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SYNTHESIS OF SBA-15 USING SONOCHEMICAL METHOD WITH NA_2SIO_3 PRECURSOR EXTRACTED FROM LAPINDO MUD

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Abstract

Lapindo mud contains 49.9% silica, making it a potential alternative source of silica precursor for the synthesis of SBA-15. In this study, SBA-15 was synthesized using the surfactant Pluronic P123 through a sonochemical method, which utilizes ultrasonic waves to enhance mixing, accelerate mass transfer, and promote the formation of mesoporous structures. Characterization of the synthesized material was carried out using FTIR and XRD techniques. The FTIR results confirmed the successful formation of silica networks, indicated by the presence of Si–O–Si functional groups at wavenumbers of 1110 cm^{-1}. Furthermore, the variation of precursor volume influenced the structural quality of SBA-15. The FTIR spectra revealed characteristic Si–O–Si vibrations in both precursor variations, indicating the formation of a silica framework. Differences in absorption band characteristics suggest that the precursor volume influences the chemical features of the synthesized SBA-15. Overall, the study demonstrates that Lapindo mud-derived silica can be effectively utilized to synthesize SBA-15 with desirable structural and functional properties.

Keywords: SBA-15, Lapindo mud, Sonochemical

I. INTRODUCTION

Lapindo mud is hot mud originating from the earth's interior that contains volcanic material mixed with gas and can be classified as a mud volcano. Lapindo mud has the potential to serve as an alternative silica source, with a silica content of 49.9% [1], as well as other components such as Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O, and SO_2 [2], [3]. The elemental composition of the Lapindo mud, based on distance from the center of the Lapindo mudflow, is as follows: iron (Fe) at 34.2% (within a 500-meter radius), 33.9% (within a 1,000-meter radius), and 36.4% (within a 1,500-meter radius). The second-highest element content is silicon (Si) at 32.7% (500 m radius), 33.0% (1,000 m radius), and 31.1% (1,500 m radius) [4].

This high silica content makes Lapindo mud a potential source of alternative silica precursors. Silica extracted from Lapindo mud not only has potential as a conventional silica material but can also be developed into mesoporous silica materials. Silica-based mesoporous materials are characterized by a high specific surface area, uniform pore diameters, and thick silica walls. Mesoporous silica nanoparticles have pore diameters ranging from 2 to 50 nm and a maximum total diameter of 1 µm [5].

One of the most widely developed mesoporous silica materials is SBA-15 (Santa Barbara Amorphous-15). SBA-15 has a regular hexagonal pore structure, a high specific surface area, and relatively thick pore walls, giving it good structural stability. Several studies have utilized alternative silica sources such as rice husk ash, beach sand, sugarcane bagasse, and petung bamboo leaves. However, Lapindo mud has greater potential due to its abundant availability and relatively high silica content.

In addition to the selection of the silica source, the synthesis method is also an important factor that can influence the characteristics of the resulting SBA-15 material. One method that can be used is the sonochemical method, a synthesis method that utilizes ultrasonic waves to generate acoustic cavitation within the reaction

system. This cavitation can enhance mixing, mass transfer, and interactions between the silica precursors and the template, thereby potentially accelerating the process of material structure formation. The mechanism of SBA-15 formation involves several stages, as shown in **Figure 1**.

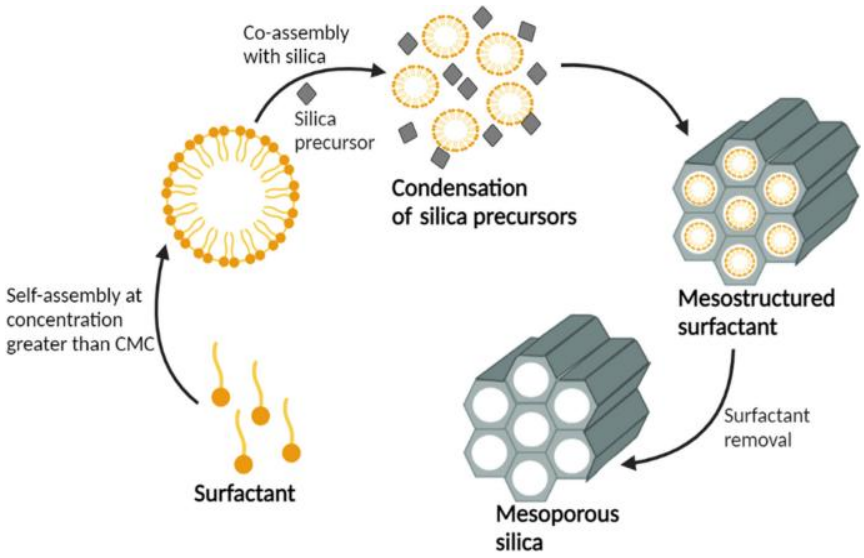


Figure 1. Mechanism of Hexagonal Structure Formation by Pluronic P123 Micelles [6]

