

Demethylation of Waste Alkali Lignin for Rapid and Efficient Ammonia Adsorption

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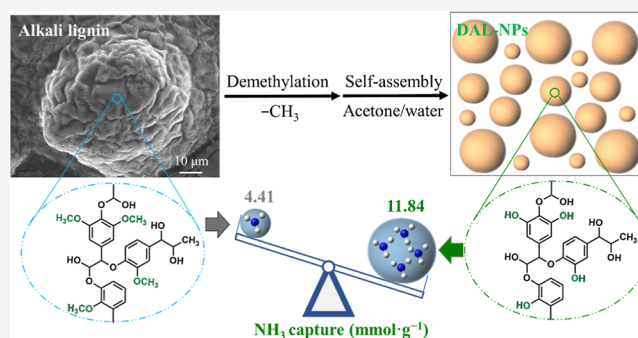
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ABSTRACT: Alkali lignin, a significant waste resource from the pulp and paper industry, is produced in large quantities each year, but its utilization remains limited. This study focuses on the cleavage of $-OCH_3$ groups attached to the aromatic ring in alkali lignin to obtain demethylated alkali lignin nanospheres (DAL-NPs) enriched with aromatic hydroxyl (Ar-OH) groups. These DAL-NPs are intended for the efficient adsorption of NH_3 . Qualitative and quantitative characterization of alkali lignin, both pre- and post-demethylation, was performed using Fourier transform infrared spectroscopy and 1H NMR. The analysis revealed a reduction in methoxy groups and an increase in Ar-OH groups in the DAL-NPs. The NH_3 adsorption experiment demonstrated that the DAL-NPs exhibited a rapid and high adsorption capacity, reaching an NH_3 uptake capacity of 10.0 mmol g^{-1} within 0.42 min at 298 K and 1.0 bar . In addition, the DAL-NPs displayed superior selectivity based on the ideal adsorption solution theory for $10\%\text{ NH}_3/90\%\text{ N}_2$ (3252) and $10\%\text{ NH}_3/90\%\text{ CO}_2$ (175) at 298 K and 1.0 bar . The DAL-NPs also demonstrated reversibility and stability during 10 adsorption–desorption cycles. The DAL-NP adsorbent possesses exceptional properties, such as abundance and low cost, rapid and efficient adsorption, reversibility, and stability, making it a promising solid adsorbent for effective NH_3 adsorption in the ammonia process.



1. INTRODUCTION

Alkali lignin, the primary component derived from the soda pulp industry, accounts for over 85% of the world's industrial lignin generation, with an annual yield of 40–50 million tons.^{1,2} Despite its significant production volume, alkali lignin remains severely underutilized in current practices due to its recalcitrant and complex structure. As a result, it is often disposed of as wastewater or incinerated without reuse. This leads to significant resource wastage and contributes to environmental issues, such as river pollution and high greenhouse gas emissions.^{3,4} Therefore, it is essential to explore strategies for modifying alkali lignin by equipping it with specific functional groups to enable practical applications. For instance, alkali lignin can be tailored into functional polymers capable of removing heavy metal ions and dyes,^{5–9} or it can be employed as dispersants.^{10,11} Additionally, it shows potential for developing lignin-based nanomaterials through physical or chemical methods.^{12,13}

Ammonia (NH_3) is a crucial substance in the chemical industry, serving as a feedstock in the manufacture of nitrogen fertilizers, nitrous oxide, alternative fuels, plastics, and dyes.^{14,15} However, NH_3 is also a corrosive air contaminant, posing significant challenges. When released into the atmosphere, NH_3 reacts with NO_x and SO_x , resulting in the formation of

secondary pollutants such as NH_4NO_3 and $(NH_4)_2SO_4$. These compounds contribute to the generation of PM 2.5 and acid rain, which can cause significant harm to both human health and the environment.^{16–19} With an estimated global annual production of almost 200 million tons of NH_3 , the widespread use of this compound increases the risk of potential leakage during production processes.^{20,21} Therefore, there is an urgent need to remove NH_3 from air and control its concentration to levels that are relatively safe.

