



Efficient and selective absorption of SO₂ by low-viscosity matrine-based deep eutectic solvents



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ABSTRACT

Designing excellent liquid absorbents with low viscosity for efficient and selective absorption of sulfur dioxide (SO₂) from flue gas is important but still challenging. Herein, matrine-based binary deep eutectic solvents (DESs) were prepared by mixing matrine (Mat) with three different amides including *N*-methylformamide (MFA), *N*-ethylformamide (EFA), and *N*-methylacetamide (MAA). It is found that the viscosities of these three DESs were lower than 20 cP at room temperature, which is significantly lower than other previous DESs or ILs that have been reported. Moreover, the results of SO₂ capture showed that the absorption rate of SO₂ by the Mat-MFA (1:2) DES was very fast (<3 min) to achieve a relatively large absorption capacity (0.84 g/g at 293.2 K and 1 bar). Mat-MFA (1:2) DES also displayed very high selectivity (576) for the SO₂/CO₂ (10/90, v/v) mixture, which is higher than most of the liquid absorbents and solid adsorbents currently reported. Furthermore, breakthrough experiments verified the good separation performance of Mat-MFA (1:2) DES in capturing 2% SO₂ in the mixture of SO₂/N₂ or SO₂/CO₂, respectively. The results illustrated that the features of low viscosity, fast absorption rate, and high selectivity of SO₂ capture make the matrine-based DESs have great potential for flue gas desulfurization.

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1. Introduction

Currently, fossil fuels (e.g., coal) are the main source of energy in the world. Fossil fuels contain a certain amount of sulfur and therefore produce the pollutant sulfur dioxide (SO₂) during combustion. If SO₂ is directly emitted into the atmosphere, it has a detrimental effect on human health and the environment [1,2]. As a result, the effective control of SO₂ emissions has become an urgent issue. Flue gas desulfurization (FGD) is a traditional method for effective control SO₂ emissions in the industry [3,4]. Among them, large-scale solid absorbents, such as calcium desulfurization have been applied to react with SO₂ in the flue gas to achieve the purpose of desulfurization. However, a large amount of gypsum-like solid waste by-products will be generated, which can cause secondary pollution [5,6]. Distinguishing from solid absorbents, liquid absorption methods are becoming increasingly more popular to capture SO₂. Currently, organic amines are the mainstream used in industry to capture SO₂ from flue gas, after desorption, a sulfur resource can be achieved, but organic amines are volatile, easy to produce secondary pollution, and causes waste of resources [7,8]. Therefore, exploring new absorbents with low volatility and

proper strength of interaction with SO₂ is significant for the advancement of SO₂ capture technology.

Ionic liquids (ILs), as a salt composed of organic cations and organic or inorganic anions in a liquid state at room temperature [9], is a promising gas absorbent for SO₂ capture for their low volatility, good thermal stability, and designable structure [10–16]. For example, Jiang et al [17], prepared several ether-linked diamines protic carboxylate ILs and used them for SO₂ capture, it was found that a high uptake capacity of 1.04 mol_{SO2}/mol_{IL} was achieved by 50 wt% [BDMAEE][L]₂ aq at 313.2 K. After, Wang and his co-workers [18] synthesized a series of functional anions ILs for SO₂ absorption. It was found that the uptake capacity of [P₄₄₄₂][PyO] IL could reach 4.71 mol_{SO2}/mol_{ILs} at 293.2 K and 1 bar. These results indicated that SO₂ gas can be dissolved in ILs in large quantities, and the IL after absorbing SO₂ gas can be heated or depressurized to achieve regeneration. However, ILs are often expensive along with a complex preparation process, making them not green enough for benign SO₂ capture. Besides, insufficient absorption rates and inadequate selectivity for competing gases (e.g., CO₂), making them difficult to be applied in practice.

