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SILICA EXTRACTION FROM RICE HUSK AS A SLOW RELEASE FERTILIZER USING MICROWAVE ASSISTED EXTRACTION

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ABSTRACT
The utilization of waste as raw materials is one of the alternatives in applying the "zero waste" principal. Rice husk is one of the wastes of rice plantation which has very high silica content at around 80-90%. The extraction of silica from rice husk can be used to make slow release fertilizer. Slow release fertilizer is a fertilizer with a nutrient release mechanism that periodically follows the pattern of nutrient absorption by plants. This study aims to optimize rice husk tablets formulation by extraction using Microwave Assisted Extraction (MAE) and to determine the physical and chemical properties of the results of rice husk extract tablets. This study used a Completely Randomized Design (CRD) with two factors, namely the concentration of KOH with variations in solvents 2.5%, 5%, and 7.5% and warm-up durations of 5, 7.5 and 10 minutes. The largest hardness value was 3,356 kgf in 7.5% KOH with 10 minutes in microwave. The largest yield of silica was 7.5% KOH with the longest heating time of 10 minutes of 5.30%. Through XRF analysis, it was found that the largest silica value was 6.51% at 5% KOH with a heating time of 5 minutes. Based on the comparison with the conventional heating method, the results of extraction did not reach optimum results of silica extract (21.8%), whereas in this research the yield obtained was 6.51%. Thus, based on these results, heating using microwave did not produce optimal silica results.

KEY WORDS
Extraction, rice husk silica, slow release fertilizer.

Rice husk, or often called husks, is one of the layers wrapping rice seeds that will be separated and become rarely used thus generates waste. The main content of rice husk is silica. Silica rice husk has a high-water resistance to chemical compounds of fertilizer. Silica has a role to increase productivity and strengthen plant growth; therefore it is resistant to pest attacks. The silica contained in soil is depleted every year because there is no return of silica (Si) into the soil. The potential for silica loss from tropical soil reaches 54.2 kg per ha every year. These problems can be overcome by making a slow release fertilizer. The application of the silica pellet method is closely related to the method of slow adsorption of nutrients on the soil. Not all fertilizers given to plants can be absorbed; some will be carried away by groundwater or degraded, so the fertilization is inefficient. The tablet fertilizer application allows plants to absorb nutrients throughout the period of growth by minimizing the risk of nutrient loss due to leaching during irrigation and avoiding the risk of excesses dissolved salt.

MATERIALS AND METHODS OF RESEARCH

The research was conducted in January 2018 until April 2018 at the Laboratory of Food and Agricultural Processing Technology in the Faculty of Agricultural Technology, University of Brawijaya, Central Laboratory of Life Sciences (LSIH), Advanced FMIPA Mineral and Materials Laboratory, State University of Malang, Biology Laboratory, University of Muhammadiyah Malang and Laboratory Chemical Engineering of Malang State Polytechnic.

The materials used in the study were rice husk, hydrochloric acid (HCL) 1M, potassium hydroxide (KOH) 1 M and distilled water.

The experimental design used in this experiment was factorial Completely Randomized Design. The first factor is the KOH concentration consisting of 3 levels namely 2.5%, 5% and 7.5% and the second factor is the comparison of duration consisting of 3 levels namely 5 minutes, 7.5 minutes and 10 minutes. This study consisted of 9 combined treatments, where each treatment was repeated 3 times, therefore 27 units were experimented.

Firstly, the rice husk was washed. Clean rice husk was dried in an oven at a temperature of 60˚C for 24 hours. Dry rice husks were then grind using a blender, to obtain a uniform size, then sieved with a mesh size of 35 or a 0.5 mm sieve hole.

Rice husk was weighed as much as 10 grams and put into a 250 ml beaker glass. Then60 ml of potassium hydroxide (KOH) was added into the beaker glass. The concentration of KOH used is 2.5%, 5% and 7.5%. Mixture of rice husk with KOH was left for 1 hour. The next step was heating using a Microwave with 100 watts of power for 5 minutes, 7.5 minutes and 10 minutes. The sample was left for 24 hours; the rice husk was then filtered using a filter cloth to separate the sediment from the filtrate. The remaining filtrate was pH adjusted using HCl to get a pH of 7. The filtrate which reached pH 7 formed a fine powder. The fine powder formed was dried in an oven with a temperature of 110˚C for 3 hours. The extracted silica was then formed into tablet using tabletting machine.

The formation of rice husk powder to tablets was done using tabletting machine. The average weight of extracted powder was 0.5 g per tablet. Then extracted powder was put into a tablet forming device, then pressed with a hammer, which hardened and solidified the powder.

The observation parameters for the yield of extraction were carried out using XRF and the hardness was determined using a penetrometer. The method used in this study was the experimental research method by conducting experiments to see the effect of KOH and the duration of extraction using microwave. Data analysis was performed using Analysis of Variance (ANOVA).

