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Synthesis and Structure Characterization of SiO_2 from Petung Bamboo Leaf Ash (Dendrocalamus asper (Schult.f.) Backer ex Heyne)

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Abstracts

The Research about Synthesis and Characterization of SiO_2 have been worked. We have synthesized Silica from "petung"bamboo leaf ash as SiO_2 source. This step used sol gel methode. SiO_2 were characterized by Fourier Transform Infra Red (FTIR) to investigated the stucture andX-Ray Diffaction to know about structure and crystallinity. FTIR spectra show peak at 617,22 cm^{-1} area that spesific for Si-H bond, peak at 786,96 $cm^{-1}$and 1095,57 cm^{-1} area specific for Si-O-Si bonds. Difractogram of SiO_2 show that there are peak at 2θ 21,99; 31,67 and 38,88 were specific for SiO_2 that calcinated at 800^oC, while for SiO_2 that calcinated at 400^oC there were no peak at 2θ 31,67 dan 38,88. That peaks not shown may be because low crystallinity of SiO_2 that calcinated at 400^oC. Calcination temperature greatly affects the crystallinity of SiO_2.

Key words: Silica; Bamboo Ash Leaves; Structure Characterization.

1. Introduction

Silica is a chemical compound with the general formula SiO_2. These compounds can be found in nature as minerals such as sand, quartz, glass, and so on. In addition, silica can also be synthesized in the laboratory. Silica is widely used for various purposes ranging from electronics, mechanics, medical, art to other fields. Silica in nature is crystalline while artificial silica is generally amorphous. In this research, we interested in using SiO_2 material. This is because the solid can be synthesized by utilizing local natural resources in Indonesia. SiO_2 sources can be derived from minerals such as zeolites, bentonites, clinoptioites that are numerous in Indonesia. In addition, SiO_2 can also be obtained from biomass such as rice husk ash, rice straw ash, and bamboo leaf ash. So far, rice husk ash has been widely studied to obtain SiO_2 while bamboo leaf ash has not been widely studied while the SiO_2 content in it reaches 60-70%.

Bamboo leaves that fall to the ground are only considered as mere waste by our community.

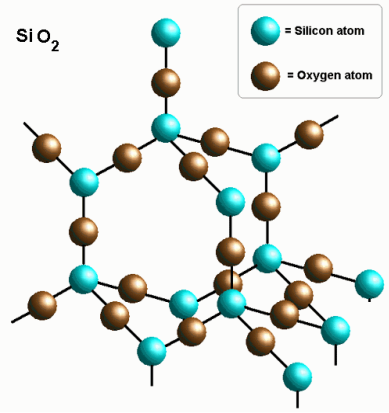


Figure 1. Tetrahedral structure of SiO_2

It is very regrettable, because in the bamboo leaf there is still a compound that can be used that is silica. According to research by Lembaga Ilmu Pengetahuan Indonesia (LIPI), the silica content of bamboo petung species reached 3.51% more than the other five bamboo species, namely 1.10% bambu tali, 2.93% bambu hitam, 1.05% bambu kuning, 1.2% bambu andong, And 1.01% bamboo ampel[1]. Indonesia is a tropical country rich in flora and fauna with variations and also varieties. One of them is bamboo. The existence of bamboo plants are found in various places, both naturally grown and deliberately cultivated. The world's bamboo population is estimated to be 1200-1300 species. There are 143 species of bamboo in Indonesia, of which 60 species are in Java [2]. Bamboo plants do not require too many requirements to grow. Bamboo can grow in wet climates until dry, from lowlands to high altitudes. Bamboo plays a very important role in the life of the people of Indonesia. People use bamboo plants usually on the stem to serve as an ingredient in the food industry, paper making, building, handicraft, and even medicine. However, the utilization of other parts of bamboo plants, such as roots, branches and leaves is still not maximized.

Bamboo petung is known by its scientific name (Dendrocalamus asper (Schult. F.) Backer ex Heyne). Bamboo petung has several names of the region such as awi bitung, pring petung and pereng petong. This type of bamboo has a clump that is a bit tight. The color of green stems yellowish. The size is bigger and higher than other types of bamboo. Stem height reaches 20 m with stem diameter up to 20 cm. The bamboo segment is quite long and thick, its length between (40 - 60) cm and its wall thickness (1 - 1.5) cm [3].

The research on the utilization of SiO_2 from bamboo ash has been studied by Ding et al., In 2008. The objective of the study was to compare the silica content found in bamboo parts, such as roots, stems, branches, and leaves and their effects To the biogeochemical cycles of silica. The result of this research is that the content of silica tends to rise from root to peak that is on leaves with percentage from 0.3% at root to 9.95% in leaf [4][5]. Furthermore, research conducted by Olugbenga O. Amu et al In 2010. The purpose of the research is to know the characteristics of bamboo ash and its influence in stabilizing to the soil of the literit on the road construction. The study provides information on bamboo leaf ash has a good potential to stabilize laterite soil on highway construction ± 38.21%, because the silica content is 75.90% [6].