In recent years, deep eutectic solvents (DESs) consisting of hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA) have attracted increasing interest from researchers in various areas for their outstanding performance [19–24]. As a class of IL ana-

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logues, DESs have many advantages over traditional ILs, such as simple preparation, reliable purity, no purification, low cost, and degradability, making them suitable for SO₂ capture [21,25–31]. For example, Han and his co-workers [32] firstly reported the use of choline chloride (CC)-glycerol (Gly) DESs as SO₂ absorbent, they found that the maximum uptake capacity of CC: Gly (1:1) was 0.678 g_{SO2}/g_{DES} SO₂ at 293.2 K and 1 × 10⁵ Pa. Later, Liu et al. [33] synthesized a variety of amide-thiocyanide DESs, showing that the acetamide-potassium thiocyanide (3:1) can reach 0.588 g_{SO2}/g_{DES} at atmospheric pressure of SO₂. That means DESs have great potential for absorption of SO₂. However, it should be noted that most DESs mentioned above are usually high viscosity and lack selectivity for the capture of SO₂ from CO₂ [34–37,47,48], especially in the flue gas (containing CO₂ and N₂). Thus, it is necessary to adjust the functionalized composition of the DESs system to regulate the force of the DESs on SO₂ and CO₂/N₂.

Herein, in this study, we prepared a series of extremely low viscosity matrine-based DESs through simple mixing of matrine with different amides in a molar ratio of 1:2 (Scheme 1). These matrine-based DESs were able to reach a rapid absorption equilibrium within 3 min under mild conditions, and the absorption capacity after equilibrium was higher than 0.81 g_{SO2}/g_{DES} at 293.2 K and 1 bar. In addition, Mat-MFA (1:2) DES had an unprecedentedly high selectivity (576) for the separation of SO₂/CO₂ (10/90 v/v) under mild conditions. The breakthrough column performance of Mat-MFA (1:2) DES was further tested to show its potential for FGD applications.

2. Experimental

2.1. Materials

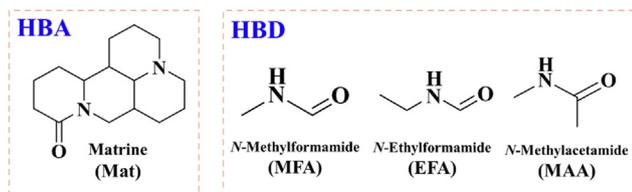
Matrine (Mat, >98%), was purchased from Shanghai Macklin Biochemical Co., Ltd. *N*-Methylacetamide (MAA, >99%), *N*-methylformamide (MFA, >99%), and *N*-ethylformamide (EFA, >99%) were purchased from Adamas-beta Co., Ltd. The gases including SO₂, N₂, and CO₂ were obtained with high grade (>99.99%) from Jiangxi Huahong Gas Co., Ltd.

2.2. Preparation of matrine-based DESs

As shown in Scheme 1, matrine-based DESs were simply synthesized by stirring and heating HBD (including MAA, MFA, EFA) and HBA (Mat) with the molar ratio of 1:2 at 333.2 K for 1 h, then a type of colorless and transparent liquid was formed after vacuum at 333.2 K for 12 h. Next, these obtained matrine-based DESs were finally obtained and named Mat-MAA (1:2), Mat-MFA (1:2), and Mat-FEA (1:2), respectively.

2.3. Characterizations

The densities of matrine-based DESs were carried out by an Anton Paar densimeter (model DMA4500) with the temperature range from 293.2 to 338.2 K. The viscosities were measured on a



Scheme 1. Chemical structures of HBA (matrine) and HBD (MFA, EFA, and MAA) used in this work.

cone-plate viscometer (Brookfield DV II+Pro) with the temperature range from 293.2 to 353.2 K, and the temperature control of ±0.05 K. FT-IR spectrum were carried on a NEXUS870 IR spectrometer. ¹H NMR and ¹³C NMR spectra were carried out by a Bruker Ascend 400 MHz spectrometer. The decomposition temperature was measured by the TG (PerkinElmer Diamond TG/DTA) from 298.2 to 653.2 K.

2.4. SO₂ absorption

All experimental data of this work were obtained by testing with self-made devices (Figure S1 shows a schematic view of the apparatus), which is the same as that used in our previous work [10,11,38]. Shortly, to a typical capture test of SO₂, a certain amount of matrine-based DES was loaded into the absorption cell. Fed partial of SO₂ into the absorption cell. Recorded the capture data after the pressure of the cell remained unchanged. The SO₂, CO₂, and N₂ absorption capacity were calculated by the pressure change before and after the test gas absorption. The separation selectivity of gas is calculated by the ideal absorption solution theory (IAST) formula [39,40]. For recycle experiment, the SO₂-saturated DES was heated at 333.2 K for 6 h under vacuum and reused for the next absorption test. Furthermore, the breakthrough experiment was carried out on a homemade apparatus for trace SO₂ separate in the mixed SO₂/CO₂ or SO₂/N₂ gases, detail breakthrough experiment and apparatus (Figure S2) are found in the Supplementary data.

