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Optimization and Characterization of Bio-Silica Extraction from Rice Straw Using RSM

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Abstract
Egypt produces about 7.86 million tons of rice straw annually. Egyptian farmers have traditionally gathered and burnt rice straws in the field in the open for rapid disposal. Environmentalists have recently attacked the practice, and legislation was passed to limit open burning, which pollutes the environment in many nations. The purpose of this research is to extract amorphous bio-silica from rice straw after burning it to produce ash, then use sodium hydroxide for an alkaline extraction to produce a sodium silicate solution. Hydrochloric acid was added to a solution of sodium silicate to precipitate silica. Temperature, time, and concentration were the three main variables that influenced alkaline extraction. To improve these values, a response surface strategy was adopted. The reaction occurred between 30 and 90 °C, extraction time between 1 and 3 h, and had a sodium hydroxide concentration of 1 and 3N. At 90°C, 3N sodium hydroxide concentration, and 3 hours, the highest yield of silica is anticipated to be 83%. The produced bio-silica was characterized using XRF, XRD, FTIR and SEM. The results indicated that precipitated bio-silica can be extracted from rice straw to produce a high yield and used in different applications.

Keywords: Rice Straw – Optimization – Response Surface Methodology – Alkaline Extraction - Silica

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1. Introduction
In the Middle East, Egypt is third in terms of the availability of agricultural waste, which totals 71.5 million tonnes [1].Agricultural waste is created by a variety of agricultural operations and includes crop stalks, jute fibres, sugarcane bags, rice husk, rice straw and wheat straw, and wheat husk. The abundance of agricultural waste contributes significantly to the contamination of the environment. Waste materials' characteristics have changed over time, causing harmful and toxic effects on humans [2]. The amount of rice straw produced in Egypt is about 7.86 million tons per year [3]. For quick disposal, Egyptian farmers have long collected and openly burned rice straws in the field. Environmentalists have criticized the practice in recent years, and legislation was given for the restriction of open burning that pollutes the environment in several countries. In rice-producing areas, managing rice straw is a difficult task. Therefore, a technique for efficient garbage disposal is required to transform this trash into something useful [3]. As a result, rice straw may be a source of renewable fuel that might replace fossil fuels, reduce CO_2 emissions, and eliminate pollution from straw burning in the open. The rice straw ash has a high silica content [3]. After the combustion of rice straw, the ash is produced, and silica can be extracted from the ash of rice straw using an alkaline solution and precipitated by acid to produce precipitated silica [4]. Precipitated silica can be utilised as an anti-caking agent in some food sectors as well as in tyres, silicone rubber, adhesives, toothpaste, and silica gel preparation [5]. The main factors affecting silica yield are Alkali content, temperature and time. A crucial factor in getting the best extraction efficiency is the alkali concentration. Since virtually all of the silica in the ash interacts with alkaline, increasing the amount of alkali will correspondingly increase the