In this paper we conduct research on the synthesis of SiO_2 from bammbo petung ash leaf. Then, characterization structure of material have been done.

2. Experiments Procedure

The leaves of bamboo petung cleaned and dried under the sun for 2 days. The dried bamboo leaves are furthermore ignored using furnaces at a temperature of 800^{o}C to obtain gray bamboo petung ash. Chemical composition of bamboo leaves ash invertigated by analysis with X Ray Fluoresence (XRF). While, SiO_2 synthesized of petung bamboo ash

with sol gel method. This method is adapted from [7]. The leaves of bamboo petung leaves are dissolved in 4 M NaOH in stop erlenmeyer. The mixture is stirred while heated at a temperature of 109 ° C. The residue is burned at a temperature of 500°C to whitish brown. The resulting substance was then dissolved in mineralized water into a yellowish brown of sodium silicate solution. The Na silicate solution is used as a precursor of silica. The solution of silicic acid is added HCl 1 M dropwise until it has pH 7. The solution is then allowed to stand for 72 hours until gel is formed. The formed gel is then washed with demineralized water and the residue is dried in an oven at 80° C. The formed silica is then crushed and sieved with 100 mesh sieve. Silica has been heated at 400°C and 800°C for 4 hours. To test whether the catalyst has been formed, the structural structure with Fourier Transform Infra Red (FTIR) and X-ray diffraction (X-Ray Diffaction) have been isvertigated.

3. Result and Discussion

Synthesis Silica from Petung Bamboo Leaf Ash

Silica is synthesized by exploiting local potency in the form of bamboo petung leaves. Nowadays, bamboo leaves left to dry and not widely used. The leaves of bamboo petung to be used are washed with running water that aims to remove the impurities and make the bamboo petung leaves clean and also as to increase the purity of the resulting silica. The leaves of bamboo petung that has been clean then through the drying stage in the sun for 2 days. The drying rate will decrease along with the decreasing of moisture content during evaporation and the drying process does not occur at once. So in sun-drying, the heat dissipation into the material takes place gradually and thoroughly so that the evaporation of water into the air is more evenly although it takes longer time to use oven oven. When the material starts to heat from the oven, the rate of drying takes place rapidly, so that when the drying rate begins to decrease, the remaining water content of the material remains[8].

The dried bamboo leaves are further ignored. The process of bamboo petrified petting is done using a furnace with a temperature of 800°C for 2 hours. At that temperature will produce good quality silica [9]. The higher the temperature of the probe, causing higher purity. Ash ash petung leaves were analyzed with XRF to determine their chemical composition. The results are presented in Table 1. The reactions that occurred during the observation were:

C, H, and Si compounds + O_2 → $CO_{2(g}$ + $H_2O_{(g)}$ + $SiO_{2(p)}$

Table 1. Chemical Composition of Bamboo Leaf Ash Leaves

Senyawa oksida*	Persen Massa(%)
Si	58.3
K	3.44
Ca	30.0
Ti	0.23
Cr	0.086
Mn	0.70
Fe	1.65
Ni	1.24
Cu	0.20
Zn	0.07
Sr	0.42
Mo	4.98
Re	0.12
Eu	0.20

The analysis shows that bamboo ash leaf is rich in silica as the starting material of support preparation. However, the black ash must be removed from its impurities forming a pure white SiO_2. Silica from petung bamboo leaves ash is synthesized by sol gel method. The sol-gel method is a method of preparing a material starting with the sol formation which then produces a three-dimensional gelation of gel (gelation) and terminated by solvent removal [10]. In general, sol-gel synthesis occurs through two stages, ie 'pre-gelation' which includes reactant hydrolysis reaction to gel formation and 'post-gelation' including

drying and calcination [11]. The sol-gel method is relatively easy to perform, does not require a long time and the interaction between solids and immobilized materials is relatively easy.

