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Extraction and Characterization of Silicon Dioxide from Coal Fly Ash as Counter Electrode Material in Dye-Sensitized Solar Cells (DSSCs)

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Abstract

The counter electrode in DSSCs must be made of a material with various advantageous chemical and physical characteristics to guarantee the cell's efficient functioning and cost efficiency. Coal fly ash is recognized for its substantial silicon dioxide (SiO_2) content and other minerals and metals. This paper provides a detailed analysis and description of extracting and characterizing SiO_2 from coal fly ash. This extraction aims to utilize SiO_2 as a material for the counter electrode in DSSCs. The extraction procedure of SiO_2 from coal fly ash involves a multi-step process that entailed the utilization of acid leaching using hydrochloric acid 1 M at 90°C for 4 h, which was subsequently followed by precipitation using NaOH 3 M at 90°C for 4 h to separate SiO_2. The SiO_2 gel was cleaned of contaminants with hot distilled water and dried at 110°C for 12 h. The SiO_2 that was obtained was analyzed utilizing a range of analytical techniques to evaluate its structural, morphological, chemical, and optical properties. The X-ray diffractometer (XRD) examination revealed that the crystal structure of coal fly ash consisted of quartz, corundum, hematite, lime, and periclase. The SiO_2 that was obtained exhibited a crystal structure that was both cubic and triclinic. The morphology is visualized using a scanning electron microscope (SEM). The study using atomic absorption spectroscopy revealed that the coal fly ash contained 52.91% SiO_2, whereas the extracted SiO_2 had a purity of 91.20%. The UV-Vis spectrophotometry investigation revealed that the SiO_2 exhibited absorbance spectra with a wide band gap of 4.17 eV, whereas the coal fly ash had absorbance spectra of 3.37 eV.

1. Introduction

Researchers have closely scrutinized and creatively explored coal fly ash in power plants in recent decades, a byproduct of coal combustion [1]. Burning coal generates many byproducts, with fly ash being the most common [2, 3]. Bottom and fly ash are the primary types of ashes generated during the combustion process, along with additional byproducts such as flue gas, boiler slag, gypsum, scrubber residue, and fluidized bed combustion ashes [4]. Coal fly ash, comprised of numerous fine particles generated during coal combustion, has garnered significant concern due to its detrimental effects on the environment, such as air and water pollution, and its potential harm to human health [5]. Although fly ash may cause pollution, it is crucial to remember that it holds significant quantities of valuable minerals like silica, alumina, and ferrous compounds [6, 7]. These three high concentrations of valuable minerals in coal fly ash are currently garnering attention from scientists, engineers, and regulators because of their exceptional properties and extensive applications, including the development of counter electrodes in dye-sensitized solar cells (DSSCs).

Since the Gratzel group invented dye-sensitized solar cells (DSSCs), these cells have attracted much attention as a promising and cost-effective means of converting solar energy into usable power [8]. DSSCs

consist of a transparent conductive substrate, a photoanode, sensitizers, electrolytes, and a counter-electrode [9, 10]. The counter electrode is essential for improving the efficiency of DSSCs. It works as an electrocatalyst to speed up the oxidation process by starting up new redox processes in the electrolyte [11] and stopping the recombination of oxidized cations with electrons in the conduction band of the photoanode material [12]. Incorporating silicon dioxide into the counter electrode offers several advantages, such as high surface area [13]. This large surface area allows for better interaction between electrolyte and counter electrode, chemical stability [14] and thermal stability [15].

One of the most common chemicals found in coal fly ash is silicon dioxide (SiO_2) [16]. Large surface area, strong catalytic support, high absorption capacity, outstanding thermal stability, and ease of reactivation are just a few of the unique qualities of solid inorganic silicon dioxide [17, 18]. Therefore, choosing the right type of material for the counter electrode is crucial, as it must possess high catalyst activity, reflection, and, most importantly, affordability [19]. It possesses significant potential for various applications, particularly as a counter electrode in DSSCs.

