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Choline chloride plus glycerol deep eutectic solvents as non-aqueous absorbents for the efficient absorption and recovery of HCl gas†

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HCl regularly yields as a byproduct in the tail gas of pharmaceutical and chemical processes, including chlorination and dechlorination reactions. It is very challenging to obtain value-added pure HCl gas by making the use of a non-aqueous absorbent for the separation and recovery of HCl with both high efficiency and excellent reversibility. In this study, a new type of choline chloride-glycerol (ChCl-Gly) deep eutectic solvents (DESs) was synthesized and used as non-aqueous absorbents for the efficient absorption and recovery of HCl gas. It was found that the ChCl-Gly (1:1.5) DES exhibited high HCl absorption capacity (0.303 g g^{-1}) at 298.2 K and 1.0 bar. In addition, the results of FTIR and ^1H NMR spectroscopies revealed that the interaction of ChCl-Gly (1:1.5) with HCl was through hydrogen-bonding, demonstrating a high reversibility and recyclability of ChCl-Gly (1:1.5) DES in twenty cycles. The present study indicates that the utilization of a ChCl-based non-aqueous absorbent is an effective methodology for the efficient absorption and recovery of HCl gas.

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1. Introduction

Hydrogen chloride (HCl) is an irritating gas, which causes harm to the environment and humans.^{1,2} On the other hand, HCl is a very important gas with valuable application in chemical production processes. For example, it can be used as both an acid catalyst and intermediate material for the synthesis of chlorine-containing downstream products, such as alkyl chloride and benzyl chloride.^{3–5} HCl regularly yields as a byproduct in the tail gas of pharmaceutical and chemical processes, including chlorination and dechlorination reactions.^{6,7} For example, in the reaction of 2,3-dichloropropene with chlorine to produce 1,1,2,3-tetrachloropropene, a large amount of HCl is produced along with the byproduct 1,2,3-trichloropropene. Therefore, a highly efficient recovery of HCl gas will not only protect the environment but also improve the utilization of HCl resources.

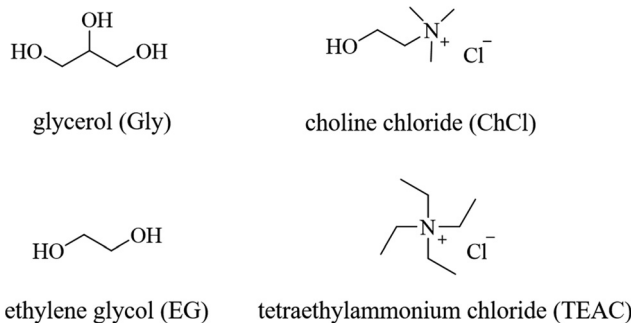
However, the traditional methodology for waste gas scrubbing through water results in a high yield of low-value and corrosive hydrochloric acid with unsatisfactory purity.⁸ An evaporation procedure of hydrochloric acid is then carried out for its purification. However, these complex processes

result in serious equipment corrosion and require a very large consumption of energy due to the high heat of vaporization of water.⁹ Therefore, to improve the high-value utilization of HCl, it is an open and significantly interesting topic to explore a green and sustainable methodology for the collection of dry and pure HCl.

The design and synthesis of a non-aqueous absorbent to display a reversible and efficient absorption and recovery of pure HCl gas is of great significance. The main non-aqueous absorbents reported currently include ionic liquids (ILs),¹⁰ polyols,¹¹ and other liquid absorbents.¹² For example, He *et al.*¹⁰ used an imidazolium IL [Hmim]Cl for the absorption of HCl with an absorption capacity of 0.384 g HCl per g IL at 298.2 K and 1.0 bar. However, subsequent studies showed that HCl gas could not be desorbed and released completely from HCl-saturated [Hmim]Cl.¹¹ The amount of residual HCl in [Hmim]Cl was as much as 35% even at the desorption temperature of 363.2 K. After that, Li *et al.*¹¹ investigated the selective absorption of HCl from SO_2 by different alcohols, such as tetraethylene glycol, ethylene glycol, and glycerol. Among these, ethylene glycol showed the HCl absorption capacity value of 0.326 g HCl per g absorbent at 313.2 K and 1.0 bar. However, these alcohols suffer from the critical drawbacks of relatively high volatility and a very slow absorption rate. Ethylene glycol is easily lost through volatilization under vacuum or high temperature,¹³ resulting in poor recyclability and reusability. Also, the above-mentioned alcohols need a long

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Scheme 1 Structures of the HBAs and HBDs in DESs.

time (*e.g.* ≥ 50 min) to reach absorption saturation. Therefore, the screening of advanced non-aqueous absorbents with good reversibility for the rapid absorption and separation of HCl gas is still in high demand.