3. Results and discussion

3.1. Characterization

These matrine-based DESs were easily synthesized by mixing matrine with amide (MFA, EFA, MAA) at the molar ratio of 1:2 (Scheme 1). The properties of these matrine-DESs were identified by FT-IR (Fig. 1), TG (Fig. 2), viscosity (η), and density (ρ) (Fig. 3), as well as ¹H NMR and ¹³C NMR (Supporting Information).

FT-IR spectra was first used to analyze and verify the structure of these matrine-based DESs absorbents. As shown in Fig. 1, the characteristic peaks at 1383, 2860, and 2940 cm⁻¹ belong to the symmetrical bending vibration, symmetric stretching vibration, and symmetric stretching vibration of C-H in the methyl group. The characteristic peak at 1666 cm⁻¹ ascribes to the stretching

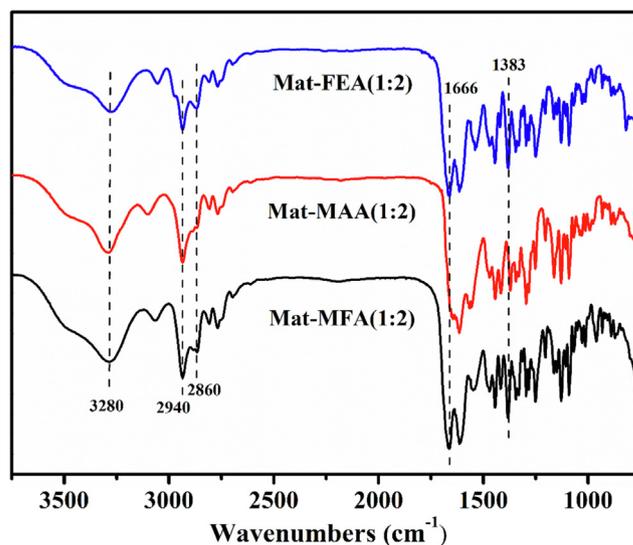


Fig. 1. FT-IR spectra of DESs Mat-MFA (1:2), Mat-MAA (1:2), and Mat-MEA (1:2).

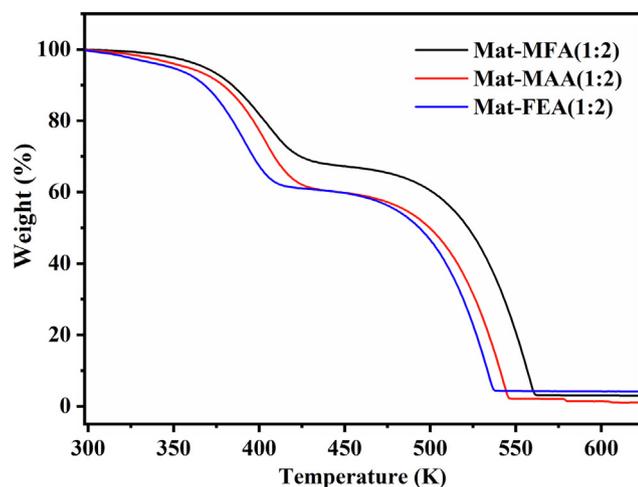


Fig. 2. TG curves of DESs Mat-MFA (1:2), Mat-MAA (1:2), and Mat-MEA (1:2).

vibration of C=O. The characteristic peak at 3280 cm^{-1} belongs to the stretching vibration of amide N-H. It is found that the characteristic peaks of the infrared spectra of the corresponding components did not shift significantly, indicating that these matrine-based DESs are successfully prepared by simply heating and mixing. The ^1H NMR and ^{13}C NMR characterization (Supporting Information) results of these three DESs were found to be in perfect accordance with their corresponding chemical structure. Based on the NMR result, no impurities were found. In addition, the TGA analysis of matrine-based DESs was further carried out as shown in Fig. 2. The decomposition temperature of these DESs is highly stable above 363.2 K , indicating that these absorbents are stable enough under the regeneration temperature (333.2 K), meeting the conditions required for heating desorption.