The precipitation approach is an excellent strategy for attaining high degrees of purity in SiO_2. This technique involves extracting these components from a natural sample. In a previous study, extraction using the acid leaching method gives a high percentage of SiO_2 purity (>96.8%) [20]. In this study, SiO_2 was synthesized from coal fly ash treated with hydrochloric acid and sodium hydroxide. The SiO_2 was employed as a counter electrode in DSSCs to enhance their efficiency in converting solar energy by optimizing the oxidation process during the regeneration of the redox couple and reducing the electron recombination, which can increase the voltage and current because of its thermal stability [21]. X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic absorption spectroscopy (AAS), and ultraviolet-visible spectroscopy (UV-VIS) were carried out to conduct comprehensive characterizations.

2. Experimental

The extraction procedure of SiO_2 from coal fly ash was conducted using a precipitation method (Figure 1), which involved reacting the coal fly ash with NaOH and hydrochloric acid.

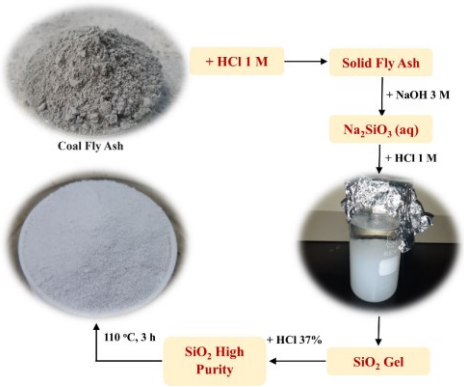

Figure 1. Schematic procedure for coal fly ash extraction

2.1. Materials and Equipment

The study used coal fly ash from PT. Semen Baturaja, NaOH (Merck), HCl (Merck), polyvinyl alcohol (Sigma Aldrich), and distilled water as materials. The research utilized laboratory glassware, an analytical balance, an oven, a hot plate stirrer, a UV-Vis spectrophotometer (Shimadzu UV-1780), an atomic absorption spectrophotometer (Shimadzu AA-6880), X-ray diffraction (Rigaku MiniFlex 300/600), and scanning electron microscopy (Tescan Vega 3).

2.2. Extraction of SiO_2 from Coal Fly Ash

An exact amount of coal fly ash (100 g) was added to 500 mL of hydrochloric acid (1 M) in a beaker glass mechanically stirred at 150 rpm and preheated at 90°C for 4 h. After 4 h of acidification, the mixture was filtered, and the residue was rinsed with hot distilled water several times and dried for 12 h at 110°C. The dried coal fly ash was dissolved with 150 mL of sodium hydroxide (3 M) and kept at 90°C for 4 h under magnetic stirring (150 rpm) to extract silica. After 4 h, it was filtered to produce a transparent filtrate of sodium silicate. To precipitate silica, sodium silicate was gradually neutralized with diluted hydrochloric acid 1 M to pH 7. After collecting the precipitated silica, it was repeatedly rinsed with hot distilled water and dried at 110°C for 12 h. Then, the precipitated SiO_2 was purified by adding it to a beaker glass containing 20 mL of hydrochloric acid (37%) and kept at 90°C for 3 h under magnetic stirring (150 rpm) to purify the precipitated SiO_2. Lastly, the solid was rinsed with hot distilled water and dried for 3 h in an oven at 110°C [22, 23].

2.3. Coal Fly Ash and SiO_2 Characterization Techniques

2.3.1. X-ray Diffraction (XRD)

The crystalline phases of coal fly ash were analyzed using XRD. This analysis was employed to ascertain the crystalline phases present in a substance, hence revealing details regarding its chemical composition through the examination of its crystal structure [24]. Phase distinction was achieved by comparing the acquired data with the information stored in reference databases [25]. The coal fly ash samples were ground into powders for analysis using powder diffraction.

2.3.2. Chemical Compositions

The mineral composition of coal fly ash and SiO_2 was analyzed using AAS. AAS is a spectroanalytical technique used to quantitatively determine the chemical elements in coal fly ash and SiO_2 by measuring the absorption of optical radiation (light) by free atoms in the gaseous phase [26].

