CHARACTERIZATION OF SILICA (SiO_2) BASED ON BEACH SAND FROM SULAWESI AND SUMATRA AS SILICON CARBIDE (SiC) BASE MATERIAL

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ABSTRACT

Silica (SiO_2) is a material that has become famous because of its profound benefits in various fields. Its presence is abundant in nature and is associated with other compounds so that silica can be produced by extraction. This study aims to characterize silica based on coastal sand from Sulawesi and Sumatra as the base material for silicon carbide (SiC). Extraction was carried out using the alkali fusion method. The tests used were X-Ray Fluorescence, Fourier Transform Infrared, and X-Ray Diffraction to determine the elemental composition of Si, functional groups, and silica crystal structure. The results show that the beach sand from Sumatra has more silica content than the beach sand from Sulawesi. Silica has silanol (Si-OH) and siloxane (Si-O-Si) functional groups. The crystal structure obtained is hexagonal. Thus, beach sand from Sumatera can be used as a source of silica as the base material for SiC.

Keywords: beach sand, silica, alkali fusion, silicon carbide.

INTRODUCTION

Indonesia has many beaches which are the source of white sand, such as the coasts of Sulawesi and Sumatra. The abundant natural resources of quartz sand have yet to be utilized better. Quartz sand characteristic in this area has white sand. It allows extensive SiO_2 content [1, 2]. The white sand is one of the natural resources that have the potential to be used as a material of economic value. Beach sand contains silica which is very useful in nanotechnology, especially in synthesizing nano silica [3 - 5].

Silica sand is one of the mineral materials whose presence in nature is abundant and can be used in various applications. For example, industrial activities are used according to their characteristics, such as glass-making products, ceramics, clean water production filters, concrete casting, sandblasting to clean machine descaling, pipes, plates, etc. [6, 7].

Silica is a chemical compound with the molecular formula SiO_2, which is the product of magma freezing. Quartz, tridymite and cristobalite are polymorphic forms of silica which can be differentiated through their stability to changes in temperature, namely: quartz, up to 870°C, tridymite, at 870 °C - 1470°C, cristobalite, at 1470°C up to 1730°C melting point. Silica is one of the most well-known crystal modifications known as quartz. One of the silica compounds that can be used as silicon raw material for the manufacture of solar panels is quartz [8]. Quartz sand contains minerals resulting from weathering, whereas the filler material for quartz sand is a mineral containing crystalline silica (SiO_2) [9].

Compounds such as SiO_2, CaO, Fe_2O_3, TiO_2, CaO, and MgO are compounds in quartz sand, with the main compound being silica (SiO_2). The impurity material acts as a colouring agent for silica sand; from this colour, the percentage degree of purity of quartz sand can be estimated. The physical properties of beach sand, in this case, quartz sand, have distinctive characteristics, namely clear white colour or other colours depending on the impurity compounds, hardness ranging from 7 (Mohs scale), density 2.65 g cm^{-3}, the melting point between 1715°C, crystal structure hexagonal, and heat conductivity between 12°C - 100°C [10].

Silica minerals are sought to be separated from other elements and improved in various high-tech applications. In order to maximize the use of these materials, high purification technology is needed. Silica purification is usually done by adding hydrochloric acid (HCl). This purification produces silica with a content of 99.99 %. Synthesis of amorphous silica from deuriet sand by reacting sodium carbonate (Na_2CO_3) has been carried out at a combustion temperature of 1030°C [8]. With the alkaline fusion method, Munasir produced silicon dioxide (SiO_2) from sloped sand with a purity of 98 % [3].

Another method that can be used to synthesize quartz sand is the co-precipitation method. The co-precipitation method is a bottom-up synthesis method used to manufacture nanoparticles. This method has the advantage that it can produce particles with tiny grain sizes and low energy consumption (< 100°C), and low cost [11].

The SiO_2 content obtained in the sand is more than 50 %, called quartz sand. SiO_2 compounds have functional group characteristics, such as Silica having silanol (Si-OH) and siloxane (Si-O-Si) functional groups [12]. Also, Si-OH groups are water molecules on the surface which are adsorbed by silica from the environment [13].

