

Effect of Reaction Parameters on CO₂ Absorption from Biogas Using CaO Sorbent Prepared from Waste Chicken Eggshell

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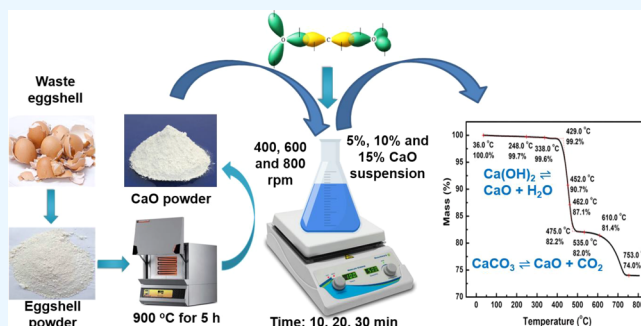
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ABSTRACT: This study provides an efficient and straightforward approach to eliminate carbon dioxide (CO₂) by absorption using a calcium oxide (CaO) sorbent derived from chicken eggshells. The sorbent concentration, stirring speed, and contact time were varied. The optimal condition for CO₂ removal was a 10% calcium hydroxide (Ca(OH)₂) suspension at 600 rpm with 20 min interaction. This optimum condition conferred the ever-highest absorption (98.71%) of CO₂ through Ca(OH)₂ suspensions from eggshell-derived CaO. X-ray diffraction was used to identify crystallographic phases and optimum conditions revealed calcium carbonate (CaCO₃) formation with the highest intensity, Fourier transform infrared spectroscopy revealed peaks for the carbonate (CO₃²⁻) group, field emission scanning electron microscopy was used to investigate the morphological and structural properties of the sorbent before and after CO₂ absorption, and thermogravimetric analysis was performed to understand the reaction mechanism. According to the kinetic analysis, the sorbent can be fully decomposed with a minimum activation energy (*E_a*) of 89.09 kJ/mol.



1. INTRODUCTION

In recent years, global warming caused by the release of greenhouse gases, most notably carbon dioxide (CO₂), has become a major global concern. The massive volume of CO₂ emissions adds more than 60% to global warming.^{1–3} Moreover, as the global population continues to expand, it is anticipated that energy demand will skyrocket.⁴ Burning fossil fuels to meet this demand has released large amounts of CO₂ into the atmosphere, escalating global warming. Continual efforts are being made to control the magnitude of forthcoming climatic changes caused by rising greenhouse gas emissions, but there are no fruitful results.^{5,6} In addition, the Copenhagen Agreement stipulates that by 2100, the global temperature increase should not exceed 2 °C above preindustrial levels. The International Energy Agency (IEA) has emphasized the need to meet the 2 °C target. If left unchecked, rising temperatures will inevitably cause sea levels to rise, thereby increasing the likelihood of storms and floods. Therefore, measures must be taken to remove CO₂ and protect against global warming.

Today, scientists worldwide consider carbon capture, storage, and utilization (CCSU) an attractive method for reducing CO₂ emissions.^{7–9} It involves the capture, compression, transport, geological storage, and use of CO₂ in various ways. Therefore, achievable and cost-effective carbon

capture methods are urgently required. Pre- and postcombustion modes offer a variety of technical choices including adsorption, absorption, membrane separation, chemical looping combustion with and without oxygen decoupling, and cryogenic separations.^{10–12} The literature has shown that water scrubbing uses a large amount of water, making it unworkable in places with a lack of water, and the membrane cost is higher with the membrane separation method, which operates at a higher working pressure (25–40 bar). The operating conditions of cryogenic separation are –100 °C and 40 bar, which are exceptionally challenging to meet.¹³ Among contemporary carbon capture technologies, absorption is the most technologically developed and has been widely used on a commercial scale for years.^{14,15}

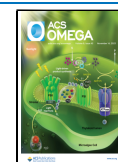
Absorption is the process of bringing a substance into contact with a solvent for separation, either physically or chemically. Physical absorption is the process of absorbing gas into a liquid without a chemical reaction.^{3,16} In contrast, a

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chemical reaction occurs when gas is absorbed into a solvent via chemical absorption.^{17,18} An efficient absorption requires a large surface area for mass transfer in the absorbent.^{19,20} A sorbent is also considered economically desirable if it has low energy requirements, is easily regenerated, has no adverse environmental effects, and is cost-effective.²¹