Recently, deep eutectic solvents (DESs) are widely recognized as a new type of IL analogs owing to their similar physical properties.¹⁴ DESs are usually mixed and formed by hydrogen-bond acceptors (HBAs) and hydrogen-bond donors (HBDs) and have been used for reversibly capturing many kinds of toxic gases,^{15–17} such as SO₂,¹⁸ H₂S,¹⁹ and HCl.²⁰ Herein, a series of DESs was synthesized by a simply mixing HBAs with HBDs (Scheme 1), and they were employed as efficient non-aqueous absorbents for the reversible capture of HCl. The HCl absorption capacity could be controlled by adjusting the ChCl concentration and absorption temperature. Moreover, the Fourier transform infrared (FTIR) spectra and proton nuclear magnetic resonance (¹H NMR) spectra of the ChCl-Gly DESs before and after HCl uptake were investigated to explore the interaction mechanism. In addition, the recyclability of the ChCl-Gly DESs in the HCl absorption-desorption process was also investigated.

2. Experimental

2.1. Materials and DES preparation

Hydrogen chloride (HCl, 99.99% v/v) and nitrogen (N₂, 99.99% v/v) were purchased from Jiangxi Huahong Special Gas Co., Ltd. Glycerol (Gly, 99%), ethylene glycol (EG, 99%), choline chloride (ChCl, 99%), and tetraethylammonium chloride (TEAC, $\geq 99\%$) were purchased from Shanghai TiTan Scientific Co., Ltd. The other chemicals were used without additional purification. The DESs were prepared by mixing ChCl and Gly at 333.2 K with different molar ratios. As the mixtures turned to transparent liquids after stirring, they were dried in a vacuum oven for 24 h at 333.2 K to remove water as much as possible.

TEAC-Gly and ChCl-EG (1:2) DESs were prepared using the same synthesis procedure as ChCl-Gly. The synthesized DESs were abbreviated as ChCl-Gly (1 : *x*), TEAC-Gly (1 : *x*) and ChCl-EG (1 : *x*), where 1 : *x* stands for the molar ratio of ChCl or TEAC to Gly or EG.

2.2. Characterization

The water content of as-prepared DESs was determined by an instrument Mettler-Toledo DL32, and the water content were found to be lower than 0.05 wt%. The viscosity of DESs before and after the absorption of HCl was measured using a cone-plate viscometer (Brookfield DV II + Pro). The preheating time in the measuring device was about 15 min with a viscosity uncertainty of $\pm 1\%$. The FTIR and ¹H NMR spectra of DESs before and after HCl absorption were recorded on a Nicolet 6700 spectrometer and a Bruker Avance 400 spectrometer in DMSO-*d*₆ with tetramethylsilane as the internal standard, respectively.

2.3. Measurement of HCl absorption

Like the previously reported literature for SO₂ absorption,^{21,22} the configuration of the apparatus for HCl absorption is shown in Fig. 1. In a typical run, 1.0 bar HCl was bubbled through a

