



Modifikasi Bentuk Silika dari *Fly Ash* Batubara dengan Polivinil Alkohol untuk Adsorben *Carbon Capture*

Silica Shape Modification Of Coal Fly Ash using Polyvinyl Alcohol for Carbon Capture Adsorbent

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ABSTRAK

Emisi CO₂ dari pembakaran batubara mendorong pengembangan teknologi *carbon capture* yang efektif dan ekonomis. Penelitian ini bertujuan mengembangkan adsorben berbasis silika dari *fly ash* batubara yang dimodifikasi menggunakan Polivinil Alkohol (PVA) untuk meningkatkan kapasitas adsorpsi CO₂. Adsorben disintesis melalui proses hidrotermal pada suhu 120°C selama 10, 14, dan 18 jam, kemudian dikalsinasi pada suhu 550°C selama 6 jam. Adsorben selanjutnya dimodifikasi dengan variasi rasio PVA dan diuji menggunakan metode *breakthrough* pada kolom adsorpsi. Karakterisasi dilakukan dengan *X-Ray Diffraction* (XRD) dan *Scanning Electron Microscopy* (SEM). Hasil menunjukkan bahwa peningkatan waktu hidrotermal dan rasio PVA memperbaiki struktur pori dan meningkatkan waktu *breakthrough* serta kapasitas adsorpsi CO₂. Sampel dengan waktu hidrotermal 18 Hours dan rasio PVA tertinggi memberikan performa terbaik. Adsorben berbasis *fly ash* termodifikasi PVA ini berpotensi sebagai alternatif material *carbon capture* yang ekonomis dan efektif.

Kata kunci: Fly Ash, Silika, Polivinil Alkohol (PVA), *Carbon Capture*, Adsorpsi CO₂.

ABSTRACT

CO₂ emissions from coal combustion require effective and economical carbon capture technologies. This study develops a silica-based adsorbent derived from coal fly ash modified with Polyvinyl Alcohol (PVA) to enhance CO₂ adsorption. Mesoporous silica was synthesized using a template-assisted hydrothermal method at 120°C for 10, 14, and 18 hours, followed by calcination at 550°C. The material was modified with varying PVA ratios and tested using a breakthrough adsorption method. Characterization was conducted using XRD and SEM. Results show that longer hydrothermal time and higher PVA ratio improve pore structure, breakthrough time, and CO₂ adsorption capacity. The 18-hour hydrothermal sample with the highest PVA ratio exhibited the best performance. The modified fly ash-based adsorbent demonstrates strong potential for economical carbon capture applications.

Keywords: Fly Ash, Mesoporous Silica, PVA, Carbon Capture, CO₂ Adsorption.

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INTRODUCTION

Fly ash waste has a large enough silica content, reaching 40.43-41.78% so that it can be used to be synthesized into CO₂ capture adsorbents (Sari et al., 2023). The use of fly ash waste can reduce the cost of implementing CCS at the PLTU Suralaya. The aim of this research is to develop adsorbents from silica for application at the Suralaya Power Plant by testing several parameters and mixing with polyvinyl alcohol. This formulation is expected to produce inexpensive adsorbents, as well as have good physical properties and separation results.

MATERIALS AND METHODS

Silicon dioxide (SiO₂) derived from coal fly ash was used as the primary adsorbent material. Cetyltrimethylammonium bromide (CTABr) served as a pore-directing agent, sodium hydroxide (NaOH) was used for pH adjustment, and polyvinyl alcohol (PVA) was applied as a modifying agent. Ethyl acetate and distilled water were used as organic and aqueous solvents, respectively.

Mesoporous silica was synthesized via a hydrothermal template method. The SiO₂ solution was mixed with CTABr and adjusted to pH 10 using NaOH, followed by hydrothermal treatment at 120°C for 10–18 h. The resulting product was filtered, washed, dried at 110°C, and calcined at 550°C to remove the surfactant and form a mesoporous structure.

