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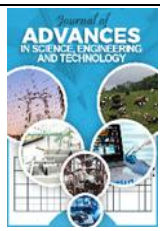

Investigation of the Amine-Functionalized Silica Material for Capture of CO_2 by Adsorption

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ABSTRACT

CO_2 emissions are of serious concern due to their negative effect on climate change. As emissions from various industrial processes continue to rise, the need to capture and utilize CO_2 becomes increasingly important. This paper examined the applicability of amine-functionalized silica material for the capture of CO_2. Commercially sourced silica gel was modified with an amino group and used as a solid support substrate. The substrate, silica, was suspended in dimethylformamide solution and reacted with a chlorinating reagent, phosphorous pentachloride, followed by substitution of the chloride atom with the amino group from ethylenediamine. The modified silica and CO_2-treated samples were analyzed using Fourier-transform infrared spectroscopy and thermogravimetric analysis. It was observed from the Thermal analysis that the silica framework was stable between 0˚C and 100˚C, and the mass loss observed near this temperature is due to the decomposition of the grafted ethylenediamine and not of the silica support. The modified silica was applied for the removal of a laboratory-prepared CO_2 gas. An excess of CO_2 was bubbled into a 1M calcium hydroxide solution, and turbidity measurements were recorded to determine removal efficiency. The regeneration of the amino-modified silica was confirmed using dilute hydrochloric acid, which stripped the attached CO_2. The contact time was evaluated, and an optimum duration of 35 min yielded the highest removal efficiency. Adsorption followed pseudo-first-order kinetics, indicating a chemisorption process. This study reported an adsorption capacity of 0.3558 g/g of CO_2, demonstrating that the modified material is effective for CO_2 capture.

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1. Introduction

The rapid increase in atmospheric CO_2 levels presents a major global challenge due to its harmful effect as a greenhouse gas on climate change (Nunes, 2023). CO_2 absorbs and traps infrared radiation in the atmosphere, causing global temperatures to rise. CO_2 is denser than air with a higher heat capacity of 0.846 kJ $kg^{-1}K^{-1}$ compared to air, which is 0.718 kJ $kg^{-1}K^{-1}$ (Evans et al., 2019).

According to the World Meteorological Organization report, the rate of the accumulation of CO_2 in the atmosphere has tripled since the 1960s, from an average of 0.8 ppm to 2.4 ppm per year. The global concentration between 2023 and 2024 and between 2023 and 2024 rose sharply by 3.5 ppm (Clark, 2025). This growth is due to human activities, widespread wildfires, and saturated natural carbon sinks. This calls for an urgent need to address carbon management practices and the advanced CO_2 capture technologies to curb further climatic deterioration.

Fossil fuels have been the major source of global energy supply for centuries. It has been asserted that fossil fuels account for a major share of global energy consumption and will continue to hold this position in the foreseeable future (Yousefzadeh et al., 2022). This dominance is largely due to their abundance, affordability, and high energy yield. Despite the global growth in renewable energy efforts to transition toward green energy, rich carbon emitter sectors such as cement, steel, and transportation still face technological and economic barriers to decarbonization (Voskian & Hatton, 2019). It has been pointed out that the emission of CO_2 from burning these fuels not only contributes to climate change, but also causes health problems, and environmental acidification, as CO_2 readily reacts with water to form carbonic acid.

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Therefore, in order to protect the health and lives of the present and future, there is a need for effective and affordable CO_2 capture and conversion technologies to mitigate these adverse impacts (Chen et al., 2022).

Adsorption is a surface phenomenon where atoms or molecules (adsorbate) adhere to the surface of a material (adsorbent) (Sakpal et al., 2012). This process occurs due to imbalanced attraction forces on the surface of an adsorbent, which creates surface energy. Adsorption could be physisorption or Chemisorption. Physisorption involves relatively weak intermolecular forces that hold particles, and chemisorption is achieved by substantial sharing of electrons between the surfaces of the adsorbent and adsorbate to create a covalent or ionic bond. Chemisorption dominates when using amine-functionalized silica to arrest CO_2; chemisorption primarily occurs. CO_2 molecules react with amine groups on the silica surface to form carbamate or bicarbonate species, depending on the presence of moisture.

