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CO₂ capture performance of graphene oxide synthesized under ultrasound irradiation

Ultrason ışınlaması altında sentezlenen grafen oksitin CO2 yakalama performansı

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CO₂ Capture Performance of Graphene Oxide Synthesized Under Ultrasound Irradiation

Highlights

- Craphene oxide (GrO) was synthesized under ultrasound irradiation.
- The CO_2 capture capacity of GrO decreased with the increase in temperature.
- The CO_2 capture capacity of GrO was reached up to 1.04 mmol.g⁻¹ at 25°C.
- ***** The Avrami (Ar) model better described the CO_2 capture process.

Graphical Abstract

The CO_2 uptake performance of the sample significantly reduced as the temperature rises, showing the exothermic character of capture. The analysis performed at 25 °C presented a much better capture capacity compared to other temperatures.



Figure. The CO₂ adsorption curves at different temperatures

Aim

The aim of this study is to determine the CO₂ capture performance of synthesized GrO under ultrasonic irradiation.

Design & Methodology

The CO₂ capture studies were applied at 25 °C, 75 °C, and 100 °C. Pf, Ps, and Ar kinetics models were used to describe the kinetic mechanism of CO₂ capture on GrO.

Originality

The originality of this study is the investigation of the CO_2 capture capacity of GrO synthesized by ultrasonic irradiation.

Findings

The CO_2 uptake performance of the sample significantly reduced as the temperature rises, showing the exothermic character of capture.

Conclusion

GrO exhibited the best CO_2 adsorption capacity at $25^{\circ}C$ (1.04 mmol g⁻¹).

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

CO₂ Capture Performance of Graphene Oxide Synthesized Under Ultrasound Irradiation

Araştırma Makalesi / Research Article

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ABSTRACT

Nowadays, CO_2 capture is a vital technology to notably reduce the uncontrolled released CO_2 emissions. CO_2 capture using graphene oxide, a derivative of graphene, has become of tremendous interest due to its unique morphology. In this present work, graphene oxide (GrO) was synthesized under ultrasound irradiation according to the modified Hummers' method and its CO_2 capture performance was examined. The X-ray powder diffraction (XRD) and Fourier transform infrared (FTIR) analyses were applied to explore the structure of the sample. CO_2 capture performance of GrO was examined by performing TG analysis under different temperatures. The CO_2 adsorption capacity of GrO was reached up to 1.04 mmol g-1 at 25°C. The experimental data getting from the kinetic study revealed that the Avrami model better described the CO_2 adsorption.

Keywords: Graphene oxide, ultrasound, CO2 capture, kinetics.

Ultrasonik Işıma Altında Sentezlenen Grafen Oksitin CO₂ Yakalama Performansı

ÖZ

Günümüzde CO₂ yakalama, kontrolsüz salınan CO₂ emisyonlarını önemli ölçüde azaltmak için önemli bir teknoloji olarak görülmektedir. CO₂ yakalamada grafenin bir türevi olan grafen oksitin kullanılması, grafen oksitin benzersiz morfolojisi nedeniyle büyük ilgi görmektedir. Bu çalışmada, modifiye Hummers yöntemine göre ultrason ışıması altında grafen oksit (GrO) sentezlenmiş ve CO₂ yakalama performansı incelenmiştir. Numunenin yapısal özelliklerinin belirlenmesi için X-ışını toz kırınımı (XRD) ve Fourier dönüşümü kızılötesi (FTIR) analizleri uygulanmıştır. GrO'nun CO₂ tutma performansı farklı sıcaklıklar altında TG analiziyle incelenmiştir. GrO'nun CO₂ adsorpsiyon kapasitesi 25°C'de 1,04 mmol g⁻¹'e ulaştı. Kinetik çalışmadan elde edilen deneysel veriler, Avrami modelinin CO₂ adsorpsiyonunu daha iyi tanımladığını ortaya koydu.

Anahtar kelimeler: Grafen oksit, ultrasonik, CO2 yakalama, kinetic.

