Synthesis and Characterization of High-Purity Silica from Silau River Sand, Indonesia using a Coprecipitation Method

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Using a coprecipitation method, SiO_2 was extracted from river sand samples obtained from the Silau River, Indonesia. The phase of each sample was determined by X-ray Fluorescence (XRF) and X-ray Diffraction (XRD). Chemical characterization was performed with Fourier Transform Infrared Spectroscopy (FTIR), while microanalysis and elemental mapping were carried out with a Scanning Electron Microscope (SEM) equipped with an Energy Dispersive Spectrometer (EDS). The thermal characteristics of the samples were examined using Differential Scanning Calorimetry (DSC). XRF data showed that after the extraction process, the concentration of SiO_2 in the river sand increased from 80.91 wt% to 99.41 wt%, demonstrating successful SiO_2 extraction. SEM images revealed that the particles were more uniform after extraction compared to those found in the river sand before the extraction process. FTIR data showed substantial changes in peak positions and intensities after extraction, indicating that the impurities had been removed and pure SiO_2 had been synthesized. The XRD results indicated that the primary component of the extracted SiO_2 was quartz, although there were some minor amorphous components. The DSC results indicated that the thermal profile of the extracted SiO_2 was nearly flat, indicating good thermal stability. In summary, the precipitation method was effective in isolating SiO_2 from Silau River sand, and the resulting product should have a variety of applications. The extracted SiO_2 was found to be of high purity.

Keywords: SiO_2 Extraction, high-purity silica, Coprecipitation Method

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River water is a vital resource for many communities, supporting domestic use, agriculture, industry, and ecosystem sustainability. The Silau River in Asahan Regency, Indonesia, is one of the region's major water sources and plays an important role in household water supply, irrigation, fisheries, mining activities, tourism, and hydroelectric power generation [1]. The river serves as a source of biodiversity and aids in maintaining the ecological health of the surrounding ecosystem [2]; it also supplies high-quality sand used in the production of construction materials such as cement [3]. While sand extraction offers significant economic benefits to those involved, it can also cause environmental damage that affects local communities reliant on the river for their livelihoods. Specifically, excessive removal of sand may lower the riverbed and alter its overall profile, negatively impacting the river's ecosystem [4].

Pure silica (SiO_2), which is used in many industrial applications, provides another economic benefit. There are several ways that silica could be used, such as a source of silicon dioxide to create glass, or as a strength-enhancing additive to concrete by serving as a reinforcing material, as a support for catalysts, and as reinforcement in composite materials [5-8]. The characteristics of silica allow it to be applied across a wide range of industries, including ceramics, glass, and electronics. Silica combines with other materials used in glass manufacturing to lower the melting point of the raw materials and produce a harder, more durable finished glass product. Silica also possesses high-temperature thermal stability and resistance. As such, silica can be applied in the ceramic industry as a base material for ceramic products. Additionally, due to its electrical insulation, silica is used in the production of semiconductor materials and components in the electronics industry because it is compatible with many other electronic materials [9].

Silica-based materials are both desirable and functional owing to their numerous properties. These include well-organized structures, a large internal surface area, and the capacity to store a large volume of chemicals due to their large pore volume. The majority of silica materials can be modified to varying degrees; many are produced via the sol-gel process [10]. The main advantage of using silica materials is that they can change their surface chemistry to function as adsorbents, catalysts, drug delivery systems, etc., in addition to being able to remove pollutants from contaminated environments [11]. As such, researchers have continued to study whether silica materials may assist in improving the efficiency of catalytic reactions, provide support for biomedical applications, treat contaminated water, and help to capture CO_2 [10].

Several silica synthesis methods have been investigated. One of the earliest techniques for producing silica gel was the hydrolysis of tetraethyl orthosilicate (TEOS). This creates hydrated silica species upon reaction with water [12], and a highly ordered structure in the solid phase that can be functionalized with sulfonic acid, providing evidence that these materials could enhance catalytic capabilities for sustainable, selective catalysis [13]. Silica has been used in antibacterial applications, with researchers finding that silica granules loaded with stable N-halamines exhibited strong antimicrobial effects against bacteria [9].

