

# **The Effect of Rice Varieties on Silica Purification from Rice Husk Ash as Adsorbent for Mn(II)**

# **Kajian Pengaruh Varietas Padi pada Pemurnian Silika Dari Abu Sekam Padi Sebagai Adsorben Mn(II)**

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### **ABSTRACT**

The influence of rice types on the purification of rice husk ash was investigated by extracting silica and purifying it with a 12% NaOH solution, followed by the coprecipitation technique. Rice husk samples were from Mbay (Nagekeo Regency) with two different varieties, Ciherang and Inpari 42, and from Soa (Ngada Regency) with Ciherang variety. The silica produced in this research was characterized using UV Vis spectrophotometry to obtain the surface area value. The surface acidity value was calculated using the acidbase titration method. Using XRF, the purity of silica was determined. Ciherang Mbay, Ciherang Soa, and Inpari Mbay had surface area values of 18.397 m2/gram, 18.347 m<sup>2</sup>/gram, and 18.491 m<sup>2</sup>/gram, respectively. The surface acidity value of the three samples was 45.1 mmol/gram, 45.5 mmol/gram, and 44.7 mmol/gram, respectively. The purity of silica from rice husk ash Ciherang Mbay, Ciherang Soa, and Inpari Mbay samples based on XRF was 98.6%, 98.3%, dan 99.2%, respectively. Silica with the highest purity (Inpari Mbay variety) was applied as an adsorbent in the adsorption process of manganese (Mn(II)) metal ion with an adsorption capacity of 212.76 µmol/gram and adsorption energy of 38.165 kJ/mol through chemical adsorption.

**Keywords**: purification, rice varieties, silica, adsorption, manganese

### **ABSTRAK**

*Pengaruh varietas padi terhadap proses pemurnian silika dari abu sekam padi telah dikaji melalui ekstraksi silika dengan metode pelarutan menggunakan larutan NaOH 12% yang dilanjutkan pengendapan dengan asam sulfat. Sampel sekam padi yang digunakan berasal dari daerah Mbay (Kab. Nagekeo) dengan dua varietas padi yang berbeda yaitu Ciherang dan Inpari 42 serta dari daerah Soa (Kab. Ngada) dengan varietas Ciherang. Silika yang dihasilkan dikarakterisasi menggunakan spektrofotometri UV Vis untuk mendapatkan nilai luas permukaan, sedangkan nilai keasaman permukaan ditentukan dengan metode titrasi asam basa serta dikarakterisasi kemurnian silika menggunakan XRF. Nilai luas permukaan silika yang dihasilkan dari sampel Ciherang Mbay, Ciherang Soa dan Inpari Mbay berturut turut adalah 18,397 m<sup>2</sup> /gram, 18,347*  m<sup>2</sup>/gram dan 18,491 m<sup>2</sup>/gram serta nilai keasaman permukaan dari ketiga sampel berturut turut adalah 45,1 *mmol/gram, 45,5 mmol/gram dan 44,7 mmol/gram. Kemurnian silika dari sampel abu sekam padi varietas Ciherang Mbay, Ciherang Soa dan Inpari Mbay berdasarkan hasil XRF berturut-turut adalah 98,6%, 98,3% dan 99,2%. Silika dengan kemurnian tertinggi (varietas Inpari Mbay) diaplikasikan sebagai adsorben pada proses adsorpsi ion logam Mn(II) dengan nilai kapasitas adsorpsi sebesar 212,76 µmol/gram dan energi adsorpsi sebesar 38,165 kJ/mol sehingga dapat diindikasikan bahwa terjadi proses adsorpsi secara kimia.*

**Kata Kunci:** *permurnian, varietas, silika, adsorpsi, mangan*

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## **1. INTRODUCTION**

The rice husk is the outermost layer of the rice grain, which has a high carbon and ash content and is resistant to weathering. Rice husk is composed of cellulose (34-44%), lignin (20-30%), ash (13-39%), and water (8-15%) (Trivana *et al*., 2015). Rice husk ash also contains silica, small amounts of alkali, and metal impurities. When burned at high temperatures (600°C) in a controlled manner, rice husks will produce silica ash which can be used in various chemical processes. Typically, the silica percentage of rice husk ash is between 94 and 96%. (Sriyanto and Darwanta, 2017).