Modification of solid support, such as silica, using amines, forms hybrid materials that retain amine reactivity while improving stability and reusability. Silica gel has been extensively used as a support material because of its large surface area, high thermal stability, and tunable surface chemistry. Amine-modified adsorbents enhance selectivity toward CO_2 capture while maintaining mechanical robustness (Watabe et al., 2013). This work critically discusses the preparation and modification of silica gel substrate material. It also examines the functionalization of the modified and treated samples. The study further examines the adsorption capacity, kinetics, and efficiency.

Fig. 1: The setup for CO_2 production.

2. Materials and Methods

All the apparatus were thoroughly cleaned first through immersion in an aqua regia bath to eliminate contaminants, then rinsed with deionized water. And they were oven-dried at 60 °C. The chemical reagents used in this study were of analytical grade and were purchased from Sigma Aldrich via their Nairobi distributor, Chemoquip, Kenya.

2.1. Sample preparation

The silica gel was used as received without further purification.

PCl_5 pre-treatment (Chlorination): The first step involves treating the silica gel with phosphorus pentachloride (PCl_5). This reaction replaces the surface hydroxyl groups (Si–OH) with chlorine atoms (Si–Cl), which are more reactive for subsequent functionalization (Zheng et al., 2006).

EDA functionalization (Amination): The resulting chlorinated silica gel is then reacted with ethylenediamine (EDA). The amine groups (–NH2) in EDA undergo a nucleophilic substitution reaction with the surface Si–Cl groups.

2.2. Chlorination and Amino group anchoring procedure

A 2.0 g mass of dry silica was suspended in 20 mL of dimethylformamide (DMF) in a three-neck flask equipped with a reflux condenser, magnetic stirrer, and thermometer. While stirring, 1.5g of phosphorus pentachloride (PCl_5) was slowly added to the resulting mixture, and the reaction was allowed to proceed for 2 hours at 80°C. Subsequently, the chlorinated samples were extensively washed with distilled water and dried in an oven at 60°C for 3 hours. With the ratio and condition above, a 10.0g chlorinate sample was prepared. A 5.0g sample of chlorinated silica gel was placed in a three-necked flask and reacted with 25.0 ml of ethylenediamine under reflux with mechanical stirring for 3 hours (Mwangi et al., 2012). The mixture was then filtered using a sintered glass crucible, and the solid residue was dried under a vacuum in a desiccator at room temperature for 24 hours (Ndung'u et al., 2021). The resulting modified silica gel was used for adsorption experiments.

2.3. Characterization

The chemical functional groups of the raw sample and modified sample were analyzed using a Fourier Transform Infrared Spectrometer (FTIR) using Tracer 100. spectrometer. The spectra were recorded in the range of 4000 to 400 cm^{-1}, at a resolution of 4 cm^{-1} with a scan speed of 20 cm^{-1}. The thermal stability of the samples was examined using Thermogravimetric Analysis (Shimadzu TGA-50). A sample of approximately 10 mg was heated to 600 °C under nitrogen flow (50 mL/min), with mass loss recorded during heating and automated cooling back to room temperature before subsequent runs.