Adsorption is widely recognized as a favorable method for the economic and efficient removal of low concentrations of NH_3 from industrial emissions.²² The choice of the adsorbent plays a critical role in the process. Various adsorbent materials, including carbon-based adsorbents,^{23–26} alumina,²⁷ silica,^{18,28} and metal-organic frameworks (MOFs),^{17,19,29–32} have been developed and modified to functionalized surfaces and porous structures, allowing for effective removal of NH_3 . For instance,

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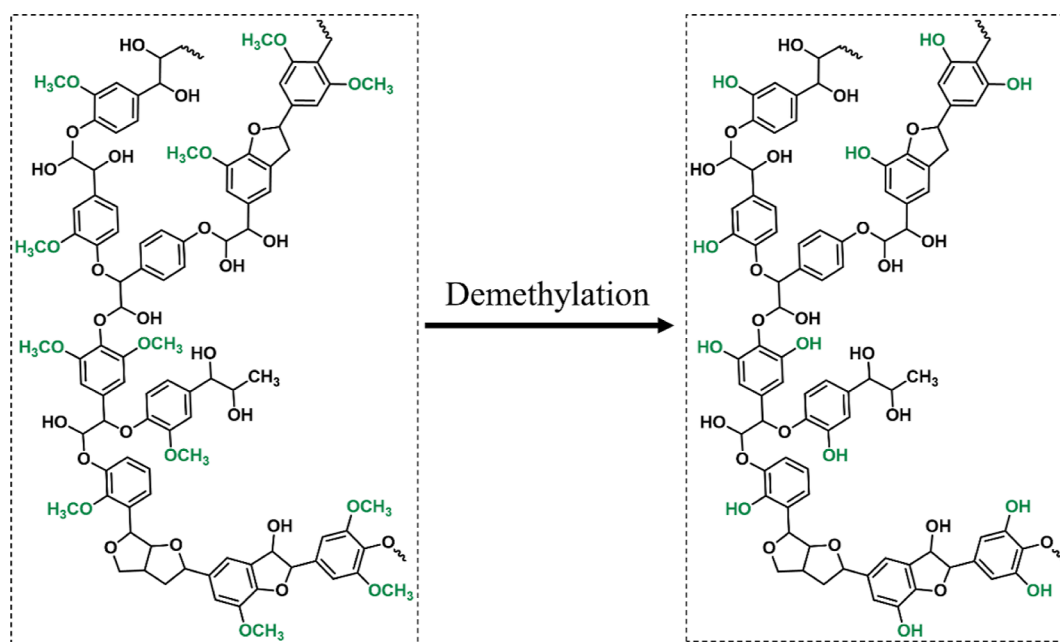
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Scheme 1. Schematic of the Production of Ar–OH-Rich Lignin by Alkali Lignin Demethylation



Wang et al.²⁹ used the MOF MIL-53-(OH)₂ as a base material and incorporated lithium chloride within its nanopore channels to achieve high-performance heterogeneous MOF adsorbents for NH₃ capture. Although these adsorbents have demonstrated favorable adsorption outcomes, they exhibit certain drawbacks such as complexity, high cost, low yield of prepared adsorbents, and limited separation selectivity, making them less suitable for industrial applications.²⁷ Our research group has recently synthesized three sulfonated phenol-formaldehyde resins that feature aromatic hydroxyl (Ar–OH) and sulfonate groups as acid sites to facilitate NH₃ adsorption.³³ Alkali lignin, which consists of H, G, and S units, contains a substantial amount of methoxy and hydroxyl groups attached to aromatic rings.^{34,35} By removing the methyl group from the methoxy group attached to the lignin's aromatic ring, an adsorbent material rich in weakly acidic Ar–OH sites can be obtained.^{36,37} This material has tremendous potential as an efficient adsorbent for capturing alkaline NH₃.

Herein, the focus of this study is the demethylation of alkali lignin to produce Ar–OH-rich lignin (Scheme 1). The resulting demethylated alkali lignin nanospheres (DAL-NPs) are then used as adsorbents for the separation and capture of NH₃. To obtain the variation of the lignin demethylation efficiency and Ar–OH content, alkali lignin before and after demethylation was characterized by Fourier transform infrared (FT-IR) spectroscopy and quantitative ¹H NMR. Then, the adsorption performance of DAL-NPs on NH₃ was systematically investigated, including adsorption capacity and separation selectivity. In addition, to explore their potential industrial applications, their recyclability and breakthrough performance in a NH₃/N₂/CO₂ mixture were also investigated.

2. MATERIALS AND METHODS

2.1. Materials. Lithium bromide (99%) and hydrogen bromide (48% by weight) were procured from Shanghai Adamas Reagent Co. Ltd. Alkali lignin and γ -valerolactone (98%) were supplied by Shanghai Maclean Biochemical Technology Co. High-purity N₂ (99.999%), NH₃ (99.999%),

and CO₂ (99.999%) were sourced from Jiangxi Huahong Special Gas Co. The experimental procedures utilized all solvents directly. The deionized water used in this study was prepared in-house in the laboratory.

