Optimasi Konsentrasi Polietilen Glikol pada Silika Mesopori Berbasis Fly Ash untuk Adsorpsi CO_2 dari Campuran Gas CO_2/N_2

Optimization of Polyethylene Glycol Concentration on Fly Ash-Derived Mesoporous Silica for CO_2 Adsorption from CO_2/N_2 Gas Mixtures

Rico Julian[1*], David Bahrin[1], Budi Santoso[1]
[1]*Department of Chemical Engineering, Faculty of Engineering, Universitas Sriwijaya, Jl. Srijaya Negara, Bukit Besar, Palembang, South Sumatra, Indonesia 30139*
**Corresponding Author: azuramerliza22@gmail.com*

Received: 28th June 2026; Revised: 7th July 2026; Accepted: 11th July 2026

ABSTRAK

Peningkatan konsentrasi CO_2 di atmosfer mendorong pengembangan material penangkapan karbon yang efisien. Penelitian ini bertujuan mengkaji pengaruh fraksi massa polietilen glikol (PEG) terhadap kinerja adsorpsi CO_2 pada silika mesopori berbasis fly ash serta mengevaluasi selektivitasnya pada kondisi simulasi pasca-pembakaran. Silika mesopori disintesis dari fly ash batu bara melalui metode hidrotermal menggunakan CTABr sebagai templat, kemudian diimpregnasi dengan PEG pada variasi fraksi massa 0%, 15%, 20%, 25%, dan 30%. Performa adsorpsi diuji menggunakan gas CO_2 murni selama tiga siklus berulang dan campuran gas 15% CO_2/85% N_2 untuk simulasi gas buang. Hasil menunjukkan peningkatan konsisten: breakthrough time meningkat dari 217 detik (PEG 0%) menjadi 460 detik (PEG 30%), setara peningkatan 111,98%. Pada kondisi pasca-pembakaran, efisiensi adsorpsi meningkat dari 7,57% menjadi 30,29%, dengan Relative Enhancement Factor (REF) sebesar 4,00 yang menunjukkan peningkatan selektivitas CO_2/N_2. Analisis XRD mengonfirmasi terjaganya struktur silika amorf, sementara SEM-EDX menunjukkan morfologi partikel teraglomerasi (1–6 µm) yang didominasi unsur silikon dan oksigen. PEG 30% ditetapkan sebagai konsentrasi optimum berdasarkan evaluasi menyeluruh seluruh parameter kinerja, yang menandakan potensinya sebagai adsorben rendah biaya untuk penangkapan CO_2 industri.

Kata kunci: *fly ash; silika mesopori; polietilen glikol; adsorpsi CO_2; carbon capture; pasca-pembakaran*

ABSTRACT

Rising atmospheric CO_2 levels has driven the development of efficient carbon capture materials. This study investigates the effect of polyethylene glycol (PEG) mass fraction on CO_2 adsorption performance of fly ash-derived mesoporous silica and evaluates its selectivity under simulated post-combustion conditions. Mesoporous silica was synthesized from coal fly ash via a hydrothermal method using CTABr as a template, followed by impregnation with PEG at five mass fractions: 0%, 15%, 20%, 25%, and 30%. Adsorption performance was assessed using pure CO_2 across three consecutive cycles and a 15% CO_2/85% N_2 gas mixture to simulate flue gas conditions. Breakthrough time increased from 217 s (0% PEG) to 460 s (30% PEG) — a 111.98% improvement. Under post-combustion conditions, adsorption efficiency rose from 7.57% to 30.29%, with a Relative Enhancement Factor (REF) of 4.00 indicating enhanced CO_2/N_2 selectivity. XRD confirmed preservation of the amorphous silica structure, while SEM-EDX revealed agglomerated particle morphology (1–6 µm) dominated by silicon and oxygen. Based on comprehensive performance evaluation, 30 wt% PEG was identified as the optimum concentration, highlighting its potential as a low-cost adsorbent for industrial CO_2 capture.

Keywords: *fly ash; mesoporous silica; polyethylene glycol; CO_2 adsorption; carbon capture; post-combustion*

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How to cite: Julian, R., Bahrin, D., & Santoso, B. Optimization of Polyethylene Glycol Concentration on Fly Ash-Derived Mesoporous Silica for CO_2 Adsorption from CO_2/N_2 Gas Mixtures. Jurnal Ilmiah Teknik Kimia, 10(2).