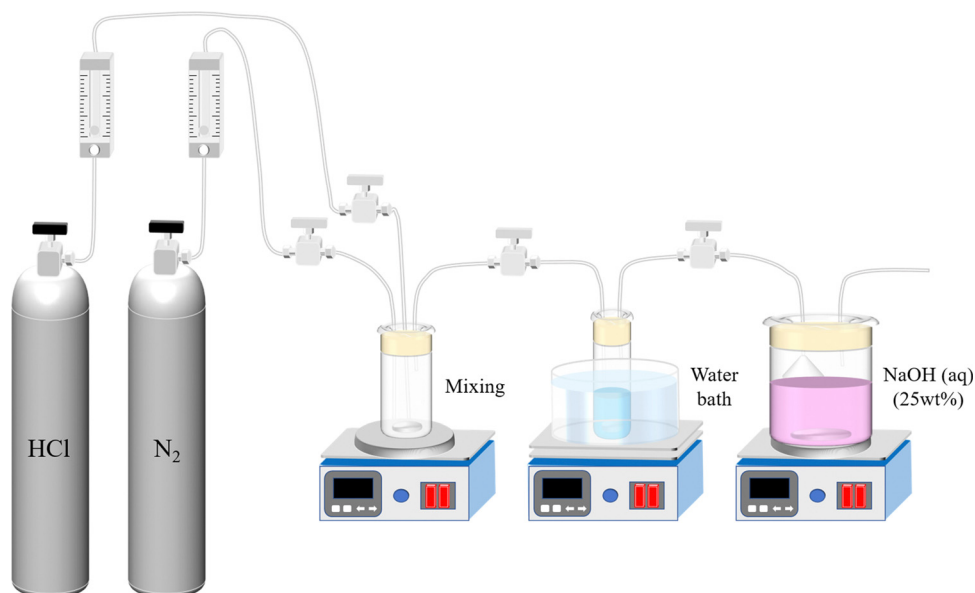


Fig. 1 Apparatus for the determination of absorption and recovery of HCl gas.

10 mL glass container containing approximately 2.0 g DESs, and the flow rate of HCl was about 60 mL min⁻¹. The glass container was immersed in a circulating water bath at a set temperature. The HCl absorption capacity was calculated until a mass difference of less than 1 mg using an electronic balance. Each experiment was repeated 15 times, and therefore, the solubility of HCl (denoted as gram of HCl per gram of DES) was obtained based on the average value. All the uncertainties of the absorption data in this study were under $\pm 0.62\%$. During the absorption of different HCl concentrations (5–100% v/v), a certain proportion of HCl and N₂ were mixed and passed into the apparatus. The loaded-HCl DESs were desorbed under vacuum at 333.2 K for 30 min for the regeneration and release of HCl.

3. Results and discussion

3.1. Absorption performance of HCl

First, we investigated the absorption effect of HCl on various DESs, which were synthesized by different ChCl-Gly ratios. As shown in Fig. 2, with the increasing molar fraction of ChCl in DES, the HCl absorption capacity of DES increased accordingly. As a result, when the ChCl-Gly molar ratio was 1 : 1.5, the HCl absorption capacity reached a maximum of 0.303 g g⁻¹. Then, we further investigated the absorption effect of ChCl-Gly (1 : 1.5) DES on HCl at various temperatures. It can be seen from Fig. 3 that as the absorption temperature increased, the HCl solubility decreased, which is consistent with literature.¹⁰ The largest HCl absorption capacity was 0.303 g g⁻¹ at 298.2 K. In addition, with the increase of absorption temperature, the equilibrium time for the absorption of HCl was greatly shortened. For example, when the absorption temperature was 298.2 K, the equilibrium time was 20 min. When the temperature increased to 333.2 K, the equilibrium time decreased to 10 min. This observation could be attributed to the fact that the viscosity of ChCl-Gly DES decreases with the increase in temperature,²³ which is beneficial to the diffusion of gas in

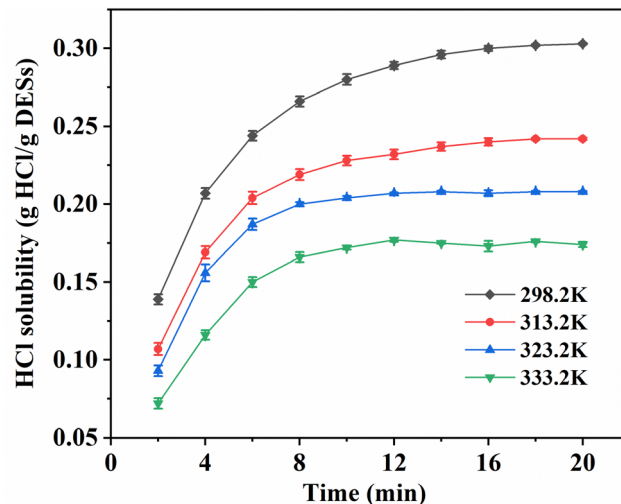


Fig. 3 HCl absorption by the ChCl-Gly (1 : 1.5) DES at different temperatures and 1.0 bar.

the absorbent reducing the equilibrium time.²⁴ Subsequently, the effect of different HCl concentrations (5–100% v/v) and temperatures on HCl absorption were further studied (Fig. 4). It was found that under a certain partial pressure, the HCl solubility had a near linear relationship with the absorption temperature. Low temperature is conducive to increase the HCl solubility. Also, it was indicated that the HCl concentration has a big impact on the HCl solubility. A higher HCl concentration would lead to a higher HCl solubility.