Silicon carbide (SiC) is a non-oxide ceramic material with good physical and chemical properties such as high hardness, melting point, decomposition temperature, and thermal conductivity. Quartz sand which contains much silica, can be used as the base material for SiC (silicon carbide). In contrast, carbon (C) can be obtained from coconut shells, coconut midribs, and also rice husks [14 - 16]. In addition to acting as a ceramic material, SiC can act as a semiconductor material and composite reinforcement to be applied to optoelectronic, abrasive, and nuclear [17, 18].

The methods used to synthesize SiC include the carbothermal reduction method, sol-gel, magnesiothermic reduction, and solid reactions. The carbothermal reduction method reduces silica with carbon at a temperature of around 1500°C - 2000°C [19]. The magnesiothermic reduction method makes SiC at low temperatures, around 550°C - 1000°C [20, 21]. Solid reaction method for manufacturing SiC with a sintering temperature of 750°C - 1400°C [22, 23]. Meanwhile, the manufacture of SiC using the sol-gel method was observed at a temperature of 1400°C - 1580°C [24].

In general, SiC can be formed at temperatures around 1200°C - 1400°C. High temperatures synthesizing SiC require much electrical energy, so the component operating costs are expensive. However, with the magnesiothermic reduction method, SiC can be synthesized at lower temperatures with the help of a magnesium catalyst which can reduce silica. Therefore, synthesizing SiC with magnesiothermic reduction can reduce energy requirements [20].

The potential of silica is helpful in various fields, so in this paper, the coastal sand from Sulawesi and Sumatra is reported to study the characterization of beach sand to produce SiO_2 compounds using the alkali fusion method. Thus, this study aims to characterize silica from beach sand as the base material for silicon carbide (SiC). The X-Ray Fluorescence (XRF), Fourier Transform Infrared (FT-IR) and X-Ray Diffraction (XRD) tests were used to characterize silica properties such as Si elemental composition, functional groups, and crystal structure.

EXPERIMENTAL

Materials

Beach sand from Sulawesi and Sumatra, HCl 20 % (hydrochloric acid), 5 mol dm^{-3} NaOH (sodium hydroxide) and equates.

Sample Preparation

Beach sand with distilled water and cleaned or separated from impurities and then dried for 1 - 2 days in direct sunlight. The beach sand was ground using a mortar and pestle and then sieved using a 40 mesh sieve [25].

Identification of Beach Sand Content

A total of 50 g of sieved beach sand powder was soaked with 100 cm^3 of 20 % HCl in a beaker glass for 14 hours to dissolve various impurities in the sand. The choice of 20 % HCl with a concentration of 6.5 mol

dm^{-3} follows previous research by Langi et al. [25]. 100 cm^3 of 20 % (6.5 mol dm^{-3}) hydrochloric acid solution was obtained by diluting 37 % (11.9 mol dm^{-3}) hydrochloric acid with a volume of 54.05 cm^3. Next, the sand is washed using distilled water until the washing water becomes neutral, and then the sand is dried. Furthermore, XRF, FTIR, and XRD tests were carried out to obtain information on the beach sand samples' Si content, functional groups, and crystal structure.

Silica Powder Extraction

The extraction process uses the alkaline fusion method [26]. This method dissolves 10 g of purification with 100 cm^3 of 5 mol dm^{-3} NaOH. Next, samples were stirred for 4 hours and heated at 80°C to form sodium silicate (Na_2SiO_3) or until white gels were formed. The gel solution was titrated using 2 mol dm^{-3} HCl (acid) until the solution had a neutral pH.

Further, the sample was washed with distilled water to remove the NaCl formed during the synthesis process, filtered using filter paper and dried at room temperature. The dried gel was then ground to get a powder. Furthermore, the extracted samples were tested for XRF, FT-IR, and XRD to obtain information on Si content, functional groups, and crystal structure of beach sand samples.