As shown in Fig. 3, the physical properties of three matrine-based DESs were tested and analyzed. It is found that the density of matrine-based DESs shows a linear decline with the increase of temperature, and the viscosity decreases exponentially with increasing temperature. Interestingly, the viscosity of the three matrine-based DESs is very small ($<20\text{ cP}$) at 298.2 K . That means the mass transfer resistance in the absorption process can be ignored, which is beneficial to speed up the absorption of SO_2 gas by the absorbent. Furthermore, the viscosities and densities data of these three matrine-based DESs were fitted by Equations (1) and (2), respectively.

Relevant viscosity and density fitting data can be consulted in Table S1 in Supporting Information.

$$\rho = A + BT \quad (1)$$

$$\eta = \eta_0 * \exp\left(\frac{D}{T}\right) \quad (2)$$

3.2. Absorption of SO_2

As shown in Fig. 4, the absorption rate of SO_2 by matrine-based DESs is very fast, which can reach equilibrium within 3 min. Generally, the absorption rate of SO_2 is usually reverse of the viscosity of the absorbent, i.e., the lower the viscosity, the faster the absorption rate. Hence, the low viscosity of these DES (below 20 cp) allows them to show a very fast SO_2 uptake rate. Furthermore, the equilibrium absorption capacity between these DESs is close and large enough ($0.77\text{--}0.84\text{ g g}^{-1}$) at 298.2 K and 1 bar , suggesting that these formamides on matrine-based DESs can improve SO_2 uptake capacity. Moreover, the influence of SO_2 partial pressure and temperature on the SO_2 uptake capacity of three matrine-based DESs were further investigated, as shown in Fig. 5A. It is found that the mass capacity of SO_2 in matrine-based DESs continuously increased from 0.00 to 0.77 g g^{-1} with the increased pressure from 0 to 100 kPa , and the capacity of these three DESs was very close. Fig. 5B shows that the absorption capacity of the Mat-MFA (1:2) DES for SO_2 increased with the decrease of temperature, indicating that low temperature is favorable to SO_2 absorption. In addition, as shown in Fig. 5, the result found that the chemical uptake capacity was litter increased at the pressure increased from 0.0 to 0.3 bar and approached a flat when the pressure was higher than 0.3 bar . In comparison, the physical absorption significantly increased with the further increase of SO_2 pressure. This is consistent with the absorption of most absorbents reported in the literature [14,41,42], and the absorption of SO_2 by Mat-MFA (1:2) is primarily physical absorption.

The solubilities of SO_2 in these matrine-based DESs are compared with other typical DESs reported in the references, as listed in Table 1. In comparison, all three matrine-based DESs were found to have good absorption capacities at 1 bar and 0.1 bar . Although some of the DES had higher absorption capacities than those of matrine-based DESs, their excessive viscosity affected the mass transfer process and resulted in slow absorption rates. For example, Zhao et al [35], reported an imidazole-based DES (EmimCl-AA (2:1)) that has a higher SO_2 uptake capacity (1.39 g g^{-1}) at 293.2 K , while the high viscosity (1400 cp) gives a long absorption equilibrium time (over 40 min) at 293.2 K . By contrast, the absorp-

