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Synthesis and Characterization of Nanosilica (SiO₂) Volcanic Rock of Mount Batur in Bali

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Abstract

This research was conducted to synthesize and characterize silica minerals (SiO₂) from volcanic rocks in the active volcano in Bali, namely Mount Batur. The synthesis carried out on five different color variants of this rock sample is by the coprecipitation method which begins with the process of taking rock samples on Mount Batur, crushing the rock until it becomes powder with a size of 100 mesh, washing with distilled water and drying, immersing the rock powder in the solution. 2 M HCl for 12 hours, then the results of the soaking were reacted again with 7 M NaOH solution as a hydrolysis process to obtain pure SiO_2 in the sample. In the form of sodium silicate precursor (Na₂SiO₃), the sample was titrated with a 2 M HCl solution to obtain silica gel which was then washed and dried until amorphous silica powder was produced. The results of the XRF analysis showed that the SiO₂ mineral content in the sample after going through the synthesis process was 94.9% and the Si element was 89.9%. The XRD characterization results show that the phase formed from the sample has a quartz structure with the highest peak at an angle of $2\theta = 23.07^{\circ}$, then decreases and levels out at an angle of $2\theta = 32.94^{\circ}$ which is characteristic of an amorphous structure and with a silica grain size of 8.47 nm - 8.65 nm.

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Introduction

Nanotechnology has attracted many scientists who are considering the potential use of particles on a scale of 1-100 nanometers [1]. At the nanoscale, physical, chemical, and biological properties differ from the properties of individual atoms and molecules of bulk matter. Therefore, this provides opportunities for the development of new research related to advanced materials that meet the demands of high-tech applications. The rapid development of various applications of nanomaterials in several fields has been observed in recent years [2]. Among several applications, nanosilica is a widely used nanomaterial with applications in adhesive polymers, optical fiber strands, inks, paints, coatings, cosmetics, food additives,

and cement-based building materials [3]. Nanosilica is one of the most common nanofillers used in manufacturing nanocomposites. Silica sources with low silica value are converted into value-added types of silica by various processes. The resulting silica products are generally classified based on their properties including nanosilica, fumed silica, precipitated silica, colloidal silica, and silica gel [4]. Nanosilica applications ultimately depend on surface chemistry, molecular structure, morphology, and porosity [2]. Therefore, modification of silica nanoparticles to improve surface chemical and physical properties is the key to successful application of the material. The best solution possible to produce nanosilica is to utilize natural resources or natural materials that are abundant on earth [5].

Rocks are a collection of one or more minerals which are the main ingredients in the formation of the earth's crust. Rocks or minerals are natural resources that are much needed and used in human life and also as basic materials in the industrial sector. Most people sell rocks raw from mining with very simple processing without knowing the other contents or minerals contained in the rocks. Rocks can be found in various places, both in rivers and mountains. Bali is one of the provinces that has the potential to be a source of volcanic rock through two currently active volcanoes, namely Mount Batur and Mount Agung. Mount Batur is an active volcano in Kintamani District, Bangli, Bali. One of the phenomena that occurs in volcanoes is the varying colors of the mountains. This is caused by differences in the materials contained in these mountain rocks. If the material is iron, the dominant color is red, if the material is coal then the dominant color is black, if the material is bronze then the mountain is greenish and so on [6].

