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EFFECTS OF PROCESS CONDITIONS ON THE PRODUCTION OF SILICA FROM RICE HUSK

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Abstract -The impact of process variables on the silica production from rice husk is presented in this article. Proximate analysis was used to characterize the rice husk. Fourier transform infrared spectroscopy was also utilized to identify the functional groups of the generated silica. Next, the impact of process factors on the yield of silica was ascertained. In addition, the kinetics of the process were assessed, and the yield was adjusted using response surface methods. From the analyses of the results, compositions of the rice husk in terms of carbohydrate, ash, crude fiber, moisture content, crude fat, and protein) were revealed. Ash content of 28.33% was recorded for the rice husk, indicating high mineral content. The moisture content of the dried rice husk (9.38%) is within the acceptable limit. There are presence of hetero atoms and double bond structures in the produced silica. This is an indicator that silica is a probable precursor for making adsorbent. Additionally, the silica that was created had Si-O bond stretch or silicate. It demonstrates that silica may be used as an adsorbent in wastewater treatment. Temperature, duration, and stirring rate all affect the silica yield. It rose as the temperature, stirring rate, and time increased until the maximum point was attained. The second-order model fits the silica manufacturing process the best, according to the kinetic analysis.
Keywords: Silica, Rice Husk, Ash, Agro Waste

1 Introduction

One agricultural waste product produced during the processing of rice is rice husk. It is a by-product that is typically burned or discarded, resulting in environmental pollution. Rice husk exhibits a significant ash content ranging from 18% to 20%. The primary constituent of rice husk ash is silica, which varies within the range of 85% to 95% (Mehta et al, 2015).

Rice seeds are produced by the monocot plants Oryza sativa (Asian rice) and Oryza glaberrima (African rice). This plant is commonly grown as an annual, although it can grow as a perennial in tropical regions and continue to give an annual crop for up to 30 years. Since rice provides more than one-fifth of the calories consumed globally by humans, it is the most significant grain in terms of human nutrition and calorie intake. In contrast, maize crops are planted largely for purposes other than human consumption. The maximum height of a rice plant is 1.8 m (3.3 5.9 feet), yet depending on the quality of the soil, some kinds can grow up to 1.8 m (Tejinder, 2000). Its long, thin leaves have a length of 50–100 cm (20–39 in) and a width of 2–2.5 cm (0.79–0.98 in) in width. The tough outer layer that protects rice grains is called rice husk. The coating that covers the grains, or seeds, of the rice plant is called rice hulls. Opaline silica and lignin are two examples of the rigid components that comprise the hull that shield the seed during the growth season. Rice husk has a wide range of applications, including as a fuel for brick kilns, furnace, and rice mills for the parboiling process (Tejinder, 2000).

The most common substance in the Earth's crust is silicon dioxide (SiO2), while rice husk (RH) is a widely available agricultural waste. husk and rice husk ash (RHA) are interesting sources of large amounts of high-quality silica that can be used for a variety of applications (Ugheoke et al., 2013). On an industrial scale, silica is produced by energy-intensive thermal processes at high temperatures involving large amounts of acids in mechanical, physical, chemical, and physical processes. Large amounts of wastewater are produced by these procedures. Ma et a., (2012) draw attention to the fact that the traditional technique of producing silica relies on the reaction of quartz and sodium carbonate at elevated temperatures. After sulfuric acid and sodium silicate are produced, silica is precipitated. On the other hand, this process generates a lot of greenhouse gases and liquid wastewater while also consuming a lot of energy. This paper addresses the production of silica from the rice husk.

2.1 Materials Used

The materials needed for this study are as follows: rice husk, distilled water, pH meter, laboratory drying oven, magnetic stirrer, electronic muffle furnace, electromagnetic sieve shaker, electric weighing balance, volumetric flasks, grinding machine, conical flasks, hot plate, agitator, measuring cylinders, beakers, spatula, Fourier transform infrared spectroscope (FTIR) and centrifuge.

2.2 Sample collection and preparation

For this investigation, rice husk was gathered from the Umejit rice mill in Ugbawka, Enugu State, Nigeria, and crushed using a grinding machine into smaller pieces.

2.3 Proximate Analysis of Rice Husk

2.3.1 Determination *of moisture content*

The proximate analysis of the rice husk was carried out using the Association of Official Analytical Chemists (AOAC method, 1990). Five grams of rice husk were weighed and then put into crucibles, which were then baked in an oven set at 105 degrees Celsius for four hours to determine the moisture content. After being taken out of the oven, the sample was cooled and weighed. After constant drying was completed, the weight was observed and recorded. Equation (1) was then used to compute the moisture content.

$$\% \text{ moisture} = \frac{A-B}{A} \times \frac{100}{1} \qquad (1)$$

where A is the initial mass of the sample (rice husk), while B is the weight of the dried sample.