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concentration of silica (SiO_2). As the alkali concentration rose, the proportion of SiO_2 progressively climbed. High temperatures can improve leaching efficiency and silica production, however, it seems that their employment with alkali is constrained given their low boiling points. High temperatures cause evaporation, which lowers the reaction's solubility. As extraction duration increases, silica yield rises until it achieves equilibrium and doesn't alter as time increases [6][7]. Numerous studies have recommended producing silica from various agricultural wastes, as demonstrated by Amibo et al. [8] used RSM to study the effect of different parameters on silica extracted from teff straw using the sol-gel method. These parameters are extraction temperature, time, rotational speed, and NaOH concentration. They studied the characteristic of teff straw ash, they found that ash contains about 93% silica. They extracted silica using varied times (55 to 115 min), the concentration of sodium hydroxide (1 to 3M), rotational speed (350 to 550 rpm), and temperatures (80 to 100℃). They found that the optimum condition to obtain the maximum yield (85.85%) of amorphous silica at a temperature of 94.98℃, time of 109.99 min, 1.5M of sodium hydroxide, and rotational speed of 499.57 rpm. While Llovera et al. [9]investigated the maximum recovery of silica production from the combustion of wheat straw. They optimized the factors affecting silica extraction and found that the maximum yield obtained was about 82% at pH a temperature of 85℃, pH varied from 9 to 10 and precipitated pH from 5 to 5.5 with high silica purity. On the other hand Gun et al. [10] characterized the silica extracted from rice husk and its application in the adsorption of safranin dye solution. They studied the effect of time, temperature, and the ratio of potassium hydroxide to rice husk ratio. It was found that the maximum obtained yield was 55%. The optimum condition of safranin adsorption uses an adsorbent dose of 1 $g.l^{-1}$ with an initial concentration of 25 $mg.l^{-1}$, contact time of 60 min, and pH of 8. Also, Azhar et al. [11] investigated the extraction of silica using the acid leaching method from sugar cane bagasse. They studied the parameters that affected the extraction of silica which are acid concentration ranging from 1 to 2 M and leaching time ranging from 30 to 90 min. They found sugar cane bagasse is an alternative source of silica production and also found that increasing the leaching time increases the percentage of silica obtained. Using the Box-Benhken design, Olawale [12] investigated the optimization of several factors, including duration (2–7 h), number of bamboo leaves (2–6 g), and fire temperature (400–700°C), that influenced the extraction of silica from bamboo leaves. At a temperature of 600 °C, for 4.5 hours, and with a mass of 5 g of bamboo leaves, he discovered that they were the optimum conditions for silica extraction. The technique of turning discarded bamboo leaves into highly reactive silica, which is utilised in the production of pottery, is also one he discovered to be valuable.

Currently, one of the major agricultural wastes generated during the harvest of rice is rice straw. The objective of this study is to use response surface methods to extract biosilica from rice straw to reduce the harmful emissions created when burning rice straw in an open field that have an impact on both humans and the environment. Also, study the effect of different parameters on silica yield and optimize the conditions to obtain maximum silica yield.

2. Materials and Methodology

2.1 . Raw Materials

The main raw materials used in silica extraction were rice straw collected from farmland, in Al Sharqia governorate, Egypt. The chemicals (hydrochloric acid and sodium hydroxide) used in silica extraction were kindly supplied from Alpha Chemika, Egypt.

2.2 . Sample Preparation

Rice straw (RS) was washed with hot water to remove any impurities and then dried in an oven at 105℃ for 3h. After drying, (RS) was burned in a muffle furnace at 700℃ for 3h to produce (RS) ash. The ash was stored in a small container to prevent any contamination of ash. The production of silica after the combustion of (RS) can be illustrated according to the following chemical reaction [13]:

$$C, H, Si, O_2, \text{impurities} \rightarrow CO_2\,(g) + H_2O\,(g) + SiO_2\,(s) + \text{metal oxides}\,(s) \qquad (1)$$

2.3 Characteristics of Raw Materials

Thermogravimetric analysis (TGA) of rice straw (RS) to study the effect of raising the temperature on the weight of (RS) and show the thermal stability of (RS). Thermogravimetric analysis is used with model Q500 in The Egyptian Academy for Engineering and Advanced Technology with a heating rate equal to 7°C per minute at a temperature range of (25-900)℃. On the other hand, Rice straw ash was analyzed using X-ray fluorescence spectrometry (XRF) to identify the chemical composition of the raw materials which was performed using an AXIOS, PANalytical 2005, wavelength dispersive (WD-XRF) sequential spectrometer placed at the National Research Center. Mineralogical analysis (XRD) was used to determine if a sample has crystalline or amorphous phases of a wide range of materials, often for mineralogical examination and material identification. The mineralogical composition is evaluated in the Science and Technology Center of Excellence using an X-ray diffraction Bruker D8 advanced computerized using X-ray with monochromatic radiation.