The process of making silica is done by making precursornya first, sodium silicate. A total of 20 grams of petung bamboo ash leaves were dissolved in 160 mL NaOH 4M in stop erlenmeyer. The process aims to dissolve the base or basic destruction. The mixture was stirred while heated at 105 ° C for 90 minutes. The residue was burned at a temperature of 500oC for 30 minutes to accelerate the process of changing the leaves of bamboo petals into sodium silicate (Na_2SiO_3). The solid is brown whitish. The obtained solids were dissolved in 200 mL demineralized water and allowed to stand for 3 nights to form a sodium silicate solution. The formed solution is then filtered using Whatmann 42 filter paper aims to separate the insoluble brown precipitate. The resulting sodium silicate solution is brownish yellow. The reactions that occur during this process are:

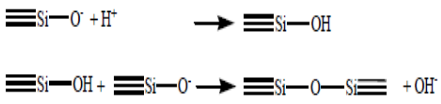
$SiO_{2(s)} + 2\ NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(aq)}$

High temperatures cause the NaOH to melt and dissociate completely into sodium ions and hydroxide ions. In SiO_2, the high electronegativity of the O atom causes Si to be more electropositive and form an unstable [SiO_2OH] intermediate, then dehydrogenation and the second hydroxyl ion binds to hydrogen to form water molecules. Two Na ions will balance the negative charge formed and interact with the SiO_3^{2-} ions thus forming sodium silicate (Na_2SiO_3). The sodium silicate solution is then added HCl 1 M dropwise while stirring using a magnetic stirrer to have a pH of 7. The addition of 1 M HCl to sodium silicate solution by stirring technique can increase the resulting silica content [12]. The addition of HCl to pH 7 in sodium silicate solution occurs H_2SiO_3 formation, followed by the reaction of the formation of $Si(OH)_4$ acid sol. The reaction can be described as follows:

$NaSiO_{3(aq)} + 2HCl_{(aq)} \rightarrow H_2SiO_{3(aq)} + 2NaCl_{(aq)}$

$H_2SiO_{3\ (aq)} + H_2O_{\ (l)} \rightarrow Si(OH)_{4\ (aq)}$

The addition of 1 M HCl to Na_2SiO_3 solution resulted in a decrease in pH, so that the concentration of H^+ in Na_2SiO_3 increased. This causes silicates to turn into silicic acid (H_2SiO_3) which causes some of the siloxane (S -O-) groups to form silanol groups ($Si(OH)_4$). $Si(OH)_4$ is polymerized by forming a crosslinkage of ≡Si—O—Si≡ to form silica gel through a condensation process, according to the following reaction Equation:



The formed gel is then allowed to stand for 72 hours. The formed gel was washed with demineralized water and the residue was dried in an oven at 80 ° C for 3 hours. The silica powder formed was then crushed and sieved with a 100 mesh sieve. The silica powder was then calcined using furnace at 400°C and also 800 ° C for 4 hours.

Characterization of Silica from Petung Bamboo Ash Leaves

The formed SiO_2 is then characterized to see which SiO_2 is made as desired. The SiO_2 sample is characterized by FTIR to see the bonds that occur in the sample. SiO_2 spectra is shown in Figure 1. In the spectra there is a peak in the region of 617.22 cm^{-1} is a Si-H bond, peak at 786.96 cm^{-1} and 1095.57 cm^{-1} is Si-O-Si bond. The peak at 3425.56 cm^{-1} shows the Si-O-H bond. This result proves that the synthesized SiO_2 is suitable. Furthermore, characterization of the structure using XRD to know the structure and crystallinity. This characterization is performed on calcined SIO_2 at two different temperatures ie 400°C and 800°C.

The synthesized SiO_2 diffractogram is shown in Figure 2 and 3. In Figure 2 (a) shows a calcined SiO_2 difractogram at a temperature of 400° C. The diffractogram is similar to the standard SiO_2 listed in the Joint Committee for Powder Diffraction Standard for SiO2 (Figure 2b).