Minerals are a resource that can be found in rocks which are abundant in Indonesia. If processed they will produce metals and various materials needed in industrial processes and have economic value [7]. One of the minerals that is often contained in rocks is the mineral silica (SiO₂) ranging from 44.89-47.76% and 71.62%, as it is known that silica is formed from the weathering of rocks that contain main minerals, such as guartz and feldspar. which is in the form of white powder [8]. Silica can be obtained from minerals, vegetables and synthesis. This compound is a raw material for producing glass and ceramics [9]. Silica is the most abundant oxide compound available in nature. This compound exists in amorphous (irregular) or polycrystalline (different crystal forms) form. Mineral silica is usually obtained through a mining process, while vegetable silica can be obtained from nature, especially plants such as rice husks and bamboo leaves [10]. Because mineral silica and vegetable silica are difficult to obtain with a high level of purity, an alternative that can be used to obtain pure silica is through a synthesis process. Synthetic silica with a high level of purity can be obtained using several synthesis methods and can use natural materials as basic materials in its manufacture such as rock powder or sand [11]. Silica powder can be obtained from natural sand or rock powder via the solid-liquid extraction method (coprecipitation) [12]. The mineral content of rocks can be determined using XRD (X-Ray Diffractometer) and XRF (X-Ray Flourescence). The XRD (X-Ray Diffractometer) method is used to determine the characterization and minerals in rocks and can also determine hydrothermal minerals or clay minerals contained in rock samples [13]. Meanwhile, the XRF (X-Ray Flourescence) method is used to analyze the elemental or metal content in rock samples and has several advantages including relatively low cost, fast analysis, multielemental, and the analysis results are qualitative and quantitative [14].

Based on the background above and several relevant research results, it indicates that hill or mountain areas which have the physical characteristics of dark or light gray colored rocks contain quite high levels of silica and have different silica mineral content (SiO₂) in each area depending on conditions. the geology. The state of the art of this research was carried out to synthesize and characterize silica minerals (SiO₂) from volcanic rocks in the active volcano in Bali, namely Mount Batur. By carrying out the synthesis and characterization of the silica minerals of volcanic rocks on Mount Batur, it is hoped that it will provide information on the characteristics of abundant rocks so that they can be utilized more optimally. The sample synthesis results were characterized using XRF and XRD. XRF characterization is used to determine the composition of mineral chemical elements in the sample, while XRD is carried out to determine the mineral phase, structure and particle size [15]. The XRD characterization results were analyzed using the ORIGIN2023 program and also the Scherrer equation, while the characterization results were analyzed using chemical stoichiometry and relevant research comparisons [16]. The novelty of this research produces an advanced and renewable material, namely nanosilica, which can be used as a basic material in the glass industry, cement industry, absorbent for synthetic dyes, biomedicine in the form of wound treatment, and a basic material for making solar panels in the future.

Theory and Calculation

In rocks there are many elements which are composed of mineral and mineraloid elements so that they form a single unit [10](SaFitri and Muh Arafatir, 2018). Important minerals found in rocks include Quartz (SiO₂), Alkali Feldspar (KAlSi₃O₈), Plagioclase Feldspar $((Ca, Na)AlSi_3O_8))$, Foida, Muscovite Mica $(K_2Al_4(Si6Al_2O_{20})(OH, F)_2)$, Mica Biotite K₂(Mg,Fe)₆Si₃O₁₀(OH)₂, Chalcedon, Calcite, Amphibole (Na,Ca)₂(Mg,Fe,Al)₃(Si,Al)₈O₂₂(OH), Pyroxine (Mg,Fe,Ca,Na) (Mg,Fe,Al) Si₂O₆, Olivine (Mg,Fe)2 SiO₄, Orthoclase (KAlSi₃O₈), and Graphite [8]. Silica (SiO₂) is the most abundant compound in the earth's crust today, accounting for 60.6% [17]. Silica can be obtained from sand, stone and siliceous soil which is abundant in Indonesia or from glass and glass crushing waste, as well as from organic materials such as rice husk ash and sugar cane ash [18]. Based on its molecular structure, silica is divided into two parts, namely crystalline silica and amorphous silica. Crystalline silica is silica whose molecular arrangement forms a certain pattern (crystals), while amorphous silica is silica whose molecular arrangement is irregular. Silica as a compound found in nature has a crystalline structure, while as a synthetic compound it is amorphous. Synthetically, silica compounds can be made from silicate solutions or from silane reagents [19].