2.4. Kinetic studies

CO_2 gas was prepared in the lab through the reaction of different masses of $CaCO_3$ (1 to 5g) with dilute hydrochloric acid. The resulting gas was allowed to interact with the modified silica sample in a flask, and the excess, unreacted gas was reacted with calcium hydroxide in the nephelometer. The turbidity of the samples was noted down at various time intervals and was graphically presented as shown in Fig. 5. Following the optimization of the adsorbate dose as shown in Fig. 5, this work resolved to use 3g of the gas as the adsorbate dose since nephelometric measurements are most accurate in the range of 0-40 NTU (O'Dell, 1996). The procedure was repeated, with the resulting turbidity measurements noted as presented in Fig. 6. Fig. 1 illustrates the setup, which shows carbon dioxide production, the interaction of the CO_2 with the adsorbent, and finally, the interaction of the resulting gas with calcium hydroxide in a nephelometer

Based on the findings of Hikita et al. (1977), who reported that CO_2 reacts with organic amines to form stable compounds, subsequent experiments were conducted separately, using a constant quantity of laboratory-prepared CO_2 gas adsorbed on varying masses of modified amine adsorbent. The excess (unabsorbed) CO_2 was bubbled into a 1.0 M calcium hydroxide solution. This produced a suspension whose turbidity was proportional to the quantity of the gas it interacted with. Table 4 shows the results obtained.

3. Results and Discussion

3.1. IR Characterization

The initial silica material and its modified forms were analyzed using the FTIR method, and the results of the parent and modified forms of the respective products are presented in Fig. 2.

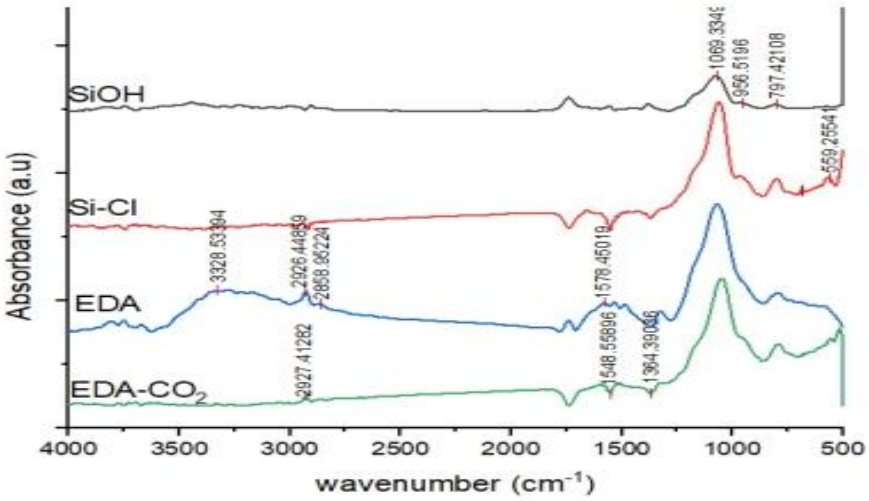

Fig. 2: The spectra of the parent silica, chlorinated silica, and aminated silica, respectively.

The results for the parent silica material show a weak signal at 956 cm^{-1}, which is attributed to the Si-O stretching vibration of Si-OH groups on the surface of the silica gel. The intensity of this band determines the concentration of silanol (Si-OH) groups in the material (Ellerbrock et al., 2022).

The weak peak at 797 cm^{-1} and the strong signal at 1069 cm^{-1} are associated with the stretching vibration of Si-O-Si bonds. Similar findings were reported by Darmakkolla et al. (2016) when they derivatized silanol functional groups with alkyl functional groups. Upon chlorination, a new peak emerged at 559 cm^{-1} associated with Si-Cl. When the resultant was aminated, the signal at 559 cm^{-1} disappeared, while broad peaks appeared at 3328 cm^{-1} and 1578 cm^{-1}, indicating successful amination of the silica (Turke et al., 2021), and the peak at 956 cm^{-1} decreased in intensity, indicating the substitution of the silanol (Si-OH) group with an amino group (Lang & Morrow, 1994).