2.3.2 Determination *of protein content*

The Standard method (Kjeldahl method) was used to calculate the crude protein content. The technique project and analysis specify nitrogen as the primary protein content in rice husk by estimating the total nitrogen in the sample and analyzing the nitrogen's conversion to protein. The actual proportion of protein in the sample was determined by multiplying the percentage of crude protein by the percentage of nitrogen, using a conversion ratio of 6.25. Using 0.01M standard HCl, titration was performed to produce the first pink color.

% Nitrogen =

$$\frac{Titration vol. \times 0.014 \times M \times 100x}{wt. of sample} \frac{100}{10} \qquad (2)$$

where M is the molarity of standard HCl.

2.3.3 Determination *of crude fibre content*

150ml of hot 0.128M H2SO4 was added to a 500ml beaker containing 5g of the rice husk sample. After 30 minutes of heating at 70°C, it was filtered and cleaned with hot distilled water. After that, the residue was moved to a beaker and heated to 70 degrees Celsius for 30 minutes using 150 milliliters of 0.223 milligrams KOH. After filtering and hot water washing, the washings were made to become alkaline. After three acetone washes, the residue was dried for one and a half hours at 105 degrees Celsius in an oven. Following a desiccator chill, it was weighed and cooked for four hours at 500°C in a muffle furnace. After cooling in a desiccator, the ash was weighed.

$$\% \text{ Crude fibre} = \frac{W_2 - W_3}{W_1} \times \frac{100}{1} \qquad (3)$$

where W_1 is the sample weight, W_2 is the dry residue weight, and W_3 is the ash weight.

2.3.4 Determination *of fat content*

Five grams of rice husk were measured, transferred into a roll of filter paper, and then put within the extraction thimble to ascertain the fat content. The extractor was filled with the thimble. Petroleum ether (180 ml) was added to the extraction flask. The extractor was attached to the flask and the condenser. The petroleum ether was recovered after the entire unit was heated for three hours on a mantle. The oil obtained in the flask was dried in an oven at 105°C. After that, it was weighed, and Equation (4) was used to determine the proportion of fat.

$$\% \text{ fat} = \frac{C - A}{B} \times \frac{100}{1} \quad (4)$$

The weights of the initial sample (B), the empty flask (A), and the flask with oil (C) are indicated.

2.3.5 Determination *of ash content*

To ascertain the sample's ash level, 5g of finely crushed rice husk was weighed into porcelain crucibles that had been cleaned, dried at 100°C in an oven, chilled in a desiccator, and then weighed again. It was then heated in a muffle furnace for three hours at 600 degrees Celsius. After that, it was taken out to cool in a desiccator and its ash content was measured by weighing it again. Equation (5) was used to calculate the amount of ash.

$$\% \text{ Ash} = \frac{A - B}{C} \times \frac{100}{1} \quad (5)$$

where B is the weight of the crucible, C is the weight of the original rice husk sample, and A is the weight of the crucible plus ash.

2.3.6 Determination of* carbohydrate *content

Total carbohydrate content was calculated in Equation (6).

Total carbohydrate =100 - (protein + fat + crude fiber + ash + water) (6)

2.4 Production of the Silica

To get rid of dirt, the rice husk was cleaned with tap water. It spent three days being sun-dried.

It was kept in a porcelain cup and calcined at 700^{o}C for 4 hours using a muffle furnace. The furnace was left open to allow more air for complete combustion. The furnace was allowed to cool, and the ash was formed. The method used for the synthesis of the silica is the sol-gel method. 20g of the ash was mixed with 2.5N NaOH (160ml) in a reflux setup. To dissolve the silica from the ash, the ash was heated for three hours in a 250 ml Erlenmeyer flask while being constantly stirred. Whatman No. 1 ashless filter paper was used to filter the sodium silicate that was created, and boiling distilled water was used to wash away any leftover residue. It was decided to let the filter cool to room temperature. To bring the pH of the filtrate (sodium silicate) to neutrality, 5N H2SO4 was added while stirring continuously. One day was spent aging the gel that the sol produced. Following aging, the slurry was centrifuged for five minutes at 400 rpm after the soft gel had been gently broken. After discarding the supernatant, the gel was put into a beaker and dried for 24 hours at 80 degrees Celsius. It was sieved and ground.

2.4.1 FTIR Characterization *of the Silica*

Functional groups of the silica were identified by Fourier transform infrared (FTIR) spectroscopy. It was done by the transmittance method with a range of 4000 - 650 wavenumber.

2.5 Determination of the Effects of Process Variables on the Silica Yield

Effects of process parameters on the silica yield were determined based on the 0ne factor at a time.