Solid substances that do not have a crystal structure are known as amorphous substances or amorphous substances (a: no, morph: form) [20]. The particles of this amorphous substance do not have a specific shape and are permanent. Crystalline and non-crystalline (amorphous) silica is often found in nature, such as in soil, rocks and sand [21]. These two forms of silica are widely used in the glass, building and electronics industries. However, because crystalline silica is carcinogenic to humans, its use is very limited. Therefore, noncrystalline silica is safer and is widely used in industry. Amorphous silica has been classified as a non-toxic material [22]. Unlike crystalline silica, amorphous silica for a long time. The coprecipitation method can be used to synthesize silica. The working principle of the coprecipitation method is that the basic material is dissolved in an acid solution to form a metal salt and a precipitating solution is added in the form of an alkaline solution to produce a precipitate with high homogeneity. This method produces a product in the form of powder. The powder from the precipitate has high purity, small particle size and a tendency to agglomerate. The deposition process involves controlling pH, temperature and stirring speed (rpm). This deposition method has a simple process in separating the precipitate at low temperatures and a relatively short time [23]. The following is the reaction that will be used to synthesize silica:

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_3 + H_2O$$
(1)

Then for the synthesis and precipitation reactions, use the reaction equation:

$$\begin{aligned} \text{Na}_2\text{SiO}_{3(s)} + 2\text{H}_2\text{O}_{(aq)} + 2\text{HCl}_{(aq)} \rightarrow \text{Si}(\text{OH})_4 + 2\text{NaCl} \end{aligned} \tag{2} \\ \text{Si}(\text{OH})_4 \rightarrow \text{SiO}_2 + 2\text{H}_2\text{O} \end{aligned} \tag{3}$$

The synthesis results will be characterized using XRF and XRD instrument tests. The results of the XRF characterization test were analyzed using chemical stoichiometry, while the results of the XRD characterization test used Bragg's Law analysis. Bragg's Law states that the difference in the path of the X-ray diffraction beam must be a multiple of the wavelength [22]. The incident ray hits a point in the first plane and is scattered by the P atom. The second incident ray that reaches the next plane is scattered by the Q atom which travels a distance SQ + QT if the two rays are parallel and in phase (reinforce each other). This travel distance is a multiple of (n) wavelength (λ), so the equation becomes (Bragg's Law):

$$n \lambda = SQ + QT$$

$$n \lambda = d \sin \theta + d \sin \theta$$

$$n \lambda = 2d \sin \theta$$
(4)

The width of the diffraction peak provides information about the grain size. The relationship between grain size and the width of the X-ray diffraction peak can be calculated using the Schrerer equation.

$$D = \frac{K\lambda}{\beta\cos\theta}$$
(5)

Where D is the particle size, K is Scherrer's constant (0.94), λ is the X-ray wavelength used, and β is the width of the half peak diffraction maximum or Full Width Half Maximum (FWHM), and θ is the position of the diffraction peak.

Experimental Method

This research was carried out in four stages, namely: (1) preparation, (2) synthesis, and (3) characterization.

1. Preparation

The sampling location was Mount Batur. Before the samples are taken, the sampling points are first determined using the Global Positioning System (GPS) and the distance of each point is measured, 20 meters long, in the cardinal directions of north, east, south, west and the center point. After obtaining 5 sample points, then rock samples were taken in the form of rock chunks using a hammer/hammer to a depth of 50 cm. The stone chunks were washed with distilled water to remove adhering dirt, then dried in the oven for 1 hour at 100°C. Then the rock samples are crushed using a hammer into small chunks, then the small

chunks of rock are crushed using a mortar and pestle to become a fine powder. In fine powder form, the samples were subjected to preliminary tests using XRF to determine the element content in volcanic rocks.