Surface modification was carried out by mixing mesoporous silica with PVA at mass ratios of 1:0, 1:0.15, and 1:0.3 in ethyl acetate, followed by stirring at 60°C for 8 h. The mixture was then dried at room temperature for 24 h and further dried at 110°C to obtain the SiO₂-PVA composite.

The prepared adsorbent was shaped into packing material and tested in a fixed-bed adsorption column using pure CO₂ at a constant flow rate of 15 mL/min. Adsorption performance was evaluated using the breakthrough method to determine CO₂ adsorption capacity and efficiency.



Figure 1. Adsorption Tools Combined (Adsorber Column, Gas Flow meter, and Gas Tank)

The adsorption capacity was calculated from the breakthrough curve using the following equation:

$$q = \frac{QC_0}{m} \int_0^{t_s} \left(1 - \frac{C_t}{C_0}\right) dt \quad (1)$$

Where q is adsorption capacity (mg/g), Q is the gas flow rate, m is the adsorbent mass, C_0 is the inlet CO₂ concentration, and C_t is the outlet CO₂ concentration.

Material characterization was conducted using Scanning Electron Microscopy (SEM) to observe surface morphology and X-Ray Diffraction (XRD) to analyze crystalline structure. The data were analyzed both qualitatively and quantitatively to evaluate the effects of hydrothermal time and PVA ratio on adsorption performance.

RESULTS AND DISCUSSION

In this experiment, the flow of CO₂ gas was set to remain stable at a rate of 15 mL/min. The gas used is pure CO₂, with an initial level of 990 ppm entering the adsorption column. To evaluate the performance of the adsorption process, the output side of the column is equipped with a flow meter and a concentration sensor that functions to record the flow rate and CO₂ level after passing through the adsorbent medium.

The method used is a *breakthrough* test. A *breakthrough* test in the adsorption process is a dynamic method used to evaluate adsorbent performance by monitoring changes in the concentration of *adsorbate* in the column output over time when the fluid is continuously flowed through the adsorbent medium (Al Kindi et al., 2023). The resulting



breakthrough curve is usually plotted as the ratio of output concentration to input concentration over time, reflecting the transition from the full adsorption phase to the adsorbent saturation phase (Fernandez et al., 2023).

The observation data are presented in the tables and figures below

Table 1. Research Result (same for all variable)

Input		
Gas Flow (mL/min)	CO ₂ Concentration(ppm)	%CO ₂
15	990	100
15	990	100
15	990	100

Table 2. Research Result without Adsorbent

Output			
Time	Gas Flow (mL/min)	CO ₂ Concentration (ppm)	%CO ₂
1 Min	14	470	47,47
2 Min	12	518	52,32
2 Min 24 Seconds	12	990	100

Table 3. Research Result Using Adsorbent

Output			
Time	Gas Flow (mL/min)	CO ₂ Concentration (ppm)	%CO ₂
10 Hours No PVA			
1 Min	14	458	46,262
2 Min	11	483	48,787
2 Min 24 Seconds	11	551	55,656
2 Min 40 Seconds	11	990	100
10 Hours 0,15 PVA			
1 Min	13	456	46,06
2 Min	11	462	46,667
2 Min 24 Seconds	10	488	49,292
3 Min	10	781	78,889
3 Min 7 Seconds	10	990	100
10 Hours 0,3 PVA			
1 Min	14	464	46,868
2 Min	10	468	47,272
2 Min 24 Seconds	10	471	47,575
3 Min	10	503	50,808
3 Min 38	10	990	100

