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Production of Magnesium Silicate from Rice Husk with Variation of NaOH Concentration and Reaction Time Length Using Sol-gel Method

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Abstract

Rice husk is a by-product of rice production where as much as 9.9 million tons become organic waste. Rice husk ash, which is rich in silica, can be processed into materials that have high selling value. Magnesium silicate is one of the silica-based composite materials with the chemical formula MgSiO3 with an example of its application as a material in the manufacture of magnesium cement. The manufacture of magnesium silicate is done by sol-gel method with NaOH solvent where this method is relatively easy and uses simple equipment. This study was conducted to know the best NaOH concentration and length of reaction time to produce optimal magnesium silicate. The NaOH concentration variables used were 3 M, 5 M, 7 M, and 9 M, while the variable length of reaction time was 90 minutes and 150 minutes. The best NaOH concentration is 7M with 90 minutes of reaction time. This variable gave 49,9% yield of magnesium silicate compared to 3M with 90 minutes reaction time where it gave 17,4% yield. The analytical tests carried out were FITR where this test was carried out to confirm the formation of magnesium silicate.

Keywords: *Magnesium silicate; NaOH; Rice husk; Sol-gel.*

Introduction

Rice husks are the residual result of rice production which, according to Badan Pusat Statistik, estimated 9,9 million tons to be organic waste. Until now the utilization of rice husk has been limited to animal food, fuel, and plant-growing media. According to study that have been done Fatriansyah, Situmorang and Dhaneswara (2018), rice husk can be utilize into high-selling value material as the content of rice husk are 38% cellulose, 22% lignin, 20% ash, 18% pentose, and about 2% other organic compounds. In order for the rice husk to be substance rich in silicate, it is needed to burn so that the rice husk turn into ash. Rice husk ash (RHA) containing high amounts of silica about 86,9 – 97,3% therefore can be used as potential materials (Mujiyanti, Ariyani and Paujiah, 2021). Silica produced from rice husk ash have some advantage compared to silica from mineral, those are realtively inexpensive, more reactive, fine grain size, and abundant (Agung M, Hanafie Sy and Mardina, 2013).

Silica contained in RHA can be extracted with alkali solution which are Potassium Hydroxide (KOH) and Sodium Hydroxide (NaOH). Agung M, Hanafie Sy and Mardina (2013), reported they have succeeded in extraction silica from RHA with KOH 10% solution while having yield as high as 50,97%. While study conducted by Fatriansyah, Situmorang and Dhaneswara (2018) reported extraction of silica with NaOH solution can be done with having yield as high as 84%. Therefore, extraction silica from RHA with NaOH is more efficient. The scope of this study is production of magnesium silicate from rice husk with sol-gel methode. The objectives of this study are to know the effect of NaOH solution concentration against the production of magnesium silicate and to know the effect of reaction time of RHA and NaOH against the production of magnesium silicate.

Research Methods

There are 3 stages to obtain magnesium silicate from rice husk which are rice husk ash preparation, synthesis of sodium silicate, and synthesis of magnesium silicate. To obtain rice husk ash, rice husk needed to be burned with furnace at 600ºC for 4 hours and grind it to fine grain. Synthesis of sodium silicate begin with mixing of rice husk ash and NaOH solution (3M, 5M, 7M, and 9M) with ratio of 1:6 stirred for 90 minutes and 150 minutes while heated at 100ºC and then filtered with filter paper, afterward will be baked at 300ºC for 4 hours. In this stage the chemical reaction according to (Kristy and Zainul, 2019) is :

$$SiO_2 + 2NaOH + H_2O \rightarrow Na_2SiO_3 + 2H_2 \quad (1)$$

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Last stage is the synthesis of magnesium silicate where natrium silicate that have been obtained is reacted with magnesium chloride ($MgCl_2$) with ratio 1:1,5 at 110°C for 1 hour until white suspensions form. The chemical reaction for this stage should be :

$$Na_2SiO_3 + MgCl_2 \rightarrow MgSiO_3 + 2NaCl \quad (2)$$

The formed suspensions will be washed with aquadest and then baked at 100°C for 2 hours thus the magnesium silicate is obtained. Afterward, in order to evaluate and characterize, some of samples were analyzed using *Fourier Transform Infrared Spectroscopy* (FTIR).

Result and Discussion

The result obtained from the production of magnesium silicate with the variable of reaction time and NaOH concentraion are presented in Table 1.