2. Synthesis

The synthesis process begins with the fine powder resulting from the preparation being extracted using a permanent magnet to remove heavy metals and obtain the desired silica powder. The fine powder that has successfully passed the extraction stage is then sieved using a 100 mesh sieve until a smaller powder size is obtained. 10 grams of the sieved sample were taken, then placed in a beaker soaked in 2 M HCl solution (synthesis process) for 12 hours [12]. The synthesis process aims to dissolve heavy metal elements remaining from the extraction process from rock powder. The results of the synthesis process will produce a precipitate in the form of silica. The results of the soaking in the form of silica precipitate were washed with distilled water 5 times until neutral (pH~7), then filtered using filter paper as the filter [12]. The precipitate that has been filtered is then heated further using an oven for 1 hour at a temperature of 100°C until the sample becomes dry and microsilica is produced.

Microsilica powder was reacted with 7 M NaOH and stirred using a magnetic stirrer at a speed of 650 rpm at a temperature of 225°C for 2 hours. This stage is called the hydrothermal process. The results of this process were dissolved again using 200 ml of distilled water and stirred for 30 minutes. Then it is filtered using filter paper to obtain sodium silicate precursor (Na₂SiO₃).

 $SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_3 + H_2O$ (6)

The filtered sodium silicate solution is then coprecipitated through a titration process by dripping 2 M HCl solution. This titration process is carried out until the pH of the solution changes from the original between 13-14 to pH 7, which is the stage for the formation of silica gel. The solution which was initially liquid and clear turns white, cloudy and thick because a gel forms. The sample was allowed to settle for 24 hours [12]. In the form of a gel precipitate, it was then washed using 600 ml of distilled water and stirred manually. Washing is carried out until the NaCl salt content has disappeared from the resulting product. The salt formed appears as a result of a chemical reaction between NaOH and HCl. The sample was then filtered and dried using an oven at a temperature of 100°C for 2 hours until amorphous silica powder was produced.

$Na_{2}SiO_{3(s)} + 2H_{2}O_{(aq)} + 2HCl_{(aq)} \rightarrow Si(OH)_{4} + 2NaCl$	(7)
$Si(OH)_4 \rightarrow SiO_2 + 2H_2O$	(8)

3. Characterization

The amorphous silica was characterized using XRD and XRF. Characterization uses XRD to determine the mineral phase and structure of minerals (SiO₂) in rocks, as well as particle size. Characterization of rock samples is carried out using XRF to determine the chemical element composition of minerals in the samples. Sample characterization using XRD instruments was carried out at BRIN Indonesia with the type of instrument being the Rigaku SmartLab series. Meanwhile, sample characterization using an XRF instrument was

carried out at the Integrated Laboratory, State University of Malang, Indonesia with the instrument brand being PANalytical type Minipal 4.

Result and Discussion

1. Synthesis Results of SiO₂ Nanoparticles

Mount Batur volcanic rock has been successfully synthesized into SiO_2 nanoparticle compounds using hydrolysis and coprecipitation methods. As shown in Figure 1 which is characterized by a white powder with a light mass. Physically, this indicates that there are compounds from SiO_2 nanoparticles as shown in the following picture.



Figure 1. Synthesis Results of SiO₂ Nanoparticles

Nanoparticle powder was obtained SiO2 from the synthesis using the coprecipitation method was 0.66 grams.

2. XRF Test Results

Based on the results of preliminary XRF characterization tests, from four rock sample points it was found that the silica content of each volcanic rock from Mount Batur was 31.6% - 41.1%. These results indicate that there is silica content in volcanic rocks and it is sufficient to continue with the synthesis process. Complete XRF characterization test data can be seen in the following table.