Despite the abundance of silica-rich sand in the Silau River, a limited number of studies have addressed the feasibility of extracting high-purity silica from this source while considering environmental sustainability. However, if silica is to be obtained from this source for use in water treatment systems or other applications, it should be extracted using environmentally feasible and manageable techniques to avoid damage to freshwater resources and the stability of the river. Pure silica can be obtained from sand using different methods. One of the most common approaches to extract very high purity silica involves two major processes. First, the powdered sand is treated with HCl. Second, the sand is treated with NaOH to produce sodium silicate, and then with HCl to produce high-purity silica [14]. The simplicity, low cost, and ability of the coprecipitation method to deliver extremely high-purity silica have led to its description as a simple, cost-effective method [15]. Furthermore, this method yields the isolated product more quickly and at lower temperatures than most traditional methods [15].

Many reports have demonstrated the effectiveness of coprecipitation in synthesizing silica-based materials [16]. For example, Saleem et al. [17] synthesized silica-coated nanoparticles via coprecipitation and proved their effectiveness for removing pollutants from wastewater. In addition, the coprecipitation method was used for the synthesis of the composite material Fe_3O_4/SiO_2/Alginate [18]. The coprecipitation method was also successfully used to produce SiO_2 particles with the desired morphology and a well-ordered pore structure [19]. The method is also widely used for the extraction of silica from various sources (rice husk ash and volcanic rocks) [20-22], enabling the isolation of high-purity silica from natural samples [22].

This study investigates the feasibility of utilizing Silau River sand as a source of high-purity silica and determines if it can be achieved in a manner that is environmentally safe and sustainable. We seek to develop a methodology to optimize silica extraction from the river, with minimal negative environmental impact on the river's ecosystem and its inhabitants, and which can also be performed in a sustainable manner at other locations.

EXPERIMENTAL SECTION

Materials and Equipment

The materials used were: sand from the Silau River, Asahan Regency, Indonesia, 37% hydrochloric acid (HCl, Merck) and sodium hydroxide (NaOH, Merck). The equipment used in this study included a 230-mesh sieve, filter paper, magnetic bar, hot plate stirrer, oven, beaker, glass, spatula, digital balance, and pH meter. Analytical and characterization instruments used were: Scanning Electron Microscope-Energy Dispersive X-ray Spectrometer (SEM-EDS; FEI Inspect S50), Fourier Transform Infrared Spectrometer (FTIR; Shimadzu IRPrestige-21), X-Ray Diffractometer (XRD; PANalytical X'Pert PRO), X-Ray Fluorescence Spectrometer (XRF; PANalytical MiniPal 4), and Differential Scanning Calorimeter (DSC; Shimadzu DSC-60 Plus Series).

Extraction of SiO_2 from Silau River Sand

Extraction of SiO_2 from Silau River sand was carried out using the coprecipitation method. 100 g of river sand was sieved through a 230-mesh sieve to obtain a homogenous particle size. A total of 50 g of the sieved sand was soaked in 3 L of 2 N HCl at 80 °C for 2 h with continuous stirring (300 rpm), and then filtered [23]. The sand was then washed and dried in an oven at 110 °C for 12 h [24]. The dried sand was dissolved in 500 mL of 7 M NaOH and heated at 90 °C for 120 minutes. The mixture was filtered to obtain a sodium silicate solution. This solution was placed in a glass beaker, and 8 M HCl was added dropwise until a white gel formed at pH 7 [25]. The silica gel was allowed to precipitate out of the solution for 24 hours, and then filtered and extensively rinsed with deionized water.