For comparison, we examined the absorption of HCl by pure glycerol at 298.2 K and 1.0 bar (Fig. 5). The result indicated that the HCl absorption capacity in glycerol was 0.27 g g⁻¹, but the absorption equilibrium time was long, about 2 h (Fig. 5a). On the contrary, ChCl-Gly (1 : 1.5) only needed 20 min to rapidly approach absorption equilibrium, which may be attributed to its low viscosity.^{25,26} Notably, it can be seen from Fig. 5b that the viscosity of ChCl-Gly (1 : 1.5) dramatically reduced to 205 cP

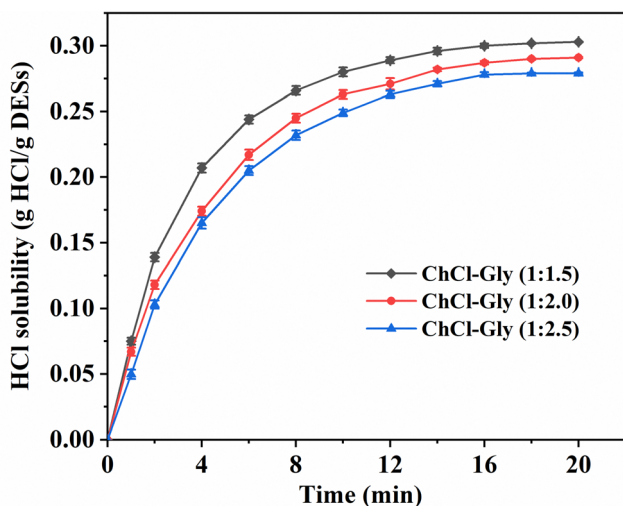


Fig. 2 HCl absorption by three ChCl-Gly DESs at 298.2 K and 1.0 bar.

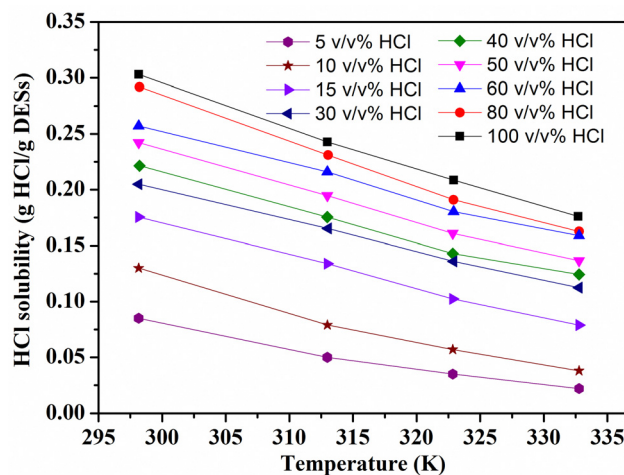


Fig. 4 HCl absorption by the ChCl-Gly (1 : 1.5) DES at different temperatures and HCl partial pressures.