2.4 . Extraction of Silica

The alkaline extraction method is the most common method used in silica extraction. 10 g of rice straw ash was added to 100 ml of sodium hydroxide solution with a liquid-solid ratio (LSR) of 10 (v/w). The sample was placed in a water bath and heated at various temperatures ranging from (30-90)℃, a constant stirring rate with different contact times ranging from (1-3) h and different concentrations of sodium hydroxide varied from (1-3) N. After a certain time, the solution was cooled to room temperature and then filtered to separate the residue of ash from the sodium silicate solution using a filter cloth. Hydrochloric acid with 1 N was added to sodium silicate solution to precipitate silica gel to adjust pH varied from (7 to 9). The precipitated gel was aged for 24 h then filtrated and washed using distillate water to remove salts from silica. The silica was dried using an oven at a temperature of 105℃ for 4 h to remove moisture content till the weight become constant, the experimental procedure was illustrated as shown in Fig 1. The yield of silica can be determined using the following Eq (2):

$$Yield(Y)\% = \frac{mass\ of\ silica\ extracted}{weight\ of ash} \times 100 \quad (2)$$

The extraction of silica was done according to the following chemical reactions [10]

$$SiO_2 + 2\,NaOH \quad \rightarrow \quad Na_2SiO_3 + H_2O \quad (3)$$

$$Na_2SiO_3 + 2HCl \rightarrow SiO_2 + 2NaCl + H_2O \quad (4)$$

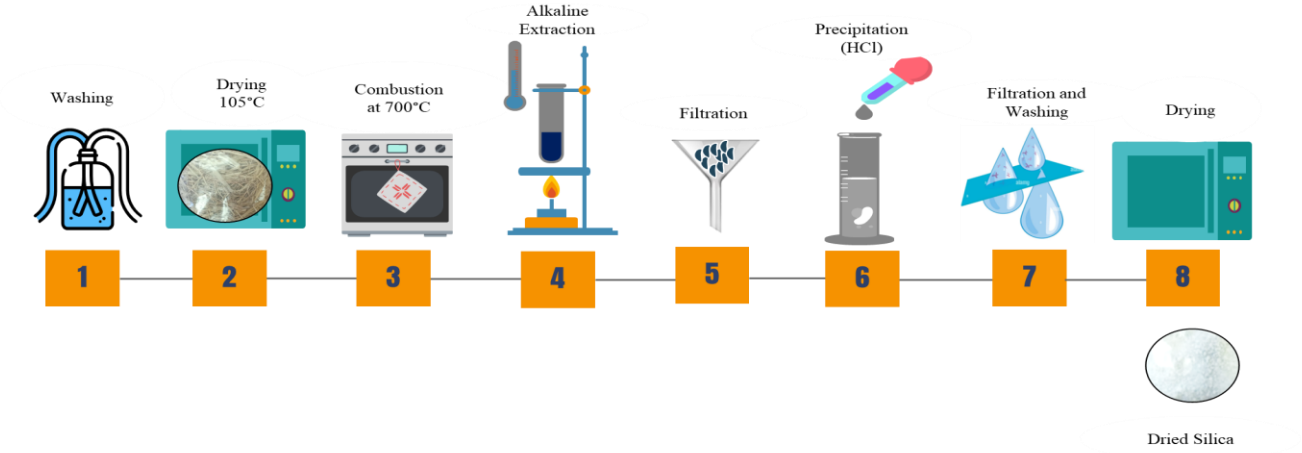


Fig. 1: Scheme of Silica Extraction

2.5 Experimental Design Using Response Surface Methodology

Experiments were performed to determine the range of the extraction variables to be optimized by determining silica yield. Different factors affecting on the extraction of silica from (RAW) ash. The main three factors are temperature, time, and concentration of sodium hydroxide. These factors have a great effect on the yield of extracted silica. Box-Behnken design is used to calculate the optimum conditions for silica extraction using three different independent variables. These variables are time-varying from (1 to 3) h, temperature from (30 to 90) ℃ and concentration of sodium hydroxide (1 to 3) N as mentioned in Table (1). The three independent variables were coded as follows: (-1) minimum, (0) average, and maximum (1).

