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## Enhanced $\text{NH}_3$ uptake and selectivity at low pressure in monolithic MOF-808 metal–organic gels incorporating $\text{CuCl}_2$

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The capture and separation of trace  $\text{NH}_3$  from industrial processes or polluted air remains a significant challenge. Herein, we report a monolithic  $\text{CuCl}_2$ @G808 metal–organic gel achieving an  $\text{NH}_3$  uptake of  $2.23 \text{ mmol g}^{-1}$  at  $298 \text{ K}$  and  $0.002 \text{ bar}$ : a  $79\%$  enhancement compared to pristine G808. The ideal adsorbed solution theory (IAST) selectivity reaches  $2.8 \times 10^3$  for  $\text{NH}_3/\text{N}_2$  and  $4.9 \times 10^5$  for  $\text{NH}_3/\text{H}_2$  at  $298 \text{ K}$ , ranking among the highest reported values. *In situ* FTIR and XPS analyses reveal that the excellent performance mainly originates from two synergistic mechanisms: (i) coordination between  $\text{NH}_3$  and  $\text{Cu}^{2+}$  sites, and (ii) hydrogen bonding between  $\text{NH}_3$  and  $\text{Cl}^-$  sites.

## Introduction

Ammonia ( $\text{NH}_3$ ) is an important chemical feedstock, mainly produced by the Haber–Bosch process, and widely used in refrigeration, energy, and fertilizer industries.<sup>1,2</sup> At the same time, ammonia is a highly toxic and corrosive gas, mostly derived from the inadvertent or intentional emission, which causes severe damage to human health and the environment even at low concentrations.<sup>3</sup> Therefore, efficient capture and separation of trace  $\text{NH}_3$  from the  $\text{NH}_3$  production process or polluted air is of great significance for improving  $\text{NH}_3$  production efficiency and reducing energy consumption, and alleviating impacts on the environment and human health.

Metal–organic frameworks (MOFs) adopt structural designability and diversity, abundant active sites, and high porosity, enabling them prospective candidates for real-world  $\text{NH}_3$  capture and separation. Several representative MOFs such as  $\text{LiCl}@\text{MIL-53-(OH)}_2$ -43.4,<sup>4</sup>  $\text{LiCl}@\text{G66-OH-35.7}$ ,<sup>5</sup>  $\text{IL}@\text{MIL-101(Cr)}$ ,<sup>6</sup>  $\text{Mg}_2(\text{dobpdc})$ ,<sup>7</sup>  $\text{Ni\_acryl\_TMA}$ ,<sup>8</sup>  $\text{Cu}_2\text{Cl}_2\text{BBTA}$ ,<sup>9</sup> MOF-303( $\text{Al}$ ),<sup>10</sup> MOF-253( $\text{Al}$ )- $\text{NiCl}_2$ -2,<sup>11</sup> MFU-4,<sup>12</sup>  $\text{Cu}(\text{cyhdc})$ ,<sup>13</sup>  $\text{Co}(\text{NA})_2$ ,<sup>14</sup> MFM-300( $\text{V}^{IV}$ ),<sup>15</sup>  $\text{Cu}(\text{BDC})$ ,<sup>16</sup>  $\text{UiO-66-Cu}^{II}$ ,<sup>17</sup> DUT-6-( $\text{OH}$ )<sub>2</sub>,<sup>18</sup>  $[\text{Mn}_2\text{Cl}_2\text{BTDD}]$ ,<sup>19</sup> Fe-soc-MOF,<sup>20</sup> and MFM-300( $\text{Sc}$ )<sup>21</sup> have demonstrated outstanding  $\text{NH}_3$  capture performance even at low pressure. However, their connatural limitations such as low  $\text{NH}_3$  adsorption capacity and separation ability at extremely low pressure, high production cost, limited stability, and powder state problem preclude their potential application for

$\text{NH}_3$  efficient capture and separation. Therefore, there is an urgent need to fabricate granular high-performance MOFs adsorbents possessing remarkable  $\text{NH}_3$  adsorption and separation ability at ultra-low pressure and facile preparation process.

