



Scaling Up of the Pyrolysis Process to Produce Silica from Rice Husk

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Abstract. Rice husk is a potential alternative source of silica and silicon. The mechanism of heating (pyrolysis) for the decomposition of rice husk is an important factor in obtaining silica of high purity. Medium-scale pyrolysis to produce silica from rice husk serves as a bridge to connect laboratory-scale production to industrial-scale production. The purpose of this study was to model and scale up the pyrolysis process as a guidance for industrial-scale production. The research method used was experimentally based. An experimental investigation was undertaken in five stages. 1) Analyzing rice husk mass conversion using thermogravimetry analysis (TGA); 2) pyrolysis modeling based on a laboratory investigation using COMSOL Multiphysics version 5.3; 3) medium-scale experiments according to the modeling results; 4) validation of the modeling results by carrying out a medium-scale experiment; 5) silica purity analysis using SEM-EDX. The medium-scale pyrolysis simulation of silica manufacture from rice husk obtained a heating rate of 1.5 °C/min. There was an increase in the heating rate of 1 °C/min when compared to the laboratory-scale process. The pyrolysis of rice husk for the production of silica affects the mass conversion and selectivity of the resulting silica product. The mass conversion produced was 13.33% to 17.87% and the purity of silica produced was 63.99% to 82.74%.

Keywords: *modeling; pyrolysis; rice husk; silica; upscaling.*

1 Introduction

Indonesia's rice production in 2015 was 75.40 million tons of dried unhulled grain [1]. The rice husk waste produced by Indonesia in 2015 reached 15.91

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million tons. This has the potential to produce 5.45 million tons of rice husk and 3.84 million tons of husk ash [2]. Husk charcoal can be processed to produce silicon dioxide (silica) by pyrolysis, giving a yield of 16.85% [3]. Husk charcoal contains about 72.1% of silicon oxide, which can be enhanced to 94.95% after heating at 700 °C for 6 hours [4]. Dry rice husk ash contains about 80-90% of silicon oxide [5, 6]. Therefore, charcoal and rice husk ash is an alternative raw material for silica and silicon production [7-9]. Silica is widely applied for manufacturing various industrial products with silica specifications depending on the product to be created. Some authors have produced silica-rich rice husk ash by applying calcination for the construction of insulation or ceramic materials [10]. Other applications include catalysts (for example in paper, paint, rubber, polymer processing) and additives, abrasives, insulators and dampers of pollutants/adsorbents [11, 12].

Amorphous silica ash is beneficial as a substitute for cement or additives [13] in the production of zeolites [14] and other ceramic applications [15]. Numerous studies on the production and application of rice husk ash have been conducted, but there are still knowledge gaps regarding the optimization of treatment conditions and their relation to the nature of the rice husk ash product. In this case, to produce pure high-reactivity silica some conditions are essential for obtaining an amorphous structure and absence of reacting carbon [4].

Pyrolysis is the chemical decomposition of organic matter through heating with no or little oxygen or other reagents so that the chemical structure of the raw material will go into gas phase [16]. The novelty of this research was to create a medium-scale pyrolysis model for making silica from rice husk, after which the results of the medium-scale modeling were validated with experimental results on a medium scale. The mass conversion produced was 13.33% to 17.87% and the purity of the medium-scale silica pyrolysis products was about 62.99% to 82.74%. The medium-scale modeling showed that the pyrolysis results were valid with an error value of 9.375%.

2 Experimental Method

An experimental investigation was carried out in 5 stages: 1) analyzing rice husk mass conversion using thermogravimetry analysis (TGA) to obtain the reaction kinetics values from the pyrolysis process; 2) pyrolysis modeling based on a laboratory investigation using COMSOL Multiphysics version 5.3; 3) medium-scale experiments. A flow chart of the pyrolysis process is shown in Figure 1.