Silica is a compound commonly found in minerals such as quartz sand. Sand has high crystallinity and contains many impurities, reducing its ability as an adsorbent (Mujiyanti and Kunarti, 2010). Therefore, silica was synthesized from rice husk to increase its qualities and replace silica derived from nature. Silica extracted from rice husk is also utilized in producing other silica-based substances. Wogo and Ndoen (2020) synthesized a composite of immobilized silica EDTA-Ag and chitosan to manufacture antibacterial plastics that could kill Escherichia coli and Staphylococcus aureus bacteria up to 99.99%. Ngatidjo *et al*. (2011) also utilized silica from amine-modified rice husks as an adsorbent for copper metal ions. In addition, research on silica from rice husks was carried out by Valentine *et al*. (2019), who produced chitosan membranes by adding rice husk ash silica as an additive to a casting solution to reduce Cu heavy metal.

Silica purification from rice husk ash is essential due to its wide range of applications. Additionally, the quality of the generated silica affects its applicability. Trivana *et al*. (2015) synthesized sodium silicate from rice husk. Based on EDS, the sodium silicate did not contain impurities in the form of C atoms and metal impurities.

Harimu *et al*. (2019) obtained silica from processing rice husk ash using NaOH  $(89.09\%)$  and  $H_2SO_4(94.94\%)$ . Sriyanto and Darwanta (2017) investigated the influence of acid type on the purification of rice husk ash, where  $H_2SO_4$  (99.48%) purification is superior to HCl (99.35%) and HNO<sub>3</sub> (99.38%) purification.

Silica is one of the important elements for rice growth and is mostly taken from the growing medium. Hence, the Si content in each rice variety can vary. In addition to variations, the soil type affects the Si content in rice. Lede *et al*. (2021) have proved that changes in cultivars and soil types can affect the Si content in rice husk ash. The silica content of the Ciherang variety sample from Tarus (99.1%) and Pariti (98.4%) was higher than that of the Inpari variety sample from Pariti (98.1%). The samples taken from two rice-producing locations in Kupang Regency reveal that the soil type impacts the Si content in rice. The soil type in the Tarus area is vertisol soil with a rather high Si content.

Mn is one of the essential heavy metals because it builds bone structure and helps bone metabolism and enzyme work. Nonetheless, if the concentration within the body is excessive, it will be poisonous and cause harm to the central nervous system and lungs. Manganese consumption should not exceed 11 mg per day because it can produce signs of nervous system diseases. In dust, the manganese concentration should not be more than 5 mg/m3 because it might induce respiratory diseases (Syuhada, 2018). In addition, high quantities of manganese in the environment can contribute to air, water, and soil pollution. Therefore, improper handling of this substance can be dangerous to humans, animals, plants, and the environment. This research uses silica as an adsorbent to reduce Mn pollution in the environment.

## **2. MATERIALS AND METHODS**

## *2.1. Materials*

The materials were rice husks of the Ciherang variety from Mbay (sample 1) and Soa (sample 2) and the Inpari 42 variety from Mbay (sample 3),  $H<sub>2</sub>SO<sub>4</sub>$ , NaOH, PP indicator, methylene blue solution, MnSO<sub>4</sub>.4H<sub>2</sub>O, HCl and distilled water. The equipment used was glassware, sieve, magnetic stirrer, analytical balance, oven, furnace, UV-Vis Spectrophotometer, X-Ray Fluorescence (XRF), and Atomic Absorption Spectrophotometer (AAS).

#### *2.2. Preparation of Rice Husk Samples*

Rice husks were collected from Mbay (Nagekeo Regency) and Soa on Flores Island, East Nusa Tenggara Province, Indonesia (Ngada Regency). There were two rice varieties taken from Mbay, namely the Ciherang variety (sample 1) and Inpari 42 (sample 3), while from Soa, the Ciherang variety (sample 2) was used. Each rice husk sample was cleaned, washed, rinsed, and dried at  $110^{\circ}$ C for  $\pm$  1 hour. Furthermore, the husks are roasted until they turn black and then roasted using a furnace for  $\pm$  4 hours at 700°C. The sample was then pulverized and passed through a 100-mesh sieve. Fig. 1 depicts photographs of rice husks from each sample.