Metal–organic framework gels (MOGs), as a novel self-shaping material, have come to prominent attention owing to its adjustable aperture from micropore to mesopore/macropore, high adsorptive capacity, and easy large-scale preparation.<sup>22</sup> MOGs can be facilely fabricated by regulating reaction conditions such as metal source, reactant concentration, solvent, and temperature.<sup>23,24</sup> Up to now, MOGs have demonstrated excellent capture performance in many fields such as methane storage,<sup>25,26</sup>  $\text{CO}_2$  capture and storage,<sup>27,28</sup> volatile organic compounds capture,<sup>29–31</sup> water remediation,<sup>32,33</sup> chemical warfare agents decontamination,<sup>34,35</sup> toxic chemical filtration.<sup>36–38</sup> However, there are few reports on the efficient capture and separation of  $\text{NH}_3$  using MOGs, especially under ultra-low pressure.

$\text{CuCl}_2$  possesses an exceptional  $\text{NH}_3$  uptake, but its application is greatly limited due to its powder state. Based on the above advantages of MOGs, herein, MOF-808 metal–organic framework gel (labeled as G808) is chosen as the platform due to its excellent stability to  $\text{NH}_3$ . Thus,  $\text{CuCl}_2@\text{G808}$  composite is prepared in water by a facile impregnation strategy at  $80 \text{ }^\circ\text{C}$ .  $\text{CuCl}_2@\text{G808}$  containing  $2.55 \text{ wt\%}$  of  $\text{CuCl}_2$  shows excellent low-pressure  $\text{NH}_3$  uptake ( $2.23 \text{ mmol g}^{-1}$ ) at  $298 \text{ K}$  and  $0.002 \text{ bar}$ , which displays enhancement of  $79\%$  than that of the pristine G808. Notable, highly selective adsorption of trace  $\text{NH}_3$  was also obtained at  $298 \text{ K}$ . Furthermore, molecular-level insights into the adsorption mechanism were elucidated through combined spectroscopic analyses.

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## Result and discussion

### Structure and morphology of $\text{CuCl}_2@\text{G808-X}$ composites

G808 is firstly synthesized according to our previous work.<sup>39</sup> Then,  $\text{CuCl}_2@\text{G808}$  composite is obtained by a facile impregnation strategy in water solution containing  $\text{CuCl}_2$  at 80 °C for 24 h. The content of  $\text{CuCl}_2$  loaded on G808 is 2.55% measured by ICP-OES. As observed in Fig. 1a, the PXRD pattern of G808 matches well with the simulated MOF-808 (CCDC: 1002672), as evidenced by the characteristic peaks at  $2\theta = 4.4^\circ$  and  $8.6^\circ$ .<sup>40</sup> Furthermore, the crystallinity of  $\text{CuCl}_2@\text{G808}$  decreases compared with that of G808, suggesting that the crystal structure of the composite collapses after loading  $\text{CuCl}_2$ . SEM results also further confirm this conclusion (Fig. S1). In addition, no significant additional peaks are observed in the PXRD of the composite, indicating that  $\text{CuCl}_2$  is evenly anchored in the nanopores of G808. Meanwhile, the uniform distribution of Cu and Cl elements in  $\text{CuCl}_2@\text{G808}$  is verified by energy-dispersive X-ray spectroscopy (Fig. S2). Fig. 1b illustrates the TGA profiles of the samples. The initial mass reduction (303–373 K) is due to residual solvent volatilization, followed by two distinct degradation stages: framework collapse at 473–673 K and organic ligand decomposition at 673–1073 K.<sup>40</sup> Compared with G808, the thermal stability of  $\text{CuCl}_2@\text{G808}$  slightly decreases from 743 K to 703 K, indicating that the introduction of  $\text{CuCl}_2$  has a certain influence on the thermal stability of the composite. This result is in coincidence with that of XRD. Comparative FT-