2.2. Preparation of Demethylated Alkali Lignin Nanoparticles. The method for preparing demethylated alkali lignin nanoparticles (DAL-NPs) has been refined based on previously reported methods.^{38,39} In a typical procedure, a hydrothermal reactor was charged with 6.1 g of lithium bromide and 3.9 g of water. The mixture was stirred for 2 min until the lithium bromide was completely dissolved, resulting in a colorless solution. 0.5 g of alkali lignin and 1.0 mL of HBr (1.48 g, 48 wt %) were added to the solution. The reaction mixture was heated and maintained at 373 K for 4 h. After completion, the hydrothermal reactor was cooled, and the solid residue at the bottom was collected by centrifugation. It was then washed three times until the pH of the supernatant reached neutrality. The solid sample was solubilized in a solvent mixture of γ -valerolactone and water (4:1, v/v) with a mass (mg)/volume (mL) ratio of 40:1. Subsequently, 1.0 mL of the resulting lignin solution was slowly added dropwise to 50 mL of deionized water, gently stirred at 303 K for 2 h, and centrifuged at 8500 rpm for 12 min. The resulting brownish solid was washed three times with fresh deionized water and subjected to lyophilization, ultimately yielding DAL-NPs.

2.3. Characterization Methods. The detailed characterization techniques and procedures are given in the Supporting Information. The ¹H NMR characterization of lignin samples underwent an acetylation reaction following demethylation to enhance the solubility of lignin in the deuterium solvent (DMSO-*d*₆) and enable accurate analysis.⁴⁰

2.4. NH₃ Adsorption Procedure. The isotherms of NH₃ capture were investigated at different temperatures and pressures by using a two-chamber volumetric method developed in-house. Further details can be found in the Supporting Information (Figures S1 and S2). The experimental setup involved monitoring of the uptake process with a

digital indicator (NI). Gas adsorption used approximately 0.1 g of the adsorbent.

3. RESULTS AND DISCUSSION

3.1. Characterization of Lignin. Figure 1 shows the FT-IR spectra of alkali lignin and DAL-NPs. The spectrum showed

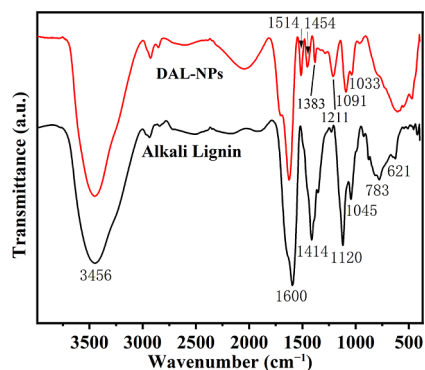


Figure 1. FT-IR spectra of alkali lignin and DAL-NPs.

a broad signal absorbance at 3456 cm^{-1} , which is indicative of the presence of O–H stretching in alcoholic and phenolic hydroxyl groups.⁴¹ The spectra display distinct peaks corresponding to the aromatic ring C=C bonds at $1450\text{--}1600\text{ cm}^{-1}$ and the C–H bonds in the region of $700\text{--}850\text{ cm}^{-1}$.⁴² The peaks observed at 1045 and 1120 cm^{-1} are mainly due to the stretching of the C–H bonds of the $-\text{OCH}_3$ group. The peak at 1414 cm^{-1} may be attributed to sodium hydroxide residues present in alkali lignin or the aromatic ring itself. Upon analysis of the demethylated alkali lignin nanoparticles in comparison to the alkali lignin, a significant reduction in the characteristic peaks related to sodium hydroxide (1414 and 783 cm^{-1}) was observed. This suggests that the demethylation process effectively eliminates sodium hydroxide impurities. Upon removal of sodium hydroxide, two previously obscured peaks (1514 and 1454 cm^{-1}) became visible. These peaks correspond to the C–C and C=C stretching vibrations of the lignin aromatic skeleton and the C–O stretching vibration peak of the benzene ring linking the methoxy Ar–OCH₃.^{42,43} Additionally, the FT-IR spectra show a notable reduction in the $-\text{OCH}_3$ peak (1045 and 1120 cm^{-1}) of DAL-NPs, while the peak corresponding to the Ar–OH group at 1211 cm^{-1} was increased.⁴⁴ These results suggest that demethylation decreases the quantity of $-\text{OCH}_3$ groups and enhances the characteristic peaks associated with Ar–OH groups in the DAL-NPs material.