Table 1. Initial mineral composition of volcanic rocks									
Compound	SiO ₂	Fe ₂ O ₃	CaO	Al_2O_3	K ₂ O				
White Rocks	31.60%	23.40%	7.57%	13.00%	0.98%				
Black Rock	41.10%	25.40%	14.9%	14.00%	1.29%				
Ash Rock	40.30%	22.60%	9.44%	12.00%	1.40%				
Red Rocks	34.10%	29.80%	13.70%	13.00%	1.30%				

Fable 2. Initial elemental corr	position of volcanic rocks
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Element	Si	Fe	Ca	Al	K
White Rocks	22.90%	34.00%	10.00%	10.00%	1.50%
Black Rock	29.00%	35.60%	18.50%	10.00%	1.80%
Ash Rock	29.40%	33.60%	12.50%	9.40%	2.08%
Red Rocks	23.40%	39.40%	16.40%	9.60%	1.74%

The results of the synthesis of volcanic rock silica using the coprecipitation method obtained XRF characterization results with a SiO_2 mineral content of 94.9% and a Si element content of 89.9%. The XRF characterization test data from the results of this synthesis can be interpreted through the following image.



Figure 2. Final Test Results for XRF Characterization of Volcanic Rock Powder Samples

The data from the XRF measurements above is in the form of a two-dimensional spectrum source with the x-axis being energy (keV) and the y-axis being the intensity of the X-rays emitted by each element. Based on this graph, the content of compounds and elements in the samples represented in table 3 and table 4 can be analyzed.

Table 3. Results of Elemental Component Analysis Through XRF Energy Spectrum Characterization

	Test											
Ele- ment	Si	K	Ca	Ti	v	Cr	Fe	Ni	Cu	Zn	Yb	Re
Conc- Unit	89,9%	0,27%	1,90%	2,69%	0,11%	0,057%	4,70%	0,03 %	0,096%	0,01%	0,05%	0,2%

 Table 4. Results of Compound Component Analysis Through XRF Energy Spectrum Characterization

 Test

Com- pound	SiO ₂	K ₂ O	CaO	TiO ₂	V_2O_6	Cr ₂ O ₃	Fe ₂ O ₃	NiO	CuO	ZnO	YbO	Re ₂ O ₇
Conc- Unit	94,9%	0,13%	1,16%	1,28%	0,092%	0,027%	2,26%	0,01%	0,046%	0,005%	0,001%	0,01%

3. XRD Characterization Test

The XRD characterization test is used to determine the mineral phase, crystallinity structure and grain size of the particle. Figure 3 is the result of XRD characterization which forms a diffraction pattern of the crystallinity of the SiO₂ mineral. It can be seen that in these results, the phase formed from the sample has a quartz structure with the highest peak at an angle of $2\theta = 23.07^\circ$, then decreases and levels out at an angle of $2\theta = 32.94^\circ$ which is characteristic of an amorphous structure. The formation of amorphous silica powder is due to the breaking of chemical bonds during the coprecipitation process. From the results of characterization using XRD, particle size can be determined using the Scherrer equation. The calculation results show that the silica particle size is 8.47 nm – 8.65 nm. From the results of these calculations it can be seen that silica nanoparticles consist of nanocrystalline, where the particle size is ≤ 100 nm



Figure 3.X-Ray Diffraction Pattern of SiO₂ Nanoparticles

XRF is an instrument used to characterize the content of metal elements and metal oxides in samples. There was a color change in the inside of the sample before and after extraction. Before extraction, the volcanic rock powder sample was gray, and after extraction, the Mount Batur volcanic rock powder was white. Table 1 and table 2 show that the SiO₂ content of volcanic rocks is 31.6% - 41.1% and the Si element content is 22.90% - 29.40%. This shows that the volcanic rock of Mount Batur can be used as raw material to produce pure SiO₂. The chemical process is carried out using the coprecipitation method to obtain SiO₂. It is a successive method for obtaining metal deposits and removing impurities from a material or sample. Following are the reactions that occur in the volcanic rock powder sample during extraction using the coprecipitation method.