IR analyses reveal negligible spectral differences between pristine G808 and  $\text{CuCl}_2@\text{G808}$  (Fig. 1c). Compared with G808, the peak of  $\text{CuCl}_2@\text{G808}$  stemmed from the carboxylate ( $-\text{COO}^-$ ) group undergoes some degrees of red shift from 1572  $\text{cm}^{-1}$  to 1562  $\text{cm}^{-1}$ , possibly through partial charge transfer from  $-\text{COO}^-$  groups to  $\text{Cu}^{2+}$ .<sup>41</sup> Like G808, the  $\text{CuCl}_2@\text{G808}$  composite also display characteristic IV isotherms for  $\text{N}_2$  adsorption (Fig. 1d), indicating the existence of microporous and mesoporous features. This result is also validated by the pore size distribution (Fig. S3 and S4). Compared to G808, pore volume, surface area, and pore width of  $\text{CuCl}_2@\text{G808}$  composite dramatically decrease due to the occupation of the pore space by  $\text{CuCl}_2$  (Table S1).

### $\text{NH}_3$ capture and separation

To study capture performance,  $\text{NH}_3$  adsorption isotherms (Fig. 2) of G808 and  $\text{CuCl}_2@\text{G808}$  composite are collected at 298 K and 1 bar. The isotherms show obvious hysteresis loops, suggesting the existing of strong interaction between  $\text{NH}_3$  and active sites. Compared with G808 (9.6  $\text{mmol g}^{-1}$ ), the  $\text{NH}_3$  capture capacity of the  $\text{CuCl}_2@\text{G808}$  (8.1  $\text{mmol g}^{-1}$ ) slightly decreases due to the decrease of porosity at 298 K and 1 bar. However, the  $\text{CuCl}_2@\text{G808}$  manifests a significantly enhanced uptake at low pressure. For example, the  $\text{NH}_3$  adsorption capacity of  $\text{CuCl}_2@\text{G808}$  can reach approximately 5.44  $\text{mmol g}^{-1}$  at 0.1 bar, 3.65  $\text{mmol g}^{-1}$  at 0.01 bar, and 2.23  $\text{mmol g}^{-1}$  even at 0.002 bar (Fig. 2a), Compared to pristine G808, these