Permalink/DOI: 10.32493/jitk.v10i2.61500

INTRODUCTION

Rising CO_2 levels are a primary driver of climate change. Coal-fired power plants are major CO_2 point sources, thus priority targets for mitigation. Post-combustion carbon capture offers retrofit potential into existing plants (Obi et al., 2025).

Adsorption-based capture offers low energy penalty, high selectivity, and regenerability. Mesoporous silica (e.g., MCM-41, SBA-15) offers high surface area, tunable pores, and modifiable surfaces (Han et al., 2019; Valeev & Kondratiev, 2022), with silanol-rich surfaces serving as anchor points for functional modification (Li et al., 2021).

Polyethylene glycol (PEG) is an effective modifier for enhancing CO_2 adsorption on silica. Unlike amine-based modifiers (e.g., PEI) which rely on energy-intensive chemisorption, PEG operates via physisorption (H-bonding, Lewis acid-base), offering milder regeneration (Li et al., 2021; Moon et al., 2024). PEG-modified silica also enhances CO_2/N_2 selectivity through interactions between CO_2's quadrupole and PEG's ether groups, unlike activated carbon or zeolites (Navik et al., 2024; Grushevenko et al., 2025). However, most studies used fixed PEG loading without optimization; excessive PEG can cause pore blocking (Moon et al., 2024; Güzel Kaya, 2021).

Indonesian coal plants generate substantial fly ash and bottom ash; PLTU Banjarsari and Tanjung Enim alone produce 250 t/day bottom ash and 200–1000 t/day fly ash (Pataras et al., 2023). Rich in amorphous silica (50–60%) and alumina (20–30%), this waste remains underutilized, yet is a promising low-cost silica precursor (Pataras et al., 2023; Valeev & Kondratiev, 2022). Fly ash can yield mesoporous silica with surface areas up to 490 m^2/g (Gao et al., 2023) and serve as a support for polyethylenimine in CO_2 capture (Jia et al., 2024). This approach reduces costs and supports circular economy by converting waste into value-added materials.

Most prior work on PEG-silica adsorbents used pure CO_2, not reflecting post-combustion flue gas (10–15% CO_2 in N_2) (Moon et al., 2024). Mixed-gas testing is essential to assess selectivity, as N_2 can compete for adsorption sites.

This study addresses these gaps by: (1) systematically optimizing PEG mass fraction (0–30 wt%) on fly ash-derived mesoporous silica; (2) evaluating adsorption performance under both pure CO_2 and simulated post-combustion gas (15% CO_2/85% N_2) conditions; and (3) characterizing the optimum adsorbent via XRD and SEM-EDX to elucidate structure-performance relationships.

MATERIALS AND METHODS

Materials

The materials used were fly ash-derived mesoporous silica synthesized with cetyltrimethylammonium bromide (CTABr, ≥98%, Sigma-Aldrich), PEG-1000 (≥99%, Merck), ethyl acetate (≥99.5%, Merck), NaOH 1 M (≥98%, Merck), and CO_2 and N_2 gases (≥99.9%, CV. Delapan Pilar Agung, Palembang). PEG-1000 was chosen for balanced chain length, ensuring CO_2–ether interactions and solubility while avoiding high viscosity of larger PEGs and volatilization of smaller ones during regeneration (Moon et al., 2024; Güzel Kaya, 2021). Equipment included a hydrothermal reactor, oven, furnace, magnetic stirrer, mass flow controller, adsorption column, and CO_2 sensor.

Mesoporous Silica Synthesis

Silica precursor was dissolved in water (1:10 w/v) and stirred with CTAB (0.45 g/50 mL) for 4 h at pH ~10 (NaOH 1 M). The mixture was hydrothermally treated at 120°C for 18 h. The product was filtered, washed, dried (110°C, 3 h), and calcined (550°C, 6 h) to remove the template.

PEG Modification

PEG was loaded at 0–30 wt% (5 wt% increments). For each variation, PEG was dissolved in ethyl acetate, mixed with silica, stirred (60°C, 8 h), left for 24 h, then dried (110°C, 4 h). The resulting adsorbents were stored in sealed containers. Regeneration was performed at 150°C for 1 h to break the physical interactions between CO_2 and PEG's ether groups without degrading the polymer,

thus maintaining cyclic stability (Güzel Kaya, 2021; Moon et al., 2024).