#### *2.3. Silica Extraction of Rice Husk Ash* a. Purification with NaOH Solution

Twenty grams of sample 1 were combined with 120 mL of 12% NaOH and heated at 85°C for 90 minutes with magnetic stirring. The sample was filtered, and the filtrate was neutralized with  $0.5$  M  $H<sub>2</sub>SO<sub>4</sub>$ until a gel precipitate formed, followed by 18 hours of incubation. The gel precipitate was filtered, washed with 1000 mL of hot distilled water, and dried in a 110°C oven for five hours. The identical procedure was applied to samples 2 and 3.

#### b. Precipitation with H2SO4 Solution

One gram of silica purified with NaOH for each sample was added with 10 mL of 10.88%  $H<sub>2</sub>SO<sub>4</sub>$ . The solution was then left for 120 minutes and washed using 200 mL of distilled water. The solution was then filtered, and the precipitate was dried in an oven at 110°C for 2 hours and then weighed. The yield was determined.

$$
\frac{\%Yield = \frac{Weight \ of \ silica}{Weight \ of \ sample}}{100\%}
$$

#### *2.3. Characterization of Silica*

Determination of the surface acidity of silica was carried out by the titration method (Wogo *et al*., 2014). The surface area of silica was measured using a spectrophotometric technique employing methylene blue as an adsorbate (Wogo *et al*., 2014). Silica purified from rice husk ash samples 1, 2, and 3 were analyzed using an XRF instrument to identify the purity of Si in the sample (Sapei *et al*., 2015). XRF analysis was also carried out on rice husk ash from the three samples.

# *2.4. Silica Applications for Adsorption of Mn Metals*

A beaker was filled with 0.1 grams of silica from sample 3 (Inpari 42). Using a batch system, adsorption was performed by adding 50 mL of  $MnSO<sub>4</sub>4H<sub>2</sub>O$  solutions with concentrations of 10, 20, 40, 80, 100, 150, and 200 mg/L. The solution was stirred for one hour before being centrifuged at 2,000 rpm for 30 mins to separate the filtrate from the adsorbent. AAS was utilized to determine the amount of metal ions adsorbed by the filtrate. The adsorption capacity was calculated using the Langmuir adsorption isotherm equation, and the adsorption energy was calculated using the Gibbs free energy equation under standard conditions.



**Figure 1.** Rice husk of each variety (a) sample 1, (b) sample 2, (c) sample 3



Figure 1. Rice husk of each variety (a) sample 1, (b) sample 2, (c) sample 3

#### **3. RESULTS AND DISCUSSION**

*3.1. Preparation of Rice Husk Samples*

In order to assist the pyrolysis process, sample preparation was performed. The coagulation procedure consisted of roasting or controlled burning until the rice husk changed color from brown to black to break down organic components, lowering the temperature and accelerating the ashing procedure. The following process was to remove the remaining organic compounds that makeup rice husks and completely oxidize all carbon to  $CO<sub>2</sub>$  gas and hydrogen to H2O vapor. The white color of the blasted ash suggests a high concentration of silica.

## *3.2. Extraction of Silica from Rice Husk Ash*

a. Purification with NaOH solution

NaOH was used in purifying silica from rice husk ash because silica has a high solubility at a pH above 9. This process produces a filtrate which is a solution of sodium silicate. When exposed to a sodium silicate solution, the skin will feel slippery, which is an early indicator of the solution's

formation (Ngatidjo *et al*., 2011). The reaction for the formation of sodium silicate is as follows:

$$
SiO_{2(s)} + 2NaOH_{(aq)} \longrightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \tag{1}
$$

After the sodium silicate solution was formed, it was followed by silica gel formation by adding  $0.5$  M H<sub>2</sub>SO<sub>4</sub> so that a neutralization reaction occurs, which formed silicic acid monomers and allowed the formation of a gel. The resulting gel was then allowed to stand and neutralized with hot distilled water. The following describes the neutralization process with sulfuric acid and washing with distilled water:

 $Na_2SiO_{3(aq)} + H_2SO_{4(aq)} \longrightarrow H_2SiO_{3(1)} +$  $\text{Na}_2\text{SO}_{4(\text{aa})}$  (2)

 $H_2SiO_{3(s)} \longrightarrow SiO_{2(s)} + H_2O$  (3)

Neutral silica gel was again dried to remove water. Silica purified using NaOH is presented in Fig. 2.



