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Synthesis and evaluation of a hyperbranched amino-cellulose composite for carbon dioxide capture

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ABSTRACT

Reduction of CO_2 emissions is a major challenge in the transition towards more sustainable energy systems. The conventional methods of acidizing involve the use of powerful mineral acids, which could lead to environmental problems and operational challenges. This study aims to develop an alternative approach and solution to these problems by creating a hyperbranched amino-cellulose composite that can act as an absorbent in capturing CO_2 for future uses. This composite material was created by performing amine functionalization and cross-linking of cellulose in order to have effective CO_2 absorption. The composite material was analyzed using FTIR, XRD, and FESEM. The effect of CO_2 pressure, absorbent time, and concentration on the absorption capacity was studied. The composite synthesized at 70 °C exhibited the highest absorption performance among the samples under consideration. An increase in CO_2 pressure led to an improvement in adsorption, but adsorption equilibrium was achieved after 45 min. an optimum adsorbent dosage of 2 g/L was obtained, whereas higher dosage did not result in a significant improvement in absorption performance. The enhanced absorption performance can be attributed to the introduction of hyperbranched amino functional groups which provide additional active site for CO_2 capture. In general, the obtained hyperbranched amino-cellulose composite showed good performance in CO_2 capture and could be considered as a sustainable material for use in integrated carbon capture.

Keywords: CO_2 capture, absorption, TEPA, hyperbranched amino-cellulose composite, pH.

INTRODUCTION

One of the major problems in the issue of achieving environmental sustainability is the reduction of carbon dioxide (CO_2) emissions. One way in which the oil and gas industry can be involved in addressing this problem is through the geological storage of CO_2 emissions in underground reservoirs. The increase in atmospheric CO_2 concentration has been one of the greatest environmental concerns around the globe in recent times. Increase in CO_2 concentration is largely a result of fossil fuel usage, industries, cement manufacture, and deforestation. CO_2 concentration in the atmosphere contributes significantly to global warming, greenhouse effect, ocean acidification, and other environmental changes. Consequently, considerable amount of research has been done on the development of sustainable, efficient and environmentally friendly absorbent material for capturing CO_2 (Nunes, 2023). Massive mitigation of CO_2 requires the utilization of materials that are economically feasible, highly selective, and available in large quantities. Among many materials considered, cellulose-based materials have gained much attention because of their abundance in nature, biodegradability, environmental friendliness and easy processibility. Additionally, cellulose is mechanically strong with a porous structure; therefore, it is a good candidate for chemical modification to improve its ability for CO_2 capture and for incorporation of metal-organic framework (MOF) crystals (Adegoke et al., 2022). Chemical absorption using amines is

one of the widely used and practical techniques for capturing CO_2. However, enhancing the performance of amine absorbents with lower energy required for their regeneration has remained a challenge. Consequently, much effort has been directed towards modification of amine absorbents in order to enhance their performance for CO_2 capture and application (Wu et al., 2024). There is a way to improve the performance of cellulose-based adsorbents using the proper choice of chemical modifiers and the right conditions of synthesis. The surface modification process is responsible for the appearance of functional groups that are mostly amines that serve as active sites for CO_2 adsorption. This kind of modification increases the physicochemical characteristics of cellulose and provides additional interaction with CO_2 molecules, improving the adsorption rate and selectivity. Hence, surface modification of cellulose has become one of the ways of creating highly effective adsorbents for carbon capture technologies (Patra et al., 2025). Tetraethylenepentamine (TEPA) is usually applied as a modifier to increase the effectiveness of CO_2 adsorption of porous materials. It has two primary and three secondary amine groups that provide plenty of active sites for interaction with CO_2. Besides, low viscosity helps better dispersion of this compound on the surface of the adsorbent (Oday and Al-jendeel., 2024). Hyperbranched polymers are three-dimensional polymers that have a highly branched structure and many terminal functional groups, which can be modified for the achievement of specific properties. Hyperbranched polymers are less viscous, more soluble and thermally and salt resistant than traditional linear polymers owing to their unique molecular structure. These properties have led to their use in different applications, such as oil field chemicals and adsorbents. In the last years, there has been increased interest in the study of hyperbranched polymers owing to their versatile structure and numerous possibilities for functionalization (Zhang and Yu, 2022). Epichlorohydrin (ECH) is a crosslinker commonly used for biopolymers, such as cellulose, starch, and chitosan. In the presence of alkaline, epoxide group of ECH is opened, and ECH forms covalent bonds with hydroxyl groups of biopolymers. The crosslinking process results in the improvement of pore size distribution, mechanical strength, and adsorption-desorption properties and thus makes ECH a promising crosslinker for the preparation of effective CO_2 adsorbents (Chen et al., 2023).

To enhance the adsorbing capability of the CO_2 from the atmosphere by the cellulose materials, many researchers have employed various amination processes. For instance, (Qi et al., 2025) have developed an aminated microcrystalline cellulose aerogel as a sustainable material for CO_2 capture through the incorporation of amino-silane functional groups on the cellulose material. The resulting material has been characterized via FTIR, SEM, and BET techniques. This material exhibited a high CO_2 adsorption capacity and cyclic stability, thereby suggesting that it can be employed as an efficient and environmental-friendly adsorbent for CO_2 capture.