In recent years, numerous researchers have investigated the use of solid waste as sorbents.^{22,23} Interest in clean technology has also attracted the use of solid waste (eggshells) as a focus of research.^{24–26} Eggshells are a virtuous source of calcium oxide (CaO), which has a very good ability to capture CO₂.^{27,28} The eggshells contain approximately 90% calcium carbonate (CaCO₃) that is converted to CaO by calcination. Furthermore, eggshells are a biodegradable organic waste material that is safe for the environment, of low cost, and can be easily regenerated. Yolanda et al. showed that calcium hydroxide (Ca(OH)₂) is suitable for a flexible CO₂ capture system for backup power plants.²⁹ This author also reported that Ca(OH)₂ is sufficient for CO₂ capturing from the ambient at room temperature.³⁰ Han et al. revealed that Ca(OH)₂ aqueous solution was appropriate for CO₂ capture from a highly concentrated CO₂ gas mixture.³¹ Considering all of these factors, the absorption process and eggshell-derived CaO are the best for CO₂ reduction.

In this study, a chemical absorption approach using CaO derived from chicken eggshells was used as a sorbent to remove CO₂. The sorbent concentration, absorption time, and stirring speed were measured at room temperature. To the best of our knowledge, this is the first approach to use CaO derived from eggshells to absorb the CO₂ from Biogas.

2. EXPERIMENTAL SECTION

2.1. Materials. For the experiments, a CaO sorbent was prepared in the laboratory from discarded chicken eggshells collected from a local restaurant in Dhanmondi, Dhaka, Bangladesh. Biogas supplied from the biogas plant at the Institute of Fuel Research and Development, Bangladesh Council for Scientific and Industrial Research, Dhaka, Bangladesh was used as the source of CO₂. Deionized (DI) water was used to prepare all of the solutions.

2.2. Methods. The experiments were conducted at the laboratory scale. The setup is divided into three sections: sorbent preparation, absorption, and analysis.

2.2.1. Calcium Oxide (CaO) Sorbent Preparation. Waste chicken eggshells were first washed with normal tap water and then with DI water to remove any dirt and impurities. The eggshell was dried in an electric oven (Memmert, UN 55) at 105 °C for 8 h. Then, the eggshell was ground into a fine powder using a blender and passed through a sieve. The sieved powder was then placed in a muffle furnace (Carbolite Gero, 30–3000 °C) and heated to 900 °C at a rate of 20 °C min⁻¹ before being calcined for 5 h.³²

2.2.2. Carbon Dioxide Absorption. The experiment was performed batchwise with respect to the absorption phase. Figure 1 represents the schematic diagram of the experimental setup. A 200 mL aliquot of simulated biogas (source of CO₂) was pumped into the gas balloon, and the analysis was performed using a portable gas analyzer (Geotech Biogas 5000), Geotechnical Instruments Ltd. (UK). It was then introduced into the absorption flask for further observation. 200 mL of the biogas was used for each cycle of the experiment. The raw gas collection balloon and CO₂ absorption flask were connected by a 5 mm diameter and a

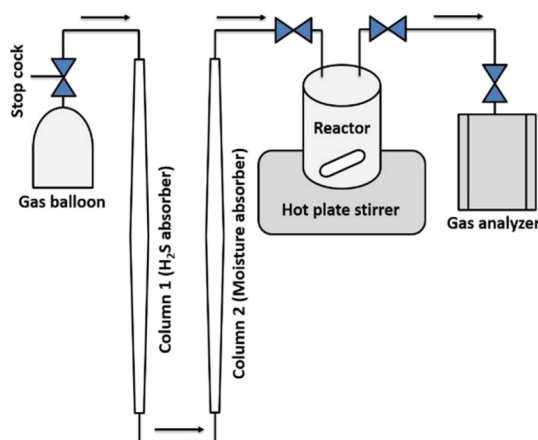
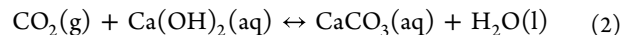


Figure 1. Schematic diagram of the experimental setup for CO₂ capture.

300 mm long hose pipe. To achieve the desired results, the chemical absorption method employed an aqueous chemical solution or suspension in which CaO was converted into Ca(OH)₂ using DI water.³³ The chemical reaction between CaO, water, and CO₂ is exothermic and increases moisture content.³⁴ When CaO was mixed with water, it produced a Ca(OH)₂ suspension according to the following reaction:



When exposed to a CO₂ source, the Ca(OH)₂ suspension reacts with CO₂, resulting in the formation of CaCO₃, as shown in the following reaction:



To inspect the effects of each parameter (contact time, concentration, and stirring speed), 27 experiments were carried out. The temperature was set at 24 °C (room temperature). The contact times were set to 10, 20, and 30 min. The stirring speeds were chosen to be 400, 600, and 800 rpm for the different experiments. Every experiment was performed in triplicate.