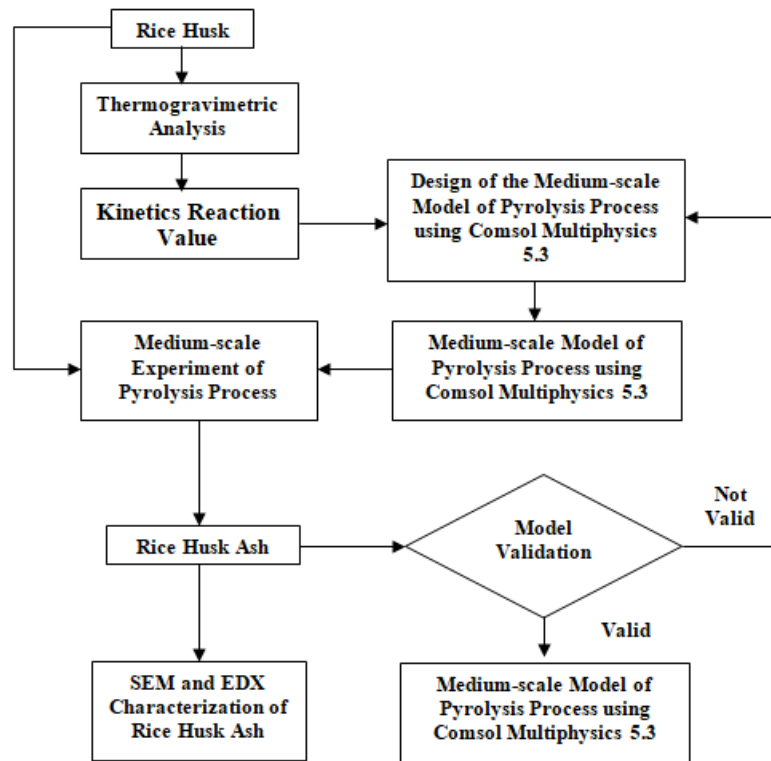


Figure 1 Pyrolysis process flow chart.

Rice husk ash production was carried out in a medium-scale furnace with a capacity of 15 kg. Rice husks were burnt at a heating rate of 1.5 °C/minute to a temperature of 900 °C. The process of rice husk burning was carried out at temperatures of 400 °C and 900 °C for 1 hour. The rice husk ash was washed using 3% acid solution (HCl). The washing process was intended to reduce impurities in the husk ash to obtain silicon dioxide. The process of washing the husk ash was done using 3% technical grade HCl (i.e. 12 ml 3% technical grade HCl for 1 gram ash) and was followed by heating on a hot plate at 200 °C and stirring using a magnet stirrer at 240 rpm for 2 hours [17]. Then the husk ash was washed with hot distilled water repeatedly until it was free from acid (tested using litmus paper) and filtered with a cow-free filter paper. The filtering results (residues + filter paper) were heated in a furnace at a temperature of 1000 °C to remove the remaining silicon dioxide. Samples were cooled in a kiln (furnace temperature equal to room temperature). 4) Validation of the modeling results by carrying out a medium-scale experiment. 5) Characterization of the silicon dioxide surface structure was done using scanning electron microscopy

(SEM) and silicon dioxide purification tests were carried out using energy dispersive X-ray spectroscopy (EDX).

3 Results and Discussion

3.1 Thermogravimetry Analysis (TGA)

The TGA curve describes the process of losing mass by temperature in the pyrolysis process with various stages of phase change of the material due to the heating process. The TGA rice husk curve in Figure 2 shows mass loss as a function of temperature with a heating rate of 1 °C/min. The phase change in the pyrolysis process occurs in 4 stages.

Stage 1 occurs at a temperature of 30.9-126.8 °C with a mass loss of 10.415% and an average mass loss of 0.108 %/min. At this stage, water vapor is removed from the material.

Stage 2 occurs at a temperature of 126.8-222.7 °C with a mass loss of 0.464% and an average mass loss of 0.0048 %/min. This stage is considered a transitional phase, where the mass loss is made possible by the loss of water vapor (if any), CO₂ gas, and others.