An additional investigation by (Panahi et al., 2026) was aimed at the development of amine-functionalized cellulose-chitosan composite with TEPA as the effective agent to be used in CO_2 capture technology. As a result of the optimization, it was possible to obtain an adsorbent with the maximum CO_2 adsorption capacity equal to 358.44 mgg^{-1} at 9 bar. As it was reported by (Boonmatoon et al., 2025), the adsorbents of amine-functionalized silica have the potential to be used in the process of CO_2 capture because of the high efficiency of adsorption. Although silica has high surface area and pore volume, its application in the process of CO_2 adsorption is constrained by such characteristics as moisture sensitivity and low CO_2 selectivity. Functionalization of silica with amine groups contributes to the improvement of adsorption performance because of the interaction with CO_2 by means of the reversible carbamate formation. Moreover, the results obtained by (He et al., 2017) revealed that hyperbranched polyamine adsorbent for CO_2 capture can be synthesized by grafting amino-terminated hyperbranched polyamine onto polypropylene fibers. The synthesized adsorbent had the maximum CO_2 adsorption capacity equal to 5.64 mmol g^{-1} at 25 °C. Also, the high efficiency of amine utilization and high regeneration performance were noted. Despite the impressive progress in the development of amine-functionalized cellulose-based adsorbents for CO_2 capture, almost all research works concentrated on the increase in adsorption capacity and material characterization. However, there is still a need for the development of efficient cellulose-based adsorbents with a higher density of active amine sites and systematic evaluation of their adsorption performance under various conditions (Akaya et al., 2025).

The aim of this study is to synthesize a hyperbranched amino-cellulose composite using TEPA and epichlorohydrin (ECH) as a sustainable adsorbent for CO_2 capture. The synthesized composite is characterized using FTIR, XRD, and FESEM to investigate its chemical structure, crystalline properties, and surface morphology. Furthermore, the effects of CO_2 pressure, contact time, and adsorbent dosage on CO_2 adsorption capacity are systematically investigated to determine the optimum conditions for effective CO_2 capture and evaluate the potential of the developed composite for sustainable carbon capture applications.

MATERIALS AND METHODS

Materials

The materials and chemicals utilized in the present work including their chemical formula, suppliers and purity are summarized in Table 1.

Methodology

Hyperbranched amino cellulose solid absorbent was prepared by cellulose activation, amination by TEPA and cross-linking using epichlorohydrin as. The hyperbranched amino-cellulose solid absorbent was then purified, washed and dried. Finaly, the absorbent was suspended in deionized water followed by exposure to CO_2 at ambient temperature for different contact times from 15 to 60 minutes to allow the in-situ formation of carbonic species to enhance CO_2 absorption. The entire process of hyperbranched amino-cellulose solid absorbent preparation and activation is depicted in Figure 1.

Experimental work

Preparation of solid absorbent

Ten grams of cellulose were dispersed in 200 mL of distilled water, then 1 M of NaOH solution was added dropwise until the pH of the solution reached 11. The mixture was then heated to 60 °C and stirred for 1 hour at 500 rpm to promote the deprotonation and activation of the hydroxyl (–OH) groups of cellulose. After that 5 ml of TEPA was added dropwise to the cellulose suspension, the reaction was continued for 5 hours at 500 rpm while maintaining the pH at approximately 11 (Helmlinger et al., 2018) by the dropwise addition of 0.01 M HCl as required. The amine-functionalization reaction was conducted at three different temperatures (60, 70, and 80 °C) to investigate the effect of reaction temperature on amine functionalization.

After the amine functionalization process, epichlorohydrin (ECH) was added dropwise as a crosslinking agent under continuous stirring at 500 rpm. The crosslinking reaction was conducted at 80 °C for 4 h. Following the addition of ECH, the pH decreased to approximately 7.6. Therefore, 0.1 M NaOH solution was added dropwise while continuously monitoring the pH until it reached approximately 8.0. This crosslinking process resulted in the formation of a hyperbranched, gel-like structure containing amine functional groups. It is noteworthy that the amine-functionalized samples prepared at 70 and 80 °C required 3 mL of ECH to form a stable gel network, whereas the sample prepared at 60 °C required 4 mL of ECH to develop a similar gel structure.

After completion of the crosslinking reaction, the reaction mixture was allowed to cool under ambient conditions to room temperature. The resulting solid product was separated by filtration

Table 1. List of chemicals and materials

Material	Chemical formula	Supplier/manufacturer	Purity/grade
Microcrystalline Cellulose powder	$(C_6H_{10}O_5)_n$	Central Drug House (CDH), India	99%
Tetraethylenepentamine	TEPA	Sigma-Aldrich, Netherlands	≥95.0%
Epichlorohydrin	ECH	Central Drug House (CDH), India	99%
Sodium hydroxide	NaOH	Avonchem	99%
Hydrochloric acid	HCl	Riedel-de Haen, Germany	99%
Distilled water	H_2O	Prepared in the laboratory, University of Baghdad, Iraq	Laboratory grade
Ethanol	C_2H_5OH	BHD Chemicals Ltd., Poole, England, UK	99.8%
Carbon dioxide	CO_2	Al-Khalej Plant	99%