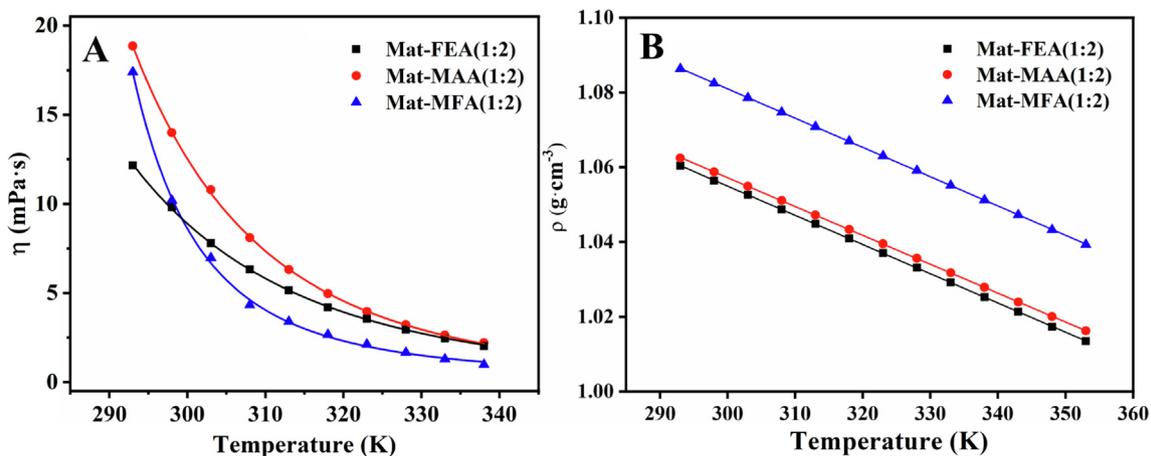


Fig. 3. Viscosities (A) and densities (B) of Mat-MFA (1:2) (■), Mat-MAA (1:2) (●), and Mat-MEA (1:2) (▲) at different temperatures.

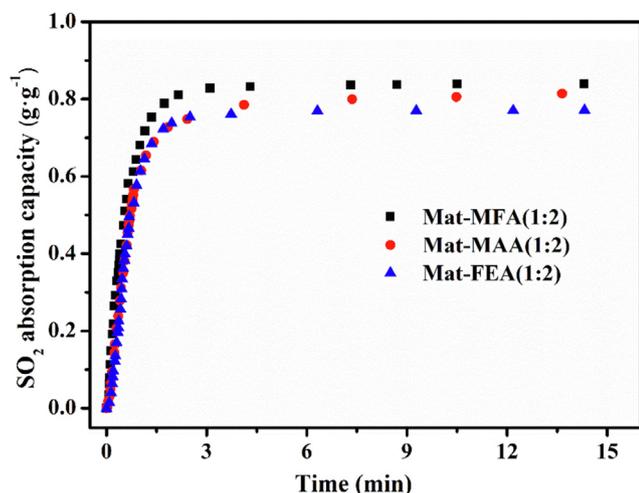


Fig. 4. Rates of SO_2 absorption in Mat-MFA (1:2) (■), Mat-MAA (1:2) (●), and Mat-MEA (1:2) (▲) at 293.2 K and initial pressure of 1 bar.

tion equilibrium time for the matrine-based DESs used in this work was significantly less than 3 min.

3.3. SO_2/CO_2 selectivity

Matrine-based DES absorbents have good SO_2 absorption performance under low pressure from the result listed in Table 1.

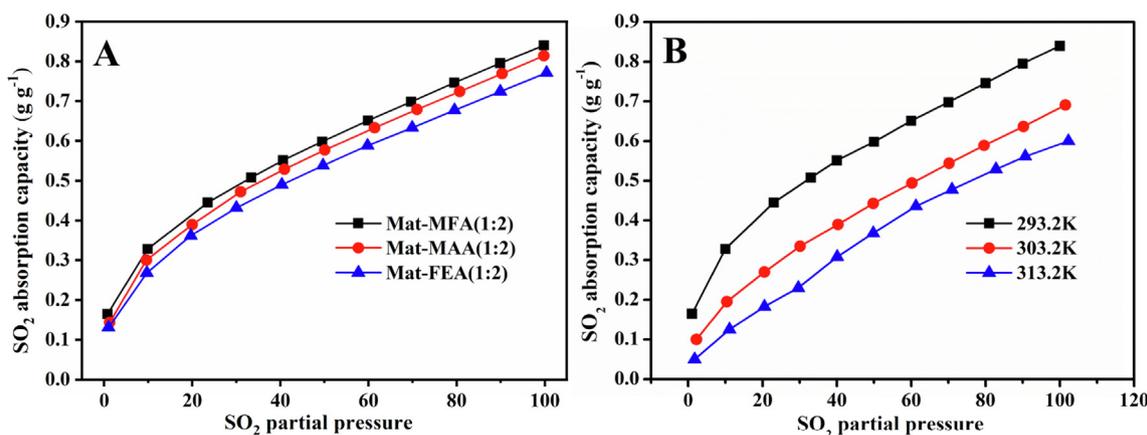


Fig. 5. Absorption of SO_2 in Mat-MFA (1:2) (■), Mat-MAA (1:2) (●), and Mat-MEA (1:2) (▲) in 293.2 K at different pressure (A), and absorption of SO_2 in Mat-MFA (1:2) at different temperatures (B).

Table 1

Comparison of viscosity and SO_2 solubilities of various DESs.

