate (FeCl<sub>3</sub>·6H<sub>2</sub>O) was purchased from Tianjin Damao Chemical Reagent Co., Ltd., China; The reagents are all analytically pure.

### The preparation of iron/alcohol amine/DES system

The DES composed of ChCl and EG with a molar ratio of 1:2 was explained in our previous works (Wang et al. 2020a). The iron/MEA/DES system was obtained as follows: FeCl<sub>3</sub> was resolved into DES with a weight ratio of 1% (identified as Fe-DES), and then, MEA was added with a molar ratio with FeCl<sub>3</sub> ranging from 1:1 to 1:9. The mixture was stirred at room temperature until the uniform solution was obtained. The prepared solutions were identified as Fe-*x*MEA-DES, where *x* is the molar ratio of MEA to FeCl<sub>3</sub>. The preparation flow of Fe-*x*MEA-DES is shown in Fig. 1. The iron/DEA/DES system and iron/TEA/DES system were obtained similarly, and identified as Fe-*x*DEA-DES and Fe-*x*TEA-DES, respectively.

### Characterization instruments

FT-IR spectra of the samples were collected using Nicolet5700 Fourier transform infrared spectrometer (Thermo Nicolet Corp., America). The EDS analysis of desulfurization product was conducted by JSM7800F energy spectrometer (Oxford Instruments Corp., Britain). XRD of desulfurization product was conducted by D8-02 7KP2025 instrument (BrukerAxs Corp., Germany). The viscosities of absorbents were measured by a NDJ—5S Digital viscometer.