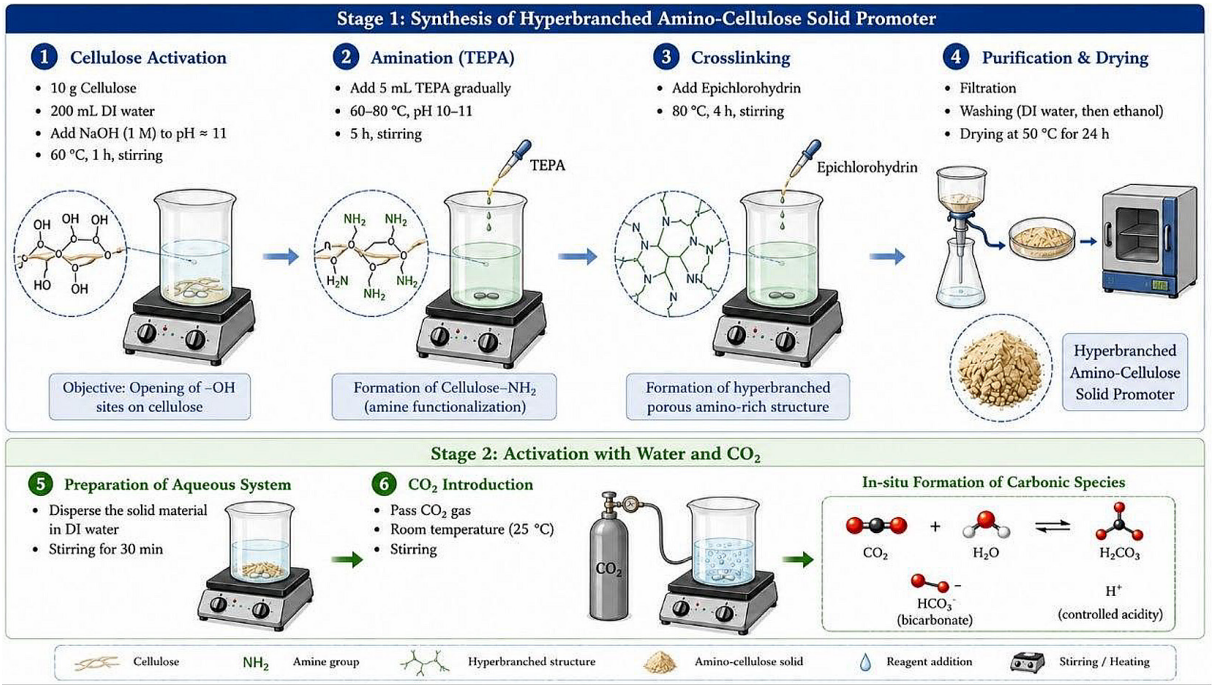


Figure 1. Schematic illustration of the synthesis of hyperbranched amino-cellulose solid absorbent and its activation in an aqueous CO_2 system

and washed twice with distilled water to remove unreacted chemicals and water-soluble by-products, followed by a final washing with ethanol to further remove residual impurities. The purified material was then dried in an oven at 50 °C for 24 h (Bernard et al., 2019) until a constant weight was achieved. The dried absorbent was stored in a sealed container until further characterization and absorption experiments.

Preparation of the aqueous suspension

An aqueous suspension was made by suspending 0.2 g of the solid sample in 100 mL of distilled water. The suspension was stirred for 30 min using a magnetic stirrer. While stirring, some drops of 0.01 M HCl solution were added dropwise to adjust the pH to approximately 7 while ensuring constant stirring to make a uniform suspension.

CO_2 absorption experiments

CO_2 absorption experiments were conducted using the experimental apparatus illustrated in Figure 2. The system consisted of a CO_2 gas cylinder (99% purity) equipped with a pressure controller and pressure gauges and connected to a high-pressure, high-temperature reactor. The reactor consisted of a 1000 mL cylindrical vessel made of carbon steel, mounted on a square base and equipped with two inlet valves, one serving as a vent valve and the other as the CO_2 inlet connected to the CO_2 cylinder. A drain valve was located at the bottom of the reactor for collecting the CO_2-activated hyperbranched amino-cellulose suspension. At the top of the reactor, a variable-speed motor (0–2500 rpm) was installed to provide continuous stirring during the CO_2 absorption experiments(Aldayyeni and Alameedy, 2026).

The prepared aqueous suspension was transferred into the reactor before feeding CO_2 gas. Experiments were performed using a pressure of 5 bar and a contact time of 45 min with continuous stirring for gas and liquid interactions. CO_2 absorption was carried out using three absorbents that have been synthesized using tetraethylenepentamine (TEPA) functionalization at temperatures of 60, 70, and 80 °C. The pH of the aqueous suspension was measured before the CO_2 feed and after 45 min of contact time. The difference in the pH were used to analyze the efficiency of the CO_2 absorption by absorbents and to select the best temperature of the TEPA functionalization process.