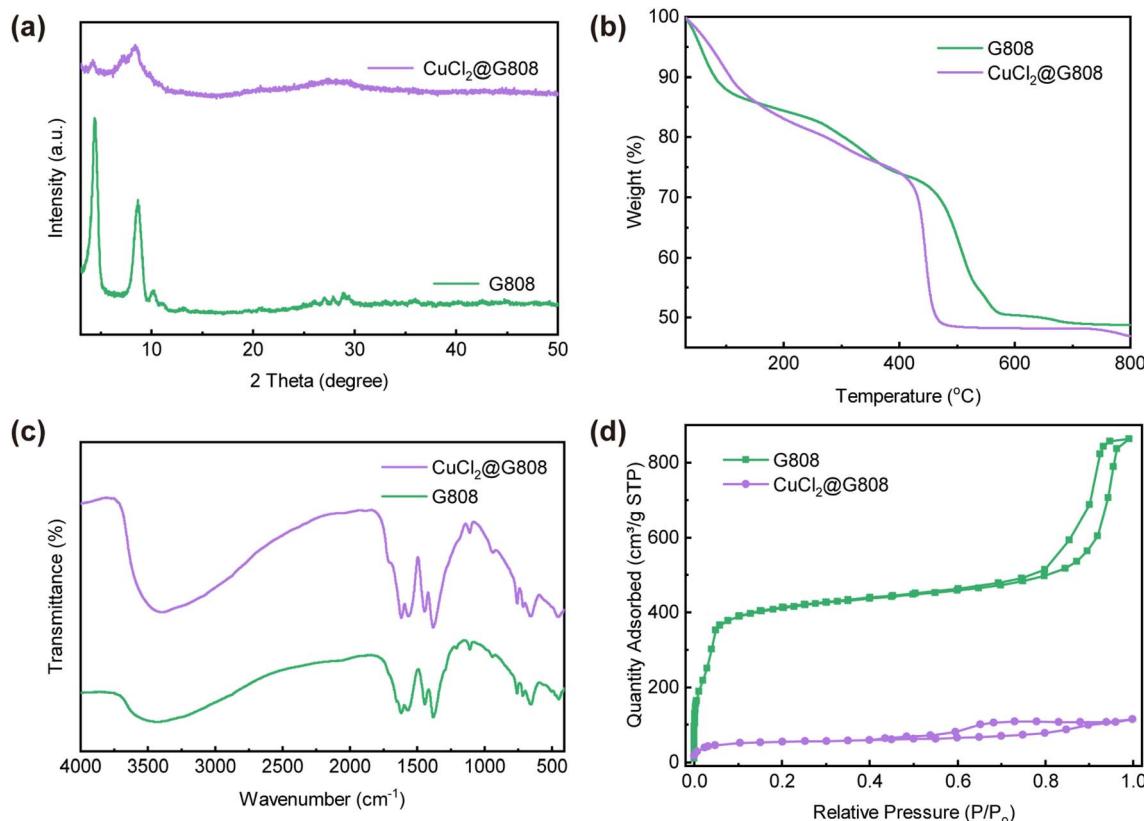


Fig. 1 (a) PXRD patterns, (b) TGA curves, (c) FT-IR spectra, and (d)  $\text{N}_2$  adsorption–desorption isotherms of  $\text{CuCl}_2@\text{G808}$ .