Analysis of XRF, FT-IR, and XRD

The samples used for XRF, FT-IR, and XRD analysis are in solid form. In XRF measurements, samples are prepared by pulverizing and forming into pellets. Next, XRF measurement using a Shimadzu Uniquant'X XRF type which works at a voltage of 50 kV. XRF analysis with PCx Uniquant software identifies elements and oxides contained in samples. The samples used in FT-IR measurements were prepared using the KBr pellet method. 1 - 10 mg. The sample is carefully pulverized with 100 mg KBr and scored into thin discs or pellets. Then, FT-IR measurements using the Shimadzu FT-IR Spectrophotometer IRprestige-21 type. This tool works on a scan range of 4000 cm^{-1} - 340 cm^{-1}, resolution 4 cm^{-1}, and scans 2 - 3 sec. FT-IR spectrum measurement and peak detection were analyzed using IR solution software.

XRD tool with the Rigaku MiniFlex II type is used for XRD measurements. This tool works at a current of 15 mA, a voltage of 30 kV, a scan rate per time of 4° per minute, a scan width of 0.02°, and a scanning interval of 5° - 90°. In XRD measurements, samples were prepared using the bulk powder method. The search and match method uses the engine's built-in software, PDXL2, equipped with a 2011 version of the ICDD (International Center for Diffraction Data) card to obtain qualitative results. Meanwhile, the RIR (Reference Intensity Ratio) method is used to obtain quantitative results [27, 28].

RESULTS AND DISCUSSION

X-Ray Fluorescence (XRF) Analysis

X-Ray Fluorescence is an analytical technique that aims to analyze the elements that make up a material. The results of the XRF analysis of beach sand samples from Sulawesi and Sumatra are shown in Tables 1 and 2. Table 1 shows that the element content in beach sand samples from Sulawesi is Ca, followed by Fe and Si. The Si content before and after extraction was 10.57 % and 10.39 %, respectively. In the form of compounds obtained SiO_2 before and after extraction, respectively,

Table 1. XRF analysis results from Sulawesi beach sand.

Oxide	Before extraction (% mass)	After extraction (% mass)	Element	Before extraction (% mass)	After extraction (% mass)
CaO	49.39	50.56	Ca	53.23	54.46
Fe_2O_3	22.43	21.48	Fe	25.93	24.82
SiO_2	17.26	16.96	Si	10.57	10.39
Na_2O	5.22	4.90	Na	5.02	4.69
Al_2O_3	2.45	2.88	Al	1.83	1.96
TiO_2	1.55	1.50	Ti	1.67	1.46
K_2O	1.35	0.88	K	1.31	1.08

Table 2. XRF analysis results from Sumatera beach sand.

Oxide	Before extraction (% mass)	After extraction (% mass)	Element	Before extraction (% mass)	After extraction (% mass)
SiO_2	87.66	87.24	Si	73.85	72.47
CaO	5.09	7.04	Ca	9.98	14.87
K_2O	3.47	3.07	K	7.89	6.88
TiO_2	1.31	0.95	Ti	2.54	1.87
Fe_2O_3	1.11	0.81	Fe	2.49	1.84

17.26 % and 16.96 %.

Meanwhile, the data from the XRF analysis of beach sand from Sumatra is shown in Table 2. Both before and after the extraction was the highest Si element content. The Si content before extraction was 73.85 %; after extraction, it was 72.47 %. Meanwhile, the SiO_2 oxide content before extraction was 87.66 %; after extraction, it was 87.24 %. The SiO_2 content is higher in the beach sand from Sumatera compared to the beach sand from Sulawesi. The high content of CaO in coastal sand from Sulawesi can be further exploited to be used as an innovative material.

Tables 1 and 2 also show that the alkali fusion method as the extraction method used in this study showed a decrease in Si content in both the coastal sand samples from Sulawesi and Sumatra, but not significantly. It is suspected that in the extraction process, with the addition of 100 cm^3 of 5 mol dm^{-3} NaOH, there is soluble SiO_2. In addition to SiO_2, oxides have decreased in content, such as Fe_2O_3, Na_2O, and K_2O. The decrease in the content of these oxides and the increasing content of oxides such as CaO, Al_2O_3, and TiO_2.