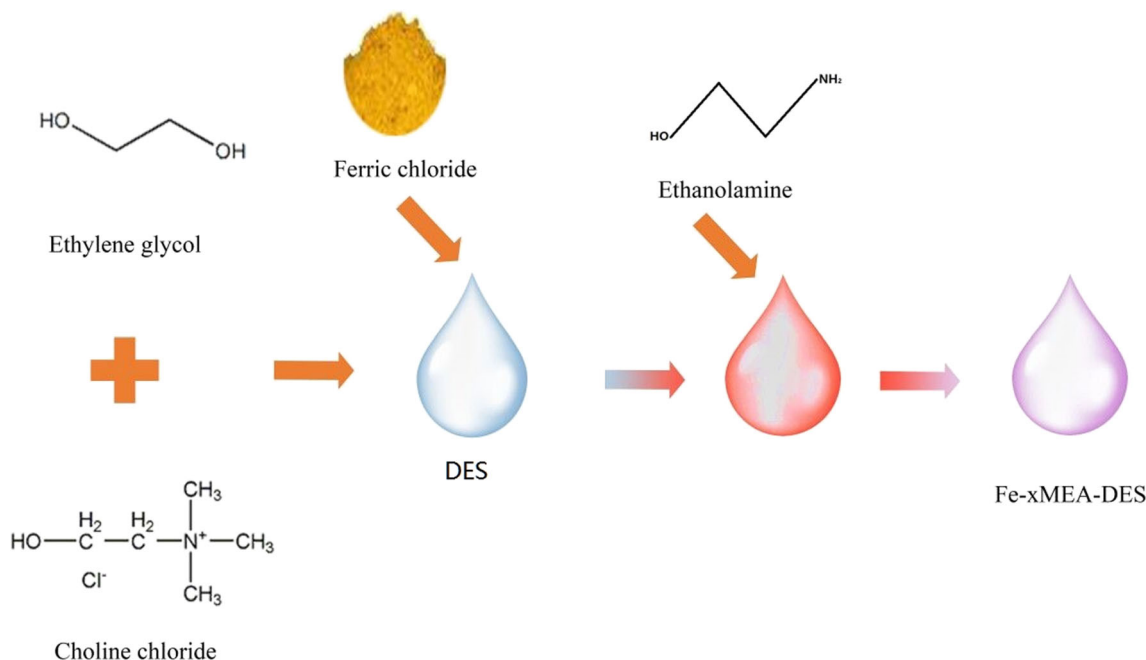
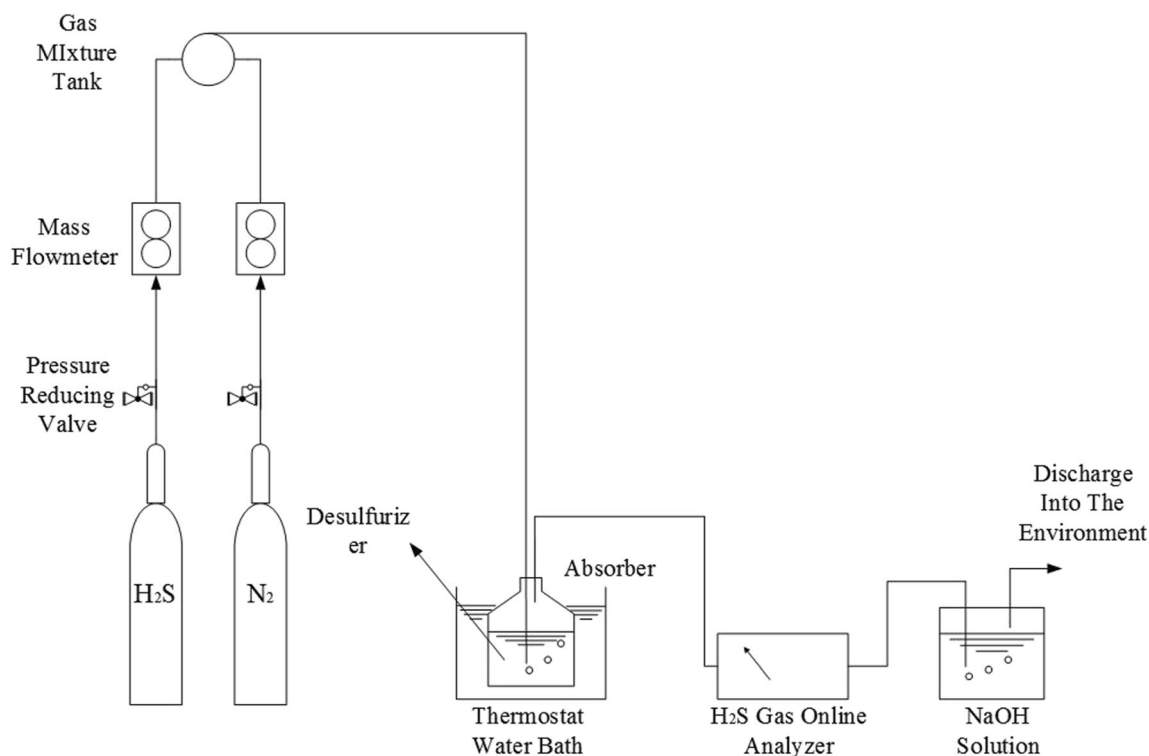


Fig. 1 The preparation flow of Fe-*x*MEA-DES



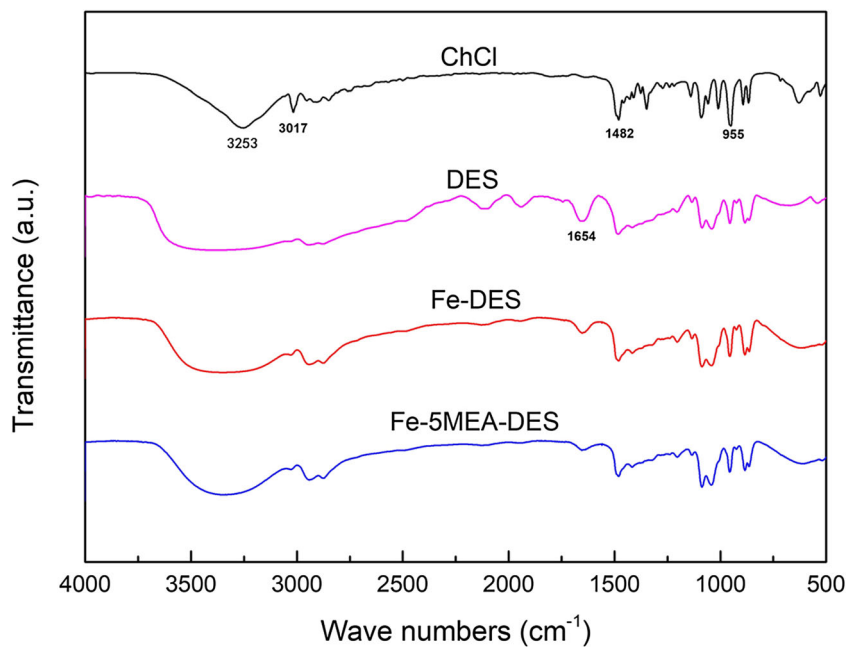
**Fig. 2** The schematic diagram for experimental apparatus