1. INTRODUCTION

 CO_2 is one of the major atmospheric greenhouse gases resulting from the increase in energy demand and several industrial activities. The dramatic increase in CO₂ emissions has become a widespread concern [1, 2]. The excessive release of CO₂ into the earth's atmosphere is identified as one of the primary causes of climate change and global warming [3, 4]. Regarding this information, to improve the living conditions for the coming years and to take decisions about the reduction of CO₂ emissions, the most emitting CO₂ sources should be determined and required measures should be taken regarding these sources [5, 6]. CO₂ capture, storage, and using technology is a promising option to attain notable reductions in anthropogenic CO₂ emissions. Considering the entire operation, CO₂ capture is seen as the most critical step, as it accounts for a high part of the total cost [7–9]. For this reason, it is expected that the preferred technology for CO_2 capture should be economical [10]. Among the CO₂ capture techniques, adsorption has

garnered interest in recent studies because of its advantages, including low operating cost, environmental friendliness with minimal energy consumption, effectiveness, excellent selectivity, and a large variety of adsorbents [11, 12]. For this purpose, considerable classes of solid materials have been evaluated as adsorbents such as metal-organic frameworks (MOFs) [11, 13], silica-based materials [14–18], calcium-based materials [5, 19, 20], zeolite [21], and carbon-based materials. Among carbon class materials, activated carbon [22, 23], CMK [24], carbon nanotubes [25] and graphene oxide [26, 27] are extensively used as solid adsorbents.

GrO, a functionalized oxidized derivative of graphene, is a two-dimensional hydrophilic material consisting of a sp²-bonded regular lattice structure. Recently, it has emerged as a possible candidate for CO₂ capture because of its significant and multifunctional morphology such as high porosity, high surface area, and thermal stability. Moreover, GrO surface possesses advantageous epoxy, carboxyl, and hydroxyl groups. These oxygen-contained functional groups make the GrO basic and it reaches a high CO₂ capture capacity by interacting with the acidic

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 CO_2 [28–30]. Apart from CO_2 capture, GrO has attention to various fields such as adsorption [31, 32], fuel cells [33], gas storage [34], electrode material for energy devices [35], and supercapacitor [36].

To the best of the authors' knowledge, any study on the evaluation of pure GrO synthesized under ultrasonic irradiation in CO_2 adsorption is not available in the literature. In this study, GrO was synthesized under ultrasound irradiation to determine the CO_2 capture capacity at various temperatures. The pseudo-first order, pseudo-second order, and Avrami kinetic models were utilized to test the fit of experimental results of CO_2 capture by GrO on the kinetics equations.

2. MATERIAL and METHOD

Graphite powder with a purity of 99.9995% was bought from the Alfa Aesar. Other chemicals used in the experimental studies were bought from Sigma Aldrich.

XRD analyses were applied in a PANalytical X'Pert-Pro XRD instrument using CuK α radiation ($\lambda = 0.15406$ nm), operating at 40 mA and 45 kV. FTIR spectrum of the sample was taken on Perkin Elmer Spectrum One FT-IR spectrometer. The CO₂ capture analyses of the sample were carried out on a Perkin Elmer Pyris Diamond thermogravimetric analyzer (TGA).

2.1 Preparation of GrO

GrO powders were prepared based on the Hummers' method explained in the previous reports, applying modifications. Firstly, H₂SO₄ was added to defined amounts of graphite and NaNO3 mixture and all materials were stirred for 1 h. Then, KMnO₄ was added slowly to the stirred mixture in the ice bath. During this process, the temperature was kept below 5°C. After the mixture was removed from the ice bath, it was taken to an ultrasonic bath for a while, and a determined quantity of deionized water was added to the reaction mixture under ultrasound. Then, H₂O₂ was introduced to the solution, and ultrasound-assisted mixing was continued until a color change was observed. The reaction was stopped when the mixture's color changed from black to brown. The resulting mixture was carefully filtered and rinsed with deionized water until it had a neutral pH. The resultant powder was dried in an oven for 24 hr and named as GrO.