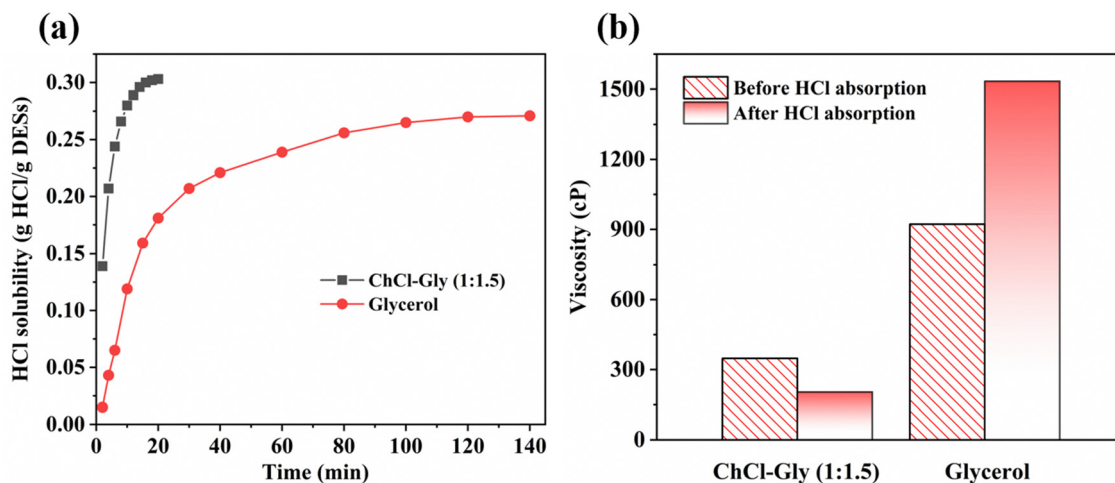


Fig. 5 (a) HCl absorption by ChCl-Gly (1:1.5) and glycerol; (b) viscosity change before and after HCl absorption at 298.2 K and 1.0 bar.

after the absorption of HCl, whereas the viscosity of ChCl-Gly (1:1.5) was 348 cP at 298.2 K before HCl absorption. This finding shows that owing to the Cl^- ion in ChCl, the HCl molecule could be easily polarized and charged in ChCl-Gly (1:1.5), which weakened the H-bonding network in ChCl-Gly (1:1.5),^{25,27} and thereby obviously reduced the viscosity of ChCl-Gly (1:1.5) after the absorption of HCl. However, the viscosity of glycerol increased significantly (up to 1534 cP) after HCl absorption compared with the viscosity (922 cP) of fresh glycerol before HCl absorption, resulting in an extremely slow absorption rate. Therefore, it is demonstrated that the low viscosities of ChCl-Gly (1:1.5) before and after HCl uptake contribute to a very rapid HCl absorption rate.

3.2. Absorption mechanism study

To further investigate the role of ChCl in the ChCl-Gly DESs for the absorption of HCl, another TEAC-Gly (1:1.5) DES was synthesized by replacing ChCl with TEAC, and the absorption results are shown in Fig. 6. It is demonstrated that the HCl absorption capacity of TEAC-Gly (1:1.5) was lower than that of ChCl-Gly (1:1.5). Moreover, the solution becomes turbid after the absorption of TEAC-Gly (1:1.5), indicating that this DES is unfavorable for the absorption of HCl gas. ChCl-Gly (1:1.5) can absorb HCl considerably well owing to the existence of hydroxyl groups in ChCl giving a clear and transparent liquid. Furthermore, to explore the H-bonding interaction of ChCl-based DES with HCl, the FTIR and ^1H NMR spectra of ChCl-Gly (1:1.5) before and after HCl uptake were examined. As shown in Fig. 7, there is no new characteristic peak appearing or disappearing in the FTIR spectra. Also, the out-of-plane bending vibration peak of the $-\text{OH}$ group in ChCl-Gly (1:1.5) before HCl absorption had two characteristic peaks at 609 and 561 cm^{-1} , respectively. After the absorption of HCl, the wavenumber of the $-\text{OH}$ group was observed to have an obvious blue-shift from 609 to 671 cm^{-1} . This is because the hydrogen bond network in ChCl-Gly (1:1.5) weakened after the absorption of HCl along with a decrease in the viscosity. As a result, the vibration frequency of the $-\text{OH}$ group in ChCl-Gly (1:1.5) increased.

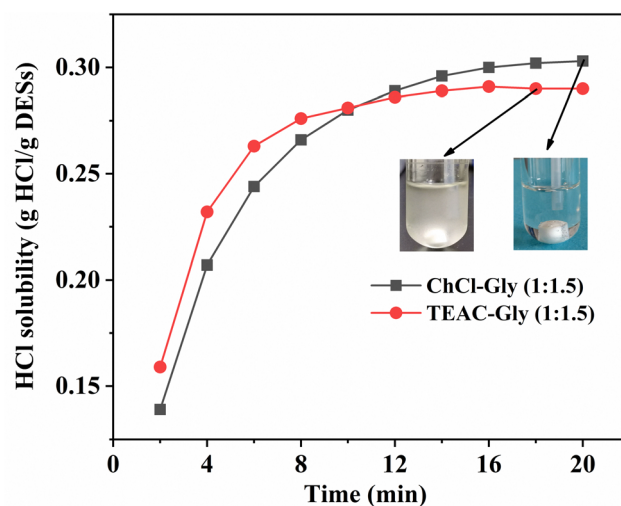


Fig. 6 HCl absorption by TEAC-Gly (1:1.5) and ChCl-Gly (1:1.5) at 298.2 K and 1.0 bar.