Based on this study, beach sand from Sumatra can be called quartz sand. This is due to the composition of its constituent oxides having a SiO_2 content that is more dominant than others or having a SiO_2 content of more than 50 %. Similar results have been obtained by Langi et al. that the beach sand from Marinsow Village, Minahasa Regency, is dominated by CaO while the SiO_2 content is low at 1.41 % [25]. Joni and Ariyanto also found sand on Camplong beach, dominantly Si, while on the coast of Nepa and Mandangin island, Ca dominant [29]. In addition, beach sand in Samadua District, South Aceh Regency, was found to be dominated by Si elements [30]. White sandy beaches or calcite sand are formed from an abundance of organisms, namely the shells of marine animals that form sand deposits.

Meanwhile, silica sand is formed from weathered rock and contains silica compounds. Then, this sand is carried away by the water and settles on the beach. Silica sand has a characteristic clear color, and consists of oxides of SiO_2, Al_2O_3, CaO, Fe_2O_3, MgO, and K_2O. The purity of beach sand varies depending on the formation process [31].

Fourier Transform Infrared (FT-IR) analysis

The FT-IR spectra of beach sand samples from Sulawesi and Sumatra are shown in Figs. 1 and 2.

The FT-IR spectra of coastal sand samples from Sulawesi before and after extraction have almost the same pattern (Fig. 1): the increase in the intensity of the absorption band after extraction at wavenumbers of 3446.79 cm^{-1}, 1643.35 cm^{-1}, 999.13 cm^{-1}, 773.46 cm^{-1}, 372.26 cm^{-1}. decreased absorption band intensity after extraction is at a wavenumber of 582.50 cm^{-1}. The absorption band at a wavenumber of 1159.22 cm^{-1} disappeared after extraction. In addition, there was a shift in the absorption peaks, namely 3458.37 cm^{-1} to 3446.79 cm^{-1}, 1633.71 cm^{-1} to 1643.35 cm^{-1}, 933.55 cm^{-1} to 999.13 cm^{-1}.

Similarly, the FT-IR spectrum on the beach sand samples from Sumatra before and after extraction has almost the same pattern (Fig. 2), decreased absorption band intensity after extraction at wavenumbers of 3435.22 cm-1, 1610.56 cm^{-1}, 1082.07 cm^{-1}, 777.31 cm^{-1}, 460.99 cm^{-1}. After extraction, absorption occurred at a wavenumber of 867.97 cm^{-1}. In addition, there was a shift in the absorption peaks, namely 3454.51 cm^{-1} to 3435.22 cm^{-1}, 1622.13 cm^{-1} to 1610.56 cm^{-1}, 1080.14 cm^{-1} to 1082.07 cm^{-1}, 779.24 cm^{-1} to 777.31 cm^{-1}, and 459.06 cm^{-1} to 460.99 cm^{-1}.

The decrease in the intensity of the absorption band

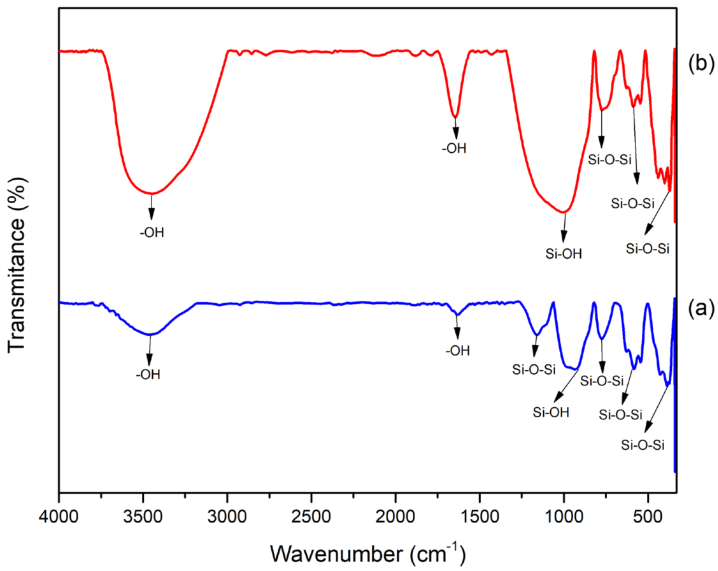

Fig. 1. FT-IR spectrum of Sulawesi beach sand (a) before extraction and (b) after extraction.