To identify functional groups containing hydrogen, we quantified lignin before and after demethylation using ¹H NMR spectroscopy. Figure 2 shows the proton signals observed during the analysis. The signal at 10.16 ppm was assigned to CHO groups, and signals at 8.47 and 8.16 ppm were allocated to the aromatic protons of the internal standard of *p*-nitrobenzaldehyde (NBA). The proton signal observed at 3.75 ppm corresponds to the $-\text{OCH}_3$ group in lignin. The two peaks at 2.26 and 2.00 ppm were identified as the aromatic and aliphatic superacetyl groups of lignin, respectively. Protons of the aliphatic portion of lignin were observed in the range of $0.8\text{--}1.5\text{ ppm}$. The analysis indicates that after the demethylation reactions, some Ph–OCH₃ groups are converted into Ar–OH groups.

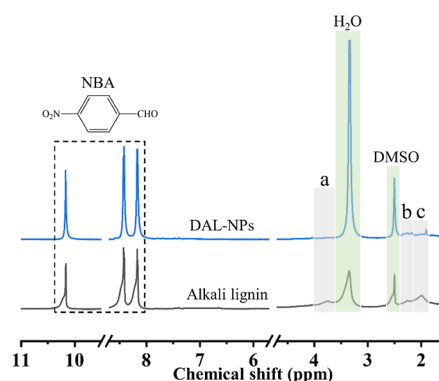


Figure 2. ¹H NMR spectrum of alkali lignin and DAL-NPs (a corresponds to the $-\text{OCH}_3$ group and parts b and c correspond to the aromatic and aliphatic acetyls, respectively).

The mass fraction of the $-\text{OCH}_3$ or Ar–OH group of lignin was determined by analyzing the ¹H NMR spectrum with NBA as an internal reference with a characteristic peak at 10.2 ppm . The results are presented in Table 1. Compared to that of alkali lignin, the content of $-\text{OCH}_3$ in DAL-NPs decreased from 1.38 to 0.59 mmol g^{-1} , whereas the Ar–OH content increased from 0.52 to 0.84 mmol g^{-1} .

Figure S3 shows that alkali lignin contains significant amounts of Na elements along with C and O elements. However, DAL-NPs do not contain any Na elements. This observation is consistent with the FT-IR characterization results, which suggest that the demethylation process effectively removes sodium hydroxide impurities from the alkali lignin through acid and water washing.

Scanning electron microscopy (SEM) was used to characterize the surface morphologies of alkali lignin and DAL-NPs. Figure 3c,d shows that the initial alkali lignin has larger clusters or aggregates, while DAL-NPs exhibit well-defined nanospheres. The nanospherical shape facilitates the diffusion and adsorption of gas molecules.

Figure 4 shows additional sorption isotherms of alkali lignin and DAL-NPs conducted under a nitrogen atmosphere. The results demonstrate that the isotherms of alkali lignin exhibit a significantly lower N₂ adsorption capacity, as well as a limited surface area of $0.95\text{ m}^2\text{ g}^{-1}$. Notably, N₂ adsorption occurs primarily at $0.9\text{--}1.0P/P_0$, indicating the existence of slit pores within the sample.

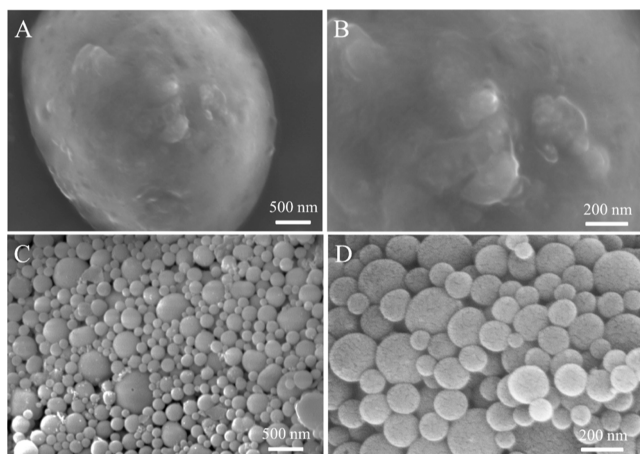
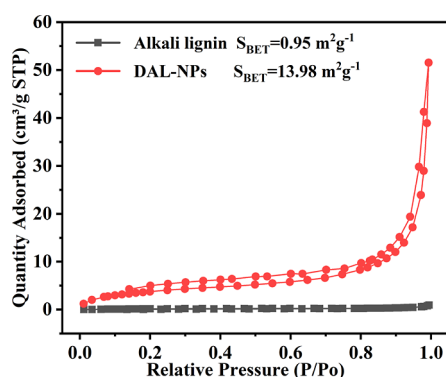
Thermogravimetry (TG) analysis was performed on alkali lignin and DAL-NPs to examine their thermal behavior, as shown in Figure S4. The analysis indicated a 5% thermogravimetric loss for both lignins and DAL-NPs when the temperature was increased from 303 to 373 K , mainly due to the removal of free water. Subsequently, as the temperature increased, the samples entered the thermal decomposition phase; during this phase, chemical bonds, mainly C–O and C–C bonds, were broken, resulting in the generation of volatiles. At 773 K , both samples completely decomposed, leaving behind coke residues. The final coke residue percentages were determined to be 36.64% for alkali lignin and 46.14% for DAL-NPs. The TG analysis indicates that both materials are stable at temperatures below 373 K .

