

**Silica Content Analysis of Siam Unus Rice Husks from South Kalimantan**

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**Abstract**

Research on analysis of rice husk content of Siam Unus with various NaOH concentrations (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M) has been done. This study aims to obtain data on the effect of variations in NaOH concentration on the purity of the silica extract from Siam Unus rice husks and silica characterization of Siam Unus rice husks using Fourier Transform Infra-Red (FTIR) and X-ray fluorescence spectrometry (XRF). The results showed that the combustion of rice husks at 200 °C as optimum temperature for 1 hour followed by combustion at 600 °C for 4 hours produces grayish-white rice husks with a yield is 20.70%. Silica functional group characterization showed that silanol (Si-OH) and siloxane (Si-O-Si) as dominant functional groups. The result of composition characterization using XRF showed that SiO<sub>2</sub> as the dominant compound with the highest percentage of SiO<sub>2</sub> is 1.5 M NaOH extract at 42.80%.

*Keywords: Siam Unus rice husk ash, extraction, silica, FTIR, XRF.*

**INTRODUCTION**

Indonesia is an agricultural country with the main agricultural product is rice which is found in various regions, including South Kalimantan Province. Based on data on the 2018 rice harvest area of 278,853 hectares and rice production of 1.14 million tons of GKG (dry milled rice) which has increased every year (Badan Pusat Statistik Provinsi Kalimantan Selatan, 2018). South Kalimantan agriculture is known as the Tidal Swamp agro-ecosystem. The local rice varieties planted were the Siamese, Bayar, Pandak, Mayang, Karang Duku, and Lemo groups. The variations of the name are named based on the shape of the grain, the taste of rice, the name of the farmer, or the special characteristics received by the farmer. The Siamese variety group is mostly found with various names at the farmer level (Khairullah & William, 2007). Farmers' preferences for local rice varieties are ease of cultivation, minimal production, high selling prices, and preferred characteristics of rice or rice (Wahdah, Langai, & Sitaresmi, 2015). The Siam Unus rice variety is one of the various types of local rice developed in South Kalimantan because it has a sweet and savory taste and a high selling value.

Siam Unus rice, apart from being a source of staple food, is also a producer of rice waste. Rice husk is a by-product of rice milling which consists of 38% cellulose, 22% lignin, 20% ash, 18% pentosane, and about 2% other compounds. The rice milling process produces 65% rice, 15-20% husks, 8-12% bran, ± 5%

groats (Fatriansyah et al., 2018). The high content of silica in rice husks 87%-97% has the potential to be used as a source of silica after going through the combustion process (Mittal, 1997).

Silica has two forms, namely morphological and crystalline silica. Silica by heating at a temperature of 700 °C is called morphological silica, while at a temperature of 800-900 °C it is called crystalline silica. The reactivity of silica is related to the structure of silica, where morphological silica is more reactive than crystalline silica due to the presence of a hydroxyl group (silanol) which is obtained after heating to a temperature of 400 °C (Mujiyanti, Nuryono, & Kunarti, 2016). According to Kalapathy, Proctor, & Shultz (2002) extraction is carried out using an alkaline solvent, namely extraction and precipitation processes at low pH using an acid solution to obtain amorphous silica. The extraction method is based on the high solubility of morphological silica in alkaline solutions such as KOH, Na<sub>2</sub>CO<sub>3</sub>, NaOH, and precipitation of soluble silica using HCl, H<sub>2</sub>SO<sub>4</sub>, CH<sub>3</sub>COOH, C<sub>2</sub>H<sub>2</sub>O<sub>4</sub> (Handayani, Nurjanah, & Rengga, 2014).