$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_3 + H_2O$	(9)
$Na_2SiO_{3(s)} + 2H_2O_{(aq)} + 2HCl_{(aq)} \rightarrow Si(OH)_4 + 2NaCl$	(10)
$Si(OH)_4 \rightarrow SiO_2 + 2H_2O$	(11)

The synthesis process begins by soaking rock powder in 2 M HCl solution for 12 hours. This process aims to dissolve heavy metal elements remaining from the extraction process from rock powder. The results of the synthesis process will produce a precipitate in the form of silica. The HCl solution functions as a precipitator for non-polar compounds. SiO₂ compounds dissolve easily in basic solutions and precipitate in acidic solutions. The coprecipitation process on the SiO₂ precipitate was carried out by reacting the silica powder into a 7 M NaOH solution and stirring using a magnetic stirrer at a speed of 650 rpm at a temperature of 225°C for 2 hours. This stage is called the hydrothermal process. The results of this process are dissolved again using distilled water and filtered to obtain sodium silicate precursor (Na₂SiO₃).

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_3 + H_2O$$
(12)

The filtered sodium silicate solution is then coprecipitated through a titration process by dripping 2 M HCl solution. This titration process is carried out until the pH of the solution changes from the original between 13-14 to pH 7, which is the stage for the formation of silica gel. In the form of a gel precipitate, it was then washed using 600 ml of distilled water and stirred manually. Washing is carried out until the NaCl salt content has disappeared from the resulting product. The salt formed appears as a result of a chemical reaction between NaOH and HCl. The sample is then filtered and dried until amorphous silica powder is produced.

$$Na_{2}SiO_{3(s)} + 2H_{2}O_{(aq)} + 2HCl_{(aq)} \rightarrow Si(OH)_{4} + 2NaCl$$

$$Si(OH)_{4} \rightarrow SiO_{2} + 2H_{2}O$$
(13)
(14)

The results of the synthesis of volcanic rock silica using the coprecipitation method obtained XRF characterization results with a SiO₂ mineral content of 94.9% and a Si element content of 89.9%. Apart from the Si element, there are several other impurity elements contained in the sample, including 4.7% Fe, 2.9% Ti, 1.9% Ca, and 0.27% K. This amorphous silica phase can occur because molarity and pH in this study function as driving forces in crystal formation [24]. Phase formation usually uses temperature treatment which requires a lot of energy consumption and takes a long time, while the coprecipitation method only takes less than 4 hours to form the sample. When compared with other methods such as alkali fusion which is usually used to extract sand or glass waste, this method is much more economical in terms of energy consumption and the synthesis results which can produce crystals with a quartz phase are not found in the alkali fusion method. This is also supported by the results of XRD characterization of volcanic rock powder which proves that no crystallinization peaks were formed.

Analysis of the mineral characteristics of SiO₂ nanoparticles based on XRD results can also be used to determine the phase, mineral structure and particle size. Determination of mineral phase and particle structure using ORIGIN2023 software. The ORIGIN2023 application is used to determine the mineral phase and particle structure, while to determine the particle size in this study the Schrerer equation is used.



Based on the diffraction graph pattern above, information is obtained regarding the

interpretation of the resulting diffraction peaks as in table 5 below.

Table 5. Discussion of X-Ray Diffraction Peak Analysis XRD Test										
No.	2θ (deg)	d(ang.)	Height(cps)	FWHM(deg)	Int. I(cps deg)	Int. W(deg)	Asym. factors			
1	07.23(7)	3.853(11)	143(35)	1.0(3)	319(40)	2.2(8)	4(5)			
2	32 94(18)	2717(14)	87(27)	1.0(4)	189(26)	22(10)	0.5(6)			