Study of operating conditions

Based on the preliminary CO_2 capture screening, the absorbent exhibiting the highest performance was selected for detailed parametric

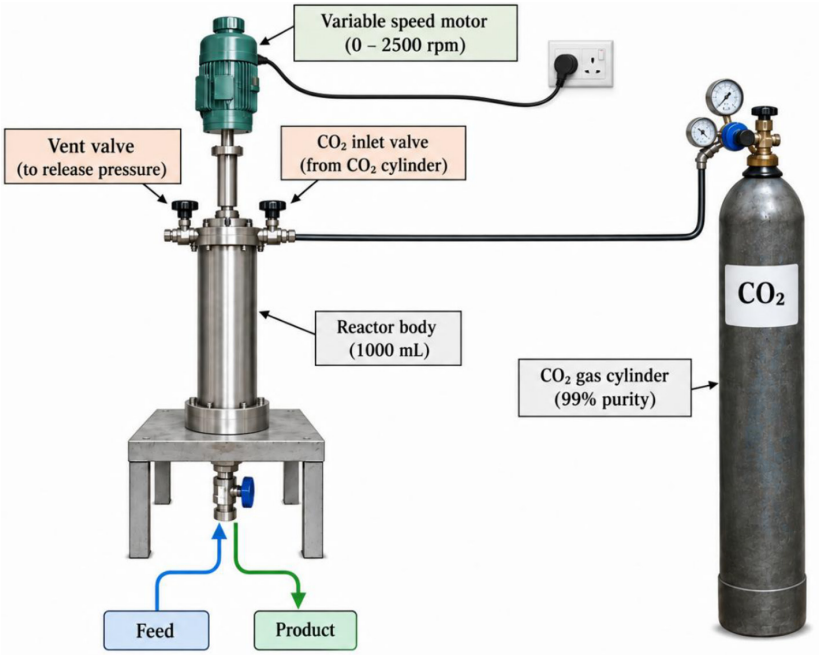


Figure 2. Experimental setup used for the CO_2 capture experiments, showing the CO_2 gas cylinder, pressure regulator, gas delivery tube, and mechanically stirred reactor

evaluation. The influence of critical operating variables on the CO_2 capture efficiency was systematically investigated. The examined parameters included CO_2 pressure (1–10 bar), contact time (15–60 min), and absorbent dosage (1–5 g/L). This parametric investigation aimed to elucidate the effect of each variable on CO_2 uptake and to establish the optimal operational conditions that maximize capture efficiency. After the initial screening tests, the best-performing absorbent was chosen for further study. The effects of main operating factors CO_2 pressure [1–10 bar], contact time [15–60 min], and dosage [1–5 g/L] on capture performance were evaluated.

RESULTS AND DISCUSSION

Characterization of adsorbents

pH analysis of amino-modified absorbents produced at 60, 70, and 80 °C

The pH of the aqueous suspensions containing the hyperbranched amino-cellulose absorbents was measured prior to CO_2 absorption. The initial pH was alkaline because of the basic amino functional groups present on the surface of the material. To reduce the initial alkalinity and adjust the pH to approximately 7 before CO_2 absorption, 0.01 M HCl solution was added dropwise to the suspensions containing the materials synthesized at 60, 70, and 80 °C, until a total volume of 1 mL was added to each suspension. Subsequently, CO_2 was introduced into each suspension at a pressure of 5 bar for 45 min, following the procedure described. After CO_2 absorption, the pH decreased to the acidic range because of the dissolution of CO_2 in water and the subsequent formation of carbonic acid (H_2CO_3). The initial and final pH values are presented in Table 2.

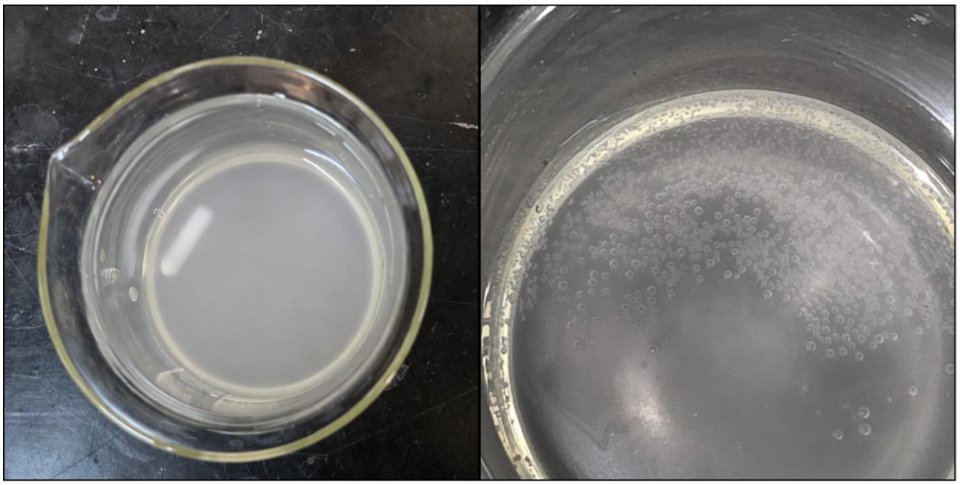
A distinct visual change was observed upon CO_2 injection. As shown in Figure 3, CO_2 bubbles were formed in the suspension, resulting in visible carbonation. While this indicates the introduction of CO_2 into the system, the absorption performance was quantitatively evaluated based on the pH measurements before and after absorption, as discussed in Table 1.