Several studies on silica extraction such as extracted silica from rice husks using 1N NaOH with a two-cycle extraction method and produced a yield of 91% (Kalapathy et al., 2002). Another study (Agung M, Hanafie Sy, & Mardina, 2013) carried out the extraction of silica from rice husks using KOH solution at various concentrations and 10% HNO<sub>3</sub> solution as a precipitate and obtained a yielding mass of 1.8690 grams from 50 grams of rice husk ash at a solution

concentration KOH 1.5% for 30 minutes (Agung M et al., 2013). Then another study extracted silica and obtained a yielding mass of 3.939 grams with 7 N NaOH for 2 hours (Sasri, Nurlina, Destiarti, & Syahbanu, 2018). Thus, in this study was conducted analysis of the silica content of siam unus rice husks south kalimantan

## METHODOLOGY

### Materials and Instrumentals

The tools used in this study were a 240 mesh sieve (Retsch), spray bottle, porcelain cup, furnace (Heraeus Hanau Type: KR-170-E), a set of chemical glassware (Pyrex), analytical balance (OHAUS model Galaxy TM 160), analytical balance (Mettler AE 160, Germany), oven (Mettler), universal pH indicator, stirring hot plate (CB 302), thermometer, X-Ray Fluorescence, XRF (Rigaku Type NEX DE) and Fourier Transform Infrared, FTIR brand (Bruker Spectrometer Type Alpha II). The materials used in this study were Siam Unus rice husk, H<sub>2</sub>SO<sub>4</sub> (Merck), NaOH (Merck), NH<sub>4</sub>OH, Whatman paper no. 4, and aquadest.

### Methods

#### Rice Husk Preparation

The rice husks used in this study were taken from the village of Kayu Bawang, Gambut District, Banjar Regency, South Kalimantan. The rice husk preparation process is carried out by cleaning the dirt or dust and soil using clean water. Then it soaked in hot water for 2 hours, then filtering and washing again three times using hot water. The cleaned husks were then dried in the sun, then continued with drying using an oven at a temperature of 110 °C for 3 hours (Pratomo, Wardhani, & Purwonugroho, 2013).

#### Ashing of Rice Husk

The dried husks were put into a porcelain cup, then quenched at an initial temperature of 200 °C for 1 hour using a furnace. After that, the temperature was increased to 600 °C for 4 hours until white ash was formed. Furthermore, the resulting ash was ground and sieved using a 240 mesh sieve (Apriliani, 2016).

#### Rice Husk Ash Silica Purification

The purification process was carried out by adding 10 grams of rice husk ash obtained in each beaker with a size of 250 mL (A, B, C, D, E). Each beaker was added with 80 mL of NaOH successively with successive concentrations of 1 M; 1.5 M; 2 M; 2.5 M; 3 M, then heated using a stirring hot plate at 95 °C for 60 minutes while stirring using a magnetic stirrer. The

next process is filtered using Whatman paper no. Then the filtrate was washed with 20 ml of warm water, then cooled at room temperature, and left overnight. The filtrate obtained is a solution of sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>). Sodium silicate was added dropwise with 5M H<sub>2</sub>SO<sub>4</sub> solution while stirring with a magnetic stirrer until gelatin was formed to pH 2 and NH<sub>4</sub>OH solution was added to pH 7 (universal indicator). The gel formed was left overnight at room temperature, then filtered, washed with warm water, and dried in an oven at 1100 °C for 5 hours, and weighed and calculated the percentage of silica yield obtained.

#### X-ray Fluorescence Analysis

The analysis to be identified is the elements contained in silica. The sample to be tested is placed in the sample holder then close the lid and run XRF. The sample will be exposed to X-rays, then it will be forwarded to the detector and then analyzed for the elements contained in the sample.

#### Fourier Transform Infrared (FTIR) Analysis

The silica analysis which will be characterized by its functional group is mixed with KBr solution and then pellets are made. Furthermore, the pellets are placed on the sample holder and placed on the FTIR beam path. The appliance is connected to a power source, then the computer and the appliance are turned on. In the next step, the FTIR tool will measure the sample and the form a graph. The resulting data is printed for discussion in analyzing the peaks formed.