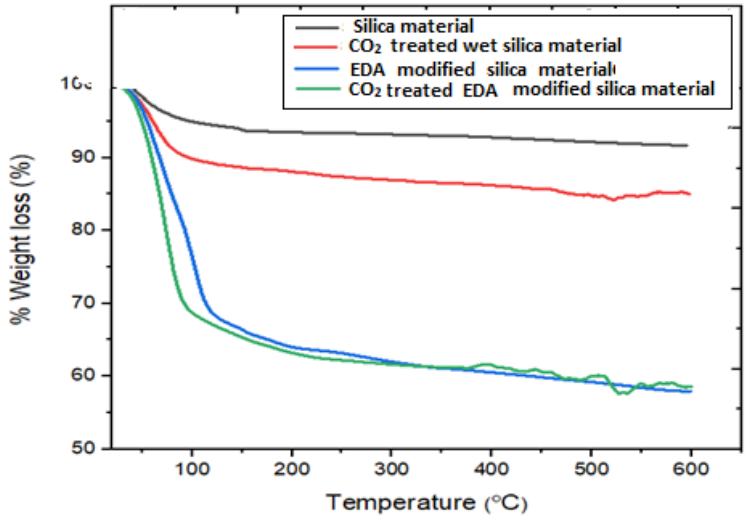

Fig. 3: TGA analytical curve for ADE-treated silica and CO_2-exposed sample.

The bands at wavenumbers 2926 cm^{-1} and 2858 cm^{-1}, characteristic of alkyl chains (C-H), were observed exclusively in the modified silica gel, with no corresponding peaks detected in the parent silica. This demonstrates the successful anchoring of amine onto the silica surface (Yoon et al., 2021). The results show new

peaks were observed at 1548 cm^{-1}, and 1364 cm^{-1}, corresponding to ammonium carbamate (NH^{+}_3 and $NCOO^{-}$) (Bacsik & Hedin, 2016).

3.2. TGA analysis of silica and its derivatives

This method was used to study the thermal stability of silica by monitoring mass changes as a function of temperature. In this study, analytical reagent (AR) grade silica was modified with ethylenediamine (EDA) to enhance its ability to adsorb CO_2 on its reactive sites. The success of the modification and subsequent CO_2 adsorption was evaluated based on the thermal stability of the resulting materials. This assessment was intended to determine whether surface modification affected the inherent stability of silica, as higher temperatures generally increase the rate of degradation of CO_2-treated EDA-modified silica material (Rao et al., 2014).

As shown in Fig. 3, all four silica derivatives studied remained stable at temperatures up to 50°C. As the temperature increases, degradation occurs at different rates. All other unmodified silica forms lose 5% of their mass when heated up to 100 °C, with no further mass loss even as the temperature continues to rise. The CO_2-treated silica loses 10% of its mass as the pyrolysis process releases gas by breaking the bond between CO_2 and the silica material. In the presence of water, silica is naturally hydrophilic, meaning its surface contains silanol (Si-OH) groups. CO_2 interacts with silanol groups on silica surfaces mainly through hydrogen bonding and catalysis, as referenced by Shi et al. (2025).

Upon exposure to heat, the ethylenediamine-modified material experiences over 70% weight loss at 100 °C, but no significant mass loss occurs as the temperature continues to rise. This is attributed to the pyrolysis of the modifying species.

For unmodified silica, TGA shows two main weight loss steps: a low-temperature phase related to physorbed water and a higher-temperature phase linked to the condensation of silanol groups. For functionalized or modified silica derivatives, TGA can measure the amount of grafted organic material by noting weight loss within specific temperature ranges, which corresponds to the organic component's decomposition. These thermal observations are consistent with the FTIR results, which further verify the presence of amine and carbonate functional groups on the modified silica surfaces.

3.3. Regeneration of the modified silica

The process of restoration of the ethylene-modified silica was investigated to establish if it was restored to its original form with its active site available for further re-use. This was done by stripping the attached CO_2 using 0.1 M hydrochloric acid. The acid-washed ethylene diamine modified silica material was then analyzed using the thermal graphic analysis method, and the results obtained are presented in Fig. 4.