2.2 CO₂ Capture

The CO₂ uptake behavior of the GrO was investigated by the TGA using high-purity CO₂ gas (> 99.99%) under atmospheric pressure. The CO₂ capture studies were applied at 25 °C, 75 °C, and 100 °C. Firstly, 10 mg GrO added in an aluminum crucible was heated in N₂ gas for 60 min with a 100 mL/min flow rate until the temperature reached 105 °C to dehumidify, and then, it was cooled to the specific temperature. In the next step, the gas flow was changed to ensure a constant flow of CO₂ gas and the GrO sample was kept under this constant flow for 1.5 h. The CO₂ capture capacity was determined based on the change in mass of the adsorbent later the initiation of the flow of CO_2 gas.

3. RESULTS and DISCUSSION 3.1 Characterization

Figure 1 indicates the XRD pattern of GrO synthesized under ultrasonic irradiation. The XRD pattern of GrO revealed a wide peak at $2\theta = 11^{\circ}$, representing the (001) crystal face of GrO. This single characteristic peak proves the formation of graphite to graphene oxide [37, 38].



Figure 1. XRD pattern of GrO

The FTIR spectrum of the GrO was presented in Figure 2. The typical wide peak at 3412 cm^{-1} was related to O-H stretching vibration in carboxyl groups due to H₂O molecule absorption. The peak appearing at 1728 cm⁻¹ resulted from the C=O stretching of carboxyl and/or carbonyl groups. The unchanged C=C bond in graphite structure was signified at 1626 cm⁻¹. The bands at 1054 cm⁻¹ and 1226 cm⁻¹ indicated the C-O stretching vibration of alkoxy and epoxy groups, respectively. The band at 1404 cm⁻¹ represented the deformation of the O-H bond [29, 37, 39].



Figure 2. FTIR spectrum of the synthesized GrO under ultrasonic irradiation

3.2 CO₂ Capture

Figure 3 displays the CO₂ adsorption capacity curves of GrO at 25°C, 75°C, and 100°C. The CO₂ uptake performance of the sample significantly reduced as the temperature rises, showing the exothermic character of capture. The analysis performed at 25 °C presented a much better capture capacity compared to other temperatures. The increase in contact time enhanced the CO₂ adsorption performance of the adsorbent. The GrO

indicated the best capacity as 1.04 mmol.g⁻¹ in 90 min at 25 °C.

To determine the CO_2 uptake capacity of GrO, the performance of other GrO-based samples conducted included in previous works in the literature is listed in Table 1. According to the reported studies, it can be stated that GrO synthesized under ultrasonic irradiation is a convenient option for CO_2 capture.



Figure 3. The CO₂ adsorption curves at different temperatures

Table 1. CO2 uptake capacities of some GrO and GrO-based materials reported in the literature

Sample	T (°C)	P (bar)	Capacity (mmol g ⁻¹)	Ref.
Mg-Al-NO ₃ LDH-NS/GrO	200	1	0.47	[24]
GrO/MWCNT(1:1)50-LDH	300	1	0.49	[25]
Amine-functionalized GrO	65	0.1	1.2	[26]
GrO – LDO-SA	200	1	0.83	[38]
Amine-functionalized GrO	25	1	0.91	[39]
GrO/ZIF-8@ZIF-67	25	1	1.12	[40]
APTES-GrO	70	1	1.5	[41]
Chitosan grafted GrO aerogel	25	1	0.26	[42]
GrO	30	1	1.1	[43]
GrO	25	1	1.04	In this study

3.3 Kinetic Analysis

To describe the kinetic mechanism of GrO, the obtained kinetic data at 25 °C were fitted to pseudo-first order (Pf), pseudo-second-order (Ps), and Avrami (Ar) kinetics models. The Pf model identifies the proportionality between the adsorption rate and the number of vacant sites present on the adsorbent surface [40]. The Ps model assumes a linear ratio between the square of the number of unoccupied adsorption sites and the rate [41]. The Ar factionary kinetic model was proposed to explain the CO₂ gas uptake rate of some solid adsorbents [44]. The Pf, Ps, and Ar kinetic models were represented by the following equations, respectively;

$$q_t = q_e \left[1 - \exp(-k_f t) \right] \tag{1}$$

$$q_t = \frac{q_\ell^2 k_s t}{1 + q_k k_s t} \tag{2}$$

$$q_t = q_e \left[1 - \exp(-(k_a t)^{n_a}) \right]$$
(3)

where q_e and q_t are the adsorption capacity at equilibrium and at a given time (mmol g⁻¹). k_f , k_s , and k_a are the rate constants of Pf, Ps, and Ar kinetic models, respectively, and n_a is the order of the equation.