As shown in Fig. 8, before HCl absorption, the ^1H NMR chemical shift for the hydroxyl hydrogen atoms (a + d, 4.5 mol H) of Gly in ChCl-Gly (1:1.5) was 4.51 ppm, while the resonance of the hydroxyl hydrogen atom (e, 1 mol H) of ChCl in ChCl-Gly (1:1.5) had a chemical shift of 5.54 ppm.^{28,29} After ChCl-Gly (1:1.5) absorbed HCl, it was found that the H signal at $\delta = 7.13$ ppm had 7.8 mol H atoms. As mentioned above, 1.0 g ChCl-Gly (1:1.5) could uptake 0.303 g HCl. By the unit-conversion calculation, that is equal to 2.3 mol HCl dissolving in 2.5 mol ChCl-Gly (1:1.5) composed of 1.0 mol ChCl plus 1.5 mol Gly. Thus, it is inferred that the 7.8 mol H atoms at $\delta = 7.13$ ppm were assigned to 5.5 mol H atoms (a + d + e) in ChCl-Gly (1:1.5) and 2.3 mol H atoms in the absorbed HCl. Owing to the H-bonding interaction of ChCl-Gly (1:1.5) with HCl, the resonance of 5.5 mol H atoms (a + d + e) in ChCl-Gly (1:1.5) was significantly shifted to the low field after HCl absorption. Further, the characterization results from FTIR and ^1H NMR spectra confirm

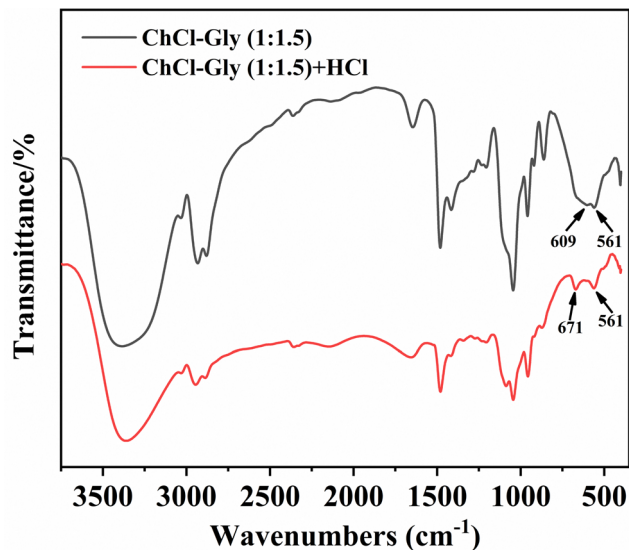


Fig. 7 FTIR spectra of ChCl-Gly (1 : 1.5) before and after HCl absorption.

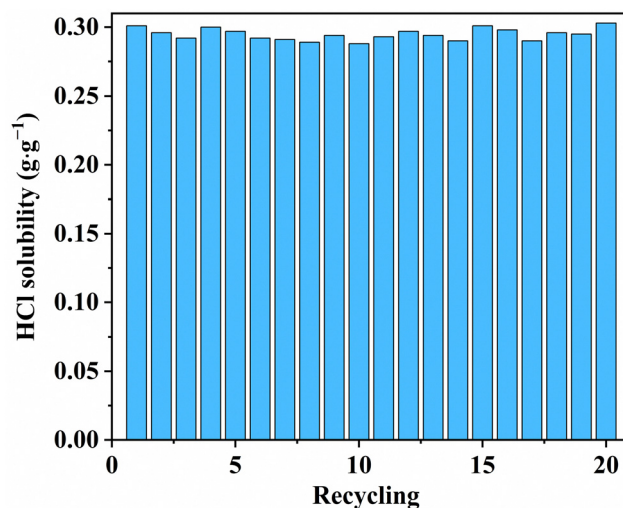


Fig. 9 Twenty cycles of HCl adsorption and desorption by ChCl-Gly (1 : 1.5).