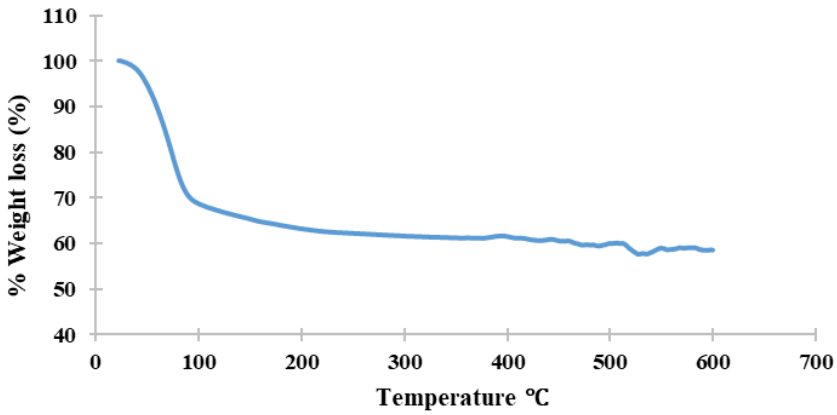

Fig. 4: TGA data for the hydrochloric acid-washed ethylene-modified silica material.

3.4. Effect of contact time on turbidity.

Contact time provides information on the reaction kinetics of the interaction between different CO_2 concentrations, which contributes to the turbidity of the calcium hydroxide solution. Fig. 5 shows the effect of contact time on turbidity at various CO_2 gas supplies. The graphs indicate consistency in the rise of turbidity for the first 35 min. This is attributed to the reaction of CO_2 with saturated calcium hydroxide, which produces a white precipitate of $CaCO_3$ (Eq. 1), which leads to the formation of a turbid suspension (Han et al., 2011). Hence, there is consistency in the rise of turbidity (O'Donoghue & Fitzsimmons, 2022).

$$Ca(OH)_{2\,(aq)} + CO_{2\,(g)} \rightarrow CaCO_{3\,(s)} + H_2O_{\,(l)} \quad (1)$$

Beyond 35 min, the limewater reaches saturation due to an excess supply of CO_2 gas interacting with the limewater, resulting in the formation of a clear solution as a result of the formation of calcium hydrogen carbonate (Kalinkin et al., 2005).

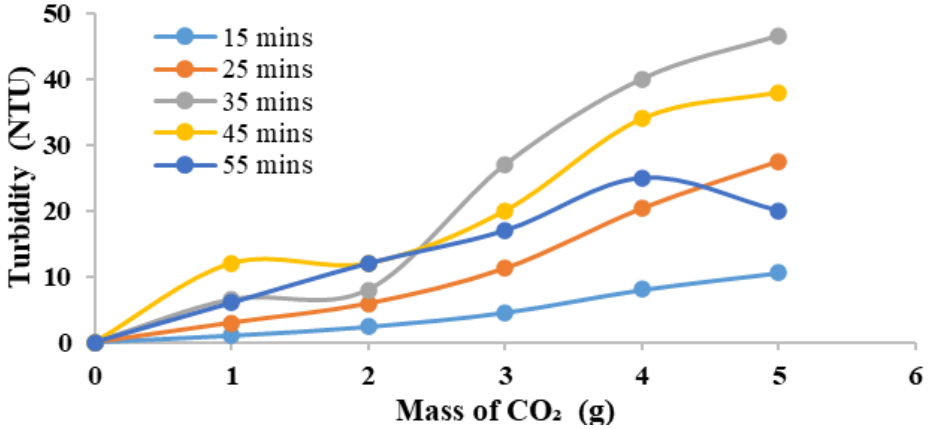

Fig. 5: Variation of turbidity (NTU) with different masses of CO_2 at various contact time, illustrating the adsorption behavior of CO_2 on amine-functionalized silica.

$$CaCO_{3\,(s)} + CO_{2\,(g)} + H_2O_{\,(l)} \rightarrow Ca(HCO_3)_{2\,(aq)} \quad (2)$$

The rate of formation and the amount of $CaCO_3$ produced largely depend on the quantity of CO_2 introduced. This indicates that increasing the amount of gas leads to a higher concentration of $CaCO_3$, as evidenced by a rapid and significant rise in turbidity. Once $CaCO_3$ forms, the reactions described in Eq. 2 are promptly initiated, resulting in a decrease in turbidity as these reactions progress.