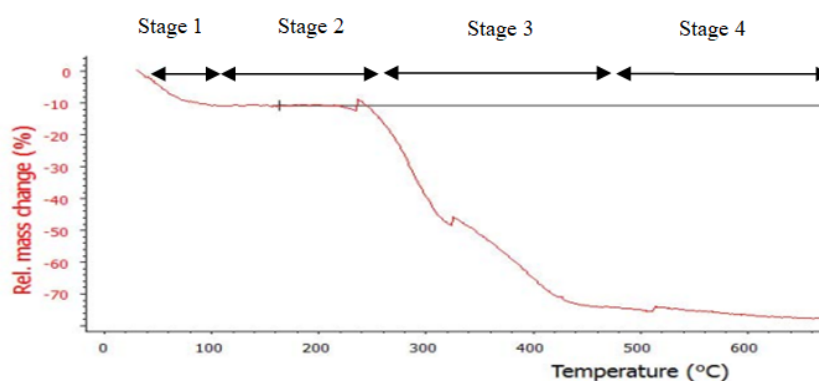


Figure 2 The TGA rice husk curve.

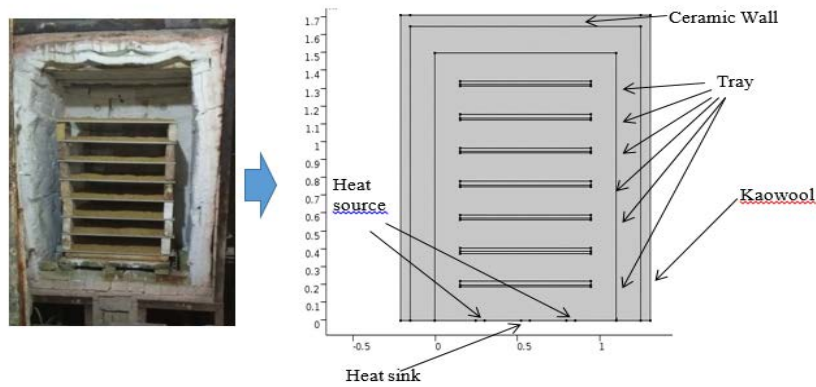
Stage 3 occurs at a temperature of 222.7-460.6 °C. At this stage there is a loss of mass of volatile material of about 62.865%. The mass loss rate of the volatile material is 0.2642 %/min. Stage 4 occurs at temperature 460.6-661.1 °C with a rather slow mass decline. At this stage the mass loss is 3.577 % with an average mass loss of 0.0178 %/min. At this stage solid carbon burning (fixed) occurs.

Table 1 Phase change phases in the pyrolysis process.

Stage	Temperature (°C)		Mass Loss		Losing Mass (% weight)	Time	Average mass loss (%/Minutes)
Stage 1	30.9	126.8	0.281	10.69	10.41	95.9	0.10
Stage 2	126.8	222.7	10.69	11.16	0.46	95.9	0.00
Stage 3	222.7	460.6	11.16	74.02	62.86	237.9	0.26
Stage 4	460.6	661.1	74.02	77.60	3.57	200.5	0.01

3.2 Modeling of Medium Pyrolysis Process

The modeling of the pyrolysis processes on a medium scale was designed based on furnace geometry, fluid flow, and heat transfer [18]. The geometry of the beam-shaped furnace is shown in Figure 3. The ceramic furnace wall was coated with kaowool to maintain the heat flow inside the furnace. At the bottom of the furnace, there was a heat source using a gas fuel, the center was placed on ceramic shelves to store the rice husks during the pyrolysis process. The rice husks were placed on a ceramic rack with a height of 2 cm.