2.3.3. Morphological Properties

SEM is an effective imaging method utilized for high-resolution examination of the surface morphology and topography of coal fly ash and SiO_2 [27]. The process involved scanning a concentrated electron beam over the surface of a sample and detecting signals [16, 27]. In this study, coal fly ash and SiO_2 were pre-deposited on the FTO glass before testing.

Table 1. XRD analysis results of coal fly ash and extracted SiO_2

Sample	Formula	Crystal size (nm)	Strain (%)	Space group	Phase	Crystal form
Coal fly ash	SiO_2	33.30	0.51	152:P3121	Quartz	Trigonal
	Al_2O_3	5.49	1.44	167:R-3c,rhombohedral	Corundum	Hexagonal
	Fe_2O_3	3.64	0.29	167:R-3c,rhombohedral	Hematite	Hexagonal
	CaO	36.00	1.09	225:Fm-3m	Lime	Cubic
	MgO	2.482	1.71	225:Fm-3m	Periclase	Cubic
Extracted SiO_2	SiO_2	40.42	0.103	1:P1	-	Triclinic
	NaCl	61.30	0.125	225:Fm-3m	-	Cubic

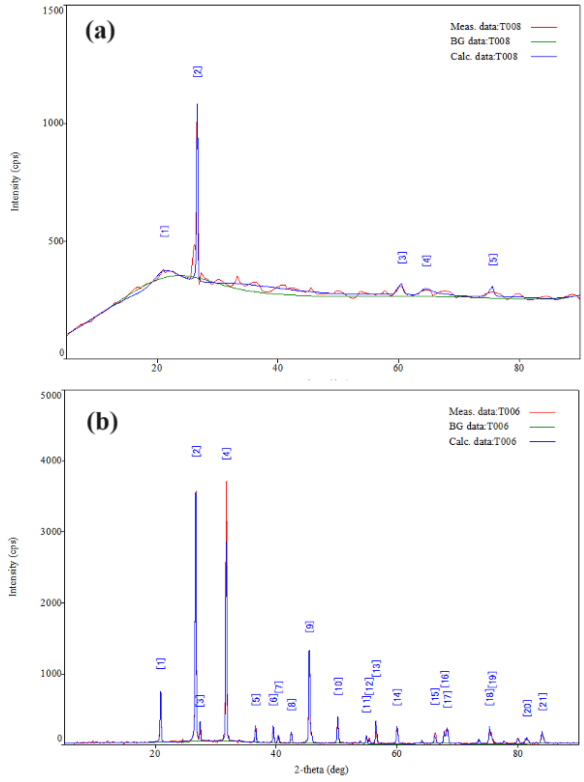

Figure 2. XRD diffractograms of (a) coal fly ash and (b) extracted SiO_2

2.3.4. Optical Properties

Optical properties refer to how a material behaves when it interacts with light. These properties encompass various characteristics of the material's absorption, transmission, or reflection of light [28]. The optical properties of coal fly ash and SiO_2 were studied using UV-Vis spectroscopy techniques. The optical analysis of coal fly ash and SiO_2 was used to calculate valence band and band gap energy [29].

3. Results and Discussion

3.1. Extraction Results

Major mineral compounds were identified in coal fly ash. These compounds include silicon dioxide (SiO_2), aluminum dioxide (Al_2O_3), iron (III) oxide (Fe_2O_3), calcium oxide (CaO), magnesium oxide (MgO), sodium oxide (Na_2O), potassium oxide (K_2O), titanium dioxide (TiO_2), and sulfur trioxide (SO_3) in different concentrations (Table 2). The composition of oxide minerals in coal fly ash depends on the coal and the oxidation process detected by its elements. Coal fly ash used as counter electrode material has a dominant oxide mineral in the form of SiO_2 of 52.91%. One of the factors contributing to its use as the primary material for silica extraction is its high silicon dioxide content. Table 2 shows the mineral composition of coal fly ash, coal fly ash after extraction, and the extracted SiO_2.