2.6 Kinetics of the methyl silica yield

The effect of temperature on the silica yield was used for the kinetic studies.

Ln(1-x) versus t was plotted in consideration of the first order model.

$$\ln(1-x) = -kt \quad (7)$$

where x is the fraction conversion of silica and t is the time.

For the zero-order model, x was plotted against t.

The rate constants K obtained from the kinetics of silica produced were used to evaluate the thermodynamic properties.

Second Order Kinetic Model

$$r_e = \frac{-dC_t}{dt} = K(C_s - C_t)^2 \quad (8)$$

Such as the integration of Eq. (9), using the boundary conditions C_t=0 at t=0 and C_t=C_t at t=t. The equation was then rearranged in a linearized form

$$\frac{t}{Ct} = \frac{t}{Cs} - \frac{1}{C_s^2 K} \quad (9)$$

As expressed by Onukwuli and Omotioma (2016), the Arrhenius equation was used to evaluate activation energy (10),

$$\text{Lnk} = \frac{-Ea}{RT} + \text{ In A} \quad (10)$$

Where k is the rate constant, Ea is the activation energy, R is the universal gas constant

($0.008314 KJmol^{-1}K^{-1}$), T is the temperature in Kelvin and A is pre-exponential constant.
Equation (3) presented a plot of various Lnk, from the kinetic study, against $^{1}/_{T}$ to obtain the activation energy (Ea) from the slope of the straight-line graph.

3.0 Results and Discussion

3.1 Characterization of the Rice Husk

The result of the proximate analysis of rice husk which was carried out using the Association of Official Analytical Chemists (AOAC method, 1990) is shown in Table 2. The compositions of the rice husk (in the order highest to lowest) include carbohydrates, ash, crude fibre, moisture content, crude fat, and protein. The high carbohydrate composition shows a highly starchy nature of the material with a very minute protein composition. Ash content of 28.33% was recorded for the rice husk, indicating high mineral content (Patil and Sharanagouda, 2017; Goh et al., 2016). The moisture content of the dried rice husk (9.38%) is within the standard limit; which is not more than 10% for long-term storage (Akubor et al, 2013; Omotoma and Akubor, 2012). This low moisture content would improve the rice husk's storage stability. This can be achieved by preventing microbial growth and reducing moisture-dependent biochemical reactions.

Table 1: Proximate analysis of the rice husk

Composition	Values
Ash (%)	28.23
Crude fat (%)	4.46
Crude fibre (%)	14.61
Moisture content (%)	9.38
Protein (%)	3.94
Carbohydrate (%)	39.38

3.2 Fourier Transform Infrared (FTIR) Spectroscopic Result of the Silica

Table 3 presents the functional groups of the silica. The FTIR spectrum of the silica is presented in Appendix A. The FTIR result shows the presence of hetero atoms and double-bond structures. This is an indication that silica is a potential precursor for producing adsorbent. In addition, Si-O bond stretch (silicate) was present in the produced silica. It shows that silica is a potential source of adsorbent for wastewater treatment.

Table 2: Functional groups of the silica

Peak	Functional Group	Class of Compound
689.6841	C = O Stretch	Amides
785.1607	C-H bend	Aromatic compounds
1025.273	=C-0-C sym.&asym.stretch	Ethers
1284.376	Si-O bond	Silicate
1387.513	CH_3C-H bend	Alkanes and Alkyls
1634.731	C=O stretch	Amides
2056.332, 2200.375	C-I stretch	Alkynes
2508.634	O-H stretch	Carboxylic Acids
2766.25	H-C=O stretch	Aldehydes
2895.117	C = H bend	Alkanes and Alkyls
3020.265	=C-H stretch	Alkenes
3184.684	O-H stretch	Carboxylic acids
3305.093	O - H stretch	Alcohols
3714.717, 3823.984	N - H stretch	Amines

3.3: Effect of Process Variables on Silica Yield.

Effects of process conditions (stirring rate, temperature, and time) on silica yield are presented in Fig.1, 2, and 3 respectively. They all display quadratic curves. In Figure 1, the plot of the stirring rate on silica yield revealed a maximum yield of 29.56%, which occurred at a stirring rate of 400rpm. The yield increased with an increase in stirring rate till the maximum was reached. For the plot of temperature on silica yield (Figure 2), the maximum yield occurred at the temperature of 700°C. The silica yield increased with a rise in temperature till the maximum point was attained (Saranga et al, 2009; Kasaai, 2015). A similar trend was recorded in the relationship between silica yield and time. Maximum silica yield occurred at the time of 4min.