Seconds

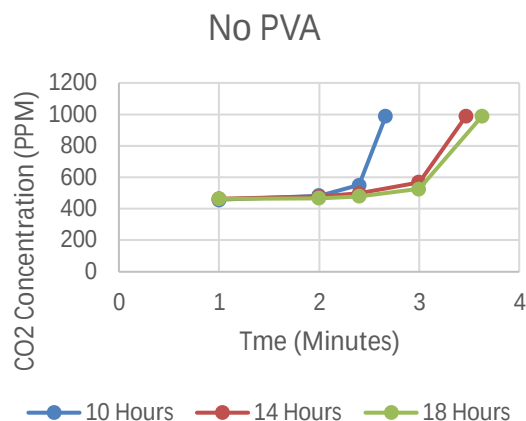
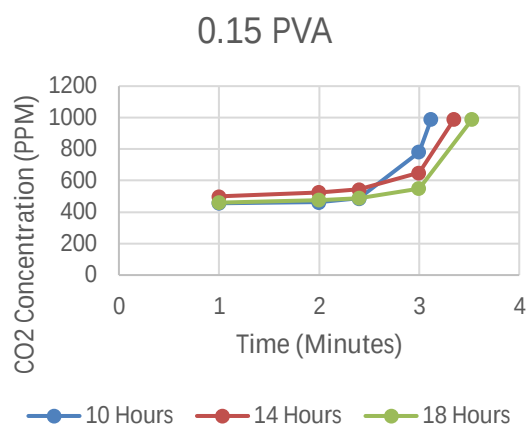
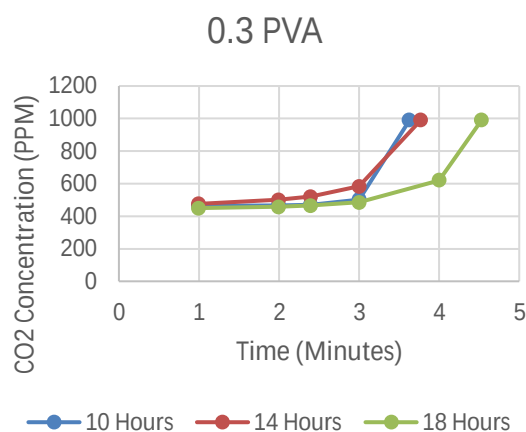
14 Hours No PVA			
1 Min	14	464	46,868
2 Min	11	478	48,282
2 Min 24 Seconds	10	497	50,202
3 Min	10	568	57,373
3 Min 28 Seconds	10	990	100
14 Hours 0,15 PVA			
1 Min	15	500	50,505
2 Min	12	525	53,03
2 Min 24 Seconds	11	543	54,848
3 Min	11	649	65,556
3 Min 21 Seconds	11	990	100
14 Hours 0,3 PVA			
1 Min	14	476	48,08
2 Min	13	501	50,606
2 Min 24 Seconds	12	521	52,626
3 Min	10	584	58,989
3 Min 46 Seconds	10	990	100
18 Hours No PVA			
1 Min	14	463	46,767
2 Min	12	468	47,272
2 Min 24 Seconds	12	480	48,484
3 Min	11	527	53,232
3 Min 38 Seconds	11	990	100
18 Hours 0,15 PVA			
1 Min	13	460	46,464
2 Min	11	477	48,181
2 Min 24 Seconds	10	489	49,393
3 Min	10	551	55,656
3 Min 32 Seconds	10	990	100
18 Hours 0,3 PVA			
1 Min	15	450	45,454
2 Min	14	458	46,262
2 Min 24 Seconds	13	465	46,969
3 Min	13	484	48,889
4 Min	12	620	62,626
4 Min 32 Seconds	12	990	100

Table 4. Research Result using Adsorbent

Adsorbed CO ₂ Concentration (ppm)	%CO ₂ Adsorbed	CO ₂ Adsorbed (mL/min)
10 Hours No PVA		
532	53,737	8,0606
507	51,212	7,6818
439	44,343	6,6515