### H<sub>2</sub>S absorption experiment

As shown in Fig. 2, the H<sub>2</sub>S absorption experiment was conducted by simulated gas which composed of H<sub>2</sub>S and N<sub>2</sub> with a H<sub>2</sub>S concentration of 1000 mg/m<sup>3</sup>. The H<sub>2</sub>S containing gas was passed through the absorbent (4 g) by bubbling with a flow rate

of 200 mL/min. The H<sub>2</sub>S concentration was detected by a TH-990FIII H<sub>2</sub>S analyzer. The absorption temperature was controlled using a constant temperature water bath. The data obtained in this experiment were measured repeated for at least three times. The error analysis in the results has been analyzed in the form of error bar in figures. The standard deviations of experi-

**Fig. 3** The FT-IR spectra of ChCl, DES, Fe-DES as well as Fe-5MEA-DES



**Table 1** The viscosity of Fe-5MEA-DES at different temperature

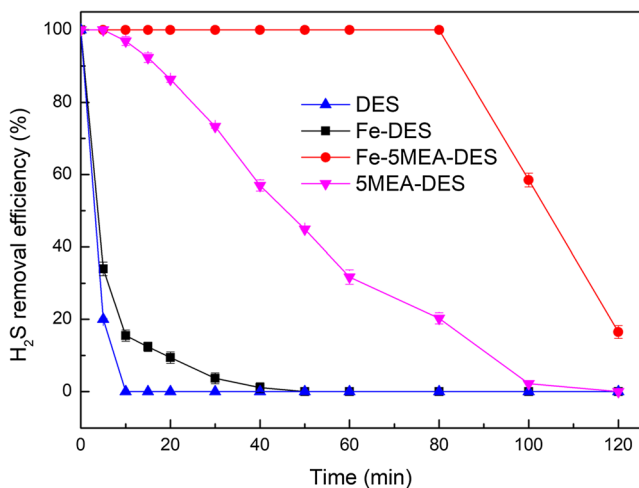
Temperature (°C)	30	60	90
Viscosity (mPa·s)	220	160	107

mental data were analyzed, and the maximum value of standard deviations is less than 5% (desulfurization rate). The results showed that the used method exhibits acceptable reproducibility.