2.3. Sorbent Characterization. At the end of the experimental run of the absorption process, the sorbent from each run was filtered. The sorbent sample was then oven-dried at 105 °C for 8 h and stored in a glass vial. Each sample was characterized by using various techniques. Crystallographic information was obtained using powder X-ray diffraction (XRD; Bruker D8 Advance, Germany) (Cu K α radiation, λ = 0.15406 nm, 40 kV and 60 mA, scanning range of 10°–60° at the rate of 10°/min) to determine crystalline phases. A Fourier transform infrared spectroscopy (FTIR; Frontier, PerkinElmer, UK) study was performed to characterize various surface functionalities in the samples with wavenumbers ranging from 400 to 4000 cm⁻¹. The surface morphology of CaO was observed using field emission scanning electron microscopy (FESEM; JSM-7610F, JEOL, Japan) with an accelerating voltage of 15 kV, and elemental composition was observed by energy-dispersive X-ray analysis (EDX). Thermogravimetric analysis (TGA; TG/DTA6300, Japan) was used to determine the thermal stability of the sample and the reaction kinetics for the formation of CaCO₃ using the Arrhenius equation by monitoring the weight change that occurred as the sample was heated at a constant rate.

3. RESULTS AND DISCUSSION

3.1. Effect of Contact Time and Concentration of Sorbent on CO₂ Removal. The interaction time between the sorbent and CO₂ was found to play an important role in the removal of CO₂. From Figure 2, it can be seen that CO₂

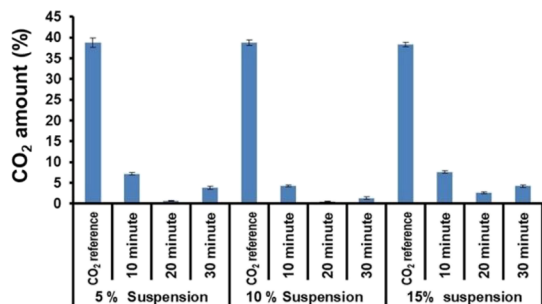


Figure 2. Effect of contact time (10, 20, and 30 min) and sorbent concentration (5, 10, and 15%) on the removal of CO₂ from biogas using CaO sorbent at 600 rotational speed including raw biogas CO₂ data.

reference (CO₂ in raw biogas) contained 38.61 ± 0.24% CO₂, and after 20 min absorption at 600 rpm, the remaining CO₂ was 0.73 ± 0.06, 0.53 ± 0.06, and 2.60 ± 0.17% for 5, 10, and 15% sorbent concentrations, respectively. Analysis data show that the concentration of CO₂ was drastically reduced with the 5, 10, and 15% sorbent suspensions by increasing the contact time with constant stirring. The maximum absorption percentage was 98.71%.

However, the analyzer data in Figure 2 show that increasing the absorbent concentration from 5 to 10% improves the removal efficiency by increasing the driving force of the reaction. Here, raw biogas contained 38.61 ± 0.24% CO₂, and after using 10% suspension at different contact times, the remaining CO₂ was 4.20 ± 0.20, 0.53 ± 0.06, and 1.30 ± 0.26%, respectively. At a 10% concentration, the absorption of CO₂ reached its highest point of 98.71%. However, after further increasing the absorbent concentration (15%), the absorption capacity decreased. This may be because of the presence of excess absorbent particles, which tend to flocculate and hinder the absorption process. When the excess solute particle stands in the way of gas molecules, the tortuosity of the diffusion path is increased and reduces the self-diffusion coefficient, resulting in the reduction of the absorption of the gas phase.^{35,36}

3.2. Effect of the Stirring Speed on CO₂ Removal. The influence of the stirring rate on the removal of CO₂ was investigated (Figure 3). It can be seen that the rate of CO₂ absorption appeared to be quite high, up to 600 rpm, and then decreased to 800 rpm. In the case of 600 rpm with 5, 10, and 15% sorbent concentration, the remaining CO₂ was 0.33 ± 0.06, 0.10 ± 0.01, and 0.17 ± 0.06%, respectively whereas the raw biogas contained 38.61 ± 0.24% CO₂. The experiment demonstrated that the stirring rate had a significant effect on the CO₂ removal. The rate of gas absorption in a gas–liquid absorption reaction is determined by the reactor dimensions, geometry, number of impellers, and stirring speed.^{37,38} The diffusional domain, which requires a large interfacial area, has a direct effect on the absorption rate. Swirling in a stirred reactor facilitates gas diffusion into the liquid phase. To prevent disruption of the planar surface, the agitation speeds were kept relatively low. It was discovered that increasing the stirring