Table 2 shows that the SiO_2 content decreases from 52.91% to 40.83%, with a 22.83% extraction efficiency. This demonstrates the success of the silica extraction process. Along with silica extraction, other mineral compounds are also reduced, likely due to the material's interaction with acid and base solutions during extraction. While some compounds decrease, others, such as sodium oxide, increase. The sodium oxide content in coal fly ash rose from 3.64% before extraction to 4.03% after extraction. NaOH binds with silica to form sodium silicate, increasing sodium oxide content. This process produces sodium silicate, a colorless, basic liquid. To obtain silica, HCl is added, causing the formation of solid particles as a gel at a pH range of 6.5–7. The extraction process yielded 37.80%. Table 2 presents the compound content resulting from the extraction.

Table 2 shows that the silica dioxide produced from the extraction process is 72.54%. Compared to the study by Caroles [30], the obtained silica dioxide has a composition of 80.42%, indicating a lower purity. The silica produced still contains many other minerals, especially the increase in sodium content in the silica material. This increase in sodium content is reinforced by the XRD results, which identify salt crystals (NaCl) in the silica material. Therefore, the silica material produced is further processed in the form of a purification process using 37% HCl to reduce the composition of salt and other mineral compounds that are still bound to the silica dioxide material to obtain silica with a high level of purity. From the purification process, a silica dioxide compound of 91.20% was obtained.

3.2. X-ray Diffraction (XRD) Analysis

The XRD graph of coal fly ash and extracted SiO_2 was obtained using the Rigaku Miniflex 600 instruments with a scanning rate of 10 deg/min and a step size of 0.02 degrees in the scanning range of 5-90 degrees. The diffractogram results are shown in Figure 2. Figure 2(a) represents the coal fly ash diffractogram. Minerals like quartz low (SiO_2), corundum (Al_2O_3), hematite (Fe_2O_3), lime (CaO), and periclase (MgO) were found in coal fly

ash, with the crystalline phase of quartz (SiO_2) being a dominant substance. Yohandri *et al.* [31] investigated fly ash as a catalyst for biodiesel production, and the ICP analysis revealed Fe and Si as primary components in the fly ash content.

Figure 2(b) represents the extracted SiO_2 diffractogram. Figure 2(b) shows the existence of SiO_2 and salt (NaCl) at the peak positions of 26.668° (with a peak intensity of 2710 cps) and 31.720° (with a peak intensity of 2831 cps), respectively. The SiO_2 that has been produced shows a crystal size of 40.42 nm. It possesses a triclinic crystal shape, with crystal parameters a = 4.9213 Å, b = 4.914 Å, and c = 5.4068 Å. The salt minerals have developed with a crystal size of 61.3 nm and consist of cubic crystals with crystal parameters a, b, and c equal to 5.6458 Å. The presence of NaCl in extracted silica has an impact on the purity of the resulting SiO_2.

In addition, the details of each diffracted sample of coal fly ash and extracted SiO_2 are tabulated in Table 1. The crystal grain size and crystal lattice distortion can be determined by analyzing the expansion of XRD peaks, specifically by measuring the full width at half maximum (FWHM) of every single peak [32]. The crystal size and lattice strain of coal fly ash and extracted SiO_2, shown in Table 1, were calculated using the Williamson-Hall method.

3.3. Atomic Absorption Spectroscopy (AAS) Analysis

The chemical components of coal fly ash and extracted SiO_2 were determined by a flame atomic absorption spectrophotometer (Shimadzu AA-6880) using an air, acetylene, and nitrous flame. Both samples were prepared using the wet digestion method. About 0.1 g of sample was accurately weighed and subjected to analysis to ascertain its chemical component. The chemical component of fly ash and extracted SiO_2 that was analyzed is shown in Table 2.

The composition of mineral oxide in fly ash depends on the coal and oxidation processes. It is mostly SiO_2, which makes up 52.91% of the fly ash (Table 2). One of the considerations for using coal fly ash as the primary material is its silicon dioxide content. Based on Table 2, the content of silicon dioxide mineral compounds found in fly ash decreases from 52.91% to 40.83%, with an extraction process percentage of 22.83%. The decrease in SiO_2 content in coal fly ash indicates the successful extraction process.