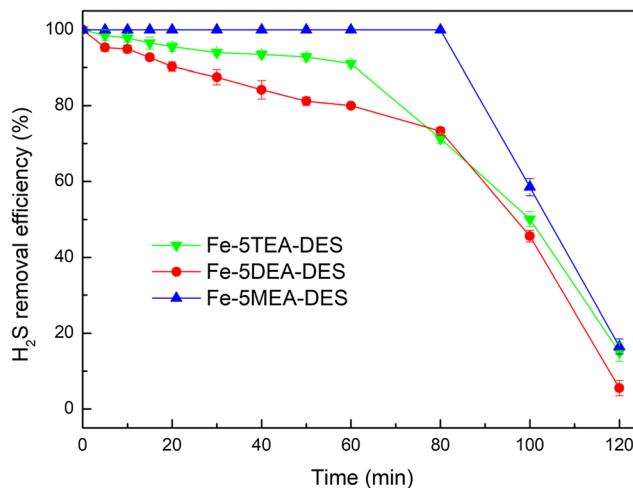
## Results and discussion

### Characterizations of absorbents

The FT-IR spectra of ChCl, DES, Fe-DES, and Fe-5MEA-DES are shown in Fig. 3. For ChCl, the peaks at 3253 and 1482  $\text{cm}^{-1}$  should be assigned to the presence of O-H bond. The peak at 3017  $\text{cm}^{-1}$  is attributed to the stretching vibration of C-H bond. The peak at 955  $\text{cm}^{-1}$  is caused by the stretching vibration of C-O bond (Huang et al. 2017). After the addition of EG, a new peak at 1654  $\text{cm}^{-1}$  was observed in the spectrum of the obtained DES, which should be assigned to the formation of hydrogen bonds (Keuleers et al. 2000). Furthermore, the spectra of DES with FeCl<sub>3</sub> only (Fe-DES) and Fe-5MEA-DES were collected. It is found that no significant change could be observed in the spectra before and after the addition of FeCl<sub>3</sub> and MEA, which revealed that the structure of DES was not destroyed after the preparation of iron/alcohol amine/DES system. Besides, after the addition of FeCl<sub>3</sub> and MEA, the viscosity of DES increased from 52 mPa·s (DES) to 220 mPa·s (Fe-5MEA-DES) at 30°C. The viscosity of Fe-5MEA-DES at different temperature is shown in Table 1. It could be found that the viscosity of absorbent decreased with the increasing of temperature.



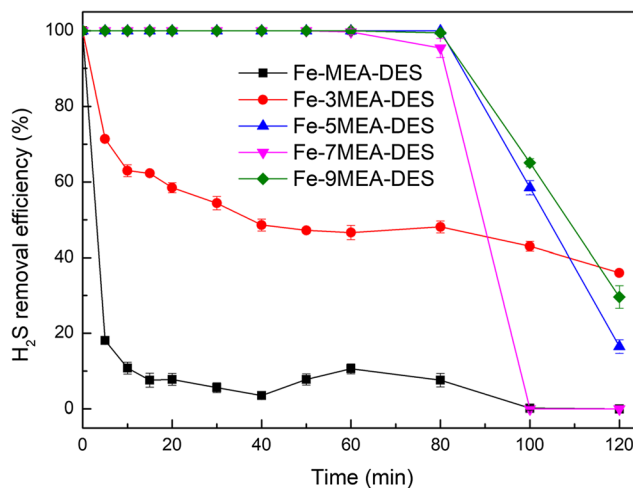
**Fig. 4** The desulfurization performances of different DES systems at 30°C



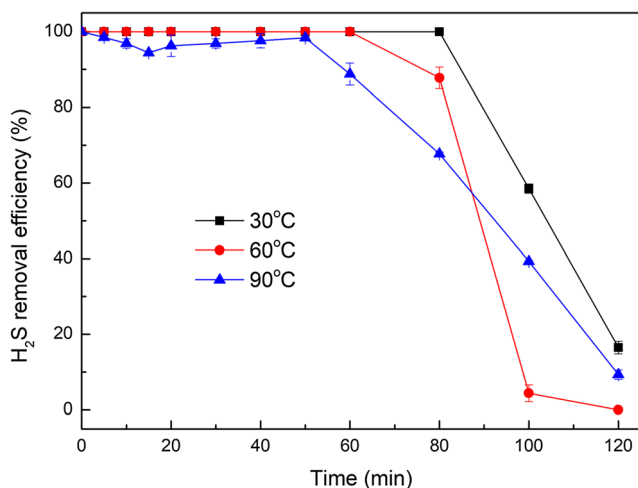
**Fig. 5** The desulfurization performances of iron/alcohol amine/DES systems with different alcohol amine at 30°C

### The desulfurization performance of iron/alcohol amine/DES systems

The desulfurization performances of different DES systems at 30°C are shown in Fig. 4. The pure DES without any additives was used for blank test. The DES without additive showed a poor H<sub>2</sub>S removal performance. The H<sub>2</sub>S removal efficiency decreased to 0 sharply at the beginning of absorption experiment. After the introduction of FeCl<sub>3</sub>, a slight enhancement could be observed, but the H<sub>2</sub>S removal efficiency is below 20% still after the absorption experiment was conducted for 10 min, which demonstrated that the addition of FeCl<sub>3</sub> alone could not make the most advantage of the oxidability of Fe<sup>3+</sup>, whereas in the presence of both FeCl<sub>3</sub> and MEA, the Fe-5MEA-DES exhibited an excellent H<sub>2</sub>S removal performance, and the H<sub>2</sub>S removal efficiency of Fe-5MEA-DES could keep 100% for 80 min. Compared with the desulfurization performance of DES with same amount of MEA only