Adsorption Testing

Adsorbents were shaped into discs (4 cm dia., 3–5 mm thick), dried (150°C, 30 min), and weighed (~2.5 g dry basis) before each run. The setup included a gas source, flow controller, adsorption column, and CO_2 sensor. Tests used (1) pure CO_2 (15 mL/min, 3 cycles) and (2) 15% CO_2/85% N_2 (40 mL/min, 1.5 bar, 29°C, 61% RH). Control runs (no adsorbent) gave C_0 = 990 ppm (pure CO_2) and 700 ppm (mixed gas) with $t_{control}$ of 2 min 24 s and 15 min, respectively.

Characterization

The optimum adsorbent (30% PEG) was characterized using X-ray Diffraction (XRD) to identify crystalline structure and diffraction patterns, and Scanning Electron Microscopy–Energy Dispersive X-ray Spectroscopy (SEM-EDX) to examine surface morphology and elemental composition.

Performance Parameters

The following parameters were calculated using the equations below:

$$Adsorption\ Efficiency\ (\%) = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

$$Capacity\ Retention\ (\%) = \frac{BT_n}{BT_1} \times 100 \quad (2)$$

$$PI\ (\%) = \frac{BT_{PEG} - BT_{0\%}}{BT_{0\%}} \times 100 \quad (3)$$

$$REF = \frac{\Delta C_{PEG}}{\Delta C_{0\%}} \quad (4)$$

$$FRR\ (\%) = \frac{Q_0 - Q_t}{Q_0} \times 100 \quad (5)$$

Where C_0 = control concentration, C_t = outlet concentration at $t_{control}$, BT_n = breakthrough time at cycle n, BT_1 = breakthrough time at cycle 1, $\Delta C = C_{control} - C_{outlet}$, Q_0 = initial flow rate, Q_t = outlet flow rate

Adsorption capacity was not determined; the focus was on breakthrough time and efficiency under fixed-bed conditions, which are more relevant to industrial design (Amponsah et al., 2025). The reported efficiency values serve as practical performance indicators for post-combustion capture. Future work should include equilibrium isotherm measurements for quantitative comparisons.

RESULTS AND DISCUSSION

CO_2 Adsorption Performance with Pure CO_2

Table 1 summarizes the breakthrough time values across five PEG concentrations and three adsorption cycles. A consistent positive trend was observed: as PEG mass fraction increased, breakthrough time increased in all cycles.

Table 1. Breakthrough Time (seconds) Across Three Cycles

PEG (%)	Cycle 1 (s)	Cycle 2 (s)	Cycle 3 (s)	Cycle 3 Retention (%)
0	217	218	197	90.78
15	235	227	202	85.96
20	282	229	203	71.99
25	387	250	240	62.02
30	460	285	274	59.57

In Cycle 1, breakthrough time increased from 217 s (0% PEG) to 460 s (30% PEG), a 111.98% improvement. The breakthrough curves (Figure 1) confirm delayed breakthrough with increasing PEG content, consistent with Table 1. The slower saturation rate of PEG 30% indicates enhanced CO_2 retention capacity.

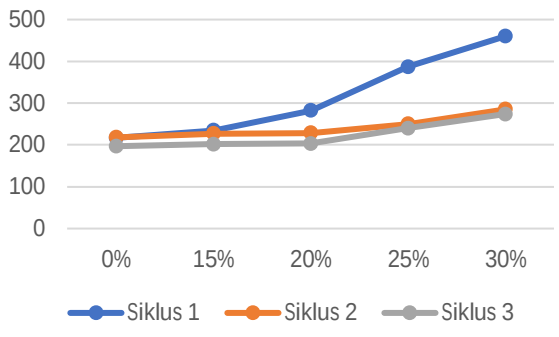


Figure 1. Breakthrough curves (CO_2 concentration vs. time) for PEG-modified fly ash-derived mesoporous silica at various PEG loadings (0%, 15%, 20%, 25%, and 30%) in the first adsorption cycle using pure CO_2 at

15 mL/min. The curves show a clear delay in breakthrough with increasing PEG content, consistent with the breakthrough time values reported in Table 1.

The enhancement in breakthrough time is attributed to the interaction between PEG's ether groups and CO_2 molecules, which act as a weak Lewis acid. The significant quadrupole moment of CO_2 enables strong electrostatic interactions with the polar ether groups, increasing adsorption affinity (Moon et al., 2024; Güzel Kaya, 2021). This is consistent with findings on PEG/silica composites, where physical interactions between ether groups and CO_2 enhanced adsorption capacity (Güzel Kaya, 2021). Similarly, PEG-functionalized silica improved CO_2/N_2 selectivity by approximately 3.8 times, confirming the role of polar groups in CO_2 affinity (Duarte et al., 2024).