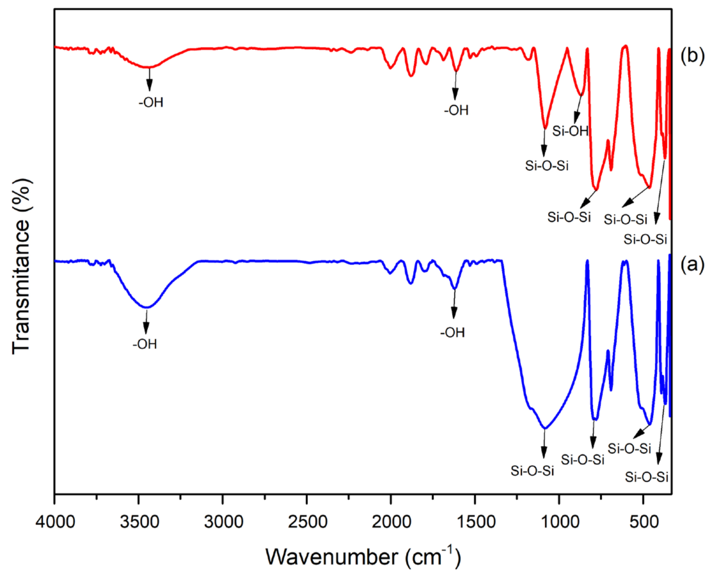

Fig. 2. FT-IR spectrum of Sumatra beach sand (a) before extraction and (b) after extraction.

Table 3. Interpretation of FT-IR spectrum from Sulawesi beach sand.

Wavenumber (cm^{-1})		Functional groups
Before extraction	After extraction	
3458.37	3446.79	–OH stretching vibration of Si-OH
1633.71	1643.35	–OH bending vibration of H_2O
1159.22	-	Si-O asymmetric stretching vibration of Si-O-Si
933.55	999.13	Si-O symmetric stretching vibration of Si-OH
773.46	773.46	Si-O symmetric stretching vibration of Si-O-Si
582.50	582.50	Si-O-Si bending vibration
387.69	372.26	Si-O-Si bending vibration

Table 4. Interpretation of FT-IR spectrum from Sumatra beach sand.

Wavenumber (cm^{-1})		Functional groups
Before extraction	After extraction	
3454.51	3435.22	–OH stretching vibration of Si-OH
1622.13	1610.56	–OH bending vibration of H_2O
1080.14	1082.07	Si-O asymmetric stretching vibration of Si-O-Si
-	867.97	Si-O symmetric stretching vibration of Si-OH
779.24	777.31	Si-O symmetric stretching vibration of Si-O-Si
459.06	460.99	Si-O-Si bending vibration
388.40	370.33	Si-O-Si bending vibration

and the lost absorption indicates a reduction in impurities in the beach sand sample. The appearance of new peaks after extraction shows the results of applying the extraction method, namely the alkali fusion method, to obtain silica with a high level of purity. The occurrence of a shift in wavenumber is influenced by impurities contained in the sample [32].

The absorption bands that appear in the FT-IR spectrum of beach sand samples from Sulawesi and Sumatra are shown in Tables 3 and 4. The alleged absorption bands identified are -OH stretching vibrations from Si-OH at a wavenumber of about 3400 cm^{-1}, -OH bending vibrations from H_2O at a wavenumber of about 1600 cm^{-1}, Si-O asymmetric stretching vibration of Si-O-Si at a wavenumber of about 1000 cm^{-1}, Si-O symmetric stretching vibration of Si-OH at a wavenumber of about 800 cm^{-1} - 900 cm^{-1}, symmetrical stretching vibration of Si-O-Si at a wavenumber of about 700 cm^{-1}, Si-O-Si bending vibration at wavenumbers around 300 cm^{-1} - 500 cm^{-1}. Based on this study, it was found that in silica, there are two types of dominant functional groups, namely the silanol group (Si-OH) and the siloxane group (Si-O-Si) [33, 34]. The reason for the existence of Si-OH is that water molecules are physically absorbed from the environment by silica [35].