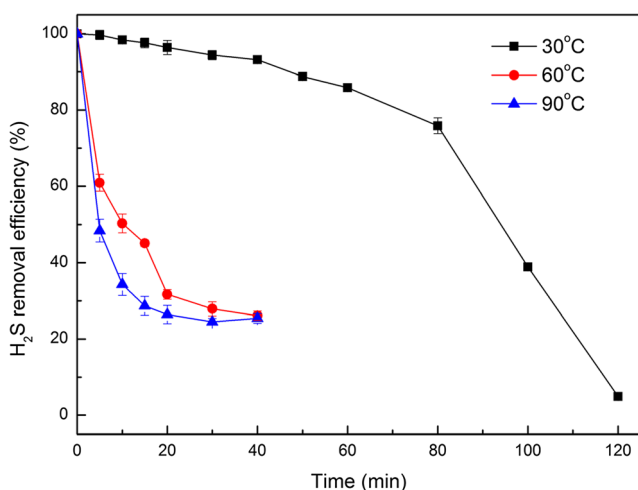
**Fig. 6** The desulfurization performances of iron/MEA/DES systems with different molar ratio of MEA and FeCl<sub>3</sub> at 30°C



**Fig. 7** The desulfurization performances of Fe-5MEA-DES under different temperature

(5MEA-DES), it is clearly that the removal of  $H_2S$  using Fe-5MEA-DES is the result of the synergistic effect of  $FeCl_3$  and MEA, rather than a simple superposition of the two substances. According to literatures (Dixon and Williams 1950; Salem 1995), the  $Fe^{3+}$  and alcohol amine would form complex compound, and then, the desulfurization performance was enhanced (Zheng et al. 1996). The results of this experiment are in agreement with the reports.

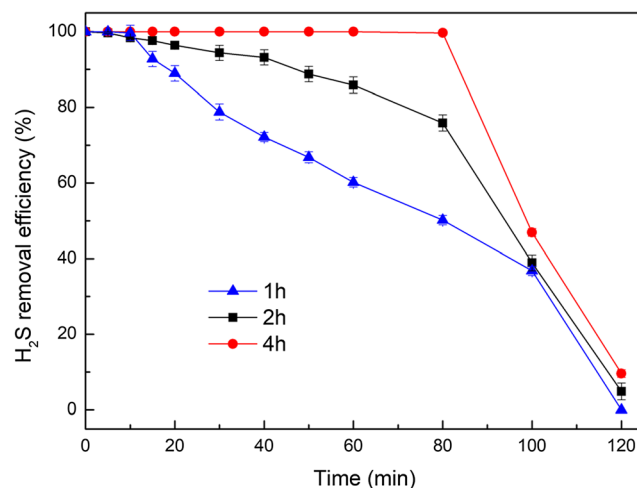
The  $H_2S$  removal performances of different iron/alcohol amine/DES systems with different alcohol amine were measured at 30°C. As shown in Fig. 5, the Fe-5MEA (DEA or TEA)-DES systems showed all excellent desulfurization performance. Among three systems, the Fe-5MEA-DES exhibited the highest  $H_2S$  removal efficiency, which could capture  $H_2S$  completely within 80 min. For Fe-5DEA-DES and Fe-5TEA-DES, the  $H_2S$  removal efficiency dropped to about 80% after 80 min. Therefore, the Fe-5MEA-DES was chosen in this work for further study.