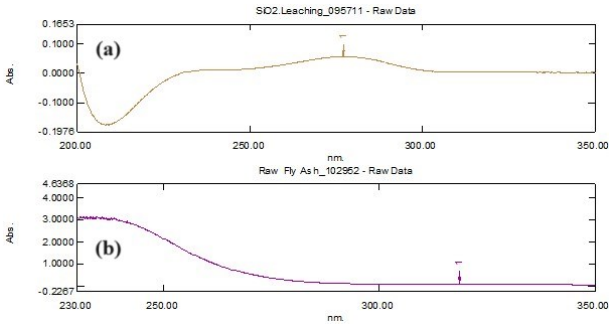

Figure 3. UV absorption spectra of (a) extracted SiO_2 and (b) coal fly ash

The chemical composition of extracted SiO_2 was also investigated using AAS to determine purity, which revealed that extracted SiO_2 had a purity of 91.20%. The AAS result confirms that most of the material was silicon; however, there are also trace amounts of sodium (4.66%), as shown by the XRD analysis (Figure 2b and Table 1). NaCl is still detected because it becomes trapped during the silica precipitation process and remains detectable even after several rinsing steps. Jumari *et al.* [33] studied the synthesis of the SiO_2/C composite from coal fly ash, and the results showed that 6% NaCl was detected in the extracted SiO_2. Rusdianasari *et al.* [34] studied the characteristics of nanosilica from rice husk ash, and the analysis revealed the presence of 6.29% sodium.

3.4. UV-Vis Spectroscopy Analysis

The UV-Vis absorption spectrum was measured using a double-beam Shimadzu Spectrophotometer UV-1780 in the 200–350 nm spectral range. A UV-Vis absorption spectrophotometer is commonly employed to investigate the optical characteristics [35] of coal fly ash and extracted SiO_2. The absorption spectra of coal fly ash and extracted SiO_2 are illustrated in Figure 3. It displays an absorption band at λ_{max} 318.50 nm for coal fly ash and 277.10 nm for extracted SiO_2 (3.37 eV and 4.17 eV).

The optical absorption method was used to accurately quantify the optical valence band and band gap values of coal fly ash and extracted SiO_2. The intercept plot on the x-axis gives the direct band gap of coal fly ash and extracted SiO_2, as illustrated in Figure 4. The valence band and bad gap of coal fly ash and extracted SiO_2 at λ_{max} are given in Table 3.

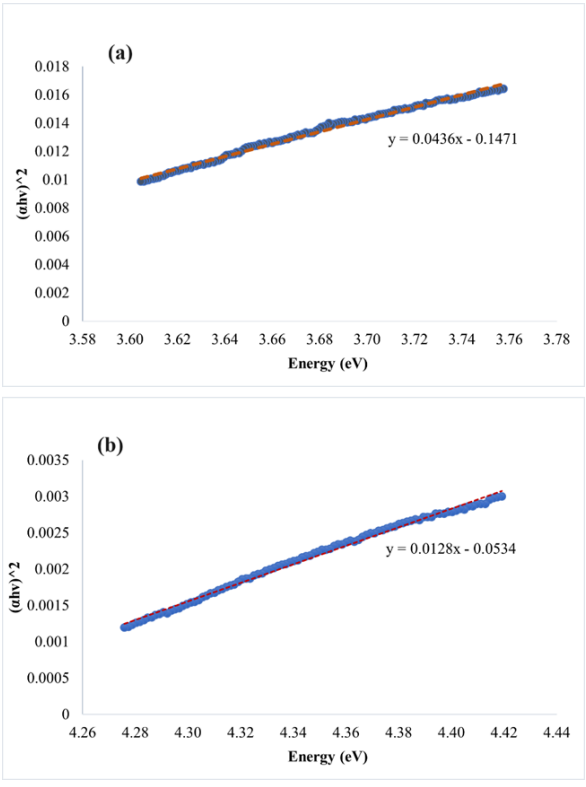

Figure 4. Band gap energy curves of (a) coal fly ash and (b) extracted SiO_2

Table 2. AAS analysis results of coal fly ash and extracted SiO_2

Composition	Percentage(%w)		
	Coal fly ash	Coal fly ash after extraction	Extracted SiO_2
SiO_2	52.91	40.83	91.20
Al_2O_3	18.21	14.12	1.03
Fe_2O_3	5.02	5.01	0.02
CaO	7.52	4.29	0.29
MgO	0.51	0.05	0.03
Na_2O	3.64	4.03	4.66
K_2O	0.33	0.24	0.19
TiO_2	0.88	0.87	0.33
SO_3	0.45	0.10	-