The cone attached to the tip of the penetrometer will be vertically straight up on the material that will be tested, then pressed into the material with a fixed compressive force. At a certain depth, the magnitude of the vertical pressure is shown to press the tool/the material and divided into a lower plate. The principle is similar to a tensile test but the method for this test is by pressing the material until the material is cracked. The results on the parameters will be obtained at the end of the process.

The recovery is a comparison between the mass of the product produced to the mass of the raw material used. Procedure for analysis of yield is: (Novari et al, 2007):

a) Weight of dry husk ingredients (grams);

b) Weight of fertilizer produced (gram).

$$\text{Yield (\%)} = [(\text{Final Mass})/(\text{Initial Mass})] \times 100\% \quad (1)$$

The sample of rice husk powder was placed on a holder (± 10 mm). Nonconductive samples need to be coated by Au-Pd, so that the sample is more conductive. The sample was then put into a SEM chamber then pumped (High Vacuum or Low Vacuum) and waited until vacuum. Samples are in the form of powder and solids. The synthesis results are then inserted into the tube and measured using X-rays.

The solubility of tablets is calculated based on the duration of the tablet dissolves into fine particles both in the soil and in water. The solubility value in water is calculated using a stopwatch. The solubility value in the soil is carried out by observing the soil that has been applied by rice husk tablets.

RESULTS AND DISCUSSION

The results of the hardness test of rice husk tablets obtained in the study ranged from 1,131-3,356 kgf. The average value of hardness obtained can be seen in Figure 1.

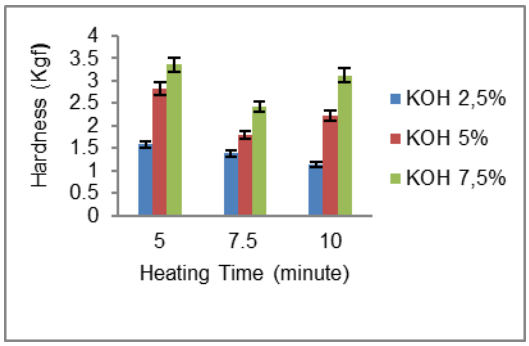

Figure 1 – Relationship between Heating Duration (minute) and Hardness Test Value (kgf)

Based on Figure 1 the trend tends to increase in each treatment. The highest hardness value of rice husk tablets increased to 3.356 kgf, at the treatment of 7.5% KOH concentration with a warm up duration of 5 minutes, while the lowest value of hardness of rice husk tablets was 1.131 kgf, at the treatment of 2.5% KOH concentration with a heating duration of 10 minutes. Based on the analysis of variance, it is showed that the KOH concentration factor and the heating time had a significant effect (P> 0.05) on the value of hardness. Hardness is the nature of a solid object in terms of its resistance to rupture due to the presence of compressive forces that are not deformed [8]. The smaller the compressive value, the greater the hardness, while the greater the compressive value, the lower the material hardness. It is because the material should be weak (Sumarmono, 2012).

The results of the extraction of silica extract from rice husk obtained in the study ranged from 1.48 to 5.30%. The average yield obtained can be seen in Figure 2.

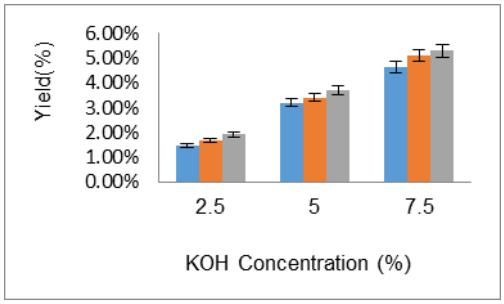

Figure 2 – Rice Husk Silica Extraction Yield

Based on Figure 2 the graph tends to increase in each treatment. The highest silica yield value is 5.3%, obtained by the treatment of 7.5% KOH concentration with a heating duration of 10 minutes, while the lowest silica yield value is 1.48%, obtained by the treatment of 2.5% KOH concentration with a heating duration of 5 minutes.

Table 1 – Anova for Rice Husk Silica Extraction Yield

Source of Variation	SS	df	MS	F	P-value	F crit
KOH concentration	49.65816	2	24.82908	57.00942	1.63E-08	3.554557
Heating time	1.309919	2	0.654959	1.503835	0.248915	3.554557
Interaction	0.092593	4	0.023148	0.05315	0.994238	2.927744
Within / Error	7.839467	18	0.435526			
Total	58.90014	26				

**F = F value, F crit = F Table.*

Based on the analysis of variance (ANOVA), it is shown that the KOH concentration factor had a significant effect on the results (P> 0.05), while the heating duration factor did not have a significant effect on the extraction value of silica (P> 0.05). According to Prasetio (2014), the higher the concentration of KOH added the more yields produced. The extraction duration also gives an effect on the yield produced. Short extraction duration will generate a

low yield because not all components are extracted properly. With longer heating time, the contact time between the husk and KOH solution will be greater, therefore resulting greater yield (Irawan, 2010).

Physical characteristics of rice husk tablets were observed by SEM (Scanning Electron Microscopy) testing and it can be seen in Figure 3.