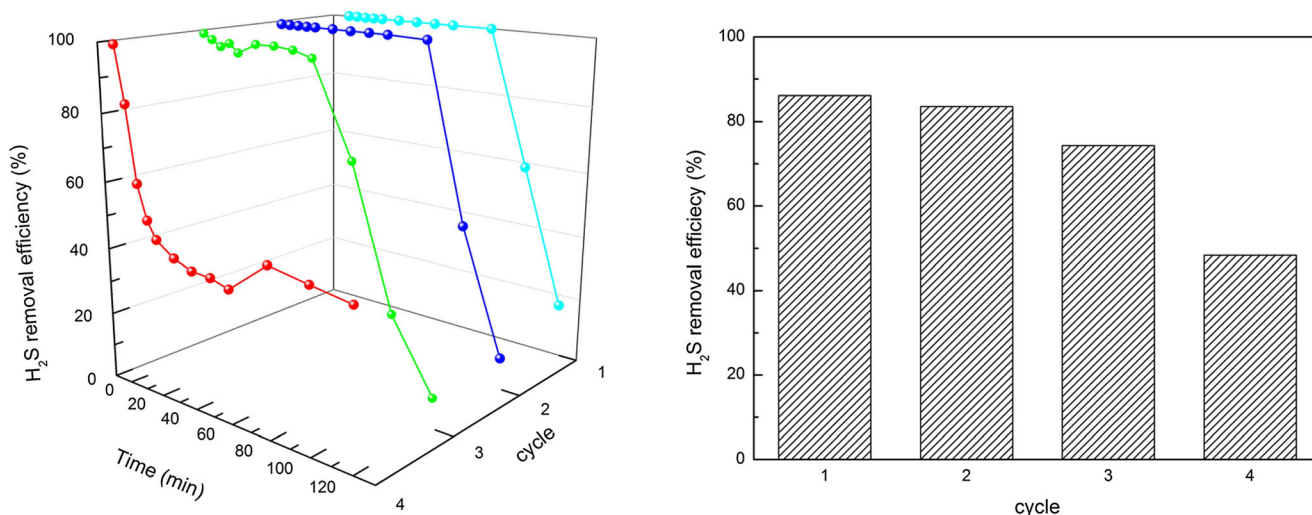
**Fig. 8** The desulfurization performances of Fe-5MEA-DES regenerated under different regeneration temperature for 2 h

The desulfurization performances of iron/MEA/DES systems with different molar ratio of MEA and  $FeCl_3$  were measured, and the results are shown in Fig. 6. The Fe-MEA-DES showed a poor  $H_2S$  removal ability, and the  $H_2S$  removal efficiency decreased to below 10% within 20 min. The desulfurization performance of iron/MEA/DES systems were enhanced as the molar ratio of MEA and  $FeCl_3$  continued to increase until the molar ratio reached 5. As the molar ratio of MEA and  $FeCl_3$  increased to 3, a great enhancement of the desulfurization performance could be observed. The  $H_2S$  removal efficiency of Fe-3MEA-DES kept about 60% for about 80 min. For Fe-5MEA-DES, Fe-7MEA-DES, and Fe-9MEA-DES, the desulfurization performance is almost the same. The  $H_2S$  removal efficiency of the three systems could all kept 100% for about 80 min, and then dropped sharply. The possible reason for this phenomenon is that when the ratio of MEA and  $FeCl_3$  reaches 5, MEA has been able to combine with most of iron ions, and even if the concentration of MEA continues to rise, limited by the concentration of iron ion, the performance would not continue to be enhanced. Considering the factor of cost performance, Fe-5MEA-DES was chosen for the subsequent experiments.

The desulfurization performances of Fe-5MEA-DES under different temperature are shown in Fig. 7. It could be seen that Fe-5MEA-DES exhibited the highest desulfurization performance at 30°C, and the  $H_2S$  removal efficiency decreased with the increasing of absorption temperature, which revealed that the absorption of  $H_2S$  in Fe-5MEA-DES prefer to a lower temperature. In general, Fe-5MEA-DES showed a relatively stable desulfurization performance, which could keep a high  $H_2S$  removal efficiency in the temperature range from 30–90°C.