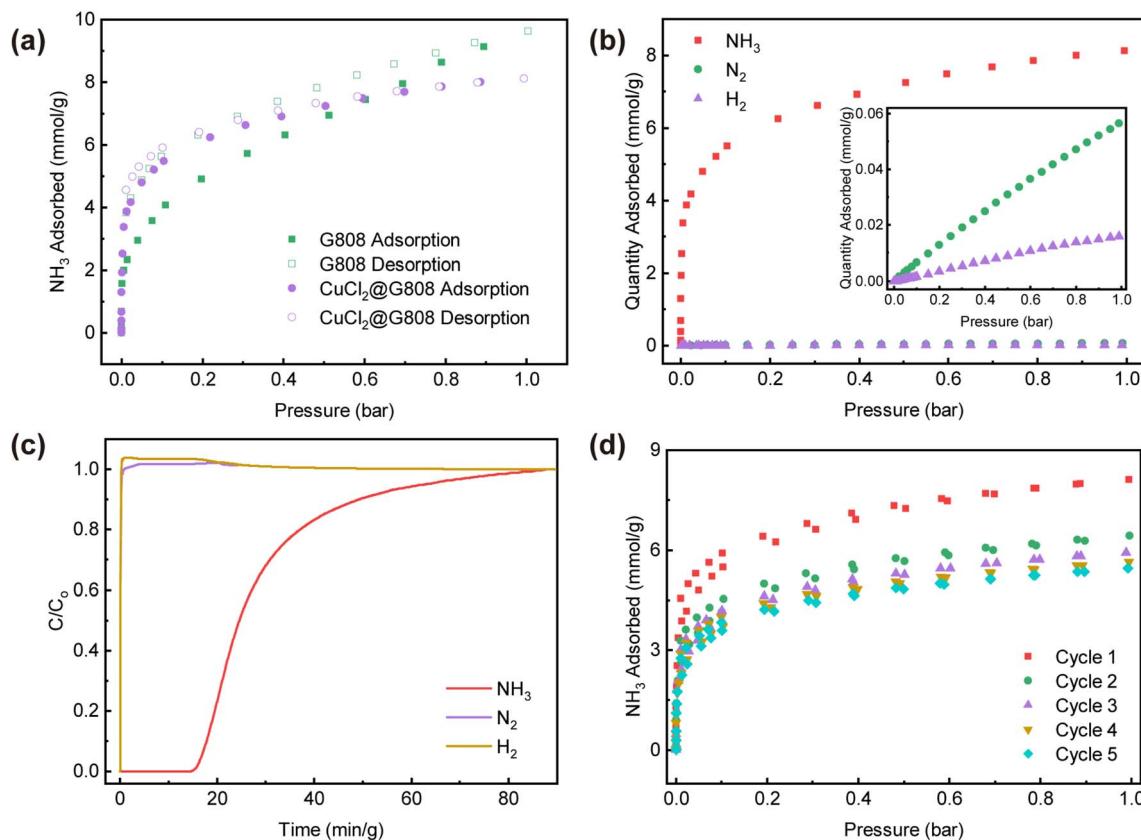


Fig. 2 (a) Adsorption–desorption isotherms of NH<sub>3</sub>, (b) adsorption isotherms of CuCl<sub>2</sub>@G808 for NH<sub>3</sub>, N<sub>2</sub>, and H<sub>2</sub>, (c) breakthrough curves of CuCl<sub>2</sub>@G808 for NH<sub>3</sub>/N<sub>2</sub>/H<sub>2</sub> mixtures, (d) NH<sub>3</sub> adsorption–desorption isotherms for different cycles of CuCl<sub>2</sub>@G808 at 298 K.

values represent enhancement of 37%, 74%, and 79%, respectively. These results are comparable to the best-behaving values reported recently (Table S2), and indicate that our composite is conducive to the capture of low concentration NH<sub>3</sub>. To validate this, N<sub>2</sub> and H<sub>2</sub> isotherms of CuCl<sub>2</sub>@G808 are measured at 273 K and 298 K (Fig. 2b, S5 and S6). Notably, the adsorption capacities of N<sub>2</sub> (0.056 mmol g<sup>-1</sup>) and H<sub>2</sub> (0.016 mmol g<sup>-1</sup>) on CuCl<sub>2</sub>@G808 at 298 K and 1 bar are significantly lower than that of NH<sub>3</sub> (8.1 mmol g<sup>-1</sup>). This may be attributed to their weak affinity with the composite originated from low  $Q_{st}$  values (Fig. S7 and S8). The IAST-predicted selectivity values of CuCl<sub>2</sub>@G808 reach  $2.8 \times 10^3$  for NH<sub>3</sub>/N<sub>2</sub> and  $4.9 \times 10^5$  for NH<sub>3</sub>/H<sub>2</sub> at 298 K, respectively (Fig. S9 and 10), surpassing most reported MOFs.<sup>11,42</sup> Furthermore, granular CuCl<sub>2</sub>@G808 exhibits excellent self-shaping ability, facile synthesis, and low production cost, demonstrating its competitive and practical potential in low concentration NH<sub>3</sub> capture and separation.