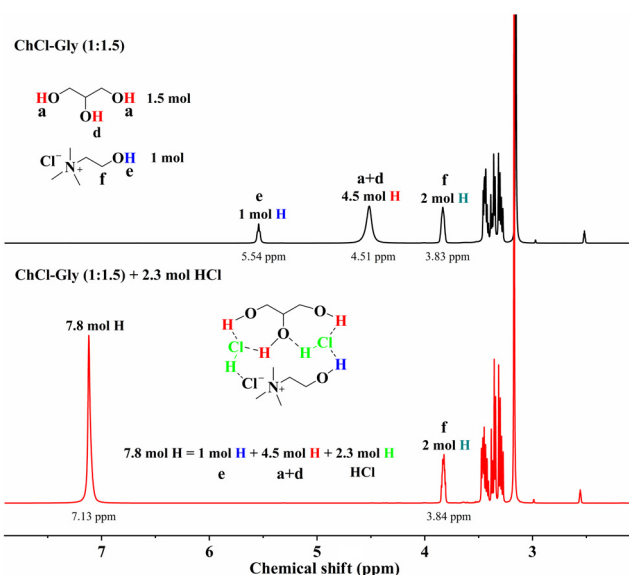


Fig. 8 ¹H NMR spectra of ChCl-Gly (1 : 1.5) before and after HCl absorption.

that the $-OH$ group in ChCl-Gly (1 : 1.5) is the active site for the absorption of HCl, and this absorption process has an H-bonding interaction behavior.

3.3. Recycle tests

To test the reversibility of ChCl-Gly DESs for HCl uptake, the ChCl-Gly (1 : 1.5) DES with dissolved HCl was placed under vacuum at 333.2 K and 30 min to desorb and release HCl. After regeneration, ChCl-Gly (1 : 1.5) was reused for the next HCl absorption process. As shown in Fig. 9, it is found that the HCl absorption capacity displayed no obvious decrease after twenty cycles, indicating that ChCl-Gly (1 : 1.5) can be completely regenerated, and the absorption of HCl in ChCl-Gly (1 : 1.5) is highly reversible. Further, Fig. 10a shows that there is no

significant change in the characteristic peaks of FTIR spectra between the fresh and regenerated ChCl-Gly (1 : 1.5). The organochlorine compounds like ethylene dichloride and 1,2,3-trichloropropane are also not detected during the absorption or desorption process of HCl by the ChCl-Gly (1 : 1.5) DES in the temperature range of 298.2 K to 333.2 K, demonstrating that ChCl-Gly (1 : 1.5) is stable enough to be recycled. Therefore, it is demonstrated that the H-bonding interaction of ChCl-Gly (1 : 1.5) with HCl endows ChCl-Gly (1 : 1.5) with excellent reversibility during twenty absorption–desorption cycles.

3.4. Comparison of ChCl-Gly (1 : 1.5) DES with other absorbents in the literature

The comprehensive performances, including HCl absorption capacity, absorption equilibrium time, and absorption reversibility and stability, for ChCl-Gly (1 : 1.5) were compared with other absorbents reported in literature.^{8,10–12} The results are listed in Table 1. It is indicated that the HCl capability of ChCl-Gly (1 : 1.5) was comparable to polyols, such as glycerol, tetraethylene glycol, and ethylene glycol. Notably, the HCl absorption equilibrium time for ChCl-Gly (1 : 1.5) was much less than in the case of polyols. Moreover, another ChCl-EG (1 : 2) DES was found to have a 4.8 wt% mass loss after regeneration and became turbid and unstable because of the evaporation of ethylene glycol (Fig. 10b). For comparison, the ChCl-Gly (1 : 1.5) DES remained in the clear liquid state after twenty runs. Both the residual HCl in ChCl-Gly (1 : 1.5) and the mass loss of ChCl-Gly (1 : 1.5) after regeneration were very small, showing the excellent reversibility and high recyclability of ChCl-Gly (1 : 1.5). In contrast, the absorbents, including [Bmim]Cl, [Hmim]Cl, and polyols, retained a certain proportion of residual HCl even at a high regeneration temperature of 363.2 K, implying their relatively poor reversibility. The absorbents water and acetonitrile have low boiling points and a huge amount of their evaporation products would be lost during the regeneration process, resulting in the poor recyclability.