**Fig. 9** The desulfurization performances of Fe-5MEA-DES regenerated at 30°C for different time



**Fig. 10** The multiple regeneration performance of Fe-5MEA-DES (regeneration condition: regeneration temperature, 30°C; regeneration time, 4 h)

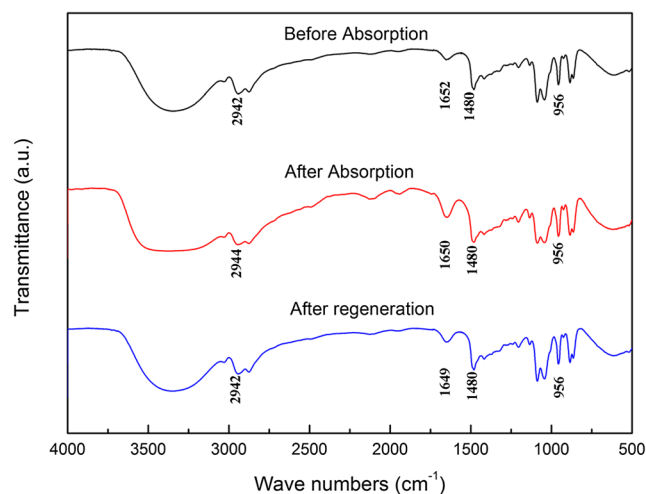
### The regeneration performance of Fe-5MEA-DES

The regeneration of exhausted Fe-5MEA-DES was carried out by air sweeping. The desulfurization performances of Fe-5MEA-DES regenerated with different temperature and time are shown in Fig. 8 and Fig. 9, respectively. The flow rate of air was 400 mL/min, and the absorption temperature was still 30°C. For Fig. 8, it is clearly that a relatively high regeneration temperature (60°C, 90°C) could hinder the regeneration. The Fe-5MEA-DES regenerated at 60°C or 90°C showed a poor desulfurization performance. By contrast, the desulfurization performance of Fe-5MEA-DES regenerated at 30°C recovered well. The regeneration of used Fe-5MEA-DES should be oxidation reaction, which is an exothermic reaction. Therefore, the regeneration of Fe-5MEA-DES should be conducted under a low temperature. As shown in Fig. 9, the effect of regeneration time on the desulfurization performance was investigated. It could be found that the desulfurization performance was enhanced with the increasing of regeneration time, which demonstrated that longer regeneration time is beneficial to the adequate regeneration of absorbent. When the regeneration time increased to 4 h, the desulfurization performance could almost be recovered. Therefore, the optimal regeneration condition was determined as: air sweeping at 30°C for 4 h.

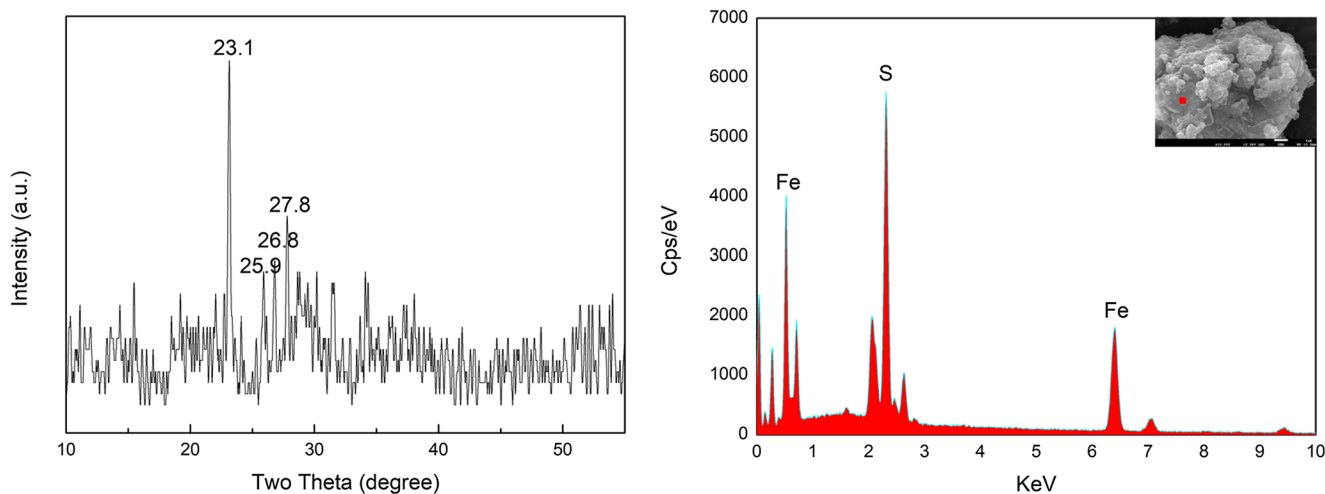
The multiple regeneration performance of Fe-5MEA-DES under optimal condition was studied, and the result was shown in Fig. 10. It could be found that the Fe-5MEA-DES could be recycled for at least three times. The H<sub>2</sub>S removal efficiency could still keep about 100% for 60 min in the third cycle. However, for the fourth time, the desulfurization performance decreased obviously, and the H<sub>2</sub>S removal efficiency dropped to about 50%. Compared with the result of other DES based desulfurizers (Liu et al. 2019b; Wang et al. 2020a), it could be found that the