Table 1. The Result of Magnesium Silicate Production

Sample	Variable		Yield $MgSiO_3$ (%)
	Reaction Time	NaOH Concentraion	
1	90 Minutes	3 M	17,4
2		5 M	38,8
3		7 M	49,9
4		9 M	46,3
5	150 Minutes	3 M	42,1
6		5 M	45,7
7		7 M	39,2
8		9 M	45,5

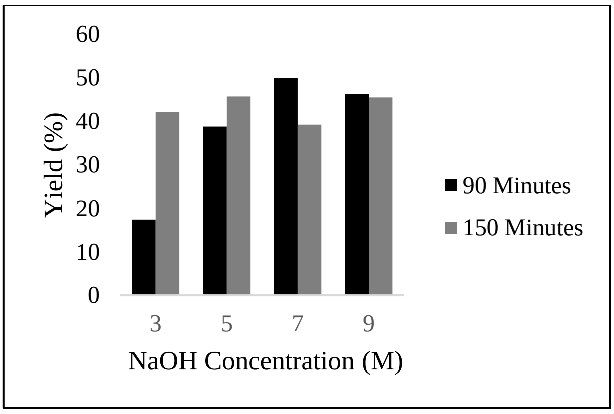

Figure 1. Yield of Magnesium Silicate Production

From the data above, can be seen that NaOH 7M with reaction time of 90 minutes gave highest yield than other variables. This was due to higher concentration implying more molecules in the solution meaning that silica will react more with NaOH before it saturated. Yield trend can be seen in Figure 1. where most variable at 40-50% yield. This could be caused by NaOH solution at 90 minutes have been saturated thus the yield can't be higher (Agung M, Hanafie Sy and Mardina, 2013).

The surface of magnesium silicate is composed of siloxane groups (Si-O-Si), silanol groups (Si-OH), and magnesiul groups (Si-O-Mg). The most reactive groups on the surface are free hydroxyl groups as these are responsible for providing the sites for pyhsical adsorption (Aysa-Martínez *et al.*, 2021). The FTIR spectra of the magnesium silicate samples show changes in the hydration process of magnesium silicate, indicating the reactions between $MgCl_2$ and SiO_2, as shown in Figure 2. The SF (SiO_2) shows absorption bands at 1050 cm^{-1}, 1043 cm^{-1} and 700 cm^{-1}, which can be assigned to asyimmetrical stretching vibration, symmetrical stretching vibration and Si-O bending vibration (Sáez, Martínez-ramírez and Blanco-varela, 2014).

The bands at 3570 cm^{-1} and 3347 cm^{-1} comes from the -OH stretching vibration due to physical adsorption of water and bound water (Sun *et al.*, 2018). The characteristic band at 2366 cm^{-1} corresponded to the O=C=O bending vibration. Another band related to O-H hydroxyl group is found at 1641 cm^{-1} and the peak at 1050 cm^{-1} shows the

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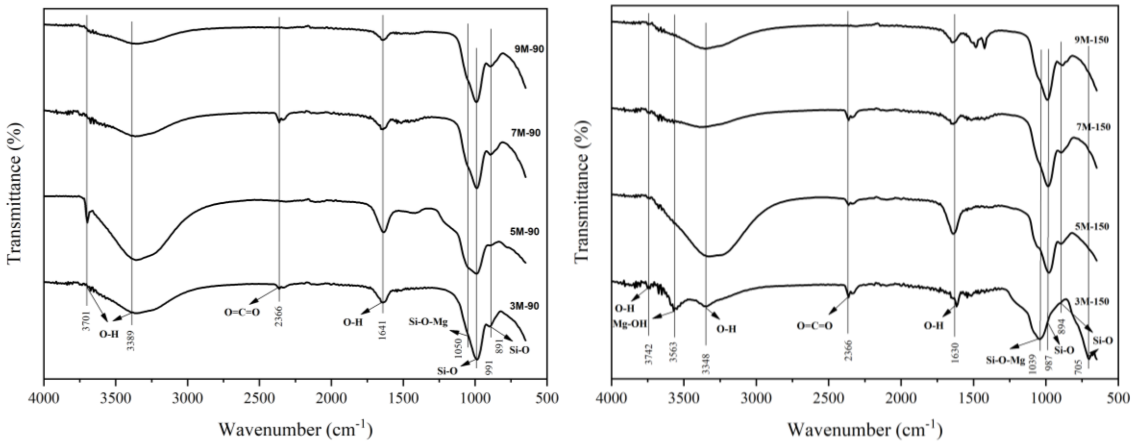
anti-symmetric stretching vibration of Si-O-Mg (Gel *et al.*, 2018), 991 cm^{-1}, and 891 cm^{-1} comes from the stretching vibration of silanol groups (Si-OH) (Li *et al.*, 2014). No significant difference was observed between sample variable.



(a) (b)

Figure 2. FTIR spectra of $MgSiO_3$ with variable reaction time (a) 90 minutes (b) 150 minutes

Similar observations were reported by Li *et al.* (2022), where slight differences between samples could be due to impurities. The peak band at 2366 cm^{-1} corresponds to O=C=O bending vibration could be due to the imperfect burning of rice husk ash. Therefore, carbon elements in rice husk ash reacts with oxygen and forms carbon dioxide.

Conclusion

According to the study result can be concluded that the more concentrated NaOH solution the more magnesium silicate formed, and the longer reaction time shows the more magnesium silicate formed. Highest magnesium silicate yield obtained by 7M NaOH for 90 minutes at 49,9% yield. FTIR analysis proof that the product is magnesium silicate.

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