Adsorption efficiency increased from 49.60% (0%, Cycle 3) to 53.84% (30%, Cycle 1) with increasing PEG content (Table 2). Although the efficiency values appear close, the breakthrough time data better reflect the cumulative effect of PEG modification. PEG loading was limited to 30 wt% to avoid pore blocking and reduced CO_2 diffusion (Moon et al., 2024; Güzel Kaya, 2021), as higher loadings may form aggregates that restrict gas transport. Future studies should explore concentrations above 30 wt% to optimize the balance between modification and mass transfer.

Table 2. Adsorption Efficiency (%) and Performance Improvement in Pure CO_2

PEG (%)	C_{outlet} (ppm)	Eff. (%)	ΔC (ppm)	REF	Flow Rate Red. (%)
0	647	7.57	53	1.00	60.0
15	600	14.29	100	1.89	60.0
20	523	25.29	177	3.34	77.5
25	491	29.86	209	3.94	75.0
30	488	30.29	212	4.00	80.0

Adsorbent Stability Across Repeated Cycles

Capacity retention declined with repeated use, more pronounced at higher PEG loadings: PEG 0% retained 90.78% of its initial capacity at Cycle 3, compared to only 59.57% for PEG 30%. This decline is due to (1) incomplete CO_2 desorption and (2) conformational relaxation of PEG chains that may block ether groups. PEG addition enhances cyclic stability by reducing degradation, though chain redistribution remains a factor affecting long-term performance (Yang et al., 2025; Miao et al., 2025).

Despite lower retention, PEG 30% still showed a longer absolute breakthrough time at Cycle 3 (274 s vs. 197 s for PEG 0%), confirming a net benefit in adsorption capacity after cyclic use. Optimization of regeneration conditions (e.g., desorption temperature, purge gas) is recommended to improve cyclic stability.

Performance Under Post-Combustion Conditions (15% CO_2/85% N_2)

Table 3 summarizes the adsorption performance under simulated post-combustion conditions. Adsorption efficiency increased progressively with PEG content, from 7.57% (0% PEG) to 30.29% (30% PEG). The ΔC values rose from 53 ppm to 212 ppm, while the Relative Enhancement Factor (REF) reached 4.00 at PEG 30%, indicating that PEG modification quadrupled CO_2 removal relative to unmodified silica under mixed-gas conditions.

Table 3. Post-Combustion Adsorption Performance (15% CO_2/85% N_2)

PEG (%)	Eff. C1 (%)	Eff. C2 (%)	Eff. C3 (%)	Improv. C1 (%)	Improv. C3 (%)
0	50.51	50.51	49.60	0.00	0.00
15	51.41	51.41	50.61	8.29	2.54
20	52.42	52.02	51.41	29.95	3.05
25	53.03	53.03	52.02	78.34	21.83
30	53.84	53.54	52.93	111.98	39.09

The improved CO_2 adsorption under mixed-gas conditions arises from selective interactions between PEG's ether groups and CO_2 molecules. CO_2 has a significantly larger quadrupole moment than N_2, enabling stronger electrostatic interactions with the polar ether groups, while non-polar N_2 has minimal

affinity (Navik et al., 2024). The REF value of 4.00 reflects this selectivity enhancement, as ether oxygen atoms act as Lewis base sites that interact with quadrupolar CO_2 via dipole-quadrupole interactions (Grushevenko et al., 2025). In contrast, non-polar N_2, with negligible quadrupole moment, cannot form such favorable interactions (Navik et al., 2024). This mechanism is supported by studies showing that specific interactions between PEG substituents and CO_2 enhance CO_2/N_2 selectivity (Grushevenko et al., 2025).

In fixed-bed systems, pressure drop directly affects energy consumption and operational costs (Najafabadi & Dehkordi, 2025). For post-combustion capture, where flue gas is near-atmospheric, even moderate pressure drops increase energy penalties and pumping costs. Optimizing particle size and packing is therefore essential for scale-up. Amponsah et al. (2025) showed that smaller particles (0.5 mm) achieve near-complete capture (100%) but high pressure drop (4.2 kPa), while larger particles (1.5 mm) reduce pressure drop (0.9 kPa) but lower efficiency to 73%. Intermediate particles (0.8 mm) offer an optimal balance (~85% efficiency, 1.7 kPa), confirming that particle size selection is key for industrial-scale column design.