## RESULTS AND DISCUSSION

### The Siam Unus Rice Husk

The rice husks taken were washed with clean water and soaked in hot water. Washing aims to remove impurities such as dust and soil. Pratomo, Wardhani, & Purwonugroho (2013) have been done a research by using the clean husks were then dried in the sun, then continued with drying using an oven at a temperature of 110 °C for 3 hours which aims to reduce the water content in the rice. The observation data of Siam Unus Rice Husk shown in Tabale 1.

### The Rice Husk Ash

Ashing proces aims to remove content such as cellulose, lignin, and hemicellulose in rice husks and convert the remaining carbon in the ash into CO<sub>2</sub>. Hydrogen gas becomes H<sub>2</sub>O and silicon becomes SiO<sub>2</sub>, while CO<sub>2</sub> and H<sub>2</sub>O gases evaporate at high temperatures, so it is expected that the ash will contain SiO<sub>2</sub> (Sriyanti, Taslimah, Nuryono, & Narsito, 2005).

Table 1. Observation data of siam unus rice husk

No.	Sample Code	Husk Mass (gram)	Ash Mass (gram)
1	A	30.00	6.56
2	B	32.55	6.98
3	C	30.23	6.96
4	D	30.26	6.34
5	E	33.90	7.22
Total Weight		156.94	34.06

Figures 1 and 2, shown that there is a change before and after the treatment of the burning process on rice husk, namely the rice husk ash produced is grayish-white. So based on Table 1, there was a reduction in the mass of rice husks by 156.94 grams to 34.06 grams of rice husk ash, due to the combustion process at high temperatures. The combustion results in the content of rice husks in the form of organic compounds being lost and turned into  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . From these data, it can be concluded that the average yield of rice husk ash is 20.70%.



Figure 1. Siam Unus Rice Husk



Figure 2. The ash result of Siam Unus Rice Rusk

In  $\text{SiO}_2$  the high electronegativity of O atoms causes Si to be more electropositive and an unstable intermediate ( $\text{SiO}_2\text{OH}$ ) is formed. This happens because of dehydrogenation and the second hydroxyl ion will bind to hydrogen to form  $\text{H}_2\text{O}$  and  $\text{SiO}_2$ - ( $\text{Na}_2\text{SiO}_3$ ) molecules to form sodium silicate as shown in Figure 3. This sodium silicate solution is then added dropwise of 5 M  $\text{H}_2\text{SO}_4$  solution to reduce the level of impurities in the form of metal oxides such as  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ , and  $\text{CaO}$  while stirring using a magnetic stirrer until silica is formed to  $\text{pH} = 2$  ( $\text{pH}$  universal indicator) and continued with  $\text{NH}_4\text{OH}$

solution until neutral (Mujiyanti, Nuryono, & Sri, 2010).

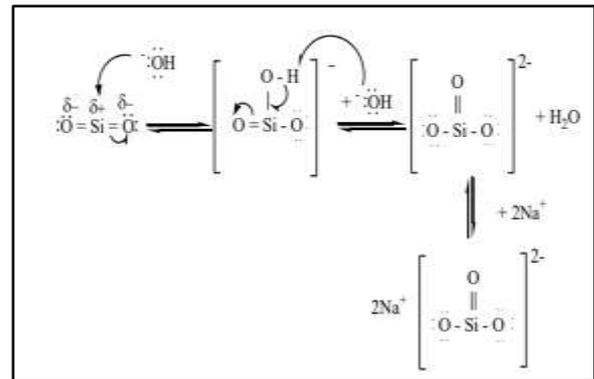


Figure 3. Reaction Mechanism of Sodium Silicate Formation (Mujiyanti et al., 2010)

The  $\text{NH}_4\text{OH}$  solution is used to raise the  $\text{pH}$  of a neutral acid, where the mini-ionized salts become  $\text{NH}_4^+$  and  $\text{OH}^-$  to neutralize excess  $\text{H}^+$  ions in the solution. The solution of 5.0 M  $\text{H}_2\text{SO}_4$  added to the sodium silicate solution will form siloxane ( $\text{Si-O-Si}$ ) and silanol ( $\text{Si-OH}$ ) groups from the interactions between species of silicate ions. The addition of 5 M  $\text{H}_2\text{SO}_4$  causes protonation of the siloxy ( $\text{Si-O}^-$ ) group to become silanol ( $\text{Si-OH}$ ). The silanol group that was formed was then further attacked by a siloxy group ( $\text{Si-O}^-$ ) with the help of an acid catalyst to form siloxane ( $\text{Si-O-Si}$ ) bond. This process occurs rapidly and continuously to form an amorphous silica network. The attack of  $\text{Si-O}^-$  on  $\text{Si-OH}$  forms  $\text{Si-O-Si}$  by condensation of an acid-catalyzed sol solution.