Fourier transform infrared spectroscopy (FTIR)

Figure 4 presents the FTIR spectra of the amino-functionalized cellulose samples synthesized at 60, 70, and 80 °C. Although the three samples exhibited generally similar characteristic peaks, noticeable differences were observed in the intensity and sharpness of several bands. In particular, the sample synthesized at 70 °C showed distinct N–H bending bands at 1573–1558 cm^{-1}. These bands were comparable to the N–H bending

Table 2. pH values of the aqueous suspension of amino-functionalized absorbents before and after CO_2 injection

Synthesis Temp. (°C)	pH before CO_2 injection	pH after CO_2 injection	ΔpH
60	6.8	5.7	1.1
70	6.7	4.7	2
80	7.2	6.8	0.4



(a) Before CO_2 injection (b) Immediately after CO_2 injection

Figure 3. Visual appearance of the amino-cellulose suspension: (a) before CO_2 injection and (b) immediately after CO_2 injection, showing the presence of CO_2 bubbles

band reported at approximately 1550 cm^{-1} for aminated cellulose nanocrystals by (D'Orsi et al., 2023). The same sample also exhibited C–N-related bands within 1369–1317 cm^{-1}, which were comparable to the C–N bands reported by (Jóźwiak et al., 2020) at 1360–1356 cm^{-1} for aminated cellulose fibers. This agreement supports the successful incorporation of amine-containing groups into the modified cellulose structure. Furthermore, the characteristic bands of cellulose remained present after modification, indicating that the main cellulose backbone was preserved. Overall, the FTIR results suggest that 70 °C provided the most favorable conditions among the investigated temperatures for TEPA grafting and crosslinking by epichlorohydrin.

The FTIR spectrum of the amino-functionalized cellulose prepared at 70 °C before CO_2 activation. A broad absorption band at 3427–3354 cm^{-1} was assigned to the stretching vibrations of O–H and N–H groups, indicating the presence of hydroxyl and amino functional groups (Bayramoglu et al., 2024). The absorption bands observed at 2968 and 2902 cm^{-1} were attributed to the stretching vibrations of aliphatic C–H groups, consistent with the C–H stretching region reported at 2920–2896 cm^{-1} (Romruen et al., 2022). The band at 1732 cm^{-1} was assigned to C=O stretching, in agreement with the previously reported range of 1732–1734 cm^{-1}, In addition, the band detected at 1635 cm^{-1} was attributed mainly to the bending vibration of adsorbed water, with possible overlap with N–H bending, and was comparable to the water-bending band reported at 1654 cm^{-1} (Sultana et al., 2024). Furthermore, the characteristic band observed at 1574 cm^{-1} was close to the N–H bending band reported at 1570 cm^{-1} for TEPA-functionalized materials. Together with the band at 1558 cm^{-1}, this observation supports the successful incorporation of TEPA and the introduction of amino groups into the modified cellulose structure (Zang et al., 2025). Furthermore, the bands observed at 1369–1317 cm^{-1} are attributed to C–N stretching vibrations, confirming the successful amination of cellulose. The strong absorption bands at 1163, 1111, 1059, and 1034 cm^{-1} correspond to C–O–C and C–O stretching vibrations of the cellulose structure, indicating that the cellulose backbone remained intact after chemical modification, which is consistent with previous studies (Bayramoglu et al., 2024). Overall, the FTIR results confirm the successful prepa-

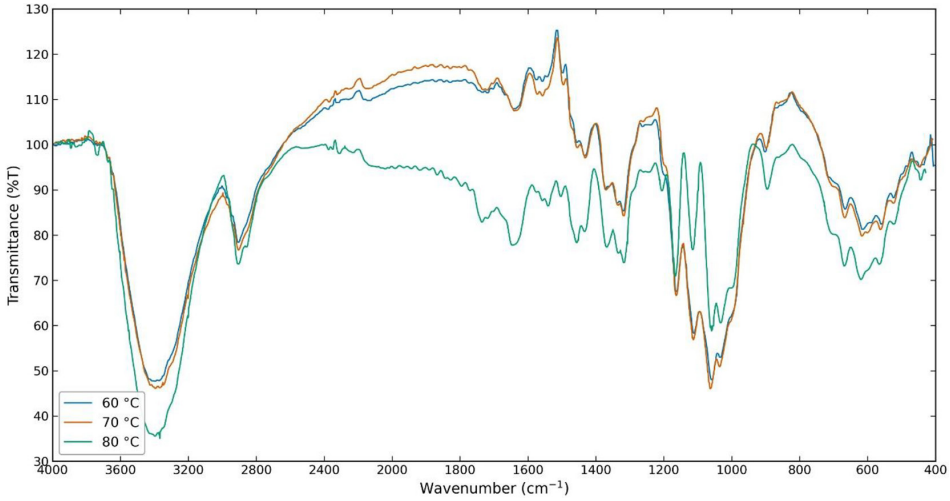


Figure 4. FTIR spectra of samples prepared at different temperatures

ration of amino-functionalized cellulose at 70 °C. Based on the pH and FTIR results, the absorbent prepared at 70 °C was selected for further XRD and FESEM analyses and subsequent operating parameter studies.