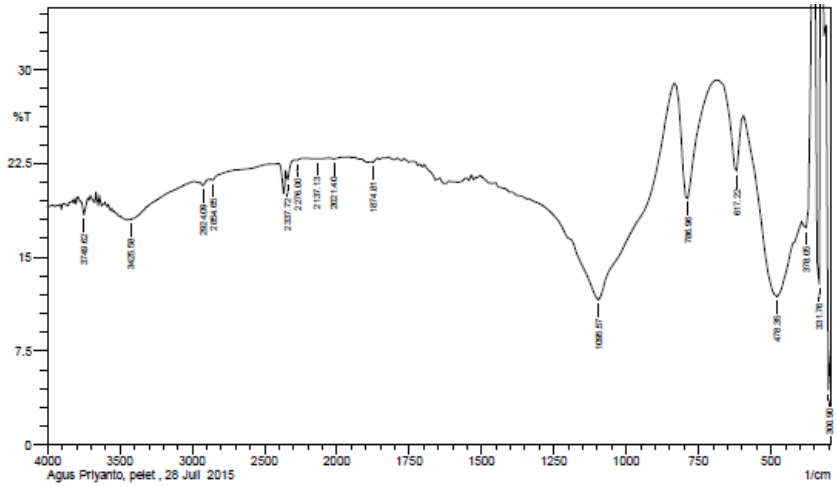


Figure 1. FTIR Spektra of SiO_2

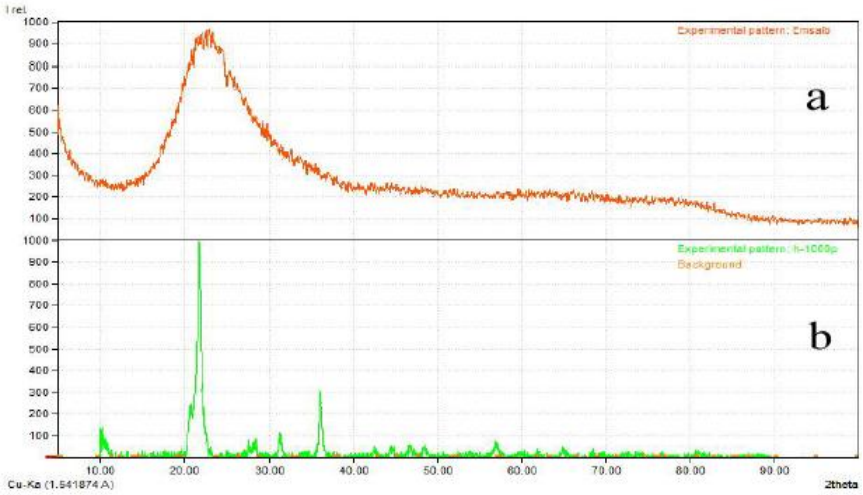


Figure 2. (a) SiO_2 difractogram calcinated at 400^{o}C (b) SiO_2 standard from JCPDS

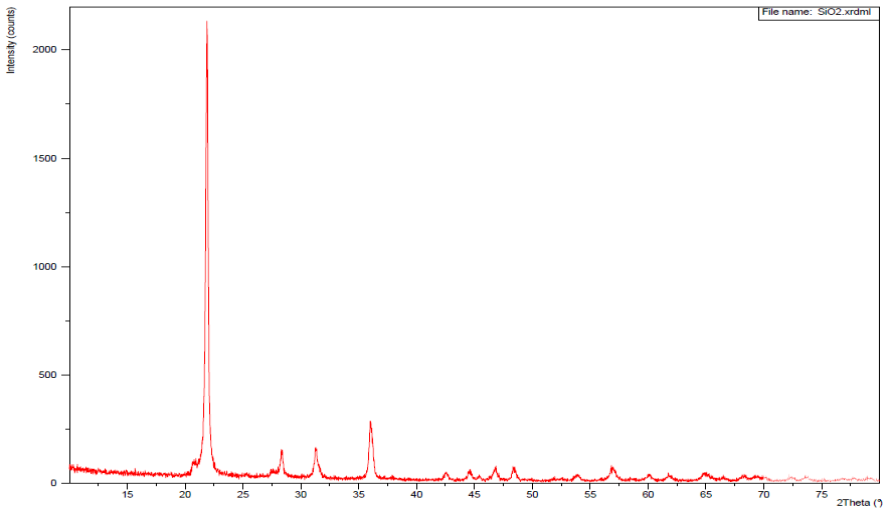


Figure 3. SiO_2 difractogram calcinated at 800^{o}C

According to JCPDS SiO_2 the specific peak standard for SiO2 lies at 2θ 21.99; 31,67 and 38,88. Diffractogram obtained showed widening peaks of several specific peaks of SiO_2 so that some peaks did not appear ie peaks at 2θ 31.67 and 38.88. This result shows the synthesized SiO_2 has very low crystallinity and degree of order [13].

Different things happen to the calcined SiO_2 of the synthesized at a temperature of 800 ° C. The SiO_2 peak is very sharp and the specific SiO_2 peak appears at 2θ 21.99; 31.67 and 38.88. The sharp peak shows that SiO_2 has a very high crystallinity [14]. This also corresponds to the research of [15] who reported that the crystallinity of a material is influenced by the temperature of calcination in the synthesis process. Based on the structure characterization by FTIR and XRD it provides evidence that the synthesized silica by sol gel method succeeded in producing SiO_2.

4. Conclusion

In conclusion, solid cilica (SiO_2) can be synthesized from daum bamboo ash by sol gel method. Characterization of SiO_2 structure with XRD seen peak at 2θ 21.99; 31,67 and 38,88 shows strong character of Silica. In other hand, characterization with FTIR appears peak at 617,22 cm^{-1} area is Si-H bond, peak at 786,96 cm^{-1} and 1095,57 cm^{-1} is Si- O-Si..

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