The silica gel was subsequently dried in an oven at 100-110 °C for 24 h [22]. The SiO_2 was now in its pure form. The mass of pure SiO_2 obtained from this process was 8.4 g (yield = 8.4 %). SEM-EDS, FTIR, XRD, XRF, and DSC analyses were used to evaluate the quality of SiO_2 obtained from the river sand.

This research was performed using green synthesis methods that included minimizing waste, making processes as efficient as possible, and using environmentally-friendly waste treatment. Concentrated HCl (acidic) and NaOH (alkaline) solutions were used during the coprecipitation-based extraction process. Any liquid waste was treated to a near-neutral pH before it was disposed of according to normal laboratory safety practices and local regulatory requirements for the disposal of institutional waste. After being treated to a near-neutral pH, all the liquid waste was treated in accordance with established environmental procedures. Although this process used strong acids and bases, the coprecipitation method required no toxic organic solvents, generated very little solid waste when producing high-purity SiO_2 relative to other extraction methods, and operated at much lower temperatures than most high-temperature extraction methods. Therefore, while the process could be described as "feasible" and "manageable", it could not be called "green".

RESULTS AND DISCUSSION

XRF Analysis

The results from Table 1 show that the purity of SiO_2 increased substantially from 80.91 % (river sand) to 99.41 % (extracted SiO_2). These results were confirmed by a previous study [26], which demonstrated that SiO_2 with a purity greater than 99 % could be achieved using a process that included an acid leach, followed by gel formation of a solution of sulfuric acid and sodium silicate prepared from river sand. Using a similar method, Al-Rubaiey and Al-Qaisi [27] successfully reduced impurity levels in their river-sediment samples by using aluminium powder and sulfuric acid, achieving a purity of 98.9 %. The SiO_2 extracted in this study had all but eliminated the impurities present in the river sand, including Al_2O_3, Fe_2O_3, K_2O, and CaO. For example, Fe_2O_3 was reduced from 3.36 % in river sand to 0.03 % in the extracted SiO_2, and K_2O from 2.98 % to 0.15 %₂. Although trace amounts of P_2O_5, Cl, and MnO remained in the extracted SiO_2, these impurities did not contribute to the purity of the extracted SiO_2.

Table 1. Chemical composition of river sand and extracted SiO_2.

Component	Percentage (wt%)	
	Sand	**Extracted SiO_2**
SiO_2	80.91	99.41
Al_2O_3	10.50	BDL
Fe_2O_3	3.36	0.03
K_2O	2.98	0.15
CaO	1.16	0.10
TiO_2	0.50	BDL
P_2O_5	0.19	0.21
Cl	0.12	0.08
MnO	0.10	0.01
Cr_2O_3	0.04	BDL
ZrO_2	0.04	BDL
BaO	0.04	BDL
Rb_2O	0.02	BDL
SrO	0.01	BDL
NiO	0.01	BDL
ZnO	0.01	BDL

Note: *BDL = below detection limit; elements reported as BDL were present at concentrations lower than the detection capability of the XRF instrument used in this study.*

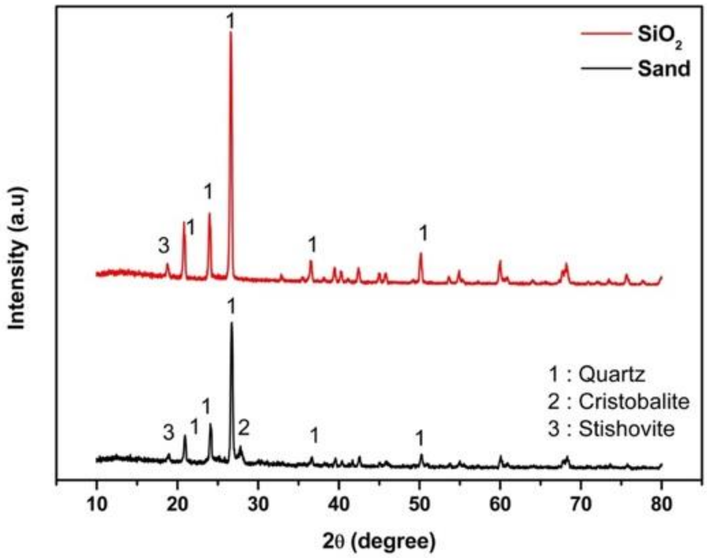

Figure 1. XRD diffractogram of river sand and extracted SiO_2 samples.