The use of sonochemical methods also presents an attractive alternative to conventional synthesis methods, which generally require longer processing times. Based on the potential silica content in Lapindo mud, the opportunity to utilize it as a source of sodium silicate, and the characteristics of SBA-15 as a mesoporous material, this study was conducted to synthesize SBA-15 using sodium silicate precursors extracted from Lapindo mud via the sonochemical method. This approach is expected to produce SBA-15 material based on an alternative silica source.

II. METHOD

A. Research Design

This study was conducted as an experimental research project at the Physical Materials Laboratory of the Department of Physics, Faculty of Mathematics and Natural Sciences, Surabaya State University. The study involved several stages, including sample preparation, sample synthesis, sample characterization, and sample testing.

Sample preparation was carried out by washing the Lapindo mud with distilled water, followed by the extraction of sodium silicate (Na_2SiO_3) from the Lapindo mud material by adding several reagents, such as 5M HCl for leaching at a 1:5 ratio at 60°C. The mixture was then decanted until the pH reached 5 and dried at 100°C until a constant weight was achieved. The calcined material was then calcined in an oven at 680°C for 1 hour. Subsequently, 13 g of the calcined silica material was mixed with 100 mL of 2 M NaOH under continuous stirring to facilitate sodium silicate formation. The resulting mixture was then separated to obtain the sodium silicate solution.

Subsequently, the resulting sodium silicate was used as a precursor in the synthesis via the sonochemical method using an ultrasonicator. A total of 4 g of Pluronic P123 was dissolved in 100 mL of 1.6 M HCl under continuous stirring with gentle heating. Subsequently, sodium silicate solution was introduced into the mixture at two different volumes (30 and 50 mL). The resulting mixture was subjected to sonication for 3 hours, followed by aging in an oven at 60 °C. The solid product was then separated by filtration and washed repeatedly with distilled water and ethanol until the pH reached 4. Finally, the collected solid was dried at 120 °C. The dried samples were subsequently calcined at 550 °C for 6 hours to remove the Pluronic P123 template and obtain the mesoporous silica material. The resulting silica nanoparticles were then

characterized using Fourier-Transform Infrared (FTIR) and X-Ray Diffraction (XRD) analyses. The sample test results consisted of quantitative data that were subsequently analyzed.

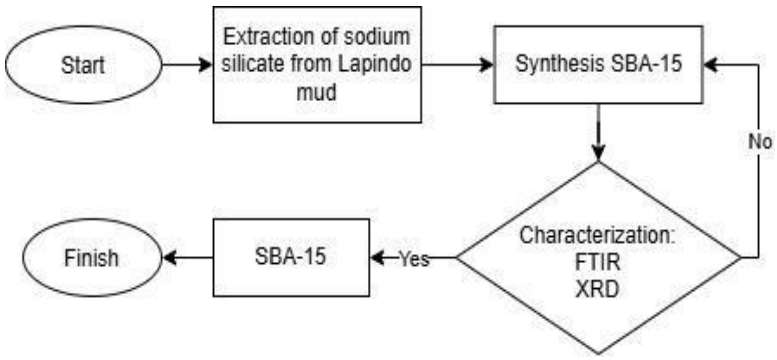
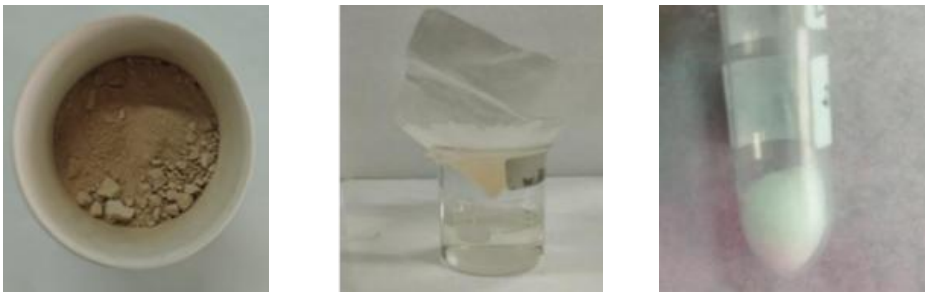


Figure 2. Research Design Flowchart

III. RESULTS AND DISCUSSION

A. Synthesis Results

The use of Lapindo mud as a silica precursor is based on its relatively high silica content, as indicated by research findings [4], which report a silica content of 33%, making it a potential alternative raw material for the synthesis of mesoporous silica materials. Extraction of the sodium silica precursor involves leaching Lapindo mud with HCl to dissolve other impurities, such as metal oxides (Fe, Al, Ca), thereby yielding microsilica [7].