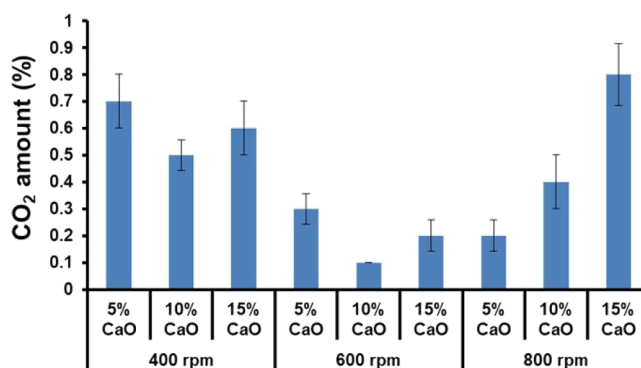


Figure 3. Effect of the rotational speed (400, 600, and 800 rpm) on CO₂ removal from biogas using different concentrations (5, 10, and 15%) of CaO sorbent at 20 min contact time.

speed boosted the CO₂ absorption to a certain level before decreasing it. This was due to a decrease in the thickness of the liquid layer, which was responsible for the resistance to the mass transfer of CO₂ gas molecules.³⁶

3.3. Characterization of the Sorbent. **3.3.1. XRD Analysis.** The XRD patterns of the calcined eggshell, raw chicken eggshell powder, and treated samples are presented in Figure 4, and there is a noticeable contrast between them, with

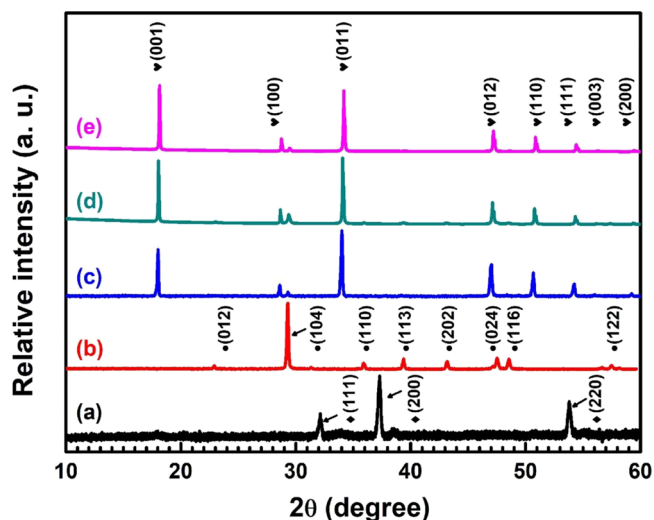


Figure 4. XRD pattern of the calcined eggshell (a), raw chicken eggshell (b), and CaO sorbent with 10% suspension and 20 min absorption time at three different rotational speeds: 400 (c), 600 (d), and 800 rpm (e).

sharp peaks at 2θ values of 32.15°, 37.29°, and 53.82° indexed to the (111), (200), and (220) planes, respectively, for cubic CaO (JCPDS card number: 37-1497) (indicated with a black diamond shape in Figure 4). Raw eggshell powder exhibited sharp peaks at 2θ values of 22.50°, 29.33°, 35.96°, 39.42°, 43.40°, 47.15°, 49.17°, and 57.40° which could be indexed to the (012), (104), (110), (113), (202), (024), (116), and (122) planes of rhombohedral CaCO₃ (indicated with a black dot shape in Figure 4) (JCPDS card number 72-1652).³⁹ The treated samples (c), (d), and (e) showed peaks at 2θ values of 18.03°, 28.62°, 34.02°, 47.02°, 50.67°, 54.21°, 56.26°, and 59.50°, which can be indexed to hexagonal Ca(OH)₂ planes (001), (100), (011), (012), (110), (111), (003), and (200) (JCPDS card number: 84-1264), respectively (indicated with a

black heart shape in Figure 4). After observing all these peaks, it is clear that although every treated sample provided absorption evidence, the (d) sample showed a sharp peak at 29.33° for the (104) plane of CaCO_3 and the best absorption of CO_2 .