Optimum PEG Concentration Determination

A composite scoring method was applied to integrate all performance parameters (adsorption efficiency, ΔC, REF as benefit criteria; flow rate reduction as a cost criterion). Each parameter was normalized and averaged to produce a total score (Table 4).

Table 4. Composite Scoring Results for PEG Concentration Selection

PEG (%)	N_{ae} (norm.)	NΔC (norm.)	NREF (norm.)	NFRR (norm.)	Total Score
0	0.250	0.250	0.250	1.000	0.438
15	0.472	0.472	0.473	1.000	0.604
20	0.835	0.835	0.835	0.774	0.820
25	0.986	0.986	0.985	0.800	0.939
30	1.000	1.000	1.000	0.750	0.938

PEG 30% was selected as the optimum concentration based on three considerations: (1) it consistently gave the highest values for all individual CO_2 capture parameters; (2) the composite score difference between PEG 25% and 30% (0.001) is negligible; and (3) the primary goal was to maximize adsorption performance. However, PEG concentrations above 30 wt% were not tested. Based on literature, excessive PEG loading beyond a certain threshold can cause pore blocking and reduce CO_2 diffusion (Moon et al., 2024; Güzel Kaya, 2021). Future studies should investigate higher PEG loadings to identify the pore-blocking threshold.

XRD and SEM-EDX Characterization

XRD analysis of the 30% PEG adsorbent revealed a broad dominant peak in the 2θ range of 20–25°, characteristic of amorphous to semi-amorphous silica (Figure 2). A minor peak at $2\theta \approx 5.38°$ emerged after PEG modification, indicating a change in diffraction characteristics without disrupting the fundamental silica structure. These findings are consistent with studies on PEG-modified silica, where organic modification of mesoporous silica may introduce low-angle diffraction peaks related to the periodicity of the modifier distribution (Güzel Kaya, 2021).

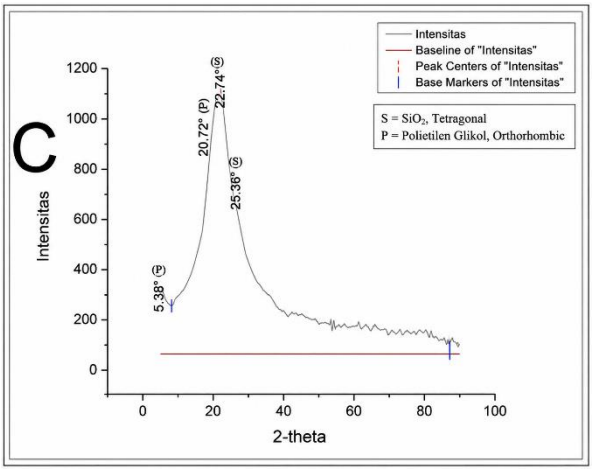
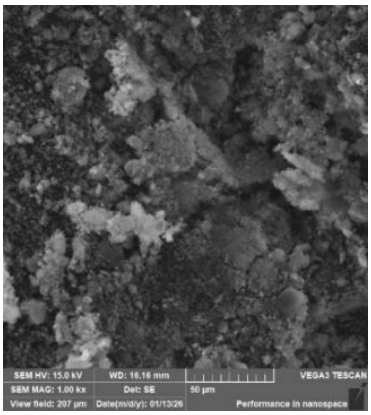


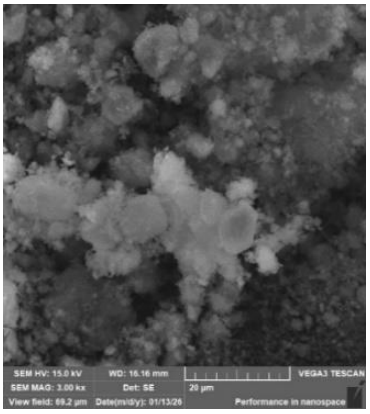
Figure 2. XRD pattern of fly ash-derived mesoporous silica modified with 30% PEG. The broad peaks at 20.74° and 25.36° (labeled S) correspond to amorphous silica, while the peak at 5.38° (labeled P) indicates the presence of PEG modification.

SEM images (Figure 3) revealed agglomerated particles (1–6 μm, mostly 1–4 μm) with heterogeneous size distribution.

EDX analysis at three points showed dominant oxygen (63.07–69.06%) and silicon (30.94–36.93%), consistent with SiO_2. Uniform elemental distribution confirmed homogeneous material composition and preservation of the silica framework after PEG modification.