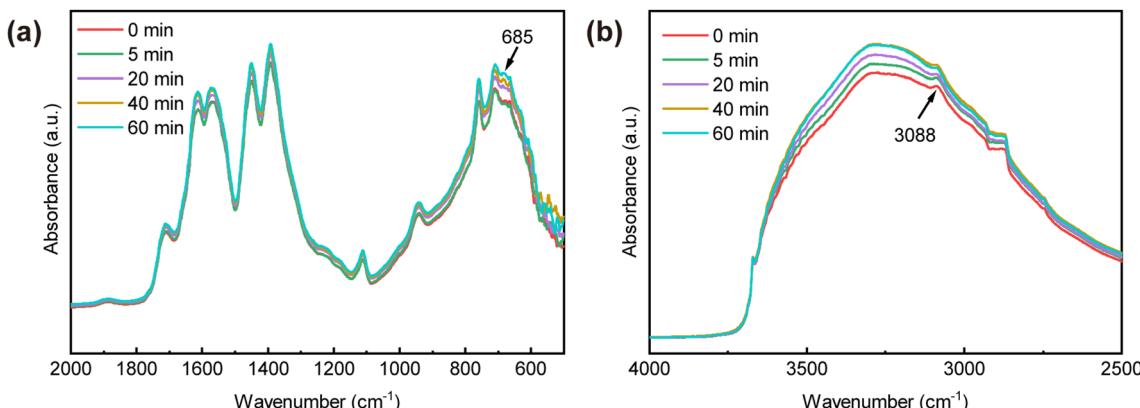
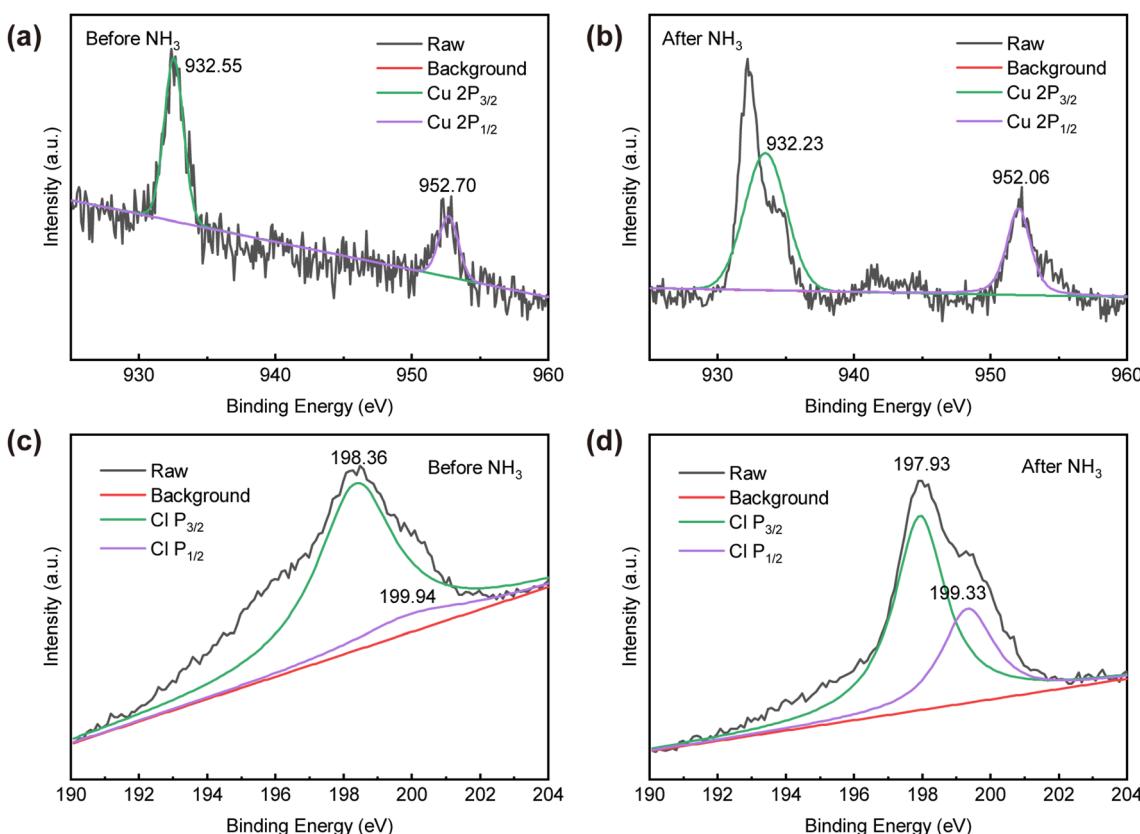
Considering the valuable NH<sub>3</sub> industry synthesis, breakthrough experiments are adopted to validate the practical separation capability of CuCl<sub>2</sub>@G808 for NH<sub>3</sub> using actual NH<sub>3</sub>/N<sub>2</sub>/H<sub>2</sub> mixtures (3% NH<sub>3</sub> in 25% N<sub>2</sub> and 72% H<sub>2</sub>) (Fig. 2c). It is clearly observed that N<sub>2</sub> and H<sub>2</sub> are immediately escaped because of their low adsorption capacity, followed by NH<sub>3</sub> after 16.8 min g<sup>-1</sup> accompanied by uptake of 3.9 mmol g<sup>-1</sup>. Thus, CuCl<sub>2</sub>@G808 can be considered as a superior adsorbent for selective separation of NH<sub>3</sub>/N<sub>2</sub>/H<sub>2</sub> at low NH<sub>3</sub> concentration.

Five recycling experiment of CuCl<sub>2</sub>@G808 is also carried out to study its practical performance (Fig. 2d and S11). NH<sub>3</sub> adsorption capacity of CuCl<sub>2</sub>@G808 can be only maintained up to 5.4 mmol g<sup>-1</sup> (67.1%) after five cycles at 298 K and 1 bar due to its partial structural collapse (Fig. S12 and S13). Meanwhile, about 50.7% of NH<sub>3</sub> is difficult to desorb due to the strong interaction between NH<sub>3</sub> and CuCl<sub>2</sub>@G808.