Absorbents	T (K)	Viscosity (cp)	SO_2 uptake (g g^{-1}) ^a		Ref.
			0.1 bar	1.0 bar	
Mat-MAA (1:2)	293.2	18.9	0.30 (1.85)	0.81 (5.02)	This work
Mat-MFA (1:2)	293.2	17.4	0.33 (1.88)	0.84 (4.81)	This work
Mat-FEA (1:2)	293.2	12.2	0.27 (1.65)	0.77 (4.75)	This work
CPL-KSCN (3:1)	293.2	1082	–	0.61 (4.14)	[33]
EmimCl-AA (2:1)	293.2	1400	0.49 (2.70)	1.39 (7.65)	[35]
EmimCl-AA (1:1)	293.2	650	0.46 (1.48)	1.25 (4.02)	[35]
PPZBr:Glycerol (1:6)	293.2	1772	0.13 (1.61)	0.35 (4.33)	[34]
ChCl-Glycerol (1:1)	293.2	–	0.15 (0.54)	0.68 (2.45)	[32]
ChCl-Glycerol (1:2)	293.2	–	0.08 (0.40)	0.48 (2.43)	[32]
Betaine:EG (1:3)	313.2	–	–	0.37 (1.75)	[47]
n _{CC} -n _{CC} (3:1)	293.2	–	–	0.53 (4.23)	[48]

^a The unit in brackets in this column is mol mol^{-1} .

Since DES Mat-MFA (1:2) has a relative higher SO_2 absorption capacity compared with the other two matrine-based DESs at both 0.1 and 1.0 bar in 293.2 K. Mat-MFA (1:2) DES was used to test the IAST selectivity of SO_2/CO_2 with varying SO_2 molar fraction in the gas phase at 1 bar. For comparison, some solid adsorbents reported in the literature are also shown in Fig. 6 and Table S2 in the Supporting Information. It is found that the Mat-MFA (1:2) has significantly higher idea selectivity of SO_2/CO_2 at different SO_2 molar fractions, especially at 10:90 fraction, a higher SO_2/CO_2 selectivity (576) was obtained at 293.2 K. That means this matrine-based DES has great promise for the selective isolation of SO_2 from mixture gases containing CO_2 . The reason is as follows: The matrine contains amides and tertiary amines, which are weakly basic according to the literature [35], and these basic sites are exactly suitable for SO_2 absorption, while they are weaker for the less acidic CO_2 . Therefore, the matrine-based DES can obtain high SO_2/CO_2 IAST selectivity. To test the actual separation performance of the matrine-based DES in the mixture of SO_2/CO_2 and SO_2/N_2 gases. A dynamic experimental breakthrough experiment was performed. The simulated mixture of gas containing 2% SO_2 and 98% CO_2 or N_2 . As it is found in Fig. 7, both CO_2 and N_2 can be rapidly eluted except SO_2 , and the SO_2 can be highly efficient elimination with a good breakthrough time are 165 and 173 min g^{-1} , respectively. This result indicates that the Mat-MFA (1:2) DES has great SO_2 separation performance with the co-existence of competitive gas of CO_2 and N_2 . This finding is highly consistent with the result of the IAST selectivity calculation. Therefore, Mat-MFA (1:2) DES has great potential for actual SO_2 separation applications.

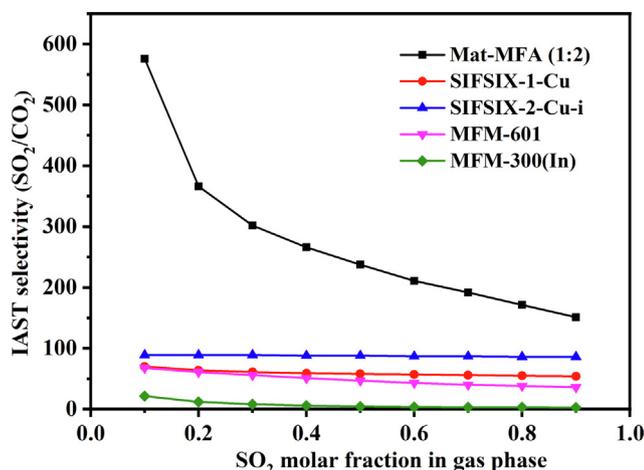


Fig. 6. The selectivity of SO_2/CO_2 in Mat-MFA (1:2) DES compared with other solid adsorbents.