0	0	0
10 Hours 0,15 PVA		
534	53,939	8,0909
528	53,333	8
502	50,707	7,606
209	21,111	3,1667
0	0	0
10 Hours 0,3 PVA		
526	53,131	7,9696
522	52,727	7,9090
519	52,424	7,8636
487	49,192	7,3787
0	0	0
14 Hours No PVA		
526	53,131	7,9696
512	51,717	7,7575
493	49,798	7,4696
422	42,626	6,3939
0	0	0
14 Hours 0,15 PVA		
490	49,495	7,4242
465	46,970	7,0454
447	45,152	6,7727
341	34,444	5,1667
0	0	0
14 Hours 0,3 PVA		
514	51,919	7,7878
489	49,394	7,4090
469	47,374	7,1060
406	41,010	6,1515
0	0	0
18 Hours No PVA		
527	53,232	7,9848
522	52,727	7,9090
510	51,515	7,7272
463	46,768	7,0151
0	0	0
18 Hours 0,15 PVA		
530	53,535	8,0303
513	51,818	7,7727
501	50,606	7,5909
439	44,343	6,6515
0	0	0
18 Hours 0,3 PVA		
540	54,545	8,1818
532	53,737	8,0606
525	53,030	7,9545
506	51,111	7,6667
370	37,374	5,6060
0	0	0

**Figure 2.** Adsorbent Absorption with no PVA**Figure 3.** Adsorbent Absorption 0,15 PVA**Figure 4.** Adsorbent Absorption 0,3 PVA

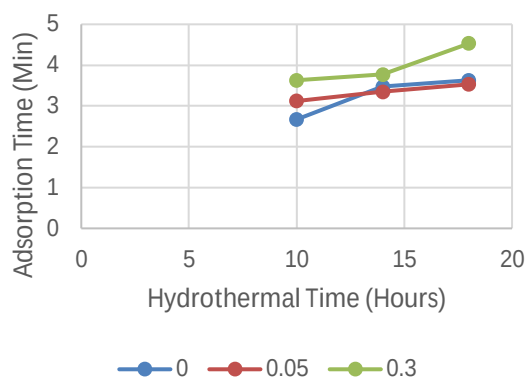


Figure 5. Effect of Hydrothermal Time on Adsorption Time

In Figure 5, the increase in CO₂ absorption time seen with increasing hydrothermal time shows that hydrothermal treatment plays an important role in the formation and maturation of the pore structure of silica-PVA adsorbents. When the hydrothermal time is extended, the process of condensation and rearrangement of silica tissue takes place more optimally, so that the pores formed become more regular and stable. However, the development of this increasingly complex pore structure also makes the path of CO₂ diffusion into the material longer, so that the time required to achieve the state of equilibrium of adsorption also increases. This pattern is commonly found in mesoporous silica materials that are synthesized with variations in the duration of hydrothermal treatment. (Juárez-Serrano et al., 2021). In addition to the influence of hydrothermal time, variations in PVA fractions also contribute to the resulting chart trends. The addition of PVA fractions increases the number of hydroxyl groups (–OH) on the surface of the adsorbent, which is polar and is able to increase the interaction between adsorbents and CO₂ molecules. This interaction supports a stronger CO₂ uptake process, but at higher PVA fractions, the presence of a polymer layer can partially

cover the silica pores or narrow access to the active site. As a result, the movement of CO₂ into the pore structure becomes slower and absorption time increases. (Zhao et al., 2022).

The adsorption capacity of the best-performing adsorbent (18 h hydrothermal treatment with 0.3 PVA) was calculated from the breakthrough curve. The inlet CO₂ concentration of 990 ppm was converted into mass concentration at 30°C and 1 atm using the ideal gas relationship (PV = nRT):

$$C_0 = \frac{990 \times 44.01}{24.87} = 1751.8 \text{ mg}/\text{m}^3$$

$$= 1.752 \text{ mg}/\text{L}$$

The normalized breakthrough data are presented below.