X-Ray Diffraction (XRD) Analysis

XRD diffractograms of beach sand from Sulawesi and Sumatra are shown in Figs. 3 and 4. The analysis results are compared for the diffraction patterns before and after extraction. The diffractogram of the obtained beach sand extraction is matched with the ICDD (International Center for Diffraction Data) contained in the PDXL2 software built into the XRD machine. The diffractogram shows that the beach sand samples from Sulawesi and Sumatra contain crystalline compounds. The diffractogram shows the sample with a crystal structure indicated by the presence of peaks at a certain angle. That means the absorption occurs at a certain angle which is the character of the crystalline phase in the natural sand sample.

Fig. 3 shows the highest intensity before extraction at $2\theta = 28.579^o$, while after extraction, $2\theta = 28.592^o$ based on the search and match results with ICDD 01-077-3162. This absorption indicates that low quartz (SiO_2) or silica compounds are found. The X-ray diffraction pattern of the extracted silica is characterized by the appearance of a 2θ angle diffraction peak at about 23° [36]. Naturally, silica is crystalline and has various crystalline forms [37, 38]. The silica content increased from 3.6 % (before extraction) to 43.3 % (after extraction) (Table 6). Table 6 also shows other phases found in natural sand samples before extraction, namely magnetite (Fe_3O_4) at an absorption peak of $2\theta = 38.488^o$, magnesium oxide (MgO) at an absorption peak of $2\theta = 52.354^o$, calcite ($CaCO_3$) at an absorption peak of $2\theta = 30.454^o$, scandium (Sc) at the absorption peak $2\theta = 48.511^o$. Other phases found in the natural sand samples after extraction were hematite (Fe_2O_3) at an absorption peak of $2\theta = 63.966^o$ and periclase (MgO) at an absorption peak of $2\theta = 42.784^o$ (Table 5). In Fig. 3, there are also absorption peaks whose phase was not identified by the database. The sample is suspected of containing still other elements that are not soluble in acid and are also not lost in the extraction process [39, 40].

Meanwhile, Fig. 4 shows that beach sand from Su-

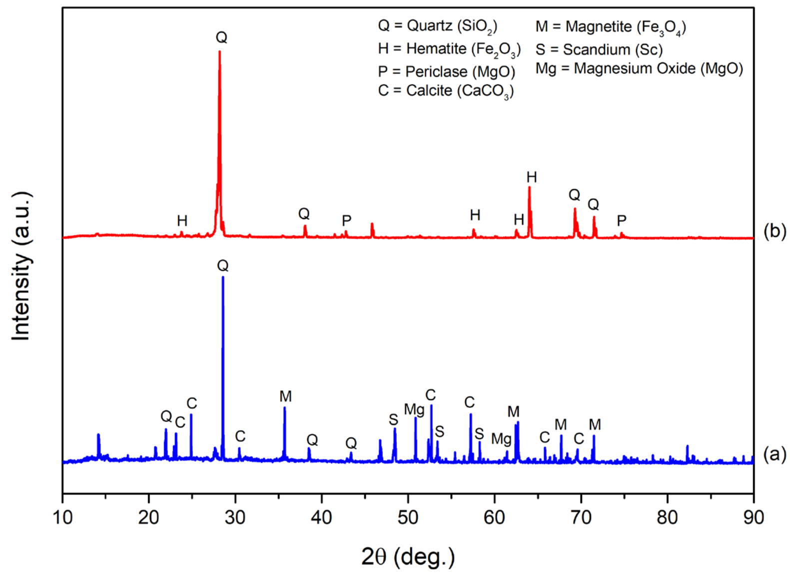

Fig. 3. XRD diffractogram of Sulawesi beach sand (a) before extraction and (b) after extraction.