3.4. SO_2 absorption mechanism

FT-IR spectra were carried out to explore the interaction between DES and SO_2 . As shown in Fig. 8, compared with the FT-IR spectra before and after absorption of SO_2 in matrine-based DES, three new absorption vibration peaks appeared at the positions of 1316 cm^{-1} , 1141 cm^{-1} , and 945 cm^{-1} . They were attributed to the asymmetric (Vas) stretching vibration of the S=O bond, the symmetric (Vs) stretching vibration of the S=O bond, and the stretching vibration peak of the S-O bond, respectively [34,43,44]. The absorption peak at 945 cm^{-1} may be the chemical interaction between SO_2 with the tertiary amine nitrogen atom of matrine, while the absorption peaks at 1316 cm^{-1} and 1141 cm^{-1} indicate that the interaction between matrine-based DES and SO_2 is not too strong [45,46]. From this result, it is evident that the high-efficiency absorption process of matrine-based DES for SO_2 is mainly through physical interaction as well as part of chemical interaction between matrine-based DES and SO_2 , which is consistent with the above result in Fig. 5. Hence, a possible absorption interaction of DES and SO_2 has been given in Scheme 2.

3.5. Recycled stability

To determine the reusability of Mat-MFA (1:2), the SO_2 capture experiment on Mat-MFA (1:2) was performed at 293.2 K and 1 bar with 8 repeated times. The SO_2 -saturated Mat-MFA (1:2) was heated to 333.2 K under 0.001 bar for 0.5 h . After degassing, the

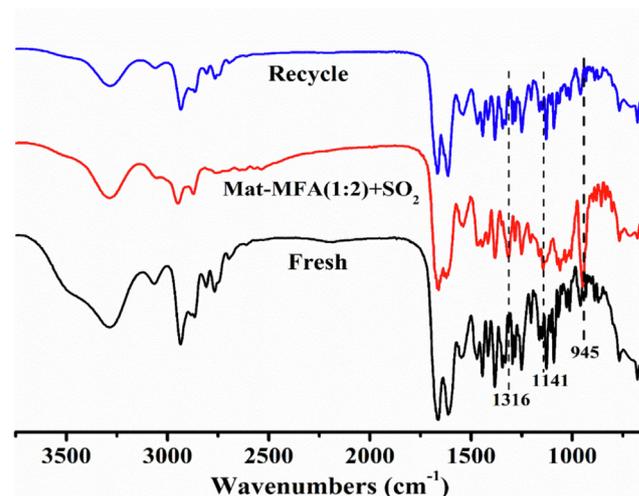
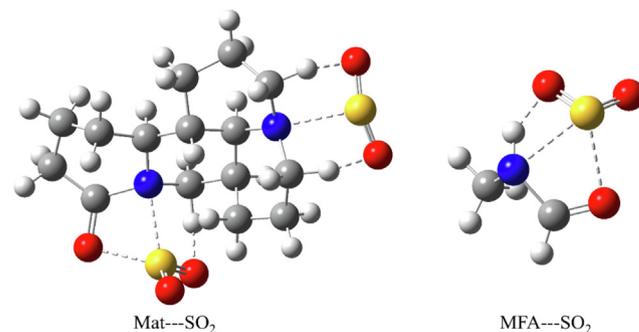


Fig. 8. FT-IR spectra of Mat-MFA (1:2) before (black), absorbed (red), and desorbed (blue) SO_2 . (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



Scheme 2. Schematic diagram of the possible absorption interaction of DES and SO_2 .

Mat-MFA (1:2) DES was reused to absorption of SO_2 for the next run. As shown in Fig. 9, it is found that the total absorption of SO_2 remains almost unchanged. There is some residual SO_2 after desorption, accounting for $\sim 15\%$ of the total SO_2 uptake capacity. The incomplete desorption of SO_2 may be the result of moderate desorption temperature because the higher desorption temperature will promote the loss of amide due to the volatile nature of amide. Therefore, the Mat-MFA (1:2) DES has good cyclic performance for the absorption of SO_2 .