3.3.2. FTIR Spectroscopy. The FTIR spectra of CaO (a), raw chicken eggshell powder (b), and treated samples 400 rpm (c), 600 rpm (d), and 800 rpm (e) were obtained by preparing pellets using KBr at room temperature, and the spectra were recorded at $400\text{--}4000\text{ cm}^{-1}$. The FTIR spectra, considering the optimum contact time, concentration variation, and rotation speed, are presented in Figure 5. The broad bands

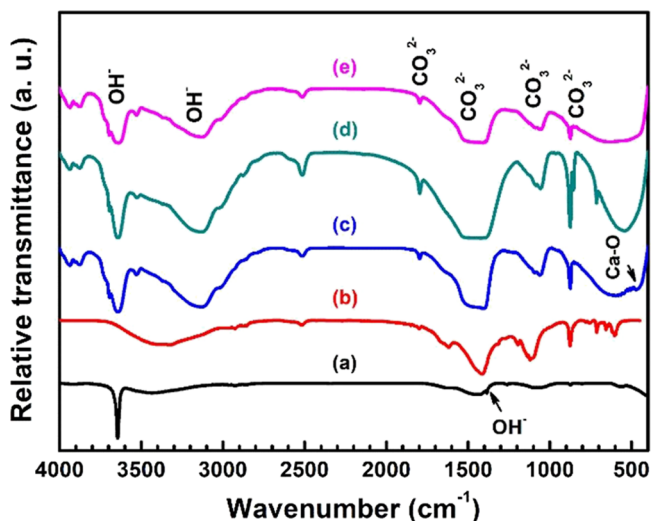


Figure 5. FTIR spectrum of CaO (a), raw chicken eggshell (b), and CaO sorbent with 10% suspension and 20 min absorption time at three different rotational speeds: 400 (c), 600 (d), and 800 rpm (e).

at 3652 and 3148 cm^{-1} can be attributed to the O–H stretching of $\text{Ca}(\text{OH})_2$. The tiny peak at 2518 cm^{-1} may be due to the CO_2 stretching vibration. CaCO_3 , on the other hand, showed peaks at 1794 and 1460 cm^{-1} which were attributed to the CO_3^{2-} group, indicating CaO carbonation. The spectra showed bands at 1073 and 862 cm^{-1} , matching the mono- and bidentate carbonate vibration modes, respectively. The strong bands at 524 and 416 cm^{-1} can be identified as the vibrations of the Ca–O.^{40,41} The intensity of the CO_3^{2-} peak increased at a 600 rpm rotational speed and then decreased at 800 rpm.

3.3.3. FESEM Analysis. The surface morphologies of the calcined eggshell and treated sorbents were investigated by FESEM and are presented in Figure 6. The surface of the calcined eggshell (Figure 6a) had a nonporous and irregular grain structure with an average grain size of $600\text{--}900\text{ nm}$. However, the sorbents after treatment with the optimum concentration and contact time (10% concentration and 20 min contact time) exhibited a variety of grain sizes and shapes at different stirring rates, covering almost the entire surface of the prepared sorbents. The sorbents at 400 rpm (Figure 6b) also showed almost the same grain size with a few small particles. This may have been due to the formation of CaCO_3 . At a stirring rate of 600 rpm, the FESEM image (Figure 6c) shows the agglomeration of small particles with an $80\text{--}120\text{ nm}$ average grain size. This may have been due to the maximum conversion of CaO to CaCO_3 . A further increase in the stirring speed (800 rpm) decreased the absorption of CO_2 onto the

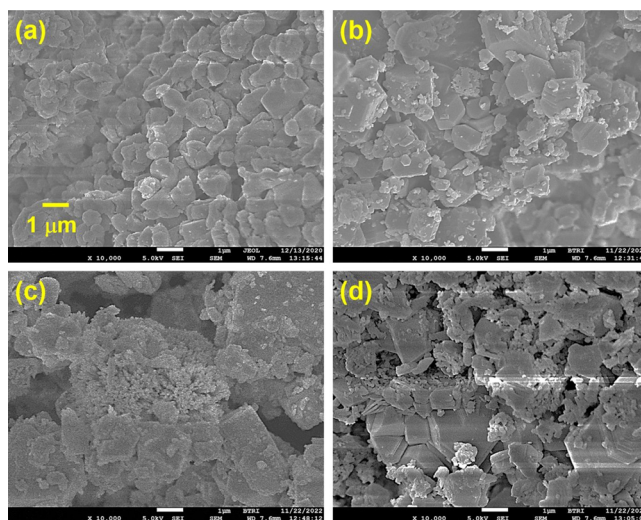


Figure 6. FESEM image for CaO (a) and CaO sorbent with 10% suspension and 20 min absorption time at three different rotational speeds: 400 (b), 600 (c), and 800 rpm (d).

CaO sorbents. The increased stirring speed may influence the accumulation of CO_2 on the surface of the absorbent.