### Mechanism of NH<sub>3</sub> adsorption

To elucidate adsorption mechanism between CuCl<sub>2</sub>@G808 and NH<sub>3</sub>, a combination of spectroscopic techniques—including *in situ* FTIR and XPS spectra are employed (Fig. S14). After adsorption of NH<sub>3</sub>, a new IR peak attributed to N–H deformation vibration emerges at 685 cm<sup>-1</sup> (Fig. 3a), suggesting potential coordination between NH<sub>3</sub> and Cu<sup>2+</sup>.<sup>43</sup> Besides, the C=O stretching vibration peak shifts from 1566 cm<sup>-1</sup> to 1574 cm<sup>-1</sup> (Fig. 3a) and the weak phenyl C–H peak at 3088 cm<sup>-1</sup> disappear (Fig. 3b) after NH<sub>3</sub> adsorption, possibly due to hydrogen bonding between NH<sub>3</sub> and these groups.

The XPS survey spectrum of CuCl<sub>2</sub>@G808 over the range of 0–1300 eV is presented in Fig. S15. Characteristic peaks corresponding to C 1s, O 1s, Zr 3d, Cu 2p, and Cl 2p are clearly observed, confirming the presence of these elements in the composite. Furthermore, XPS spectrum of CuCl<sub>2</sub>@G808 after NH<sub>3</sub> adsorption is also recorded (Fig. 4). Following NH<sub>3</sub>

Fig. 3 (a) and (b) *In situ* FTIR spectra of CuCl<sub>2</sub>@G808 during NH<sub>3</sub> uptake.Fig. 4 XPS spectra of Cu 2p (a) and (b) and Cl 2p (c) and (d) for CuCl<sub>2</sub>@G808 before and after NH<sub>3</sub> adsorption.

adsorption, the binding energies of Cu 2p<sub>3/2</sub> and Cu 2p<sub>1/2</sub> shift from 932.55 eV and 952.70 eV to 932.23 eV and 952.06 eV, respectively (Fig. 4a and b). This negative shift suggests enhanced electron density around Cu<sup>2+</sup> due to coordination with the N atom of NH<sub>3</sub>, consistent with electron donation from the adsorbate to the metal center.<sup>43</sup> Meanwhile, the Cl 2p peaks of CuCl<sub>2</sub>@G808 at 199.94 eV and 198.36 eV decrease to 199.33 eV and 197.93 eV, respectively (Fig. 4c and d), attributed to hydrogen bonding between NH<sub>3</sub> and Cl<sup>-</sup> sites.<sup>4,43</sup> These findings further validate that CuCl<sub>2</sub> incorporation enhances the NH<sub>3</sub> adsorption capacity of the composite at low pressure.

## Conclusion

In summary, we develop a granular CuCl<sub>2</sub>@G808 metal–organic gel *via* a simple aqueous-phase impregnation method. It is interesting to observe that CuCl<sub>2</sub>@G808 demonstrates an exceptional NH<sub>3</sub> uptake (2.23 mmol g<sup>-1</sup>) at 298 K and 0.002 bar, which exhibits 79% enhancement over pristine G808 and maintains 67.1% adsorption capacity after five adsorption–desorption cycles. On the other hand, CuCl<sub>2</sub>@G808 achieves outstanding IAST selectivity for NH<sub>3</sub>/N<sub>2</sub> ( $2.8 \times 10^3$ ) and NH<sub>3</sub>/H<sub>2</sub> ( $4.9 \times 10^5$ ). *In situ* FTIR and XPS spectra reveal that low NH<sub>3</sub>



pressure can be mainly explained by  $\text{NH}_3\text{-Cu}^{2+}$  coordination and hydrogen-bond networks. These findings advance the rational design of robust adsorbents for capturing and separating trace  $\text{NH}_3$ .

## Conflicts of interest

The authors declare no conflict of interest.

## Data availability

The authors declare that the data supporting the findings of this study are available within the paper and its supplementary information (SI) files. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request. Supplementary information is available. See DOI: <https://doi.org/10.1039/d5ra07740k>.

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