**Figure 3** Geometry of the furnace.

The fluid flow in the furnace room follows the continuity equation and the momentum balance as follows:

Mass balance

$$\rho \nabla \cdot (\mathbf{u}) = 0 \quad (1)$$

Balance of momentum

$$\rho (\mathbf{u} \cdot \nabla) \mathbf{u} = \nabla \cdot \left[-p\mathbf{I} + (\mu + \mu_T) (\nabla \mathbf{u} + (\nabla \mathbf{u})^T) \right] + \mathbf{F} \quad (2)$$

The turbulence formed in the fluid flow was modeled using the k- ε turbulence model expressed by the transport equation as follows:

Turbulence model

$$\rho(\mathbf{u} \cdot \nabla)k = \nabla \cdot \left[\left(\mu + \frac{\mu_T}{\sigma_k} \right) \nabla k \right] + P_k - \rho\varepsilon \quad (3)$$

$$\rho(\mathbf{u} \cdot \nabla)\varepsilon = \nabla \cdot \left[\left(\mu + \frac{\mu_T}{\sigma_\varepsilon} \right) \nabla \varepsilon \right] + C_{\varepsilon 1} \frac{\varepsilon}{k} P_k - C_{\varepsilon 2} \rho \frac{\varepsilon^2}{k} \quad (4)$$

The heat transfer occurring in the furnace is caused by conduction and convection because the furnace used in the pyrolysis process has a gap or cavity so that there will be convection heat transfer by the air or gas flow [19]. The heat transfer in the rice husks placed on the ceramic rack proceeds by conduction due to direct contact between the ceramic particles and the rice husk particles. The general heat transfer equation by conduction and convection is shown in Eq.(5):

$$d_z \rho C_p \frac{\partial T}{\partial t} + d_z \rho C_p \mathbf{u} \cdot \nabla T + \nabla \cdot \mathbf{q} = d_z Q + q_0 + d_z Q_p + d_z Q_{vd} \quad (5)$$

$$\mathbf{q} = -d_z k \nabla T$$

The heat transfer by conduction and convection in the furnace is grouped into fluids, solids, and porous media [18]. The heat transfer in fluids occurs by the air or gas flow in the furnace [20]. The gas is assumed to be an ideal gas so that the density parameter of Eq. (5) can be expressed as follows:

$$\rho = \frac{P_A}{R_s T} \quad (6)$$

Heat transfer of solids occurs in the furnace shelves and walls through direct contact of the shell particles and the furnace walls. The heat transfer equation on the rack and furnace wall is expressed in Eq. (5). Heat transfer in porous media occurs in the rice husk because the rice husk particles are large enough to create cavities between the rice husks. The conductivity and heat capacity of the media is expressed as effective parameters that are contributed by both the solid and the fluid particles that fill the pore cavities. The conductivity parameter and heat capacity in Eq. (5) are expressed as follows:

$$(\rho C_p)_{eff} = \theta_p \rho_p (C_p)_p + (1 - \theta_p) \rho C_p \quad (7)$$

$$k_{eff} = \theta_p k_p + (1 - \theta_p) k \quad (8)$$

The result of the simulation in a medium-scale furnace shows the variation of temperature escalation on each shelf. This is caused by the uneven heat flow to all parts of the furnace, as shown in Figure 4.