3.3.4. EDX Analysis. The EDX analysis revealed the elemental composition of the treated sorbents (Figure 7).

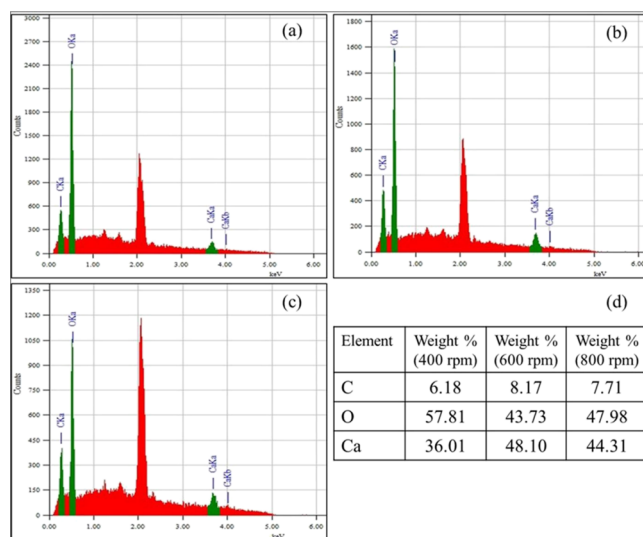


Figure 7. EDX analysis of CaO sorbent with 10% suspension and 20 min absorption time at three different rotational speeds: 400 (a), 600 (b), and 800 rpm (c) and atomic weight percent of elements of these samples (d).

The weight percentage of carbon (C), oxygen (O), and calcium (Ca) was investigated, which was varied for each sample. The maximum C weight percentage was observed at the 600 rpm stirring speed (Figure 7d), indicating the maximum formation of CaCO_3 . Which indicated the maximum absorption capacity of CaO at that condition. Therefore, stirring speed is an important regulator that maximizes the absorption of CO_2 .

3.3.5. TG Analysis. TGA is an analytical method that measures the mass loss of a sample over time as the temperature varies. It can be used to analyze the thermal durability and composition of a material. The TGA curve of

the sorbent after the absorption of CO₂ with a molecular weight of 100 amu (atomic mass units) is shown in Figures 8

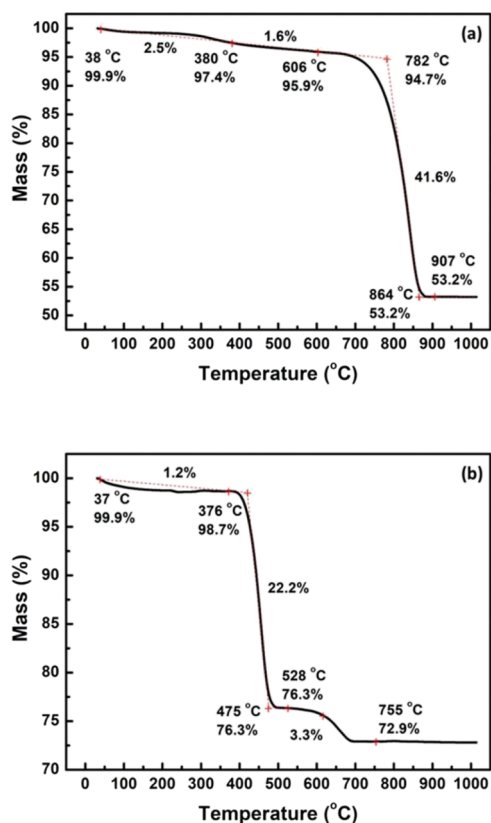


Figure 8. TGA of CaO (a) and raw eggshell powder (b).

and 9. Figure 8a represents the raw eggshell TGA curve. A 2.5% mass loss was observed from 32 to 380 °C temperature, which is due to the moisture removal from the waste eggshell powder. Moreover, 1.6% mass loss was also observed from 380 to 606 °C temperature, which may be due to the hydroxide removal from the eggshell, and then up to 864 °C breakdown of CaCO₃ occurred, and it gives exactly 41.6% mass loss. On the other hand, Figure 8b illustrates the raw CaO TGA peak. A 1.2% weight loss was found from 37 to 376 °C temperature, which may be due to the removal of moisture content from the ambient environment. However, 22.2% mass loss was observed from 376 to 475 °C temperature, which caused the breakdown of hydroxide in the sample. Moreover, a 3.3% mass loss was found for the breakdown of CaCO₃ from 475 to 755 °C.