The relative simplicity of the coprecipitation technique used in this research makes it economical and easy to use in a typical laboratory, in comparison to Boualem et al. [26], who utilized a complex method (acid treatment, gelation) demanding considerable experience and instrumentation. Al-Rubaiey and Al-Qaisi [27] used a high-temperature reduction-leaching technique, which is expensive and complicated due to the high temperature and use of reactive chemical agents.

XRD Characterization

XRD analysis of the river sand and isolated SiO_2 showed that both samples primarily contained quartz (SiO_2) [28]. The results are presented in Figure 1. The three highest intensity peaks for SiO_2 in the river sand sample were located at 2θ = 20.9°, 24.1°, and 26.7°, whereas the peaks for the isolated SiO_2 were located at 2θ = 20.8°, 24.0°, and 26.6°. The small variations in the positions of the peaks, along with differences in intensity, suggest that the leaching and purification process was successfully carried out [29]. The location of the peaks further supported the presence of silica in the isolated sample and confirmed that the prepared silica was predominantly composed of crystalline quartz, as evidenced by the sharp diffraction peaks at 2θ ≈ 20.8°, 24.0°, and 26.6° [28, 30]. In addition, the presence of a broad background signal suggested a minor fraction of amorphous silica within the material [30, 31]. It should be noted that the region between 20° and 30° is characteristic of silica, resulting from the distortion of quartz and cristobalite forms [32]. The XRD analysis therefore demonstrated that pure silica could be synthesized from river sand using a simple and environmentally friendly technique.

FTIR Characterization

FTIR spectra were recorded in the wavenumber range of 4000–400 cm^{-1}, with a resolution of 4 cm^{-1} and 32 scans per sample. The samples were prepared using the KBr pellet method prior to analysis. Figure 2 shows the FTIR spectra of the river sand and the extracted silica. In the river sand, an absorption band at 3451 cm^{-1} was observed, which corresponded to the –OH functional group [33]. The absorption band at 1615 cm^{-1} indicated the presence of Si–OH groups. Additionally, peaks at 1031 and 788 cm^{-1} were attributed to asymmetric Si–O stretching and symmetric Si–O–Si stretching, respectively [34, 35]. In the FTIR spectrum of the extracted SiO_2, a shift in the absorption bands was observed: the –OH group shifted from 3451 cm^{-1} to 3430 cm^{-1}, and the Si–OH group shifted from 1615 cm^{-1} to 1609 cm^{-1} [36]. Finally, the asymmetric Si–O stretching and symmetric Si–O–Si stretching shifted from 1031 and 788 cm^{-1} to 1103 and 791 cm^{-1}, respectively [37, 38]. These results indicated the successful extraction of SiO_2 from the river sand.