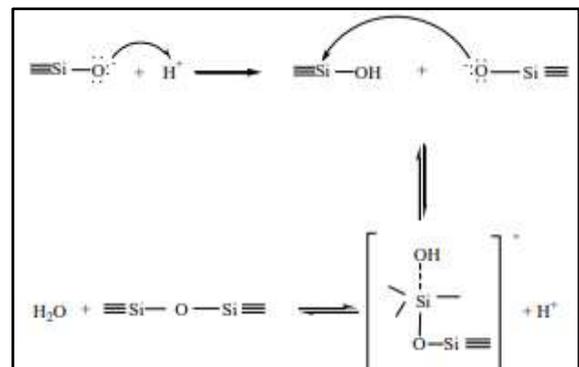


Figure 4. Reaction Mechanism of Silica Gel Formation

The expected reaction mechanism for the formation of silica gel from acidification of sodium silicate solution is shown in Figure 4. Furthermore, the maturation process is carried out for 18 hours.

Table 2. FTIR Wavenumbers Data of Silicate

No.	Functional Groups	Reference Wavenumbers	Rice Husk Ash	Wavenumbers (cm <sup>-1</sup> )				
				NaOH (M)				
				1.0	1.5	2.0	2.5	3.0
1	Symmetric stretching vibration of Si-O on Si-O-Si	801-809	809	809	801	801	792	809
2	Asymmetric stretching vibration of Si-O on Si-OH	949-966	-	966	949	949	966	958
3	Symmetric stretching vibration of Si-O on Si-O-Si	1088-1097	1088	1097	1097	1097	1097	1088
4	Bending vibration of OH on Si-OH	1637-1645	1645	1637	1637	1637	1645	1645
5	Bending vibration of Si-O-Si	2318-2468	-	2351	2342	2351	2351	2333
6	-OH groups of Si-OH	3448-3456	3448	3446	3456	3456	3456	3456

After the ripening process, the silica formed is then washed with warm water to produce Na<sub>2</sub>SiO<sub>4</sub> salt, where this solution has chemical properties that only dissolve in distilled water at a temperature of 20 °C, and helps increase the solubility of foreign substances and accelerates the filtration. Then the silica is filtered and dried. Silica drying is carried out in an oven at 100 °C for 5 hours so that dehydration occurs in the hydrogel and the formation of silica with a SiO<sub>2</sub>.xH<sub>2</sub>O structure called xerogel (silica powder) (Mujiyanti, Komari, & Sari, 2013). The final product produced is solid silica (dry silica).

#### Fourier Transform Infrared (FTIR) Analysis

Fourier Transform Infrared Spectroscopy (FTIR) method is an analytical method used to identify compounds indicated by specific peaks indicating the type of functional group possessed by Siam Unus rice husk ash and silica. The basic principle of infrared spectroscopy is the interaction between the vibrations of bonded atoms/functional groups in a molecule by adsorption of infrared electromagnetic wave radiation. The absorption of infrared radiation can cause the excitation of the vibrational energy of the molecule to a higher vibrational energy level. To be able to absorb, the molecule must have a change in dipole moment as a result of vibration (Retnosari, 2013). Figure 5 above shows the spectra of Siam Unus rice husk ash and the resulting silica. Several peaks indicate the presence of functional groups before and after the extraction of rice

husk ash with various concentrations of NaOH (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M) at wavenumbers 500-4000 cm<sup>-1</sup>.