The sorbent underwent thermal breakdown at temperatures above 400 °C, resulting in the formation of CaO and CO₂ (Figure 9).^{42,43} Once the entire sample underwent the breakdown reaction, the solid-phase CaO remained on the weighing balance, providing a final reading of approximately 73 amu (i.e., once the temperature reached 800 °C). In this study, the TGA of CaCO₃ samples after absorption reactions under optimal absorbent concentration and reaction time (10% concentration and 20 min time) with stirring rates of 400, 600, and 800 rpm were evaluated. In all cases, the sorbent started to decompose at approximately 430 °C and was converted to CaO and CO₂ at temperatures above 700 °C. In sample (a), there was an initial mass loss of water molecules at 250–390 °C. Ca(OH)₂ decomposed to CaO and CO₂ at temperatures ranging from 429.1 to 475.5 °C. CaCO₃ then decomposes until

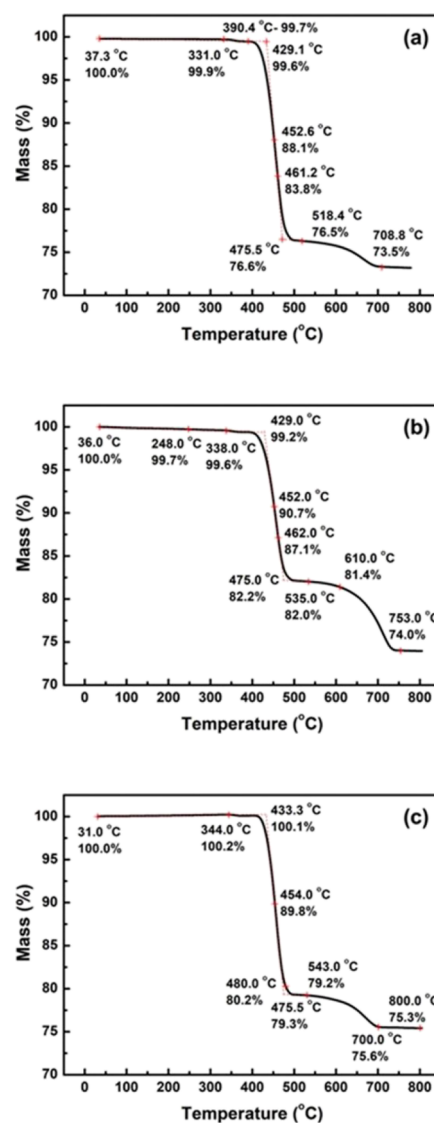


Figure 9. TGA of CaO sorbent after absorption with 10% suspension and 20 min absorption time at three different rotational speeds: 400 (a), 600 (b), and 800 rpm (c).

the temperature reaches 708.8 °C.⁴⁴ The sorbent obtained from the reaction at a stirring rate of 600 rpm started to decompose at 429 °C. At 429–753 °C, decomposing Ca(OH)₂ resulted in 17.0% weight loss and 8.2% weight loss for CaCO₃. On the other hand, sample (c) initially absorbs moisture and gains some weight and then again starts to lose weight at 433.3 °C. Ca(OH)₂ decomposes to CaO at 433.3–475.5 °C; however, CaCO₃ does not decompose completely until the temperature reaches 800 °C. The decomposition rates in accordance with the temperature change are shown in Table 1. TGA data revealed that the maximum amount of CaCO₃ was decomposed from the sample in the reaction at 600 rpm, indicating the formation of maximum CaCO₃ which means maximum CO₂ absorption.

3.3.6. Reaction Kinetics for the Formation of CaCO₃. To establish kinetic parameters, the study considers a first-order reaction, which includes the two-stage decomposition rate of the materials. The following kinetic equation (eq 3) was examined for heat deterioration, which is a simple differentiation of the first-order reaction. The carbonation rates of

Table 1. Percentage Mass Loss for Thermal Decomposition of Calcium Carbonate Obtained from Three Different Stirring Rates with 10% Suspension and 20 min Absorption Time

	400 rpm		600 rpm		800 rpm	
temperature range (°C)	429.1–475.5	475.1–708.8	429–475	475–753	433.3–475.5	475.5–800
weight loss (%)	23.0	3.1	17.0	8.2	20.8	4
total weight loss (%)	26.1		25.2		24.8	

Ca(OH)₂ were higher than those of CaO, particularly at temperatures above 400 °C. In addition, as described by eq 3, it is believed that the reaction proceeds through the formation of an interface of water molecules or OH⁻ ions at the solid surface and is controlled by an inherent chemical reaction that occurs only on the surface that is not covered by CaCO₃.^{45,46}

$$\frac{dX}{dt} = k_1[1 - (2 - n)k_2X]^{1/(2-n)} \quad (3)$$

where k_1 and k_2 represent proportionality constants and n represents the reaction order. Assuming that the carbonation reaction is of zero order for the CO₂ partial pressure,^{46,47} the temperature dependence of k_1 was calculated by applying the Arrhenius law described in eq 4. Figure 10 shows the Arrhenius diagram for k_1 .

$$k = k_0 \exp\left(-\frac{E_a}{RT}\right) \quad (4)$$

where k represents rate constants, E_a is the activation energy, R is the molar gas constant, and T is the temperature in Kelvin.