(a)



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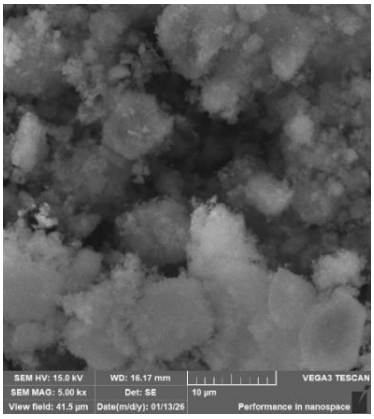

(c)

Figure 3. SEM images of 30% PEG-modified fly ash-derived mesoporous silica at magnifications of (a) 1000×, (b) 3000×, and (c) 5000×, showing agglomerated particle morphology with heterogeneous size distribution (1–6 µm)

EDX could not confirm PEG grafting due to its limitation in detecting light elements (C) in silica matrices, and FTIR, TGA, and BET were not performed due to instrument limitations. However, the improved breakthrough time, adsorption efficiency, and selectivity with increasing PEG loading indirectly support successful PEG incorporation. Future work should include FTIR to confirm PEG grafting through characteristic C–O–C (~1100–1250 cm^{-1}), C–H (~1460 cm^{-1}), and O–H (~3300 cm^{-1}) vibrations (Hsiao et al., 2025), TGA to quantify loading, and BET-BJH to evaluate surface area and porosity, which would provide a more comprehensive understanding of the structure–performance relationships.

To validate the "low-cost adsorbent" claim, the 30% PEG-silica was compared with commercial adsorbents. Under post-combustion conditions, it achieved a CO_2 uptake of ~0.69 mmol/g, lower than Zeolite 13X (2.0–4.7 mmol/g) (Parikh & Mahinpey, 2025) and amine-functionalized silica (up to 2.44 mmol/g) (Bae et al., 2025). Despite its lower absolute capacity, the PEG-silica adsorbent offers a cost-effective alternative for large-scale CO_2 capture due to its simple synthesis and use of fly ash waste as the silica source.

CONCLUSION

This study demonstrated that PEG modification of fly ash-derived mesoporous silica consistently enhanced CO_2 adsorption performance across all evaluated parameters. Key findings are as follows:

1. Increasing PEG mass fraction from 0% to 30% increased breakthrough time by 111.98% (217 s to 460 s) in the first adsorption cycle with pure CO_2, and improved adsorption efficiency from 49.60% to 53.84%.
2. Under simulated post-combustion conditions (15% CO_2/85% N_2), adsorption efficiency increased from 7.57% (0% PEG) to 30.29% (30% PEG), with a Relative Enhancement Factor of 4.00, indicating a fourfold improvement in CO_2 removal relative to unmodified silica.

3. Despite lower capacity retention in high-PEG adsorbents during cyclic testing, PEG 30% maintained superior absolute breakthrough times compared to unmodified silica across all cycles.
4. PEG 30% was designated the optimum concentration based on consistent superiority across all CO_2 capture parameters and negligible difference in composite scoring versus PEG 25%.
5. XRD confirmed preservation of the amorphous silica structure post-modification; SEM-EDX revealed agglomerated particle morphology (1–6 µm) with SiO_2-dominant elemental composition.

The 30% PEG-silica adsorbent achieved a breakthrough time of 460 s, an efficiency of 30.29% under 15% CO_2/N_2, and ~0.69 mmol/g uptake—comparable to or better than previously reported PEG/silica composites (Güzel Kaya, 2021) and polyol-modified PEI/silica systems (Moon et al., 2024). Unlike amine-functionalized sorbents that require complex synthesis and high regeneration energy, our adsorbent offers a simpler route and utilizes low-cost fly ash. These findings confirm its potential as a cost-effective and sustainable CO_2 adsorbent.

Future work should investigate PEG concentrations above 30% to identify the pore-blocking threshold, conduct FTIR and BET-BJH characterization, optimize regeneration conditions, and evaluate performance with more adsorption cycles.

ACKNOWLEDGMENTS

The authors express their sincere gratitude to Dr. Ir. David Bahrin, S.T., M.T. and Dr. Budi Santoso, S.T., M.T. for their invaluable guidance, constructive feedback, and continuous support throughout this research. The authors also thank Daniel, Arival Fikri, Aslam, and Christian for their technical assistance and helpful discussions during the experimental work.

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