Based on the pattern formed in Figure 4, the SiO₂ phase formed is amorphous because no crystallinization peaks are formed. The synthesis results show that the SiO2 formed is amorphous with a peak around the angle $2\theta = 23.07^{\circ}$, then decreases and levels out at an angle $2\theta = 32.94^{\circ}$. The formation of amorphous silica powder is due to the breaking of chemical bonds during the coprecipitation process. When silica is dissolved in a 7M NaOH solution, the chemical bonds of SiO₂ are disassembled, resulting in the formation of sodium silicate precursor (Na₂SiO₃), so that when the coprecipitation process is carried out, namely by titrating the HCl solution, silica gel will be obtained which is then dried with the structure formed being amorphous. The results of SiO₂ in amorphous form with a peak around the angle $2\theta = 22.68^{\circ}$, then decreases and flattens at an angle $2\theta = 37.50^{\circ}$ [12]. This result is in accordance with the standard pattern of silica recorded in the Standards Joint Committee on Powder Diffraction (JCPDS) which shows that amorphous silica has a hump between 15° to 35° of 2 θ (19°-21°) [24]. Apart from that, there is other supporting research that strengthens

the results of XRD characterization of Mount Batur rock powder samples, including research conducted by Munasir et al. who found a diffraction peak at $2\theta \approx 20^{\circ}$ - 24° which represents the amorphous peak in silica [22]. Research by Kadhim et al. which shows that the sample formed has a quartz structure with the highest peak at $2\theta = 22.4^{\circ}$, which is characteristic of an amorphous structure [25]. Hasanah et al. using the coprecipitation technique for the extraction and characterization of Mount Sinabung volcanic ash, a focused amorphous nanosilica peak was obtained at $2\theta = 23^{\circ}$ [26].

From the information provided by the ORIGIN 2023 application in analyzing diffraction peaks, you can also find the grain size of the volcanic rock powder samples from Mount Batur using the Schrerer equation.

$$D = \frac{K\lambda}{\beta\cos\theta}$$

Through the above equation, the grain size calculation for the peak position at an angle of $2\theta = 23.07^{\circ}$, with the respective values of K is 0.94, λ is 0.15406 nm, β is the radian of 1 = 0.01744, and θ is 11.535°, which is as follows.

$$D = \frac{(0.94)(0.15406 nm)}{radian(1)\cos\left(\frac{23.07}{2}\right)}$$
$$D = 8,47 nm$$

So, the size of the SiO_2 particles produced is 8.47 nm.

Based on the results of grain size analysis calculations using the Schrerer equation above, it can be concluded that the SiO_2 powder produced through the synthesis process using the coprecipitation method is nanosilica. This is in line with the theory in the literature review which explains that nanoparticles are dispersed particulates or solid particles whose grain sizes range from 1 – 100 nm. This also proves that using the coprecipitation synthesis method is capable of producing nano-sized silica and a high level of SiO_2 purity of 94.9% and Si element purity of 89.9%.

Conclusion

Here we report that SiO₂ nanoparticles can be synthesized using the coprecipitation method by reacting the extracted rock powder sediment with a 2 M HCl solution which is then coprecipitated into a 7 M NaOH solution to produce sodium silicate precursor (Na₂SiO₃). The sodium silicate precursor (Na₂SiO₃) is titrated with a 2 M HCl solution to obtain a saturated pH of SiO₂ (pH 4 – 5) until it forms silica gel. Amorphous silica is obtained after the silica gel is washed again using distilled water to remove the salt content formed due to the reaction of NaOH with HCl. XRF results show that SiO₂ nanoparticles powdered from Mount Batur rock produce a SiO₂ mineral content of 94.9% and Si elements of 89.9%. The XRD results provide information that the SiO₂ phase formed is amorphous because no crystallinization peaks are formed. The synthesis results show that the SiO₂ formed is amorphous with a peak around the angle $2\theta = 23.07^{\circ}$ with the resulting particle size being 8.47 nm. Based on the research results, the authors can provide suggestions for the sustainability of this research for added SEM – EDX characterization to be able to determine the distribution and morphology of SiO₂ volcanic rock powder, as well as particle size more clearly from the synthesis results.

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