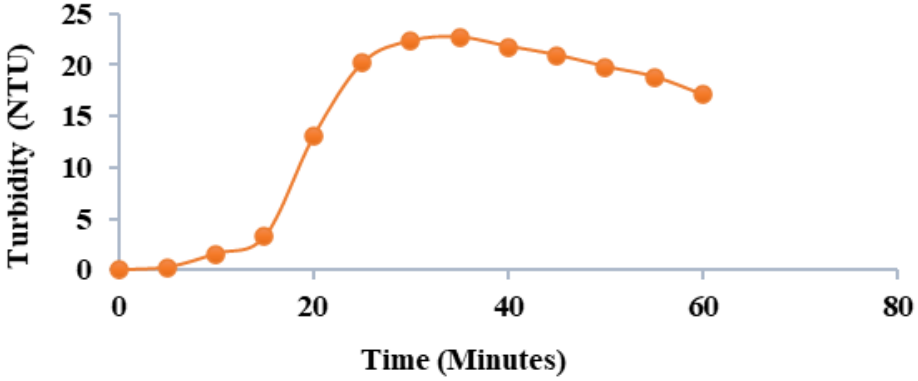

Fig. 6: Effect of contact time on turbidity for 3g of CO_2.

From Fig. 6, turbidity rises sharply from 15 to 25 min, which can be attributed to the significant supply of CO_2 produced during the experiment. However, as the experiment progressed, the reaction depicted by Eq. 2 slowed down the rate of turbidity increase until equilibrium was achieved. Once all the $Ca(OH)_2$ is consumed and no further $CaCO_3$ can form from reaction 1, the formation of $Ca(HCO_3)_2$ from reaction 2 leads to a gradual decline in turbidity. This reduction in turbidity within the cuvette caused the formation of Calcium bicarbonate, as depicted by reaction 2, which was also confirmed through visual observation during the experiment.

3.5. Calibration curve

To ensure the viability of analyzing our data graphs, it is logical to verify and determine the volume of gas corresponding to each turbidity reading. In this procedure, varying masses of the supplied sample interacted with $Ca(OH)_2$ in the presence of 1 g of the modified adsorbent. The resulting turbidity values were recorded and are presented in Fig. 7.

The mass of the supplied gas exhibits a strong linear correlation with the resulting turbidity ($r^2 = 0.9726$). This model enables straightforward prediction and determination of the amount of gas absorbed by $Ca(OH)_2$ for each turbidity measurement. The adsorption kinetic studies were carried out to determine the molecularity of the reaction.

3.6. Kinetic models

The first and second order reaction kinetics were established using Ho's pseudo first-order and second-order kinetic models, according to Lagergren (1898) and Freundlich (1907), respectively, as shown in Eqs. 3 and 4.

$$\ln[x]_t = \ln[x]_0 - k_1 t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Where $[x]_0$ and $[x]_t$ represent the initial and final concentrations of the adsorbing species, k_1 and k_2 are the rate constants for the pseudo-first- and pseudo-second-order models, respectively, q_t is the adsorption capacity at time t, and q_e is the adsorption capacity at equilibrium. The variation of concentration with time using the first and second order kinetics was calculated from Table 1, and a graphical representation of the results was generated as presented in Fig. 7(b) and Fig. 7(c), respectively.