(a) (b) (c)

Figure 3 (a) Silica extraction from Lapindo mud; (b) Na_2SiO_3 extraction from Lapindo mud; (c) SBA-15

Visually, the synthesized material underwent changes compared to the initial precursor, which was brown ash, due to the leaching process; it still contained $Fe^{3+}$and, following the addition of NaOH, became a silica precursor in the form of Na_2SiO_3. The Lapindo mud, which initially appeared as shown in **Figure 3(a)** grayish-brown color, produced a white solution after undergoing an extraction process to obtain Na_2SiO_3 and a brownish $Fe(OH)_3$ {3} precipitate, as shown in **Figure 3(b)**. These visual changes indicate differences in the physical appearance of the material before and after the extraction process. However, further compositional characterization is required to confirm the reduction of impurities and the silica content. Additionally, a powder with a finer and more homogeneous texture was obtained compared to the initial material, as shown in **Figure 3(c)**, indicating that the synthesis process proceeded successfully, resulting in SBA-15.

B. Fourier Transform Infra-Red (FTIR)

FTIR characterization analysis was conducted to identify the functional groups present in SBA-15 with different precursor variations. The FTIR characterization spectra show the relationship between wavenumber and transmittance. The results of the FTIR characterization in this study are shown in **Figure 4**.

Figure 4 presents the FTIR spectra of SBA-15 synthesized using 30mL of sodium silicate precursor extracted from Lapindo mud. The observed absorption bands indicate the successful formation of a silica framework, as evidenced by the characteristic vibrations of Si–O–Si and Si–OH bonds. The absorption band

at 1110 cm^{-1} is attributed to the asymmetric stretching vibration of Si–O–Si, representing the formation of interconnected siloxane bonds within the silica network. Furthermore, the absorption band at 798 cm^{-1} corresponds to the symmetric stretching vibration of Si–O–Si, while the band at 480 cm^{-1} is associated with Si–O bending vibrations. The presence of these three characteristic absorption bands confirms the formation of the silica framework.

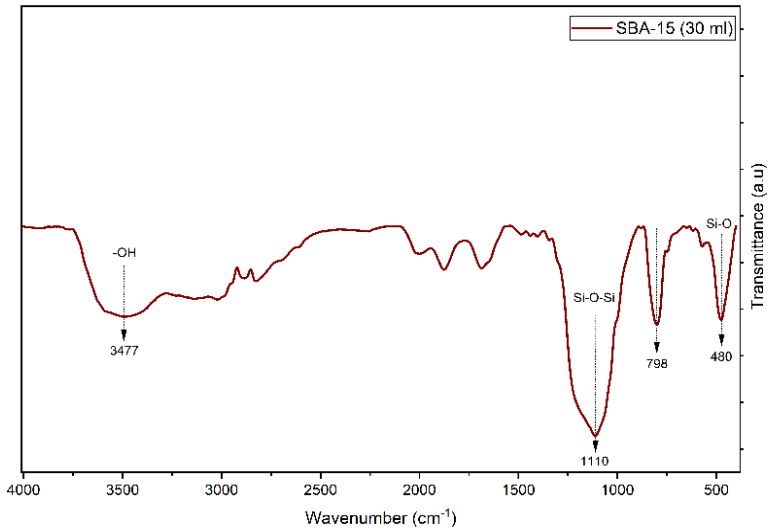


Figure 4. FTIR Spectrum of SBA-15

Additionally, a broad absorption band around 3477 cm^{-1} is attributed to O–H stretching vibrations associated with surface silanol (Si–OH) groups and adsorbed water molecules. The presence of silanol groups indicates the existence of hydroxylated sites on the silica surface, which are characteristic of mesoporous silica materials. The absorption band associated with Si–OH reported in the literature at approximately 3750 cm^{-1} further supports the identification of surface silanol groups [10].

The FTIR results demonstrate the successful formation of silica-based materials through the sonochemical method. The presence of characteristic Si–O–Si vibrations confirm the development of a silica network, while the Si–OH groups indicate the presence of hydroxyl functionalities on its surface. These findings suggest that sodium silicate extracted from Lapindo mud can be effectively utilized as an alternative silica precursor for SBA-15 synthesis.