XRD analysis

Figure 5 shows the XRD plot for the amino functionalized cellulose absorbent prepared at 70 °C, it can be shown a sharp diffraction peak at about 22° (2θ) that corresponds to the crystal structure of cellulose. Other diffraction peaks with lower intensities appear at 16°, 35°, and 43°, demonstrating that the crystal structure of cellulose was maintained during the modification process. Clarify that the cellulose's fundamental crystalline structure was preserved (the peak at 22° remains), while noting a slight decrease in crystallinity due to chemical reaction and cross-linking, which increase the amorphous region.

FE-SEM analysis

FESEM images of amino functionalized cellulose absorbent synthesized at 70° C at various magnifications are depicted in Figure 6. From the figures, an irregular morphology of fiber and sheet with a rough and heterogeneous surface is observed. The functionalized cellulose possesses interconnected fibers, fractured flakes, and pores on its surface. At high magnifications, pores with nanometer-sized dimensions are identified, which means that a porous structure has been developed upon chemical modifications. These morphological aspects would give rise to more active sites for CO_2 absorption.

Influence of operating variables on CO_2 capture efficiency

Influence of CO_2 pressure

For the current investigation, influence of CO_2 pressure on the capture efficiency of the hyperbranched amino-cellulose suspension was determined keeping constant the values of the contact time (45 min) and temperature (25 °C). Thus, for this investigation, pressure was the only variable.

Where, ΔpH is the difference between the initial pH value of the hyperbranched amino-cellulose suspension prior to CO_2 addition and pH after CO_2 addition. Higher value of ΔpH means better acidification of the solution due to CO_2 dissolution in it. Figure 7 illustrates the influence of CO_2 pressure on the ΔpH. When the pressure of CO_2 was increased from 2 to 10 bar, the value of ΔpH was increased from 1.7 to 2.4. The obtained results indicate that an increase in the CO_2 pressure contributed to increasing the solubility of CO_2 in the aqueous suspension. CO_2, which has been dissolved, reacts with the water molecules to produce carbonic acid (H_2CO_3). The carbonic acid partly dissociates to HCO_3^- and H^+ ions, leading to the decrease in the pH. Also, the aminated groups in hyperbranched amino-cellulose contributed to CO_2 capture, increasing the solubility of CO_2 and, therefore, the quantity of carbonic acid. Within the examined range of pressures, the maximum value of ΔpH was observed for 10 bar. An increase in CO_2 pressure improved the efficiency of the absorbent in the process of CO_2 absorption. It corresponds to previously published data (Panahi et al., 2025). The increased pressure leads to an improved driving force for the mass transfer.

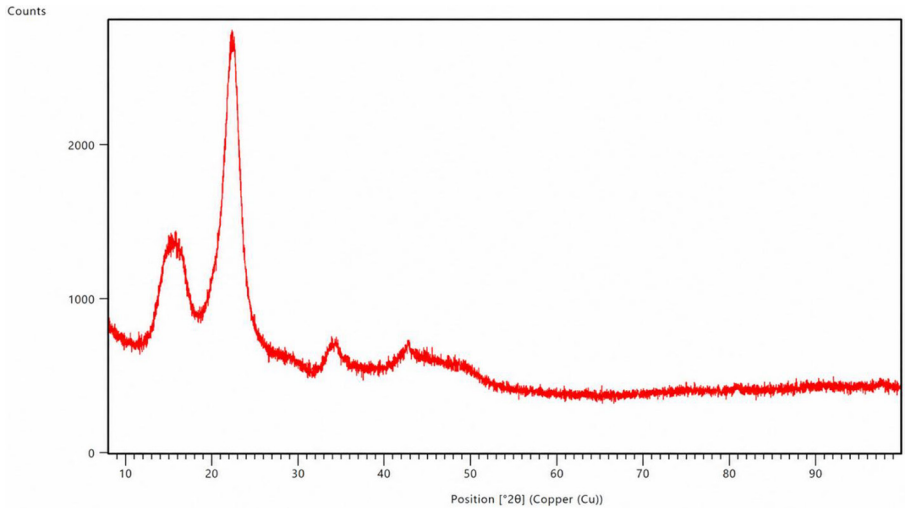


Figure 5. XRD pattern of the amino-functionalized cellulose absorbent prepared at 70 °C