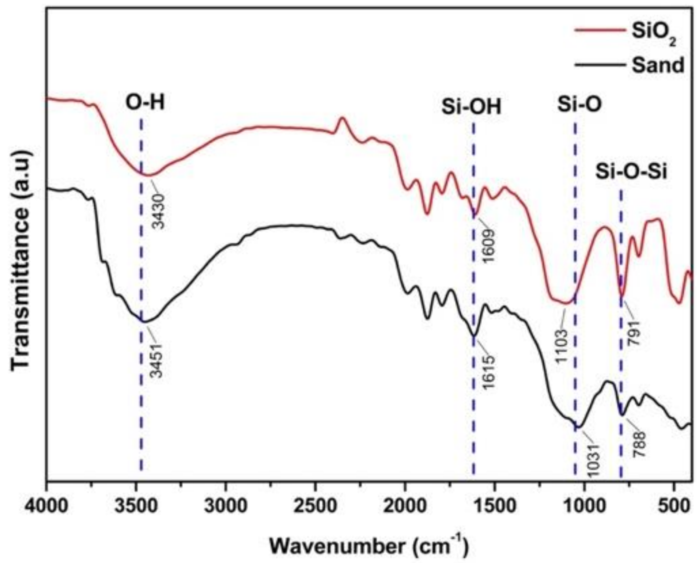

Figure 2. FTIR spectra of river sand and extracted SiO_2 samples.

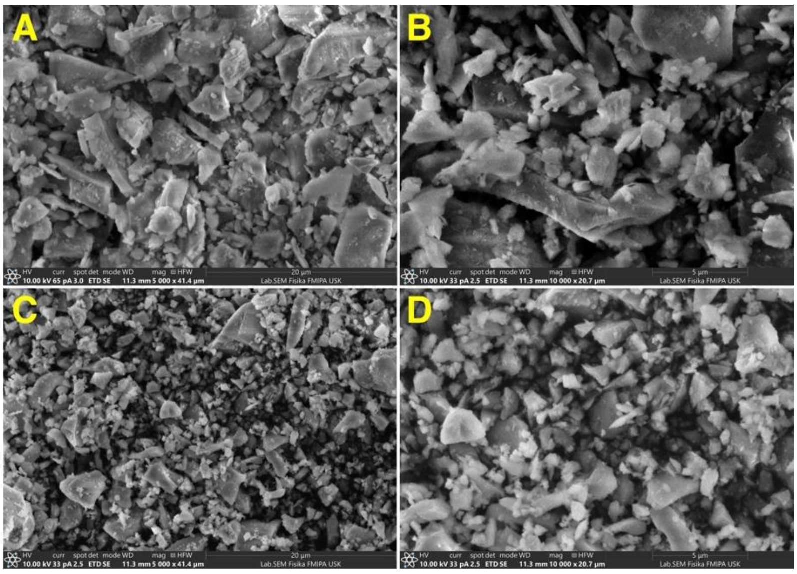

Figure 3. SEM morphology of river sand (A. 5000×, B. 10,000×) and extracted SiO_2 (C. 5000×, D. 10,000×) samples.

SEM and EDS Surface Morphological Analysis

The SEM images in Figure 3 illustrate the morphological characteristics of both the river sand and the extracted SiO_2 at various magnifications. At a magnification of 5000×, the river sand displayed an irregular, coarse morphology characterized by angular particles and a high degree of surface roughness, suggesting little to no weathering had occurred, or that it was transported for a very short distance. While the grain size distribution of the sand appeared to be fairly uniform, there were several large grains observed. The higher magnification of 10,000× provided significantly greater detail regarding the texture of the sand; all individual grains had irregularly shaped surfaces and small microcracks/crevices that appeared consistent with naturally formed sand grains, suggesting the presence of impurities/mineral inclusions throughout the sand.

Compared with the raw SiO_2, the isolated SiO_2 had a much finer, more consistent texture at 5000×. The SiO_2 particles appeared to be much smaller, more angular, and evenly dispersed throughout the sample area. At high magnification (10,000×), there also seemed to be very few surface defects on the SiO_2

particles. Overall, these images showed that the SiO_2 particles were chemically pure, and had sharp edges. The consistent structure, combined with the delicacy and homogeneity of the isolated SiO_2, indicated that the extracted material could be of high purity [21, 26, 39].