3.2. NH₃ Uptake Performance. Figure 5a shows the NH₃ uptake rates of alkali lignin and DAL-NPs at 298 K and 1 bar . Both alkali lignin and DAL-NPs exhibited rapid adsorption rates for NH₃, reaching equilibrium within 2.5 min . The NH₃ uptake capacity of 10.0 mmol g^{-1} was achieved within 0.42

Table 1. Chemical Shifts and Relative Proportions of $-OCH_3$ and $Ar-OH$ Groups in Alkali Lignin and DAL-NPs^a

lignin sample	M (mg)	M_i (mg)	chemical shift (ppm)			F_{Ar-OH} (mmol g^{-1})	F_{MeO} (mmol g^{-1})
			I_i δ 10.4–10.2	I_{Ar-OH} δ 2.5–2.17	I_{MeO} δ 4.0–3.48		
alkali lignin	20	5	2	4.16	11.04	0.52	1.38
DAL-NPs	20	5	2	6.72	4.72	0.84	0.59

^a M represents the weight of alkali lignin or DAL-NPs (mg), M_i represents the weight of NBA (mg), I_i represents the peak area of NBA, and F represents the specific content of certain groups (mmol g^{-1}).

**Figure 3.** SEM images of alkali lignin (A,B) and DAL-NPs (C,D).**Figure 4.** N_2 sorption isotherms of alkali lignin and DAL-NPs.

min at 298 K and 1.0 bar. DAL-NPs had a higher NH_3 uptake capacity (11.84 mmol g^{-1}) than alkali lignin (4.41 mmol g^{-1}) under the same conditions. This observation suggests that DAL-NPs have a higher concentration of acidic $Ar-OH$ groups than alkali lignin, which enhances their NH_3 sorption capacity. Additionally, we conducted experiments to examine the adsorption rate of DAL-NPs at different temperatures (Figure 5a). Surprisingly, it is found that temperature did not have a significant impact on the equilibrium time (~ 2.5 min) for NH_3 adsorption by DAL-NPs. However, the adsorption capacity is significantly affected by the temperature, indicating a significant decrease in adsorption capacity with increasing temperature. Similar results have been reported in the relevant literature.^{33,45–47}

To investigate the impact of the pressure on NH_3 adsorption by DAL-NPs, we conducted static adsorption experiments to measure the adsorption isotherms of DAL-NPs at various pressures. Figure 5b shows an increase in the NH_3 gas adsorption capacity of DAL-NPs with increasing pressure, indicating that NH_3 adsorption by DAL-NPs becomes more

favorable under higher pressure conditions. The isothermal heat of the reaction was determined to be within the range of -30 to -25 kJ mol^{-1} by establishing the relationship among NH_3 uptake, capacity, and temperature (see Figure S5).

In the ammonia industry, flue gases commonly contain a mixture of NH_3 , N_2 , and CO_2 . Therefore, it is important to assess the selectivity of DAL-NPs in separating NH_3 from gas mixtures. Figure 5c shows the adsorption capacity of DAL-NPs for NH_3 , N_2 , and CO_2 at various pressures and at a temperature of 298 K. It is clear that the uptake capacity of DAL-NPs for N_2 and CO_2 is significantly lower than that for NH_3 at different pressures. Furthermore, we conducted additional investigations into the IAST selectivities for NH_3/CO_2 and NH_3/N_2 at varying NH_3 molar fractions (Figure 5d). The results demonstrate that DAL-NPs exhibit exceptional selectivity for separating NH_3/N_2 and NH_3/CO_2 mixtures, with IAST selectivity values of 3252 for NH_3/N_2 (10/90, v/v) and 175 for NH_3/CO_2 (10/90, v/v). These findings highlight the significant potential of DAL-NPs as an excellent adsorbent for NH_3 capture.