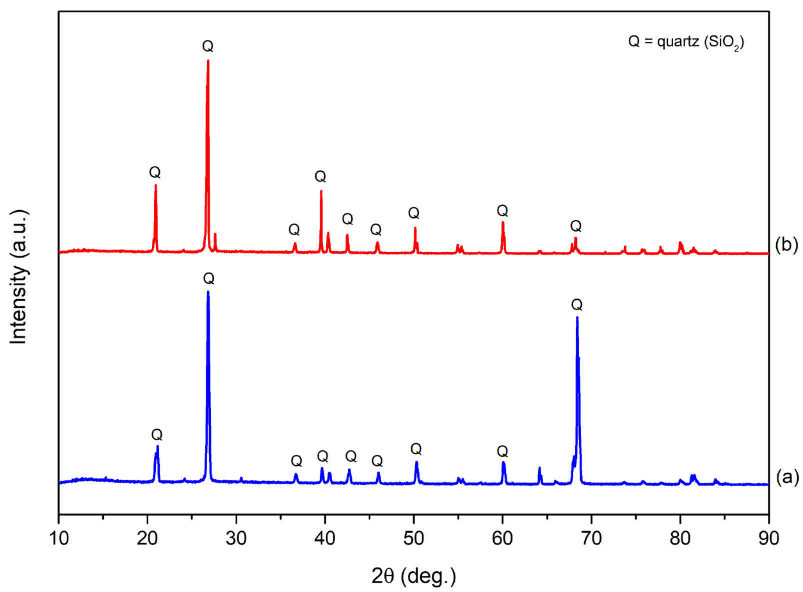

Fig. 4 XRD diffractogram of Sumatra beach sand (a) before extraction and (b) after extraction.

matra identified a single phase, namely low quartz (SiO_2) or silica phase, where the highest absorption peaks were at 2θ = 26,808° (before extraction) and 2θ = 26,818° (after extraction). The silica content in beach sand from Sumatera before and after extraction was identified as 100 % (Table 6). These results are relevant to the XRF results, which show that the SiO_2 oxide is dominant in the sample. XRD did not identify the discovery of other elements from XRF in the form of compounds, allegedly not in the database. Both beach sand from Sulawesi and Sumatra were identified as having a low quartz phase; it is suspected that the silica extraction process was applied to heating at a low temperature of 80°C. In this study, hydrochloric acid immersion treatment and extraction by alkali fusion method did not affect the crystal structure but the chemical composition. Also, it results in a hexagonal-shaped silica crystal structure where the hexagonal crystal system has the characteristic lattice parameters a = b ≠ c and α = β = 90°, γ = 120° [41]. In addition, there is a shift to the left in the XRD curve.

Table 5. Results of XRD analysis of Sulawesi beach sand.

Phase name	Formula	Content (% mass)	
		Before extraction	After extraction
Quartz	SiO_2	3.6	43.3
Hematite	Fe_2O_3	-	55.4
Periclase	MgO	-	1.29
Magnetite	Fe_3O_4	1.6	-
Magnesium	MgO	2.5	-
Calcite	$Ca(CO_3)$	86	-
Scandium	Sc	6.1	-

Table 6. Results of XRD analysis of Sumatera beach sand.

Phase name	Formula	Content (% mass)	
		Before extraction	After extraction
Quartz	SiO_2	100	100

Table 7. Silica phase lattice parameters in beach sand.

Lattice paraeters	Before extraction		After extraction	
	Sulawesi	Sumatra	Sulawesi	Sumatra
a (A)	4.66	4.89	4.74	4.90
b (A)	4.66	4.89	4.74	4.90
c (A)	4.92	5.39	4.98	5.41
α (deg.)	90	90	90	90
β (deg.)	90	90	90	90
γ (deg.)	120	120	120	120

It is due to the size of the silica crystals experiencing expansion [40]. This result follows Table 7, where silica crystals' lattice parameters a, b, and c increased after extraction.

CONCLUSIONS

Silica extraction using the alkali fusion method has been successfully carried out so that silica (SiO2) is obtained in coastal sand from Sulawesi and Sumatra. Beach sand from Sumatra contains more silica than beach sand from Sulawesi. Beach sand samples containing silica have the characteristics of silanol (Si-OH) and siloxane (Si-O-Si) functional groups. A quartz (SiO2) phase was found in the beach sand samples with a hexagonal crystal form. Thus, beach sand from Sulawesi and Sumatra has the potential as a base material to be applied as a silicon carbide (SiC) functional material.

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