Time (min)	CO ₂ (ppm)	1-(Ct/C0)
1.00	450	0.5455
2.00	458	0.5374
2.40	465	0.5303
3.00	484	0.5111
4.00	620	0.3737
4.53	990	0

Using the trapezoidal integration method,

$$A = \frac{0.5455 + 0.5374}{2} (2 - 1) + \frac{0.5374 + 0.5303}{2} (2.4 - 2) + \frac{0.5303 + 0.5111}{2} (3 - 2.4) + \frac{0.5111 + 0.3737}{2} (4 - 3) + \frac{0.3737 + 0}{2} (4.53 - 4)$$

$$A = 1.609 \text{ min}$$

Substituting the experimental data into Equation (1):

$$q = \frac{0.015 \times 1.752 \times 1.609}{0.4} = 0.106 \text{ mg/g}$$

The adsorption capacity in mmol/g:

$$q = \frac{0.106}{44.01} = 0.0024 \text{ mmol/g}$$



The sample synthesized at 18 h hydrothermal treatment with a SiO_2 :PVA ratio of 1:0.3 exhibited the best adsorption performance, with the longest breakthrough time of 4.53 min, the highest initial CO_2 removal efficiency of 54.55%, and an adsorption capacity of 0.106 mg/g (0.0024 mmol/g).

All adsorption experiments reached complete saturation within less than 5 minutes. Such rapid breakthrough suggests that the adsorption bed possessed a relatively low adsorption capacity under the present operating conditions. In addition, this behavior may indicate partial gas channeling inside the packed bed, where preferential flow paths reduce the effective contact between CO_2 and the adsorbent particles. Channeling may arise from non-uniform particle packing, insufficient bed height, or particle size distribution. Although no direct evidence of channeling was obtained, this possibility should be considered because the breakthrough curves exhibit very steep transitions from adsorption to saturation. Future work should evaluate pressure drop, packing density, and residence time to verify whether channeling occurs.

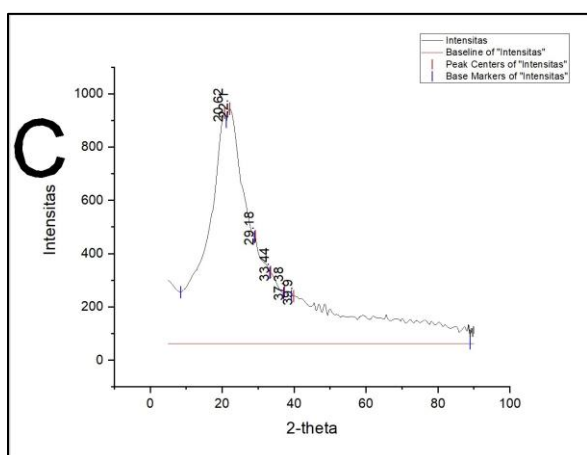


Figure 6. Graph of Analysis Results of XRD SiO_2 -PVA 18-Hours 0.3 PVA

In Figure 6, The XRD results on these silica-PVA adsorbents show that the material has a balance between the semi-crystalline phase of PVA and the amorphous phase of silica. Although it does not exhibit sharp crystalline peaks, its structural characteristics do reflect properties suitable for gas adsorption applications, such as CO_2 . The presence of amorphous silica does not significantly inhibit the formation of PVA structures, but plays a role in modifying the internal tissues of composites, thus potentially improving the effectiveness of adsorption through a combination of surface interactions and improved internal porosity.