3.3. Breakthrough Performance of DAL-NPs. To evaluate the practical performance of DAL-NPs in separating NH_3/CO_2 and NH_3/N_2 under real conditions, we studied a flue gas mixture consisting of 96% N_2 , 1% CO_2 , and 3% NH_3 . We examined the breakthrough behavior of DAL-NPs was examined at 303 K and 1 bar, with a gas streamflow rate of 20.0 mL min^{-1} . Figure 6a shows a gradual breakthrough of NH_3 on DAL-NPs, with a residence time of approximately 100 min g^{-1} . These results demonstrate that DAL-NPs can effectively and selectively eliminate NH_3 , even in the presence of N_2 and CO_2 . This highlights the potential of DAL-NPs for use in the fuel gas treatment.

3.4. Regeneration Performance. To investigate the cycling stability of DAL-NPs, we subjected the sample that had reached adsorption saturation to a vacuum desorption process at 353 K and 0.1 kPa for 2.5 h, resulting in the recovery of the desorbed DAL-NPs. We subsequently measured the adsorption capacity of DAL-NPs for NH_3 again and repeated the process 10 times to assess the cycling performance. Figure 6b shows that there was no discernible change in the adsorption capacity of DAL-NPs for NH_3 after 10 cycles. Furthermore, the FT-IR spectra of the cyclically desorbed DAL-NPs are shown in Figure S6. A prominent peak at 1393 cm^{-1} is observed, which is due to the formation of NH_4^+ resulting from the interaction between NH_3 and $Ar-OH$ in DAL-NPs.¹⁸ Additionally, no significant changes in characteristic peaks were observed in the FT-IR spectra of the cyclically desorbed DAL-NPs when compared to those of the fresh DAL-NPs. Table S1 displays the elemental composition of the DAL-NPs adsorbent before and after NH_3 desorption. The results demonstrate that the content of elemental N remains stable without significant changes, and the content of other elements (C, H, and O) is also consistent. Therefore, it can be concluded that the DAL-NP material is capable of completely

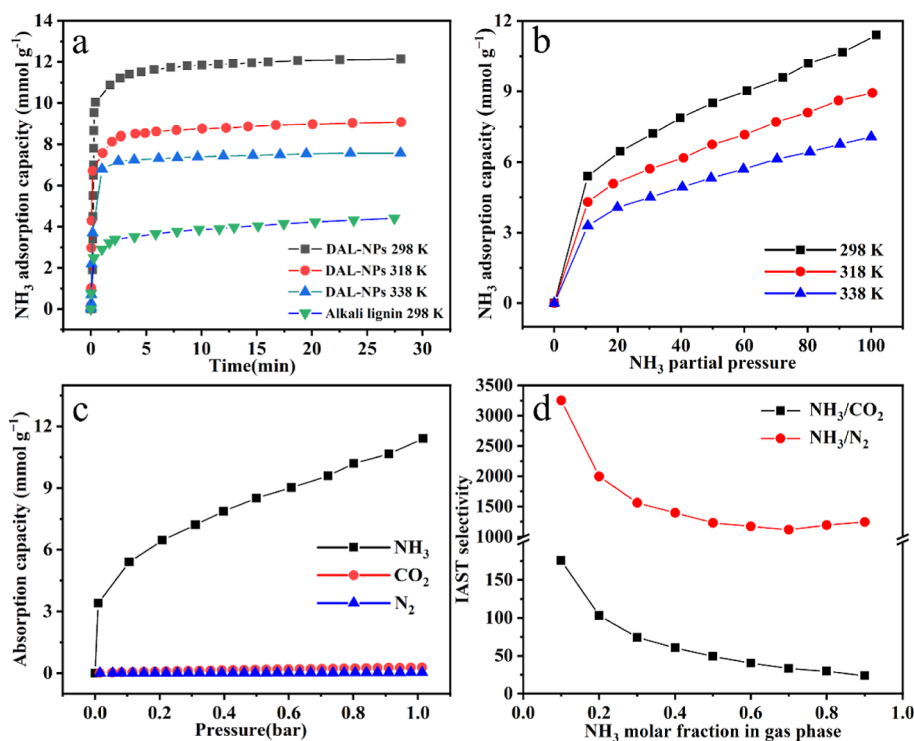


Figure 5. (a) NH_3 adsorption rate of alkali lignin and DAL-NPs, (b) influence of temperature on NH_3 adsorption through DAL-NPs, (c) NH_3 , CO_2 , and N_2 uptake isotherms of DAL-NPs at 298 K, 1 bar, and (d) IAST selectivities to NH_3/CO_2 and NH_3/N_2 .