Table 1. Identification of Functional Groups in SBA-15 Based on FTIR Analysis and Literature Data

Wave Number (cm^{-1})		Function Group	Ref.
Research (30mL)	**Reference**		
480	490	Si-O bending vibration	[8]
798	858	Si-O-Si symmetric	[9]
1110	1135	Si-O-Si asymmetric sretching	[9]
3477	3500	-OH	[8]
-	3750	Si-OH	[10]

C. X-Ray Diffraction (XRD)

The structural characteristics of SBA-15 material are generally determined using X-ray diffraction (XRD) to identify the regularity of the mesoporous structure. In this study, XRD analysis was not carried out directly. Therefore, the findings are based on reference data relevant to SBA-15 material.

As shown in **Figure 5**, the XRD diffraction pattern of SBA-15 exhibits three main peaks at low angles ($2\theta \approx 0.5–2°$), indexed as the (100), (110) and (200) planes, respectively [11]. These three peaks are characteristic of the two-dimensional hexagonal mesoporous structure with p6mm symmetry possessed by SBA-15. The (100) peak typically has the highest intensity and is related to the inter-pore distance (d-spacing), whilst the

(110) and (200) peaks appear with lower intensity as higher-order reflections indicating long-range structural order [11].

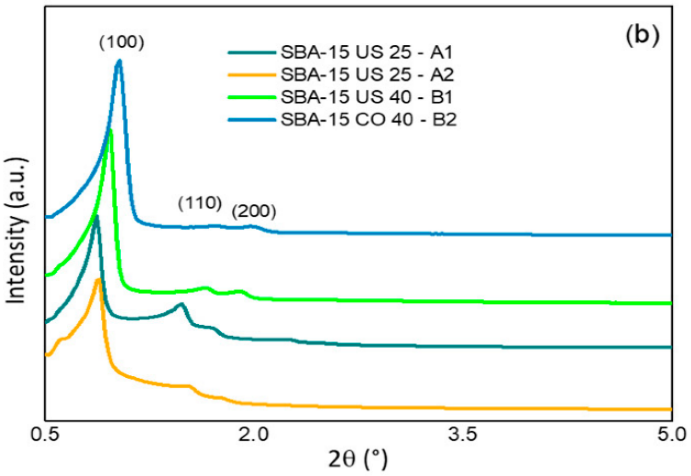


Figure 5. X-Ray Diffraction (XRD) of SBA-15 [11]

The presence of these three peaks indicates that the material possesses a regular and periodically organised pore arrangement. The intensity and sharpness of the diffraction peaks reflect the degree of mesopore structural regularity, whereby sharper and more intense peaks indicate a more homogeneous and regular structure. Conversely, a decrease in intensity or broadening of the diffraction peaks indicates a reduction in structural regularity, which may be caused by imperfections in the synthesis process or degradation of the pore walls.

Although no XRD analysis was carried out, the FTIR characterisation results in this study indicate the formation of the silica framework characteristic of SBA-15. The presence of these Si–O–Si groups is a key indicator of the formation of the silica network that forms the basic framework of the SBA-15 material. In the literature, the formation of this silica network correlates with the formation of a regular mesoporous structure, as confirmed by XRD. Therefore, although XRD data were not obtained directly, the consistency of the FTIR results with the characteristics of SBA-15 reported in the literature can be used as an initial indication of the formation of the expected material structure.

IV. CONCLUSION

A. Conclusion

Based on the results of the research conducted, it can be concluded that Lapindo mud has excellent potential as an alternative source of silica in the synthesis of SBA-15 mesoporous materials due to its high silica content. The extraction process, which uses the HCl leaching method followed by the formation of sodium silicate (Na_2SiO_3), has proven effective in reducing impurity levels such as metal oxides and improving the purity of the resulting silica. This is evidenced by changes in the color and texture of the material, which became more homogeneous after the synthesis process.

The synthesis of SBA-15 using the sonochemical method with the aid of the surfactant Pluronic P123 successfully produced a mesoporous silica material characterized by the formation of silica-specific functional groups, namely Si–O–Si and Si–OH, as determined by FTIR characterization. Overall, the sonochemical method proved effective in accelerating the synthesis process and produce Lapindo mud-based SBA-15 material with the appropriate characteristics,

B. Suggestion

Based on the research conducted, it is recommended that further characterization be performed using XRD-SAXS analysis to determine the structure and wall thickness of the SBA-15 material synthesized from the Na_2SiO_3 precursor extracted from Lapindo mud.

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