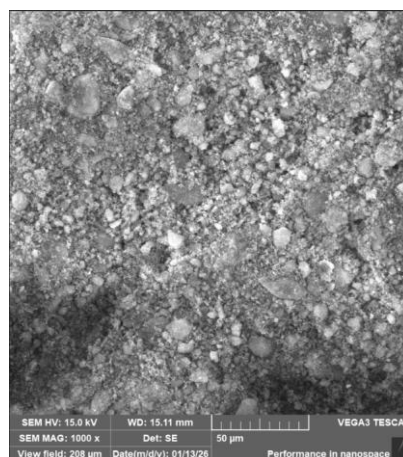


Figure 7. SEM Micrograph of PVA Modified Samples with Magnification 1000x

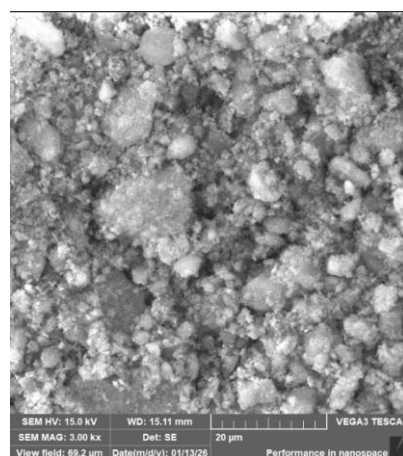


Figure 8. SEM Micrograph of PVA Modified Samples with Magnification 3000x

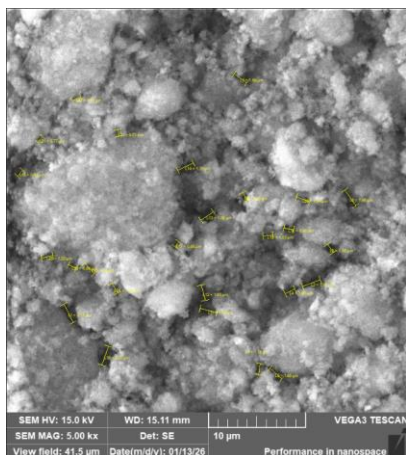


Figure 9. SEM Micrograph of PVA Modified Samples with Magnification 5000x

SEM testing was performed on the best variations, namely 18-H hydrothermal and 0.3 PVA to see the effect of the physical characteristics of the adsorbent on absorption ability. Morphological analysis of silica–PVA adsorbents was performed using Scanning Electron Microscopy (SEM) at several magnification levels. SEM shows that adsorbents have an inhomogeneous surface morphology with a non-uniform particle distribution and a tendency to form agglomerates. Granular structures with particle sizes varying from micro to submicro scales were observed, indicating the formation of a composite material between silica and polyvinyl alcohol (PVA) matrices.

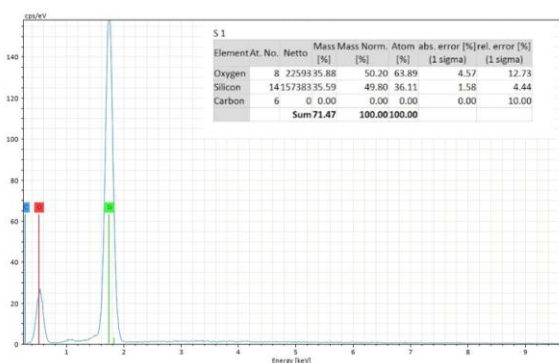


Figure 10. SEM-EDX Test Result

This condition indicates that the synthesis methods applied, including hydrothermal processes and calcination at high temperatures, are able to produce mesoporous silica material with a good level of purity. The high content of silicon in the material indicates that the silica skeleton is formed optimally and stably. This pure and homogeneous silica structure has the potential to make a positive contribution to adsorption performance, as a good SiO₂ framework generally correlates with specific surface area and higher pore regularity. The resulting mesoporous silica material has the potential to have more effective and stable adsorption capabilities of CO₂ gas in adsorbent applications.

CONCLUSION

The results of this study demonstrate that both hydrothermal time and PVA ratio significantly influence the characteristics and performance of the synthesized adsorbent. Increasing the hydrothermal duration from 10 to 18 hours improved the development of the mesoporous silica structure, leading to better pore formation. In addition, the incorporation of PVA enhanced the pore structure and adsorption performance, as indicated by longer breakthrough times and higher CO₂ adsorption capacity. The sample synthesized at 18 h hydrothermal treatment with a SiO₂:PVA ratio of 1:0.3 exhibited the best adsorption performance, with the longest breakthrough time of 4.53 min, the highest initial CO₂ removal efficiency of 54.55%, and an adsorption capacity of 0.106 mg/g (0.0024 mmol/g).

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