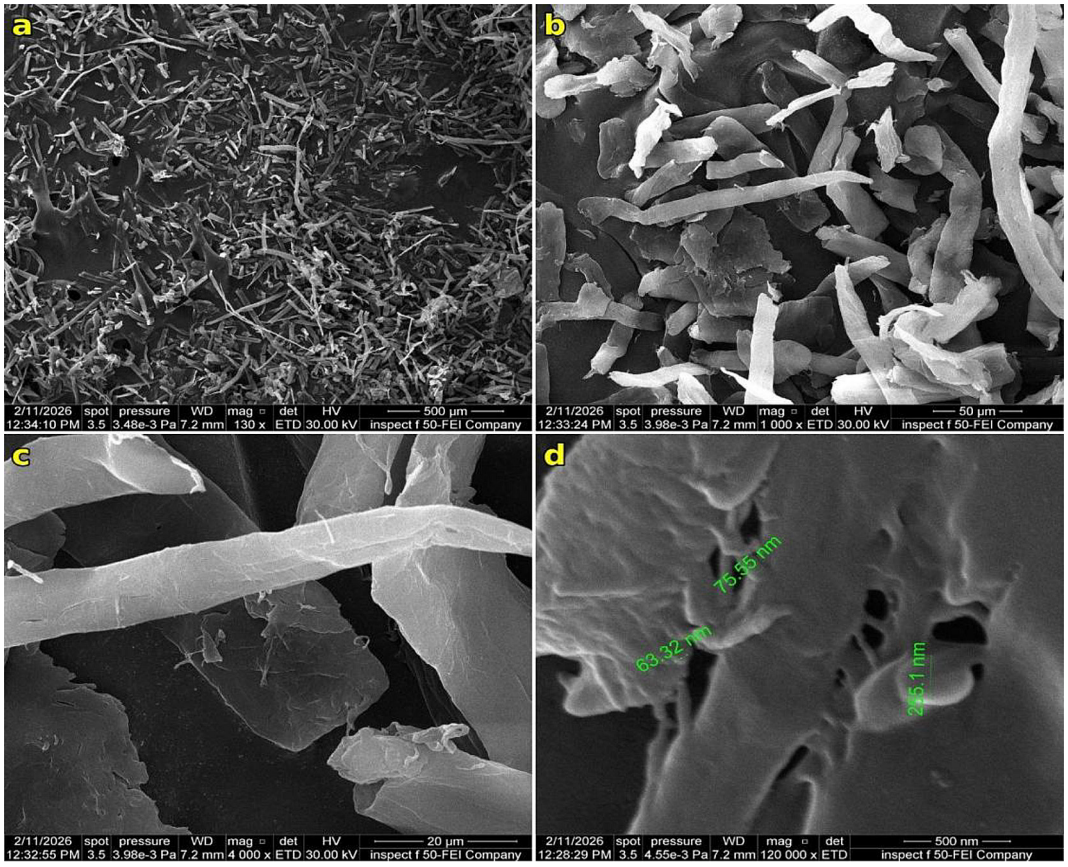


Figure 6. FESEM images of the amino-functionalized cellulose adsorbent prepared at 70 °C at different magnifications: (a) 130×, (b) 1000×, (c) 4000×, and (d) 120000×

Effect of contact time

The effect of contact time on CO_2 capture capability of hyperbranched amino-cellulose suspension was studied by keeping the CO_2 pressure at a constant value of 10 bar and temperature at 25 °C. In this case, the contact time was the single operating variable in the current experiment.

Figure 8 represents the effect of contact time on pH change (ΔpH) of hyperbranched amino-cellulose suspension. The increase of contact time from 15 min to 45 min led to an increase in ΔpH from 0.8 to 2.4, suggesting that increasing the contact time leads to CO_2 dissolution and formation of carbonic acid, thus increasing the pH decrease. On the other hand there was no further increase in

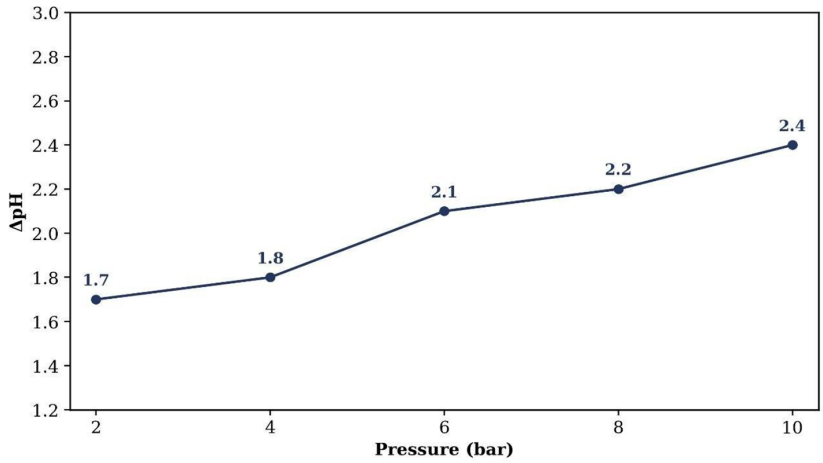


Figure 7. Effect of CO_2 pressure on the pH change (ΔpH) of the hyperbranched amino-cellulose suspension at a constant time of 45 min and (25 °C)

ΔpH at 60 min, which had a value of 2.4. This is an indication that the equilibrium state was achieved after 45 min and thus any further increase in contact time did not have a positive impact on CO_2 absorption. This is consistent with the absorption pattern that has been documented with polyethyleneimine functionalized cellulose material, where the CO_2 absorption rate within the first 45 min was faster compared to absorption that took place after 45 min, implying rapid absorption followed by a slower phase of absorption when all active sites were occupied (Vocht et al., 2022).

Effect of adsorbent concentration

The effect of absorbent concentration on the CO_2 capturing capacity of the hyperbranched amino-cellulose suspension was studied under constant CO_2 pressure of 10 bar, contact time of 45 minutes and room temperature (25 °C). Hence, absorbent concentration was the only changing operational parameter used in this experiment.