To determine the accuracy of the applied kinetic models, an error function $(\Delta q\%)$ was calculated. This error function was defined as;

$$\Delta q(\%) = \sqrt{\frac{\sum \left[\frac{q_{e(exp)} - q_{e(pre)}}{q_{e(exp)}}\right]^2}{n-1}} \times 100\%$$
(4)

where the values of the experimental and predicted CO₂ adsorption capacities, respectively, are represented by $q_{e(exp)}$ and $q_{e(pre)}$. The number of the kinetic data points was demonstrated as *n*.

The kinetic plots and calculated parameters along with these three models were given in Figure 4 and Table 2.



Figure 4. Kinetics plots for the CO₂ capture on GrO a) Pf, b) Ps, and c) Ar

Kinetic Model	Parameter	Value
	$q_{e,exp} (mmol.g^{-1})$	1.1037
	k _f	0.0474
Pf	R ²	0.9695
	$\Delta q(\%)$	0.5964
Ps	$q_{e,cal} (mmol.g^{-1})$	1.293
	ks	0.036
	R ²	0.999
	Δq(%)	6.09
	$q_{e,cal} (mmol.g^{-1})$	1.057
Ar	ka	0.046
	n _a	0.9682
	R ²	0.9917
	Δq(%)	0.596

Table 2. The calculated kinetic parameters for CO₂ adsorption on GrO at 25 (°C)

Among all of the studied kinetics, Ar factionary model presented a much lower $\Delta q(\%)$ value. As a result, the q_e value obtained with the Ar model has a value closer to the experimental q_e than the values obtained with the Pf and Ps models. The Ar kinetic model identified well the CO₂ adsorption mechanism on GrO, and this demonstrates the appearance of a complex mechanism or multiple reactions [45].

To estimate the activation energy of the CO_2 adsorption on GrO, the Arrhenius equation was used as given in Eq. (4). The kinetic coefficients at various temperatures (25 °C, 75 °C, 100 °C) may be evaluated by this equation.

$$k = Ae^{\frac{-L_a}{RT}} \tag{5}$$

The linear form of the Arrhenius equation was given by following equation;

$$\ln k = \ln A - \frac{E_a}{RT} \tag{6}$$

where A refers to Arrhenius pre-exponential factor, E_a is a term related with activation energy and R is ideal gas constant.

The activation energy (E_a) of CO₂ adsorption was obtained from the plot of ln *k* versus inverse temperature (1/T) (Fig 5). The correlation coefficient R² of the plot was found as 0.99 (more than 0.95) and this proves the linearity between ln *k* and 1/T. According to Figure 5, the E_a of the study was calculated as 8.54 kJ.



Figure 5. The Arrhenius plot used for the estimation of Ea

4. CONCLUSION

In this paper, GrO was synthesized under ultrasound irradiation following Hummers' method and its CO_2 capture performance was evaluated with TG analysis under 25°C, 75°C, and 100°C. The CO_2 capture performance of GrO decreased with increasing temperature due to the exothermic nature of the adsorption. The CO_2 uptake capacity of GrO was revealed as 1.04 mmol g⁻¹ for 90 min at 25 °C. The CO_2 adsorption kinetics of GrO were described by applying to obtain kinetic data for three different kinetic models. The Ar kinetic model has the best fit for CO_2 capture kinetic data.

DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Deniz SEZGIN: Performed the experiments and wrote the manuscript.

Müge SARI YILMAZ: Performed the experiments, analyzed the results and wrote the manuscript.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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