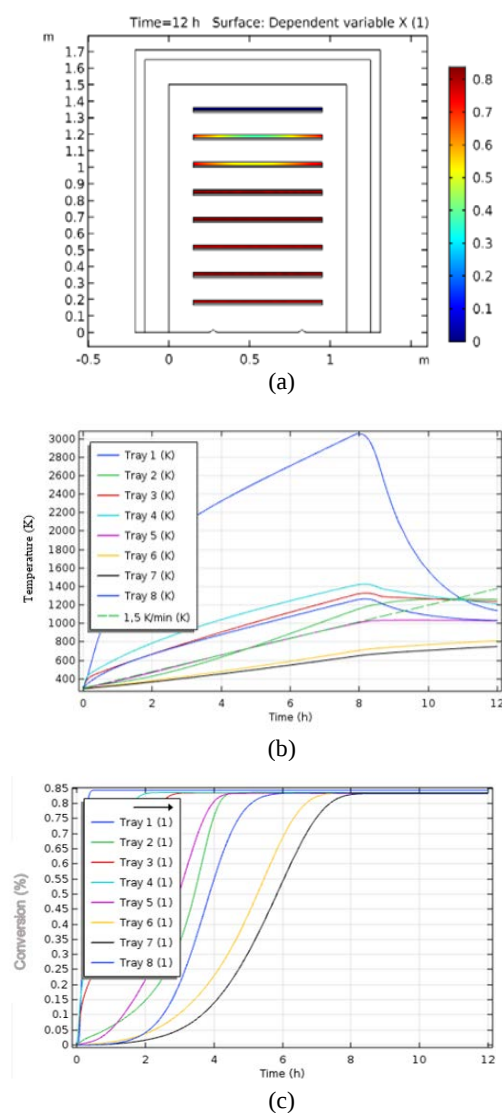


Figure 4 (a) Temperature distribution of the modeling result, (b) temperature distribution graph of the modeling result, (c) conversion graph of the modeling result.

The temperature escalation on tray 1 and tray 8 shows a very high increase because tray 1 was close to the heat source. The hot flue gas flowed to the side of the shelf and then moved upwards to the 8th tray. A laboratory furnace with a size of 17 x 11 x 12 cm was heated by connecting it to the electricity grid to achieve a uniform temperature profile of the rice husk pyrolysis process. Based on the profile data, simulations were performed for a larger-scale furnace with dimensions of 152 x 122 x 171 cm. The modeling results showed the temperature variations in the medium-scale furnace.

The lower part of the furnace, closer to the heat source, showed higher temperatures, appearing as lighter colors in the figure (blue, pale yellow, orange yellow, orange, and red, indicating the order at which the temperature is getting lower). The pyrolysis temperature based on the modeling result reached 1900 °C within 20 hours with the gas inlet velocity of the heat source at 1 m/s. The consideration for the selection of the furnace dimensions were the availability of a medium-scale furnace that was ready for pyrolysis, as shown in Figure 4. Some variable values in the furnace were adjusted to the standard values of the research results, as shown in Tables 2 and 3.

Table 2 Alumina standard values.

Property	Variable	Expression	Unit
Coefficient of thermal expansion	Alpha; α	8e-6	1/K
Heat capacity at constant	Cp	900	J/(kg K)
Density	rho	3900	Kg/m ³
Thermal conductivity	k	27	W/(m K)

Table 3 Rice husk standard values.

Property	Variable	Expression	Unit
Density	rho	(T(1/K)) (Kg/m ³)	Kg/m ³
Thermal conductivity	k	0.08	W/(m K)
Heat capacity at constant	Cp	Cp(T)	J/(kg K)

3.3 Validation

A validation test was carried out on the model's performance with a maximum deviation limit of 10% [21]. The variable used was the conversion of silica mass from the modeling and the experimental results using the absolute mean error (AME) method. The results are presented in Table 4. The results of the medium-scale experiment produced silicon dioxide with mass conversion at 13.33% to 17.87%. The results of the medium-scale modeling produced silicon dioxide with mass conversion at 15.09% to 17.33%.

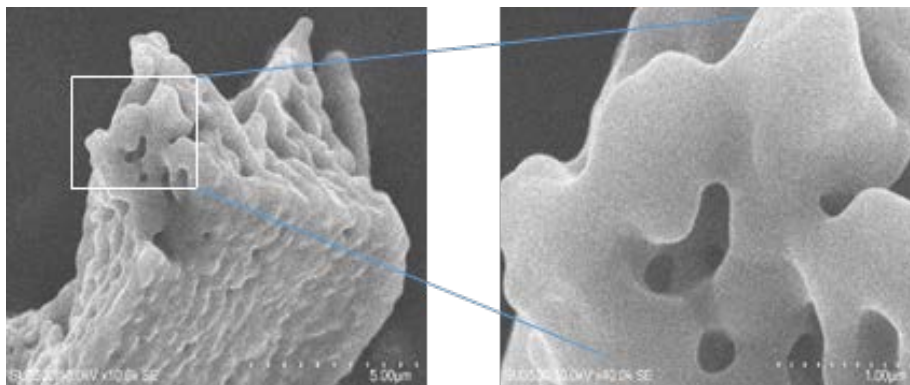
Table 4 Conversion of rice husk mass in the pyrolysis process on medium scale.