The effect of absorbent concentration on the pH change (ΔpH) is shown in Figure 9. It is seen that the ΔpH value increases from 1.33 to 2.75, as the absorbent concentration increases from [1 to 5 g/L]. The largest value of ΔpH is found between 1 g/L and 2 g/L of absorbent concentration, which indicates better CO_2 capturing due to an increase in the number of amino groups. However, raising the absorbent concentration from [2 to 5 g/L] led to only an incremental rise in ΔpH, which indicates that even though the use of a larger amount of hyperbranched amino-cellulose added some new amino groups and adsorption sites to the system, only a slight rise in CO_2 absorption was observed when using higher amounts of the

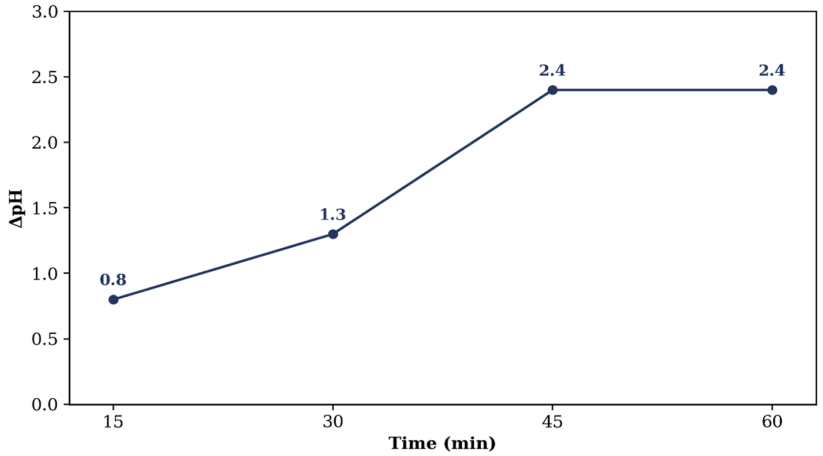


Figure 8. Effect of contact time on the pH change (ΔpH) of the hyperbranched amino-cellulose suspension at a constant CO_2 pressure of 10 bar and (25 °C)

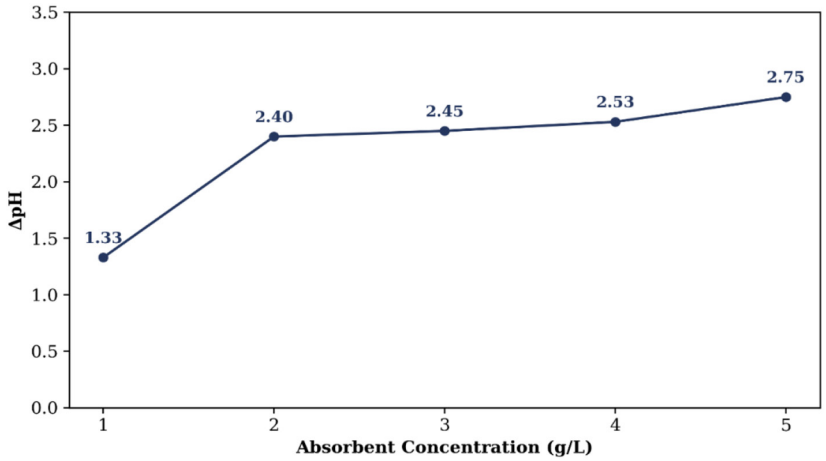


Figure 9. Effect of adsorbent concentration on the pH change (ΔpH) of the hyperbranched amino-cellulose suspension at a constant CO_2 pressure of 10 bar, contact time of 45 min, and 25 °C

adsorbent. Such behavior might suggest that the majority of the available CO_2 molecules reacted with all available adsorption sites under given experimental conditions (100 mL distilled water, 10 bar pressure, and 45 min reaction time). Hence, considering a practical point of view and taking into account economical considerations, an absorbent concentration of 2 g/L can be considered the most appropriate concentration to apply within these conditions since a higher absorbent concentration does not significantly contribute to CO_2 absorption but requires more absorbent material. In the case of amine functionalized cellulose absorbents, the same effect has been observed before: an increment in the amine loading improves CO_2 absorption due to the addition of new adsorption sites, however, an excess of amine loading decreases the efficiency of the extra sites due to diffusion restrictions and partial pore blockage (Paenmao et al., 2025).

CONCLUSIONS

A hyperbranched amino-cellulose composite material has been successfully synthesized via cellulose functionalization using TEPA and crosslinking with epichlorohydrin for use in CO_2 absorption applications. FTIR, XRD, and FESEM results have proven that successful amino group functionalization from TEPA has been achieved without losing the crystalline nature of cellulose as well as formation of a porous structure having many active adsorption sites. Out of all the prepared composites, the one prepared at 70°C showed the best overall performance. Enhanced CO_2 absorption properties of the composite have been attributed to the high presence of primary and secondary amino groups from TEPA in addition to the hyperbranched crosslinked structure from epichlorohydrin which enhanced the structural stability of the composite while maintaining the accessibility of the active adsorption sites. In addition, the dispersion of the functionalized solid into the aqueous medium ensured efficient interaction between CO_2 molecules and the absorbent.

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