The EDS spectra of the river sand and extracted SiO_2 samples are presented in Figure 4, while the corresponding elemental compositions are summarized in Table 2. The river sand was found to contain approximately 57.4 wt% (± 0.6 %) of oxygen, and approximately 22.2 wt% (± 0.3 %) of silica. Other elements present included carbon (9.7 wt% ± 0.6 %), aluminium (5.5 wt% ± 0.1 %), iron (2.2 wt% ± 0.1 %), potassium (1.6 wt% ± 0.0 %), sodium (0.9 wt% ± 0.1 %), and calcium (0.5 wt% ± 0.0 %). The sand contained significant amounts of silica (SiO_2) along with other mineral impurities such as aluminosilicates, iron oxides, and minor elements. The significant amount of C in the sand indicated that it likely contained organic material or carbonates.

In the extracted SiO_2 the amount of Si increased to 37.8 wt% (± 0.2%), while oxygen decreased to 57.0 wt% (± 0.3%) and carbon to 5.2 wt% (± 0.2%). This showed that the extraction process was successful in concentrating SiO_2 by eliminating much of the impurities. Though some carbon remained in the product after extraction, it was considerably less compared to the unprocessed sand; thus, the extraction process was effective at reducing the quantity of organic impurities and carbonates within the material. As a result, the extraction process yielded a substantial increase in SiO_2 purity.

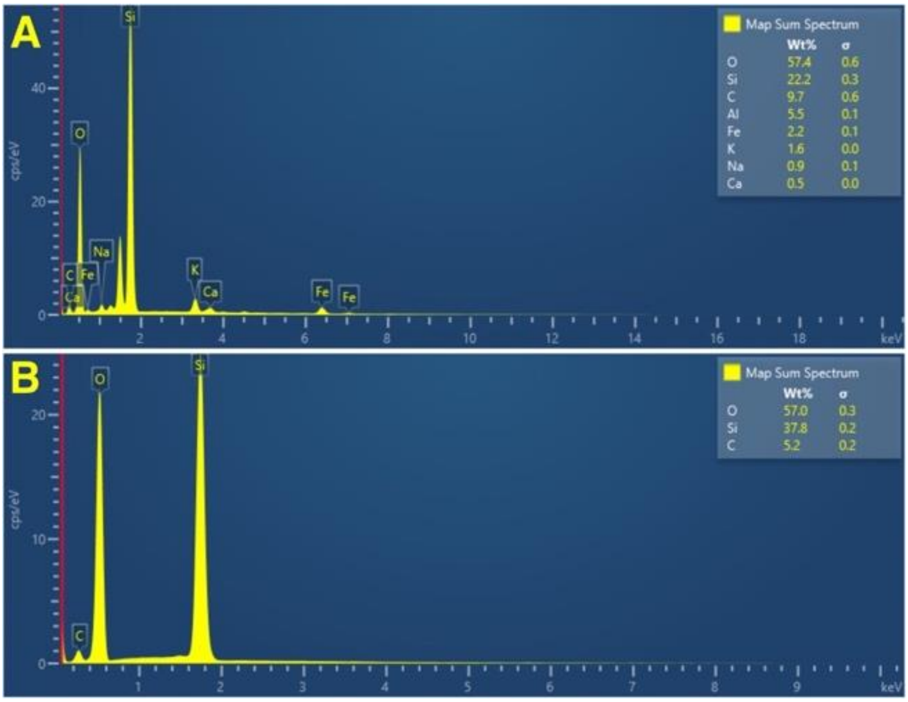

Figure 4. EDS analysis of river sand (A) and extracted silica (B) samples, with their chemical composition.

Table 2. Elemental composition of raw river sand and extracted silica as determined by EDS.