**Figure 2.** Silica purified with NaOH (a) sample 1, (b) sample 2, (c) sample 3

b. Precipitation with  $H_2SO_4$  solution

The precipitation process with a 10.88% H2SO4 solution (Harimu, 2019) aims to reduce impurities in the form of metal oxides such as  $Na<sub>2</sub>O$ ,  $K<sub>2</sub>O$ , and CaO produced in the extraction process with NaOH. The silica yield of the three samples is presented in Table 1.

Based on Table 1, the yield of silica from similar rice varieties can differ depending on the sampling location. The Ciherang variety contains the least amount of silica compared to the Inpari variety. As demonstrated in Fig. 3, silica from sulfuric acid precipitation yields powder with finer granules and a whiter hue than silica through NaOH purification. The characterization results using XRF for the three samples are presented in Table 2.

According to Table 2, silica purity increased across all treatments. Silica with the highest purity was found in sample 3, namely from the processing of rice husk ash of the Inpari variety at 99.2%. Different levels of silica purity in the three samples demonstrated a correlation between rice variety and soil nutrient content.

Based on the different varieties, sample 1 (Ciherang Mbay) and sample 3 (Inpari 42 Mbay) had different levels of silica purity even though they were planted in the same area. It is possible that they share the same nutrients. Silica is the most abundant nutrient absorbed by rice plants, and it plays a crucial role in boosting photosynthesis and plant tolerance to biotic and abiotic stressors (Putri *et al*., 2017). Where rice varieties are continually developed into superior varieties that can produce quality rice plants and increase the resistance of these plants to biotic and abiotic stresses (Tampoma *et al*., 2017), this underlies the assumption of this study that rice varieties can affect the level of silica purity produced. In contrast, the influence of soil can be seen in samples 1 (Ciherang Mbay) and 2 (Ciherang Soa), which come from different areas. In addition, the silica content of the two samples varied. According to Makarim *et al*. (2007), rice plants absorb silica from the soil at a much higher rate than other elements such as potassium, nitrogen, phosphorus, and calcium. Therefore, the silica content of the soil can also affect the silica purity produced by processing rice husk ash.

**Table 1** Silica yield

Sample	Yield $(\% )$
Sample 1	82.91%
Sample 2	83.40%
Sample 3	82.80%



**Figure 3.** Silica from the precipitation of (a) sample 1, (b) sample 2 and (c) sample 3

	Parameters $(\% )$								
Compound	Rice Husk Ash			Purification eith NaOH	Precipitation with $H_2SO_4$				
	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S1	S <sub>2</sub>	S <sub>3</sub>
SiO <sub>2</sub>	94.7	95.5	93.5	96.0	97.0	98.4	98.6	98.3	99.2
K <sub>2</sub> O	1.41	2.17	4.04	0.063	0.066	0.21	$\overline{\phantom{a}}$	-	
CaO	3.08	1.75	1.89	3.41	2.38	0.883	1.12	1.38	0.524
MnO	0.15	0.16	0.19	0.065	0.13	0.13	0.029	0.055	0.044
Fe <sub>2</sub> O <sub>3</sub>	0.369	0.19	0.18	0.23	0.13	0.11	0.11	0.073	0.065
CuO	0.043	0.052	0.059	0.043	0.050	0.055	0.031	0.033	0.033
ZnO	0.02	0.036	0.022	0.036	0.083	0.10	0.005	0.051	0.039
Re <sub>2</sub> O <sub>7</sub>	0.07	0.07	0.07	0.06	0.06	0.06	0.080	0.07	0.07
Eu <sub>2</sub> O <sub>3</sub>	0.05	0.04	0.05	0.03	0.03	0.03		0.04	

**Table 2** XRF data from rice husk ash and silica extraction using NaOH and H<sub>2</sub>SO<sub>4</sub>

#### *3.3. Silica characterization*

Surface acidity is the amount of total acid (Bronsted acid and Lewis acid) on the surface of a solid expressed as the number of mmol of acid per sample weight (Widihati, 2008). Data on the silica surface acidity of the three samples are presented in Table 3.