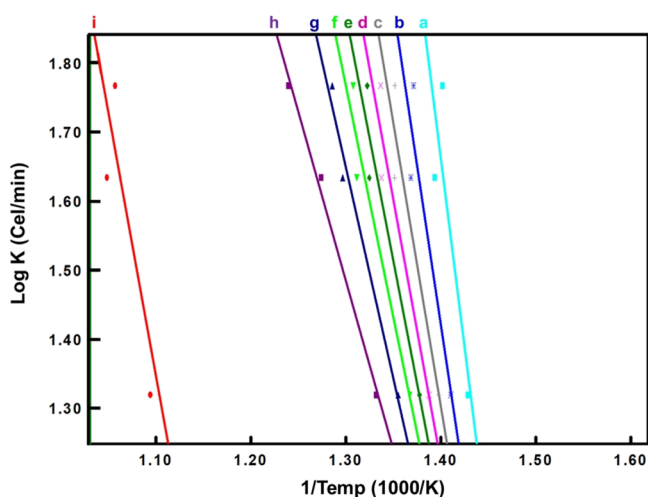


Figure 10. Arrhenius plot, Log k (Cel/min) against $1/\text{Temp}$ (1000/K), to calculate the activation energy (E_a) for the carbonation of Ca(OH)₂ at different regression factors: 10 (a), 20 (b), 30 (c), 40 (d), 50 (e), 60 (f), 70 (g), 80 (h), and 90% (i).

The apparent activation energy and frequency factors obtained by linear regression are given in Table 2.

The data indicate that deterioration at a 10% regression factor (R. F.) occurred at an activation energy of 199.07 kJ/mol, resulting in a weight loss of 2.34%. With an activation energy of 153.48 kJ/mol at the maximum regression factor, 21.49% of the weight was lost. At an R. F. of 80%, the lowest activation energy of 89.09 kJ/mol was required to degrade 19.09% of the sample.

Table 2. Calculated Activation Energy (E_a) Using Arrhenius Plot at Different Regression Factors (R. F.)

no.	R. F. (%)	E_a (kJ/mol)	TG (%)
1	10	199.07	97.67
2	20	167.71	95.27
3	30	149.63	92.88
4	40	138.15	90.49
5	50	129.69	88.09
6	60	122.15	85.70
7	70	111.44	83.30
8	80	89.10	80.91
9	90	153.49	78.52

4. CONCLUSIONS

Eggshell-derived CaO has been successfully used as an environmentally friendly alternative and an effective CO₂ sorbent. Additional characterization was conducted to determine the morphological suitability of the material for the CO₂ absorption applications. The XRD diffractogram revealed rhombohedral-phase CaCO₃ for raw chicken eggshell powder and cubic-phase CaO for calcined chicken eggshell powder. On the other hand, the treated samples showed a combination of hexagonal-phased Ca(OH)₂ and rhombohedral-phased CaCO₃, indicating the carbonation of the sorbents. The FTIR spectrum revealed the O–H group and the CO₃²⁻ group. The intensity of the CO₃²⁻ peak increased at 600 rpm and then decreased at 800 rpm. A nonporous and irregular grain structure with an average grain size of 600–900 nm was observed for the calcined eggshell using FESEM. In the case of a stirring rate of 600 rpm, the FESEM image showed the agglomeration of small particles with 80–120 nm. Finally, TGA data revealed that the maximum amount of CaCO₃ was decomposed from the sample in the reaction at 600 rpm. The experimental results illustrated that the sorbent concentration, reaction time, and stirring speed played significant roles in the CO₂ removal efficiency. The highest performance of the CaO sorbent as an absorber is achieved when the concentration of the solvent used is 10% with a stirring speed of 600 rpm and a contact time of 20 min. The kinetic study confirmed that a minimum of 89.09 kJ/mol was required to decompose 19.09% of the absorbent sample. As a result, the developed technology may be a good approach for managing the massive amount of eggshells as kitchen waste as well as a good absorbent for global CO₂ mitigation.

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Notes

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