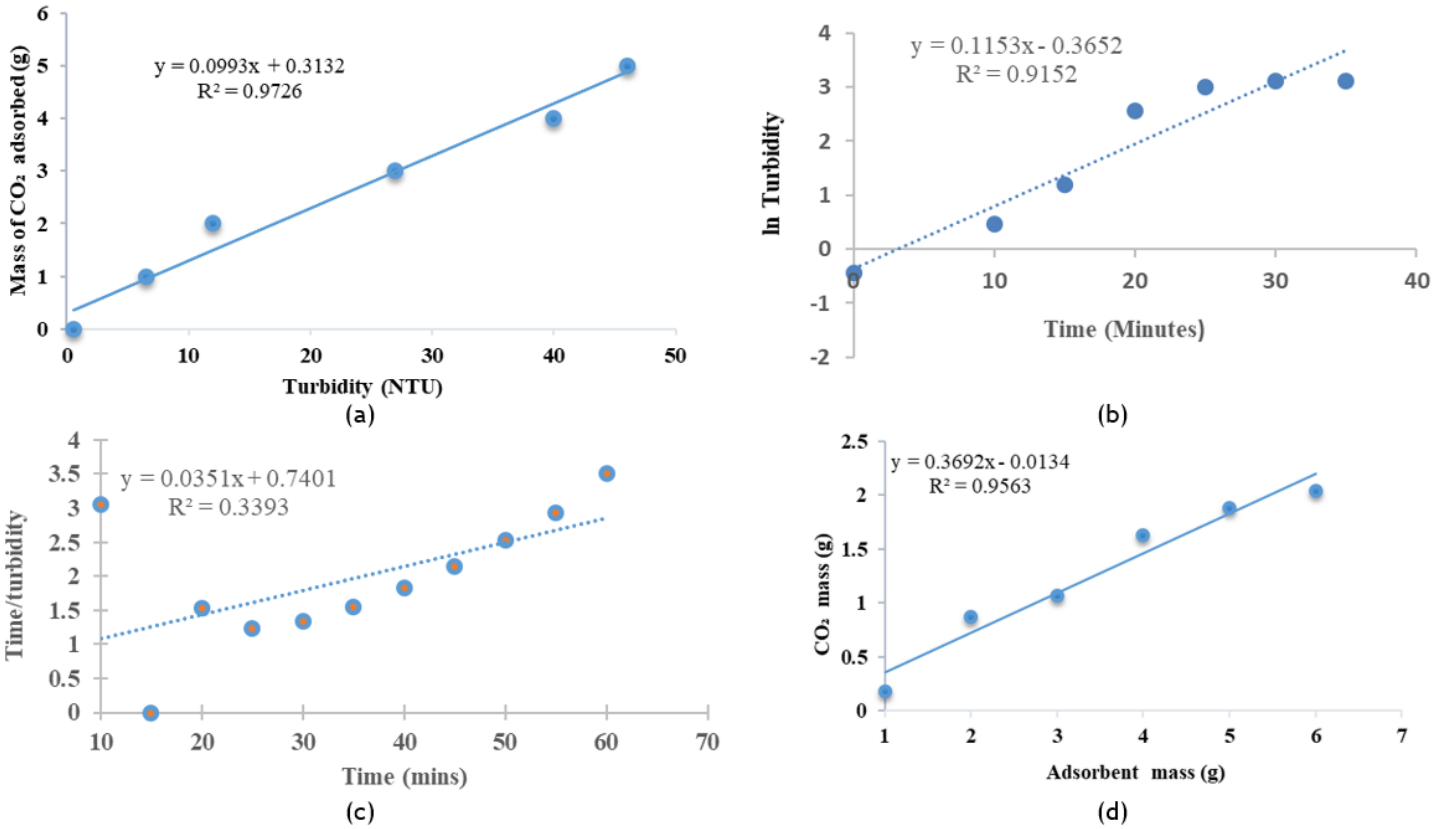

Fig. 7: (a) Calibration curve for turbidity corresponding to a given amount of CO_2, (b) The pseudo first order kinetic of the modified silica gel, (c) The pseudo-second-order kinetics of the modified silica gel, (d) The Mass of gas adsorbed versus mass of adsorbent.