removal efficiency of H<sub>2</sub>S and the temperature stability of Fe-5MEA-DES are similar to the DES composed of ChCl and FeCl<sub>3</sub>, higher than that of alkaline DES solution (DES and PEI), but the regeneration performance was relatively weak. Besides, the thermal stability of DES is lower compared with conventional ILs based desulfurizers (Liu et al. 2017a; Liu et al. 2017a; Taheri et al. 2021). However, considering the low cost and easy preparation, the DES based desulfurizers are acceptable as a new non-aqueous solution.

To further understand the absorption-regeneration process, the Fe-5MEA-DES before and after absorption, as well as after regeneration, were characterized using FT-IR. As shown in Fig. 11, no obvious change could be found from the FT-IR spectra of Fe-5MEA-DES after absorption or regeneration, which demonstrated that the structural and functional groups of MEA and DES were not significantly affected by the absorption process. The redox reaction of



**Fig. 11** The FT-IR spectra of Fe-5MEA-DES before and after absorption as well as after regeneration



**Fig. 12** The XRD patterns (left) and EDS result (right) of desulfurization product

iron ion and  $\text{H}_2\text{S}$  was not reflected in the FT-IR spectra. The solid desulfurization product was separated using centrifugation, and characterized by XRD and EDS, respectively. From Fig. 12, it could be found that the bulk of the desulfurization product was sulfur element. For the desulfurization mechanism, a problem in this work is the separation of desulfurization product. The separated solid product has been identified as sulfur. It can be confirmed that the removal mechanism of  $\text{H}_2\text{S}$  is oxidation reaction. However, because the property of DES solution is different from that of aqueous solution, the intermediate products could not be clearly separated and identified yet. Besides, according to the XRD pattern, there are many impurities in the desulfurization product. Combined the result of EDS, it could be inferred that a large amount of iron was attached on the surface of sulfur, which lead to the loss of iron from liquid. As a result, the desulfurization performance of absorbent decreased gradually in the multiple absorption-regeneration process. Consequently, the prevention of the loss of iron should be an important work in the future to improve the recycling performance of iron/alcohol amine/DES system.

## Conclusions

The iron/alcohol amine/DES system was found to be a good desulfurizer for  $\text{H}_2\text{S}$  removal. The samples were characterized by FTIR spectroscopy, and the desulfurization product was analyzed by EDS and XRD. The Fe-5MEA-DES showed a relatively stable desulfurization performance in a wide temperature range from 30–90°C, and could keep 100%  $\text{H}_2\text{S}$  removal efficiency for 80 min at 30°C. The optimal regeneration condition was determined as follows: air sweeping at 30°C for 4 h, and the Fe-5MEA-DES showed a relatively high regeneration performance for at least three times. After the fourth

time cycle, the desulfurization performance decreased by about 50%. The decrease of the desulfurization performance of absorbent in the multiple absorption-regeneration process should be attributed to the loss of iron element in the liquid. The solid desulfurization product was identified as sulfur element.

**Author contribution** Xinpeng Liu conceived and planned the whole work. Baohua Wang, Yahui Qiu, and Xiaole Dong carried out the experimental work. Baohua Wang and Yixin Song contributed to the analysis and interpretation of results. Qingmei Meng and Menghong Li were contributors in writing the manuscript. All authors read and approved the final manuscript.

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## Declarations

**Ethics approval and consent to participate** Not applicable.

**Consent for publication** Not applicable.

**Competing interests** The authors declare no competing interests.

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