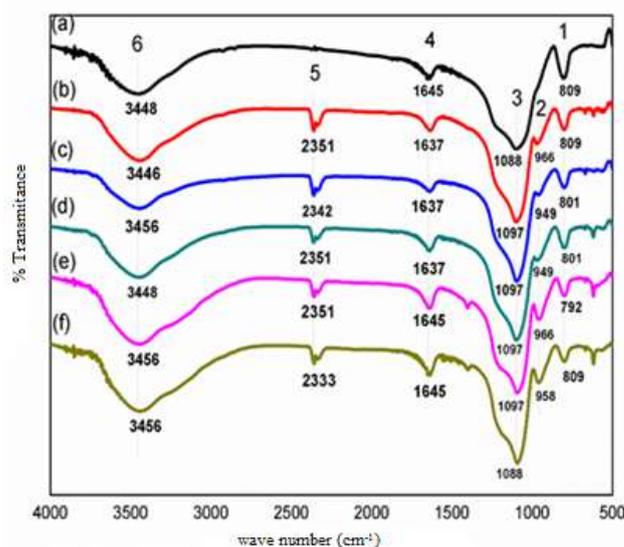


Figure 5. FTIR Spectra of (a) Siam Unus Rice Husk Ash, and silicate after extracted by NaOH variation concentration (b) 1.0 M, (c) 1.5 M, (d) 2.0 M (e) 2.5 M, (f) 3.0 M

Spectra analysis shows the results of peaks formed by functional groups because these compounds will absorb electromagnetic radiation in the infrared region. FTIR spectroscopy is measurement technique that collects infrared spectra. The identification of each functional group can be seen in Table 2. The peaks of

the five spectra are in the range of wavenumbers, namely 801-809  $\text{cm}^{-1}$  which shows the symmetrical stretching vibration of Si-O from Si-O-Si, the range of wave numbers 940-966  $\text{cm}^{-1}$  shows asymmetric stretching of Si-O from Si-O-Si, while the wave number in the range of 1088-1097  $\text{cm}^{-1}$  indicates a Si-O stretching asymmetry of Si-O-Si, in the wavenumber range of 1637-1645  $\text{cm}^{-1}$  shows the -OH bending vibration of  $\text{H}_2\text{O}$ , the wavenumber 2318-2468  $\text{cm}^{-1}$  indicates the buckling vibration of Si-O and the range of wave number 3448-3456  $\text{cm}^{-1}$  indicates the presence of the OH-group of Si-OH. Characteristic identification was strengthened by the absorption band at 470  $\text{cm}^{-1}$  which is the Si-O-Si bending vibration while the absorption band at 794  $\text{cm}^{-1}$  shows the O-Si-O bending vibration characteristic (Mujiyanti, Nisa, Rosyidah, Ariyani, & Abdullah, 2020; Sekewael, Wijaya, & Triyono, 2018)

shift in the number of waves at each peak shows the effect of varying NaOH concentrations (Pavia, Lampman, Kriz, & Vyvyan, 2013).

### X-ray Fluorescence Analysis

XRF analysis was carried out to analyze the chemical composition and the concentration of elements contained in the rice husk ash silica after purification using various concentrations of NaOH. XRF analysis is a qualitative method that provides identification of chemical species in the sample while quantitative analysis determines the number of certain components in a substance. The working principle of XRF analysis is based on the occurrence of collisions between atoms on the surface of the sample by X-rays.