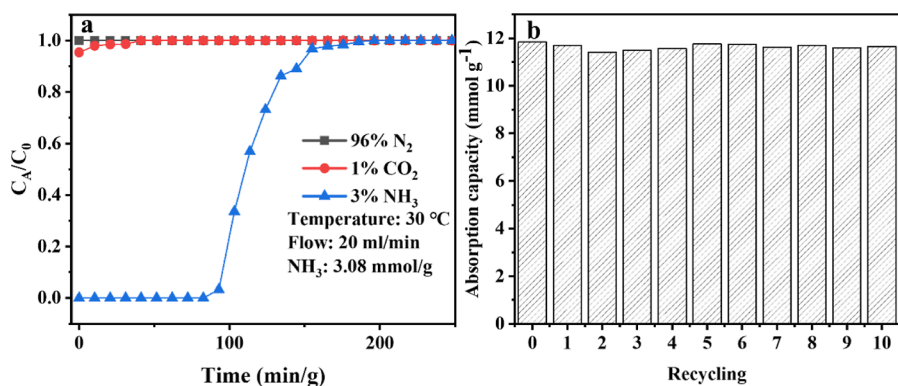


Figure 6. (a) Experimental breakthrough curves of simulated NH_3 flue gas separation by DAL-NPs at 303 K and (b) recycling performance of DAL-NPs for NH_3 adsorption and desorption at 298 K.

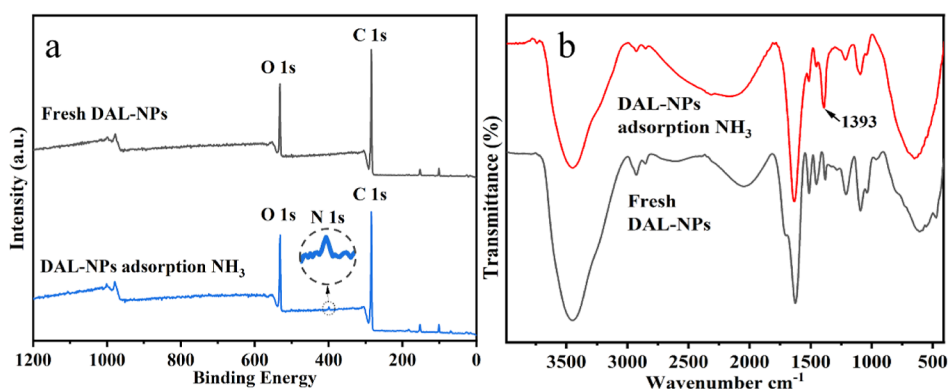


Figure 7. (a) XPS curves and (b) FT-IR images of fresh DAL-NPs and DAL-NPs adsorption of NH_3 .

desorbing NH_3 while remaining stable. The finding is consistent with the FT-IR characterization presented in Figure

S6, which shows that NH_3 can be efficiently desorbed from the Ar–OH-rich adsorbent, indicating exceptional recyclability.

Table 2. NH₃ Adsorption Capacity and Recycling Performance of the Adsorbents and Other Adsorbents in the Literature

entry	adsorbents	NH ₃ capacity (mmol·g ⁻¹)		recycling conditions	NH ₃ adsorption uptake loss	refs
		0.1 bar	1.0 bar			
1	alkali lignin	1.74	4.41			this work
2	DAL-NPs	5.40	11.84	353 K, 2 h	almost no less (10 cycles)	this work
3	organosolv lignin	2.10	5.45			this work
4	de-organosolv lignin	4.91	11.62	353 K, 2 h	almost no less (8 cycles)	this work
5	NU-300	4.05	8.28	393 K, 12 h	~35% (3 cycles)	48
6	PAA	5.73	10.70	353 K, 18 h	13% (5 cycles)	49
7	PI	4.21	9.00	353 K, 18 h	almost no less (5 cycles)	49
8	BIDC-2-700	6.24	14.26	298 K	~21% (2 cycles)	25
9	BIDC-0.5-750	3.84	11.47	298 K	~5% (2 cycles)	25
10	PIM-1-COOH	7.05	12.2	298 K	~11% (3 cycles)	50
11	Cu ₂ O ₄ /BGB-2-600-0.7	1.45	8.68	298 K, 2 h	~25% (8 cycles)	53
12	PDVB-2.0AA	4.51	8.83	353 K, 2 h	11.2% (10 cycles)	52