The adsorption kinetics were analyzed using the pseudo-first-order (PFO) and pseudo-second-order (PSO) models to evaluate the mechanism of CO_2 uptake on the modified silica surface. The PFO model had an R^2 value of 0.9152, while the PSO model showed an R^2 value of 0.3393.

The stronger correlation of the experimental data with the PFO model indicates that CO_2 adsorption onto the EDA-modified silica is primarily driven by a chemisorption mechanism involving valence forces through electron sharing or exchange between CO_2 molecules and the amine functional groups on the silica surface. The lower R^2 value for the PFO model suggests that physisorption plays a minor role in the overall adsorption process.

3.7. Exploitation of the adsorbent material

The experimental measurements and the step-by-step calculations of gas consumption are highlighted as shown in Table 1. Accounting for the gas adsorbed by the reference, the specific mass of CO_2 adsorbed by the modified silica was calculated. This relationship is graphically presented in Fig. 7(d).

Table 1: Variation of adsorbent mass with turbidity at constant mass of gas.

Mass of adsorbent (g)	Turbidity (NTU)	Average Turbidity (NTU)	Δ Turbidity (NTU)	Gas consumed by $Ca(OH)_2$	Gas consumed by adsorbent.
0	0.57, 0.58, 0.577	0.57	0.00	-	-
1	28.14, 28.75, 26.46	27.78	27.21	3.0152	0.1777
2	22.44, 22.13, 20.77	21.34	20.77	2.3220	0.8709
3	19.39, 17.56, 19.45	18.23	18.23	2.1234	1.0695
4	12.96, 13.08, 13.53	13.18	12.61	1.5654	1.6275
5	10.01,11.98,9.75	10.58	10.01	1.3072	1.8857
6	8.58, 7.97, 8.67	9.01	8.44	1.1513	2.0416

Fig. 7(d) depicts the relationship between the mass of gas consumed and the mass of the adsorbent used. The reaction shows a strong linear correlation with a coefficient of determination (R^2 = 0.9563). This high value suggests that the adsorption process is highly predictable and follows a given trend across all tested mass and, implying the distribution of amine functional groups on the silica surface is uniform, allowing for a steady increase in gas uptake as more adsorbent is introduced. From the slope of the linear regression, the calculated adsorption capacity of the modified silica gel is 0.3558 grams of CO_2 per gram of adsorbent.

4. Conclusion

This study successfully achieved functionalization of silica using an amine, and it was confirmed by FTIR

analysis. This functionalization enabled the material to chemically react with CO_2 to form carbamate species. From the Thermal analysis results, the silica framework remained stable within the temperature range of 0°C to 100°C. The observed weight loss near this temperature was due to the thermal decomposition of the anchored ethylenediamine groups as opposed to the degradation of the silica support. According to the findings of this study, the modified material can effectively be used for CO_2 removal in various industrial sectors. The findings show that the modified material can be regenerated by washing the CO_2-treated sample using 0.1 M hydrochloric acid solution. The study revealed that the modified silica chemically interacts with CO_2, as indicated by adsorption data fitting a pseudo-first-order kinetic model, which suggests a chemisorption process. An adsorption of 0.3558 g of gas took only 35 min, demonstrating its potential for various applications. Using the turbidometric method, this study confirmed that it is a reliable indicator for monitoring adsorption progress. This indicates that the modified adsorbent has potential applications in mitigating the adverse effects of CO_2 on global warming. Such cartridges could be placed at multiple points where gas adsorption is needed. In conclusion, our study shows that the highly reactive modified silica material can serve as an effective sorbent for removing greenhouse gases in the remediation of contaminated environments.

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