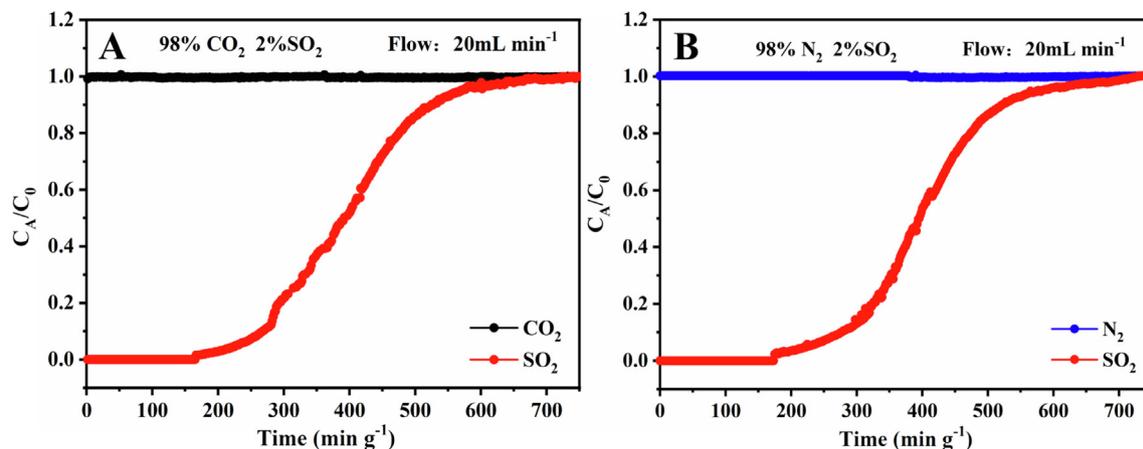


Fig. 7. Breakthrough curves for SO_2/CO_2 (A) and SO_2/N_2 (A) mixed gas absorption on Mat-MFA (1:2) DES at 293.2 K and 1 bar .

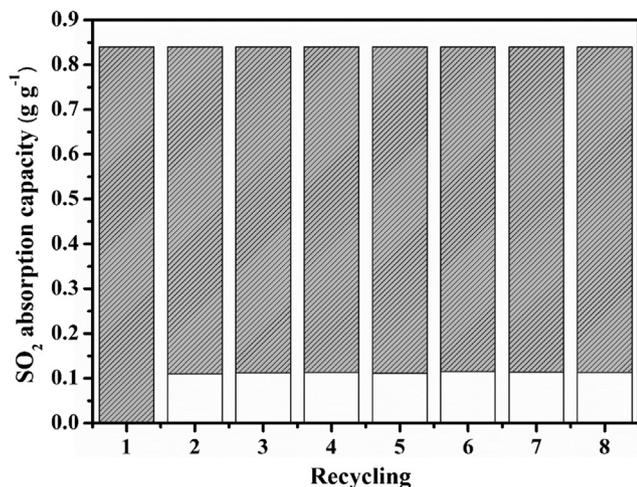


Fig. 9. SO₂ absorption/desorption cycles in Mat-MFA (1:2) DES.

4. Conclusions

In summary, three matrine-based DESs were synthesized, characterized, and used as promising absorbents for the highly selective absorption of SO₂. It is demonstrated that these DESs show extremely low viscosities (<20 cP) and displayed a very fast SO₂ absorption rate (<3 min) at room temperature. Mat-MFA (1:2) DES exhibited considerable SO₂ uptake capacity, with 0.81 g g⁻¹ at 293.2 K and 1 bar, and 0.30 g g⁻¹ at 293.2 K and 0.1 bar. At the same time, Mat-MFA (1:2) DES resulted in a very high selectivity of SO₂/CO₂ (576) for 10% SO₂ and 90% CO₂, which is higher than most liquid absorbents and porous solid adsorbents reported in the literature. Furthermore, the breakthrough experiment confirmed that Mat-MFA (1:2) DES showed exceptional performance in the separation of 2% SO₂ in the SO₂ and CO₂ mixed gas. The fast uptake, low viscosity, and high selectivity make these matrine-based DESs have great potential for a practical desulfurization process.

CRediT authorship contribution statement

Zhang-Min Li: Formal analysis, Resources, Supervision, Writing - review & editing. **Shu-Xian Zhu:** Methodology, Software. **Duan-Jian Tao:** Writing - review & editing, Supervision. **Yan Zhou:** Conceptualization, Writing - review & editing, Supervision.

Data availability

Data will be made available on request.

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.molliq.2022.120521>.

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