Element	River Sand (wt ± SD) %	Extracted SiO_2 (wt ± SD) %
O	57.4 ± 0.6	57.0 ± 0.3
Si	22.2 ± 0.3	37.8 ± 0.2
C	9.7 ± 0.6	5.2 ± 0.2
Al	5.5 ± 0.1	BDL
Fe	2.2 ± 0.1	BDL
K	1.6 ± 0.0	BDL
Na	0.9 ± 0.1	BDL
Ca	0.5 ± 0.0	BDL

Note: *BDL: below detection limit*; *elements reported as BDL were present at concentrations lower than the detection capability of the EDS instrument used in this study.*

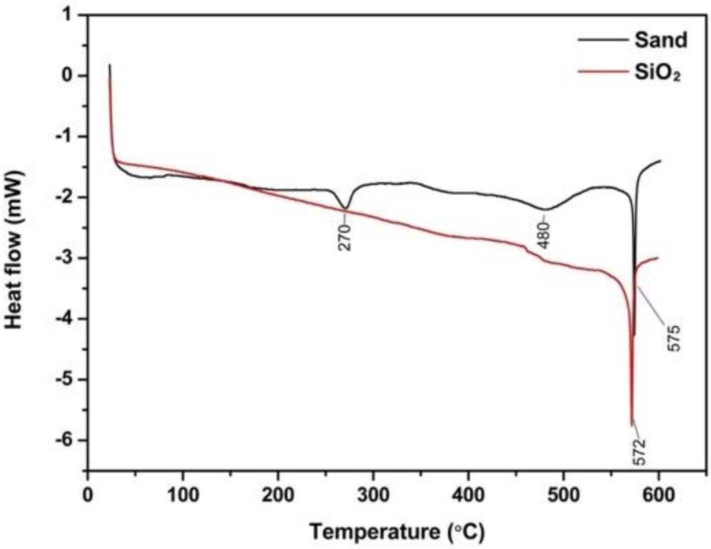

Figure 5. DSC curves of the river sand and extracted SiO_2 samples.

DSC Analysis

The thermal characteristics of the river sand and the extracted SiO_2 were evaluated by analysing their DSC curves over the temperature range of 25 - 600 °C, as shown in Figure 5. The DSC curves for both the river sand and the extracted SiO_2 were analysed at a constant heating rate of 10 °C/min to ensure comparison of their behaviours. The DSC trace of the river sand sample displayed three sequential exothermic peaks with centres at 270, 480, and 575 °C, which may be associated with the structural rearrangements and phase-related transformations of mineral components present in the sand [40, 41]. In contrast, the extracted SiO_2 showed a single dominant exothermic peak at around 572 °C, indicating a simpler thermal behaviour after impurity removal [41, 42]. Furthermore, the slight shift of the exothermic peak maxima from 575 °C for the river sand to 572 °C for the extracted SiO_2 could suggest a very minor amount of mass loss attributed to the degradation of inaccessible hydroxyls [43]. SiO_2 showed a higher heat capacity than river sand, as indicated by the heat flow rate [35, 42].

CONCLUSION

The coprecipitation method used in this study successfully isolated silica (SiO_2) from a sample of sand collected from the Silau River. The results obtained from the pure silica extracted by the coprecipitation method were better than those of the pure silica in the original sand, as demonstrated by XRF, SEM, FTIR, XRD, and DSC analyses. The extracted SiO_2 samples had a significantly higher purity (99.41 %) compared to the original sand (80.91 %). SEM images suggested increased particle uniformity after the extraction process. Analysis of the FTIR spectra showed that many absorption peaks produced by impurities which were present in the original sand were absent in the extracted samples, indicating that they were removed during the extraction process. XRD analysis confirmed that the extracted SiO_2 consisted predominantly of quartz with a minor degree of amorphous content. DSC results indicated that the extracted SiO_2 had improved thermal stability and exhibited a nearly flat thermal profile. Collectively, these results confirmed that the coprecipitation method was an efficient means of extracting high-purity SiO_2 from Silau River sand. Additionally, the physical properties of the extracted SiO_2 indicate that it may be a potential value-added product for several applications, such as industrial production or environmental remediation. Therefore, this study suggests that river sands may provide a viable source of high-quality SiO_2 for certain industries, including materials science and photocatalysis.

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