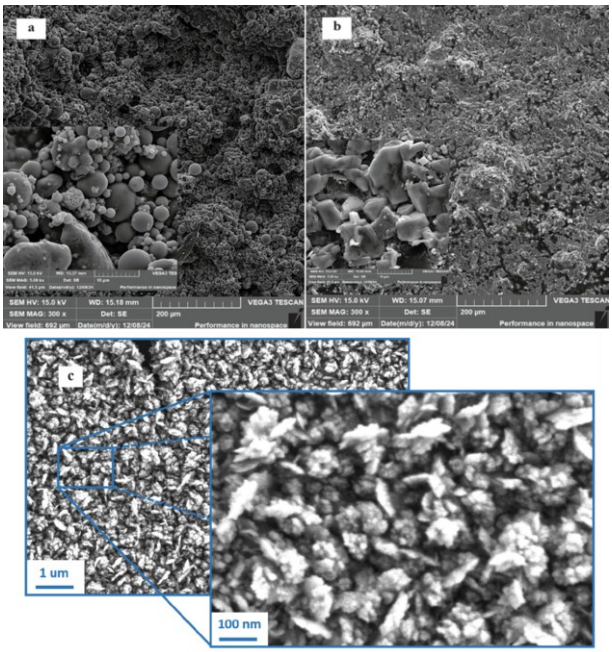

Figure 5. SEM images of (a) coal fly ash, (b) extracted SiO_2 and (c) platinum [36]

Platinum is widely used as a counter-electrode material due to its excellent electrical conductivity and catalytic properties [22]. Platinum does not have a significant band gap relevant to optical properties in the same way that SiO_2 does. While Pt offers superior catalytic performance, it is expensive and less abundant. SiO_2, on the other hand, is abundant and cost-effective, especially when derived from waste materials like fly ash. Wang *et al.* [37] investigated the optical properties of SiO_2 and SiO_2-TiO_2 and revealed that SiO_2 has a band gap of 4.01 eV. The presence of impurities (NaCl) in extracted SiO_2 can cause this study's band gap to be larger than in previous studies, thereby affecting the optical analysis results.

Table 3. Optical parameters of coal fly ash and extracted SiO_2

Sample	λ_{max} (nm)	Energy photon (eV)	Band gap (eV)
Coal fly ash	318.50	3.89	3.37
Extracted SiO_2	277.10	4.47	4.17

3.5. Scanning Electron Microscopy Analysis

The morphologies of coal fly ash and extracted SiO_2 particles are shown in Figure 5. Before completing SEM testing, coal fly ash and SiO_2 were coated upon fluorine-doped tin oxide (FTO) glass using polyvinyl alcohol (PVA). Figure 5(a) represents the electron micrograph of coal fly ash at 10 micron and 200 micron magnification. This picture substantiates the approximate spherical shape. Figure 5(b) shows the SEM image of the extracted SiO_2 particles at 300× and 5000× magnification. Results show that it tends to have complex surfaces with irregular angles and various types of crystal shapes. Figure 5(c) represents an SEM image of platinum, commonly used as a counter electrode material, alongside coal fly ash and the extracted SiO_2.

4. Conclusion

The precipitation process effectively isolated SiO_2 from coal fly ash, which was subsequently analyzed using XRD, AAS, UV-Vis absorption spectrophotometry, and SEM. XRD analysis validated that the extracted SiO_2 has a crystal size of 40.43 nm, displaying diverse crystal forms. The crystal structure of SiO_2 was determined to be triclinic. SEM analysis showed that the extracted SiO_2 has complex and irregular surfaces with uneven angles. According to the experiments, the purified SiO_2 has a purity level of 91.20%, with minor sodium impurities amounting to 4.66%. The UV absorption spectrum revealed that the extracted SiO_2 possesses a band gap of 4.17 eV.

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