According to the surface acidity table, there is no statistically significant difference between the surface acidity values of the three samples. The sample with the greatest surface acidity is number 2, with a value of 45,5 mmol/gram.

The surface area can be determined based on the sample's number of methylene blue molecules adsorbed at the optimum contact time. Surface area determination was carried out at a maximum wavelength of 660 nm and a standard curve with the regression equation,  $y = 0.0904x - 0.0017$ , with an R<sup>2</sup> value of 0.999. The surface area data of the three samples are presented in Table 4.

Based on Table 4, the surface area of the three samples was not significantly different. Surface area is known to play an important role in the adsorption process. The greater the surface area of an adsorbent, the more adsorbate can be adsorbed by the adsorbent (Wogo *et al*., 2014). The highest surface area was obtained from sample 3 of 18.491 m<sup>2</sup>/g. The resulting surface area is higher when compared to a study conducted by Giri *et al*. (2014), who used the same method by utilizing silica gel from rice husk ash modified with diphenylcarbazide (Si-DPZida) to adsorb Cr(VI) ions with a surface area value of  $4.4538 \text{ m}^2/\text{g}$ .





Contact time (minutes)	Surface Area $(m^2/\text{gram})$					
	Sample 1	Sample 2	Sample 3			
30	16.686	18.111	17.875			
40	18.275	18.121	18.418			
50	18.397	17.485	18.142			
60	17.987	18.347	18.491			
70	17.711	18.141	18.480			

**Table 4** The surface area of the sample

# *3.4. Application of silica for adsorption of Mn metal*

The silica used for the adsorption of Mn metal ions was refined silica from sample 3 (Inpari 42 Mbay) with the highest silica content based on XRF data. The adsorption process was carried out on manganese metal ions with various concentrations between 10 to 200 mg/L with a contact time of 60 minutes according to the data for determining the sample's surface area.

The adsorption capacity was determined by the Langmuir adsorption isotherm equation. The slope value was used to calculate the adsorption capacity, and the intercept value to calculate the adsorption energy value. The adsorption capacity was calculated using the Langmuir isotherm equation and the curve showing the relationship between the amount of metal ion adsorbed on the adsorbent in an equilibrium state. The adsorption isotherm pattern curve is presented in Fig. 4.

Based on the curve in Fig. 4, the value of the adsorption capacity of the purified silica from sample 3 is 212.76 µmol/gram. This relatively high adsorption capacity indicates that the higher the silica purity, the better its application as an adsorbent. Silica has active groups on its surface in the form of silanol groups (-SiOH) and siloxane groups (Si-O-Si), both of which have an O atom, which is a hard base group (Wogo *et al*., 2014). Based on the HSAB theory, the  $Mn^{2+}$  metal ion is a group of strong acids that tend to bind to stable hard bases.

The adsorption energy value was 38.165 kJ/mol, indicating that there was a chemical adsorption process that involves bonds between the adsorbate and the surface of the adsorbent. According to Adamson (1990), chemical adsorption (chemisorption) can occur if the adsorption energy produced is more than 20.92 kJ/mol.



**Figure 4.** Graph of sample adsorption isotherm pattern 3



**Figure 5.** Graph for determining the adsorption capacity value of sample 3

### **4. CONCLUSION**

Based on the research performed, it could be concluded that:

- 1. The yield of silica produced by each rice variety varies. The yield of silica was 82.91% for the Ciherang Mbay sample, 83.40% for the Ciherang Soa sample, and 82.80% for the Inpari 42 Mbay sample.
- 2. Based on the XRF, the highest silica purity was found in the Inpari 42 sample from the Mbay area, namely 99.2%. In contrast, the acidity and surface area of the three samples showed no significant difference.
- 3. The adsorption capacity for Mn metal ions was 212.76 µmol/gram and was chemical adsorption with an adsorption energy of 38.165 kJ/mol.

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