Data from XRF analysis of Siam Unus rice husk ash silica with various concentrations of NaOH as an extractor can be seen in Figure 6. Based on Figure 6,

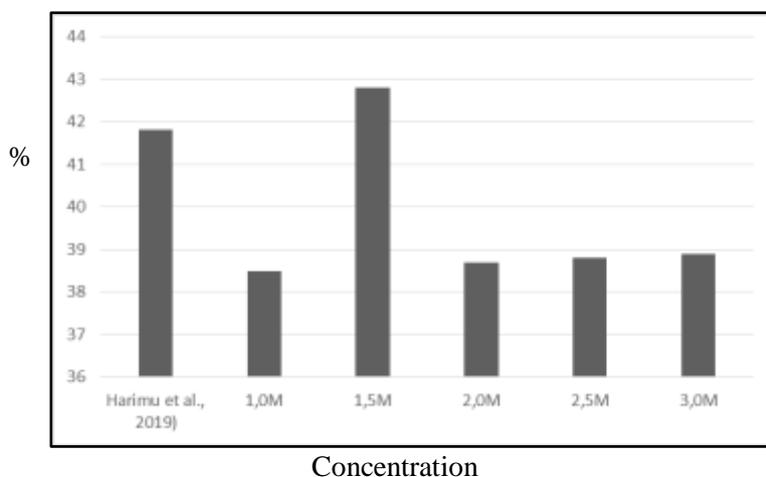


Figure 6. XRF Percentage of  $\text{SiO}_2$  Siam Unus Rice Husk

The absorption peak with a wavenumber range of 956-964  $\text{cm}^{-1}$  shows the Si-O stretching asymmetry of Si-O-Si which is only found in silica samples after passing NaOH treatment with various variations (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M), but peaks in the range of wave numbers did not appear in the husk ash samples. The peak with a wavenumber range of 2318-2468  $\text{cm}^{-1}$  indicates Si-O bending vibrations that are not visible in the husk ash, while the peak in the wavenumber range appears in the spectra of silica samples that have been treated with NaOH (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M). This indicates that silica has been extracted from the Siam Unus rice husk ash sample after undergoing treatment with NaOH (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M). From the five samples, it can be seen that the difference in wave number is not too significant. The

shows the results of XRF analysis of Siam Unus rice silica with various concentrations of NaOH (1.0 M; 1.5 M; 2.0 M; 2.5 M; 3.0 M) which is more dominating is  $\text{SiO}_2$  compound with the highest percentage than other metal compounds are  $\text{P}_2\text{O}_5$ , CaO,  $\text{Fe}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3$ . A previous study by (Harimu, Rudi, Haetami, Santoso, & Asriyanti, 2019), was found at a NaOH concentration of 3.0 M with a percentage of 41.81% using rice husk ash samples from Mataiwoi Village, Southeast Sulawesi. Silica yields from Siam Unus rice husk ash with various NaOH concentrations obtained the optimum percentage at a concentration of 1.5 M of 42.80%. This is due to the differences in the sources of rice husks used and the relatively different geographical conditions where rice plants grow (Tufaila & Alam, 2014). According to research and development of

swampland, it is known that the tidal swampland of South Kalimantan is a peat soil group because it has soil with a pH range of 4.0-4.5 and also has a land typology with high levels of aluminum and iron (Haryono, Noor, Syahbudi, & Sarwani, 2013).

In this study, the results of Siam Unus rice husk silica with the percentage of SiO<sub>2</sub> at a concentration of NaOH 1.0 M was 38.50%, then increased into 42.80% at a concentration of 1.5 M NaOH, but decreased at various concentrations of 2.0 M, 2, 5 M and 3.0 M were 38.70%, 38.80%, and 38.90%, respectively. This is because the higher the concentration and viscosity of the NaOH solution causes a decrease in ionic activity in the solution so that the number of bonds formed between NaOH and SiO<sub>2</sub> in rice husk ash decreases (Harimu, Rudi, Haetami, Santoso, & Asriyanti, 2019; Agung M, Hanafie Sy, & Mardina, 2013).

## CONCLUSION

Based on the research that has been done, it can be concluded that the extraction of Siam Unus rice husk ash which was carried out with the highest silica content was obtained at a concentration of 1.5 M NaOH which was 8.59 grams with a content of 85.97%. The results of the FTIR data analysis of the Siam Unus rice husk ash sample and the extracted silica showed the dominant functional groups were silanol and siloxane. The results of the XRF analysis showed that the highest percentage of SiO<sub>2</sub> obtained was at a concentration of 1.5 M NaOH at 42.80%.

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