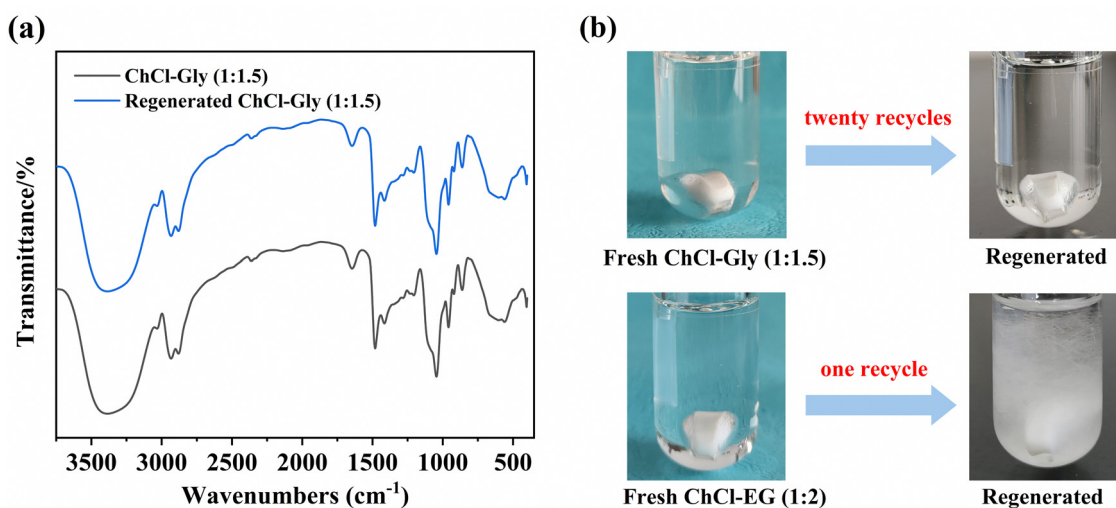


Fig. 10 (a) Comparison of the FTIR spectra of fresh and regenerated ChCl-Gly (1:1.5), (b) photos of fresh and regenerated ChCl-Gly (1:1.5) and ChCl-EG (1:2).

Table 1 Comparison of the comprehensive performances of different absorbents for the absorption of HCl at 1.0 bar

Absorbents	Temperature (K)	Solubility (g HCl per g absorbent)	Time (min)	Residual HCl after regeneration (%)	Mass loss after regeneration (%)	Reference
ChCl-Gly (1:1.5)	298.2	0.303	20	Trace at 333.2 K	Trace	This work
ChCl-EG (1:2)	298.2	0.382	20	Trace at 333.2 K	4.8 wt%	This work
[Bmim]Cl	298.2	0.458	—	21.1% at 363.2 K	Trace	10
[Hmim]Cl	313.2	0.384	50	34.3% at 363.2 K	Trace	11
Glycerol	313.2	0.199	50	1.4% at 363.2 K	Trace	11
Tetraethylene glycol	313.2	0.263	50	5.2% at 363.2 K	~5 wt%	11
Ethylene glycol	313.2	0.326	50	3.9% at 363.2 K	~10 wt%	11
Acetonitrile	293.2	0.098	—	—	Highly volatile	12
Water	293.2	0.420	—	Ionization	Highly volatile	8

Therefore, it is well accepted that the ChCl-Gly (1:1.5) DES exhibited the comprehensive performances of considerable HCl absorption capacity, fast absorption rate, good reversibility, and high stability, which shows good potential for the efficient absorption and separation of pure HCl in the tail gas.

4. Conclusions

In summary, a new type of ChCl-Gly DESs was prepared and served as non-aqueous absorbents for highly efficient and reversible absorption and recovery of HCl gas. It was found that the H-bonding interaction formed between the ChCl-Gly DESs and HCl endowed ChCl-Gly (1:1.5) with the highest HCl solubility and excellent absorption reversibility. ChCl-Gly (1:1.5) could maintain the initial HCl uptake performance during twenty absorption-desorption cycles. It is believed that the ChCl-Gly DESs are promising non-aqueous absorbents for the separation and recovery of pure HCl from industrial exhaust.

Conflicts of interest

There are no conflicts to declare.

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