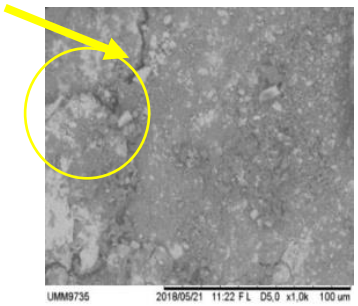

Figure 3 – Characteristics of Composition of Organic Material Rice Husk Tablets Using Scanning Electron Microscopy

Samples with 2.5% KOH concentration and 5 minutes warm-up duration, where the surface was too tenuous. Samples with 5% KOH concentration and 7.5 minutes warm-up duration had sufficient density but were not as comparable when compared to samples with 7.5% of KOH concentration and 10 minutes for warm-up time. Based on Figure 3, it can be seen in the yellow arrow that the structure of the tablet will be more tightly closed if the heating goes longer; moreover, the high concentration of KOH solution will cause the bond formed to be stronger and possess a high density. In Figure 3, there is also three visible parts in the yellow circle. The brightest part is inorganic material, for parts that are grey are organic materials and for the darkest colored parts are materials with a slow density, therefore they cannot be penetrated by waves.

From the data obtained by the sample with the best results, namely the treatment of KOH concentration as much as 7.5% with a heating duration of 10 minutes, then the chemical analysis of the sample was carried out using X- rays to find out the elements contained in the figure 4.

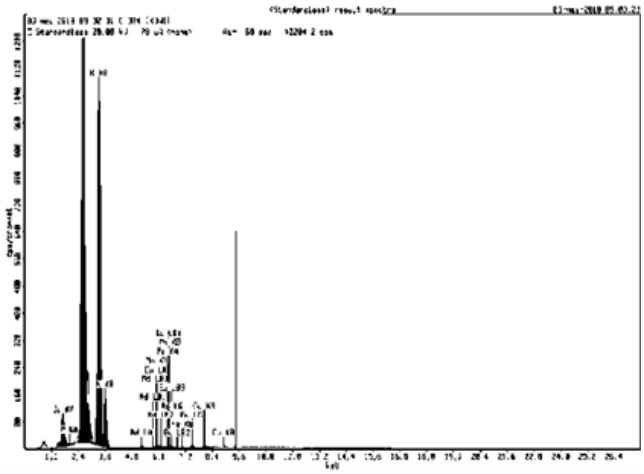

Figure 4 –XRF Rice Husk Powder with the treatment of 7.5 KOH

Based on Figure 4, it can be seen that the most dominant element is Potassium. The second most elements is chlorine and the third most element is silica. Silica is the most elements contained in rice husk, but in this study the silica obtained was only 6.22. It was possibly due to the heating duration, the KOH solvent was saturated. Therefore, at the time of operation the solubility of the KOH solvent to silica was reduced. According to Laksmono

(2002), the saturation of solvents is caused by the solubility of the solute decreases with increasing time; this is indicated by the decrease in solutes taken by the solvent. In addition, the length of operation allows the silica formed to degenerate and be settled in rice husk, thus silica in the filtrate decreases.

Table 2 – Duration of Solubility in Water

Treatment	t_1 = 5 minutes (minutes)	t_2 = 7,5 minutes (minutes)	t_3 = 10 minutes (minutes)	Time in soil soluble (minutes)
KOH 2,5%	40	40	45	40
KOH 5%	100	105	110	100
KOH 7,5%	150	150	155	150

Table 3 – Water Testing Results with Percolation Method (500 ml of water, 1 gram of rice husk tablets)

Parameter	Unit	Result
Calium	Mg/L	74.89
Clorin	Mg/L	0.11
Silica	Mg/L	16.68

Based on Table 2, the concentration of 2.5% KOH with a warm up duration of 5 and 7.5 minutes has faster solubility, i.e. for 40 minutes, while 7.5% KOH concentration with a warm up duration of 10 minutes had solubility in longer absorption for 155 minutes. The pores formed on the tablet will affect the solubility of the tablet in water (release). Spacing pores made it easier for water to enter the pores, therefore the tablet's release time can be faster. Meanwhile, the tight pores caused the water difficulty to enter the pores; therefore the tablet's release time became slower. Based on Table 2, the results of solubility in the soil were obtained. The sample with 2.5% solubility dissolved in the soil for 14 days, while the solubility of 5% can dissolve in the soil for 25 days, and for the solubility of 7.5% can dissolve in the soil for 37 days. Based on Table 3, results were obtained on percolation testing of water included in rice husk tablets. Potassium obtained in 500 ml of water was 74.89 mg / L, for chlorine obtained as much as 0.11 mg / L, and the resulting silica was 16.68 mg / L.

CONCLUSION

The results show that the extraction of silica from rice husks is found to range between 1.48-5.3%. The results of the XRF analysis show that the largest silica value is 6.51%. Based on these results, heating using microwave does not produce optimal silica results. The relationship between the concentration of KOH and hardness has a direct proportional result, while the length of heating duration with hardness is inversely proportional. The smallest results are obtained with a value of 1,131 Kgf and the largest as much as 3,356 Kgf.

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