Tray	Percentage of silicon dioxide mass		Validation test
	Experiment	Modeling	AME
Tray 1	17.07	14,65	0,14
Tray 2	13.87	15,38	0,10
Tray 3	16.00	15,55	0,02
Tray 4	14.93	15,45	0,03
Tray 5	13.33	15,66	0,17
Tray 6	14.67	15,48	0,05
Tray 7	15.47	15,76	0,01
Tray 8	17.87	15,56	0,12

The main purpose of the scale-up process is to maintain acceptable product quality. This means making the same product in larger amounts than those produced on a laboratory scale. There are several ways to increase reactor capacity [22], namely adding reactors that are identical in parallel, extending the reactor and enlarging the reactor diameter by scaling up using geometry similarity. Geometry similarity means keeping the reactor length-per-width ratio constant during scaling up.

3.4 SEM and EDX Results

The SEM images of the rice husk ash samples show a high porosity degree and a high surface area. Figures 5 to 10 show the morphology of the rice husk ash before and after leaching using an acid solution. By leaching using an acid solution, the rice husk ash breaks down, the surface of the outer epidermis peel off and several large pores are created. This suggests that the acid solution used affected the morphology structure of the rice husk ash and affected the elements contained in the rice husk ash [4, 22, 23].

**Figure 5** SEM results of Silica Tray 1 before leaching.

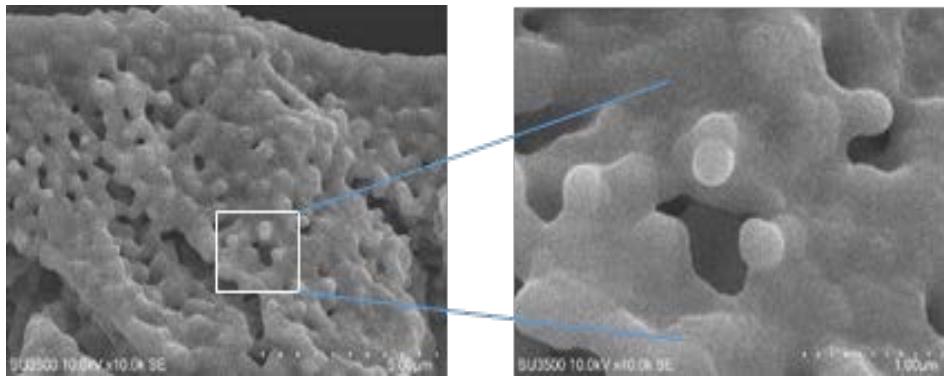


Figure 6 SEM results of Silica Tray 1 after leaching.

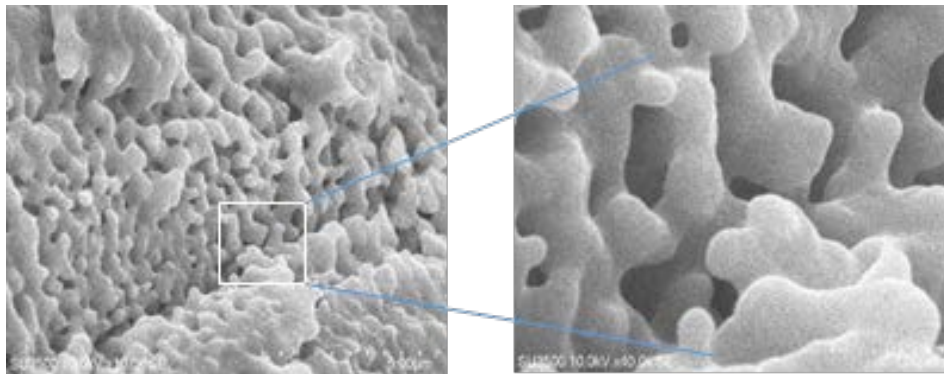


Figure 7 SEM results of Silica Tray 4 before leaching.