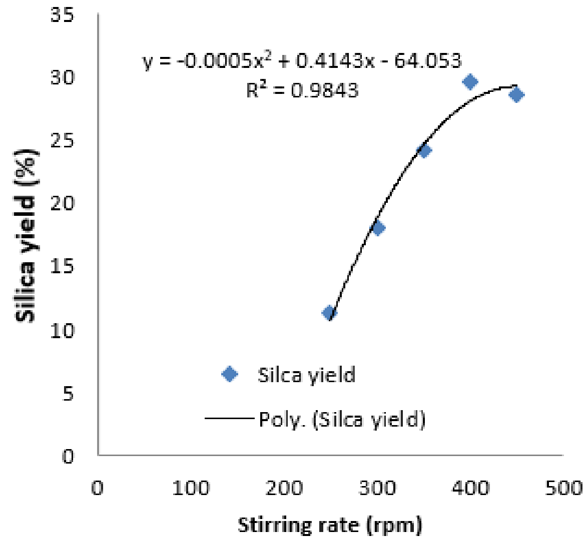

Figure 1: Effect of stirring rate on silica yield

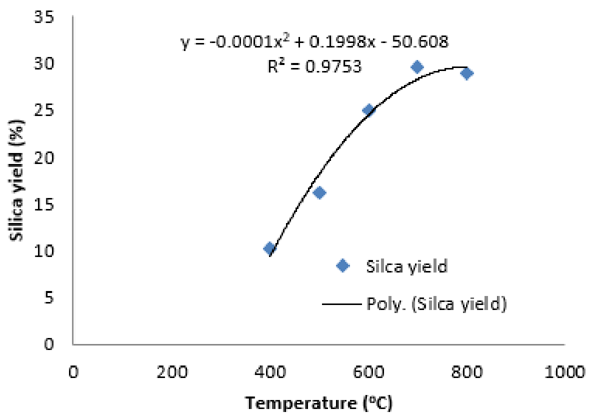

Figure 2: Effect of temperature on silca yield

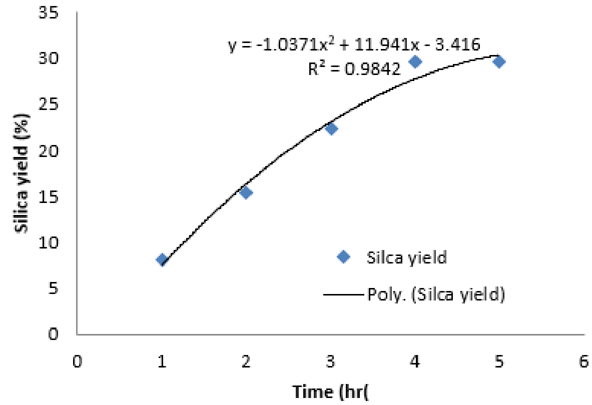

Figure 3: Effect of time on silica yield

3.4 Kinetic Parameters

The order and kinetic parameters of the methyl ester production are shown in Table 8. Zero-order, first-order, and second-order kinetics were considered at different temperatures; 773K, 873K, and 973K. The rate constant (k) increased with an increase in temperature. Values of correlation of determination (R^2) of the second-order model are closer to one compared to those of the first and zero-order models. Hence, the second-order model is the best-fitted model of the silica production process. Table 9 lists the values of the activation energy. They were determined through the use of the Arrhenius model in the graphical analyses of the experimental data. The figures represent the minimal energy needed for the formation of silica.

Table 3: Kinetic Parameters

Model	Temp. (k)	K	R^2
Zero order	773	0.0037	0.9702
	873	0.0045	0.9723
	973	0.0060	0.9851
First order	773	0.0139	0.9955
	873	0.0274	0.9963
	973	0.0458	0.9972
Second order	773	0.7973	0.9981
	873	0.9341	0.9990
	973	1.0081	0.9996

Table 4: Activation energy of the silica production

Order	Activation energy (kJ/kmol.K)
First	14939.43
Zero	37314.89
Second	7400.375

Conclusion

The following conclusions were deduced from the results analysis. Compositions of the rice husk (in the order highest to lowest) include carbohydrate, ash, crude fibre, moisture content, crude fat, and protein. The high carbohydrate composition shows a highly starchy nature of the material with a very minute protein composition. Ash content of 28.33% was recorded for the rice husk, indicating high mineral content. The moisture content of the dried rice husk (9.38%) is within the acceptable limit. There is the presence of hetero atoms and double bond structures in the produced silica. This is an indication that silica is a potential precursor for producing adsorbent. Also, Si-O bond stretch (silicate) was present in the produced silica. It shows that silica is a potential source of adsorbent for wastewater treatment. The silica yield is a function of stirring rate, temperature, and time. It increased with a continuous increase in stirring rate, temperature, and time till the maximum point was achieved. The second-order model is the best-suited model of the silica production process.

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