3.5. Mechanism of NH₃ Absorption. X-ray photoelectron spectroscopy (XPS) and FT-IR characterization of the materials before and after adsorption were conducted to investigate the adsorption mechanism of NH₃ by DAL-NPs (Figure 7). The wide XPS spectrum in Figure 7a shows a small peak at 399.7 eV in the adsorbent after NH₃ adsorption, which can be attributed to the peak of N 1s in adsorbed NH₃, compared to that of fresh DAL-NPs. In addition, the FT-IR analysis showed an extra peak at 1393 cm⁻¹ (Figure 7d), which corresponds to the N–H stretching vibration after the adsorption of DAL-NPs. This indicates that DAL-NPs successfully adsorbed NH₃. The phenolic hydroxyl group on the surface of the DAL-NPs material is widely recognized to exhibit weak acidity. Therefore, the adsorption of NH₃ by DAL-NPs materials is achieved through the interaction between the phenolic hydroxyl group with weak acidity on the surface of DAL-NPs and alkaline NH₃, which results in efficient NH₃ capture.

3.6. Comparison of DAL-NPs with Other Adsorbents in the Literature. Table 2 compares DAL-NPs and organosolv lignin from bamboo with other published porous adsorbents, including NU-300,⁴⁸ PAA,⁴⁹ BIDC-2-700,²⁵ PIM-1-COOH,⁵⁰ Cu₂O₄/BGB-2-600-0.7,⁵¹ and PDVB-2.0AA,⁵² for their NH₃ adsorption capacity and cycling performance. The NH₃ capture performance of DAL-NPs is 2.7 times higher than that of alkali lignin (11.84 vs 4.41 mmol·g⁻¹) at 298 K and 1 bar. The NH₃ uptake capacity of DAL-NPs exceeds that of NU-300, PAA, PI, Cu₂O₄/BGB-2-600-0.7, and PDVB-2.0AA. Notably, NU-300, BIDC-2-700, and Cu₂O₄/BGB-2-600-0.7 exhibit significant reductions in NH₃ uptake capacity during recycling. For example, Table 2 shows that NU-300 experiences a loss of almost 35% after three cycles due to the high interaction between their adsorption sites and NH₃, with adsorption enthalpy exceeding –50 kJ·mol⁻¹.³³ This makes complete desorption of adsorbed NH₃ challenging. In contrast, DAL-NPs exhibit almost no loss of NH₃ uptake capacity even after 10 cycles and can be fully desorbed under mild conditions. Overall, the DAL-NP adsorbent demonstrates satisfactory NH₃ adsorption capacity, excellent reversibility, and high selectivity for adsorbing NH₃ in the NH₃/CO₂/N₂ mixture.

4. CONCLUSIONS

This work successfully demethylated a significant amount of alkali lignin from pulp and paper to produce DAL-NPs. The resulting DAL-NPs were characterized and found to possess a

higher concentration of Ar–OH groups than that of alkali lignin. Efficient NH₃ adsorption is achieved using DAL-NPs as an adsorbent. The experimental results demonstrate a large NH₃ uptake capacity of 11.84 mmol g⁻¹ and rapid attainment of adsorption equilibrium within 5 min. Furthermore, DAL-NPs demonstrate exceptional selectivity for separating NH₃/CO₂ and NH₃/N₂, as demonstrated by IAST selectivity values of 175 and 3252, respectively. The breakthrough experiments conducted with simulated flue gas mixtures containing 3% NH₃ further confirm the impressive separation performance of DAL-NPs. Additionally, DAL-NPs exhibit notable structural stability and excellent reversibility over 10 cycles, indicating significant potential for NH₃ adsorption in industrial applications. This study demonstrates the effective use of waste industrial alkali lignin for the selective removal and capture of NH₃ from industrial flue gases. The reuse of waste material is of great importance, and this research provides a valuable contribution to this field.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.iecr.3c04306>.

Characterization methods, ¹H NMR characterization, NH₃ adsorption procedure, breakthrough tests, breakthrough experiments apparatus, NH₃ adsorption apparatus, apparatus for the determination of NH₃ adsorption, breakthrough experiment apparatus, XPS curves for alkali lignin and DAL-NPs, TG curves of alkali lignin and DAL-NPs, isotope heat of DAL-NPs, FTIR spectra of fresh and 10 times regenerated DAL-NPs, and elemental analysis of the DAL-NPs adsorbent before and after desorption of NH₃ (PDF)

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Notes

The authors declare no competing financial interest.

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