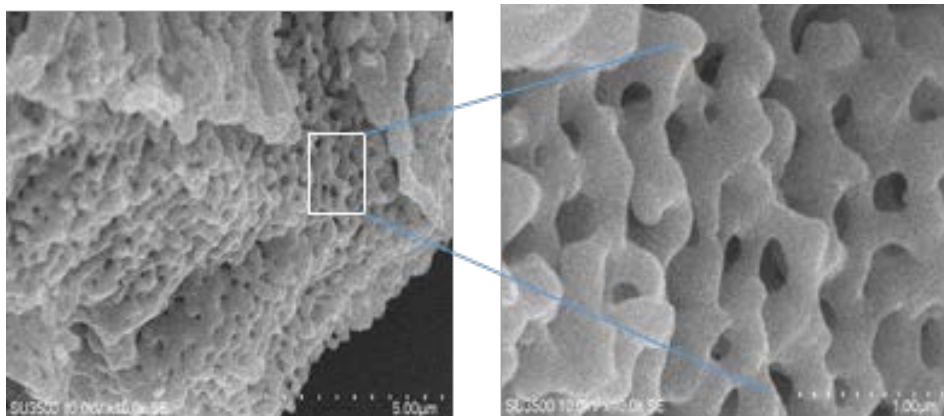


Figure 8 SEM results of Silica Tray 4 after leaching.

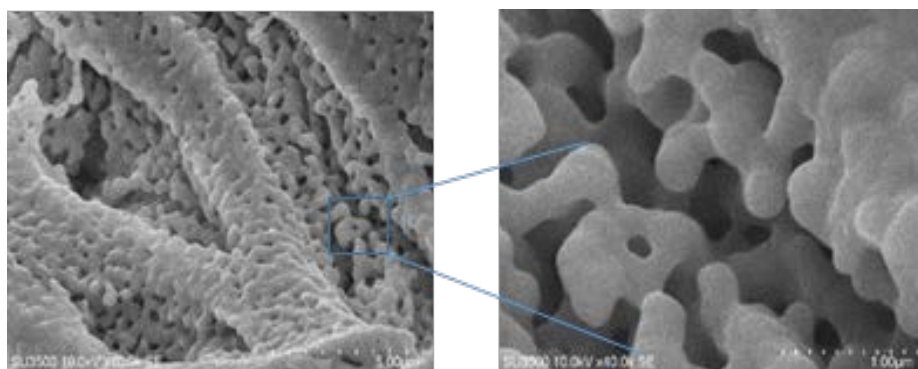


Figure 9 SEM results of Silica Tray 8 before leaching.

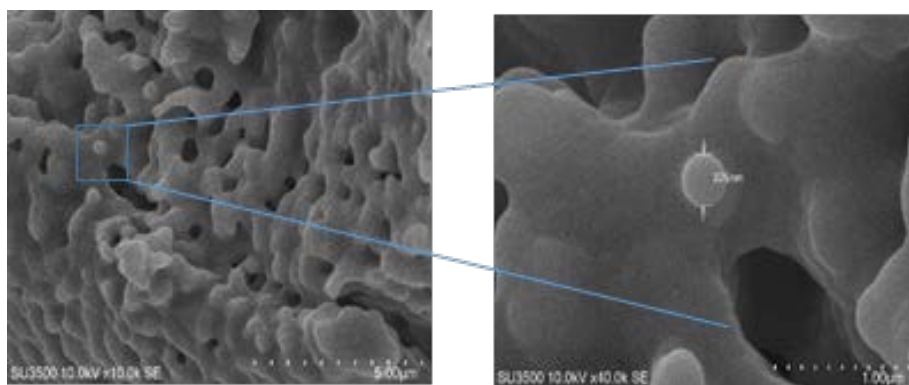


Figure 10 SEM results of Silica Tray 8 after leaching.

Figures 5 and 6 show the morphology of the rice husk ash before and after leaching using the acid solution in Tray 1. Figures 7 and 8 show the morphology of the rice husk ash before and after leaching using the acid solution in Tray 4. Figures 9 and 10 show the morphology of the rice husk ash before and after leaching using the acid solution in Tray 8. These images suggest that the acid solution affects the elements contained in the rice husk ash [4,23,24] as shown in Table 5.

According to the results of the EDX analysis of silica/SiO₂ Tray 1, 4 and 8, the purity of the silica obtained from the rice husk ash before washing and after washing was 80.79% and 82.74% for Tray 1. The purity of the silica obtained from the rice husk ash before washing and after washing was 63.99% and 80.19% for Tray 4. The purity of the silica obtained from the rice husk ash before washing and after washing was 71.55% and 76.44% for Tray 8.

Table 5 Results of EDX analysis of silica /SiO₂ Tray 1, 4 and 8.

Element	Percentage (%) of Atoms					
	Tray 1		Tray 4		Tray 8	
	Before Leaching	After Leaching	Before Leaching	After Leaching	Before Leaching	After Leaching
C	11.98	7.27	11.92	9.73	10.14	8.39
O	59.86	64.39	65.61	62.2	65.81	65.7
Na	0.47	0.52	1.13	0.44	-	0.21
Mg	0.3	-	-	0.18	-	0.23
Al	-	-	-	0.44	-	-
Si	26.93	27.58	21.33	26.73	23.85	25.48
K	0.45	0.24	-	0.29	0.2	-
Purity of Silica	80.79	82.74	63.99	80.19	71.55	76.44

4 Conclusion

Modeling the pyrolysis process on a medium scale was designed based on furnace geometry, fluid flow, and heat flow. A medium-scale pyrolysis model was produced for the manufacture of silica from rice husk with a heating rate of 1.5 °C/min. The results of the modeling were applied in medium-scale experiments. The modeling results were then compared with experimental results for validation. The average mass conversion value of the modeling results and the experimental results was 15.4%, and the silicon dioxide purity of the medium scale experiment was 63.99% to 82.74%. The error value from validation between the simulation model and the experimental results was 8.65%. Thus, the simulation model can be used as a reference in an effort to develop silica from rice husks on an industrial-scale so that it can be developed into an agro-industrial business.

Acknowledgement

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Nomenclature

C_p is the specific heat capacity at constant pressure (J/(kg·K))

C_{z1} , C_{z2} are turbulence model constants

ε is the turbulent dissipation rate (m^2/s^3)

$\mathbf{F}$ is the volume force vector (N/m^3)

$\mathbf{I}$ is the identity matrix

k is the turbulent kinetic energy (m^2/s^2)

k_{eff} is the effective thermal conductivity (W/(m·K))

μ is the viscous stress tensor (Pa)

μ_T is the turbulent viscosity (Pa)
 p is the pressure (Pa)
 P_k is the turbulence kinetic energy generation rate ($J \cdot kg/m^3 s$)
 $\mathbf{q}$ is the heat flux vector (W/m^2)
 q_o is the heat flux vector (W/m^2)
 Q contains the heat sources (W/m^3)
 Q_p contains the heat sources at constant pressure (W/m^3)
 Q_{vd} contains the heat sources at viscous dissipation (W/m^3)
 ρ is the density (kg/m^3)
 R_s is the resistivity of the material ($m^2/s^2 K$)
 T is the absolute temperature (K)
 θ_p is the solid volume fraction (SI unit: 1)
 σ_k is the turbulence constant
 $\mathbf{u}$ is the velocity vector (m/s)

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