The number of experiments can be calculated using Eq (5) [14]:

$$Number\ of\ experiments = 2K\ (K-1) + C_o \quad (5)$$

Where

K: represents the number of factors that affect the experiments.

C_o: number of replicas of the center point that equals 5 experiments.

It is shown that the required number of experiments is 17 under different conditions, but there were five experiments under the same conditions (center point) which applied to optimize the extraction of silica using (RS) ash via Design Expert Software V.12 [15].

Table 1: Coded and Actual Values of variables Levels

Variable	Symbol	Coded and Actual Levels		
		-1	0	1
Time	X1	1h	2h	3h
Temperature	X2	30°C	60°C	90°C
Concentration	X3	1N	2N	3N

2.6 Statistical Analysis and Validation of Optimized Variables

The statistical analysis of the results obtained from experiments was applied using the analysis of variance (ANOVA) via Design-Expert Software (V.12). In all systems, significance can be considered when the p-value is less than 0.05, the significance of regression coefficients is evaluated using F-test. The determination coefficient (R^2) and adjusted coefficient (R_a^2) were used to determine the accuracy of the model. The competence of the generated model was models was confirmed by applying the extraction of silica at the optimum conditions in triplicate and determining the average experimental silica yield [16][17].

3. Results and Discussions

3.1 Characteristics of Raw Materials

The characteristics of rice straw and its ash were determined in the following section.

3.1.1 Thermal Gravimetric Analysis

Fig 2 illustrated the TGA of (RS) that showed a slight weight loss of about 8% due to the removal of the physical water presence in (RS) ranging from 30 to 120°C. Cellulose has a high disorder crystalline structure while hemicellulose is a branched polymer that has a low degree of polymerization. They decomposed at temperatures varied from 200°C to 350°C. Another weight loss in the range of 350°C to 600°C is due to the gradual decomposition of lignin which has higher thermal stability with a wide range of thermal decomposition [18][19].

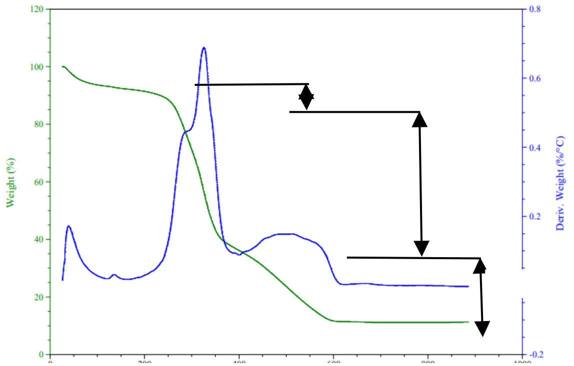


Fig. 2: TGA of Rice Straw

3.1.1 Chemical Analysis (XRF)

The mineral composition of (RS) ash is shown in Table (2). The total weight of silicon dioxide (SiO_2) in (RS) ash was 84.48% and the remaining is impurities.

3.2 Experimental Design and Statistical Analysis of Silica Extraction

Design Expert (version 12.0.3) was used to give the statistical analysis where model fitting statistics and contour statistical model graph were obtained. The results obtained were compared to the predicted results. The optimum conditions for maximum silica extraction yield were also obtained from the Design Expert.

Table 2: Chemical Analysis of Rice Straw Ash

Main Constituents	(wt%)
SiO_2	**84.48**
TiO_2	**0.06**
Al_2O_3	**0.36**
$Fe_2O_3^{tot.}$	**0.53**
MgO	**1.26**
CaO	**6.15**
Na_2O	**0.48**
K_2O	**2.06**
P_2O_5	**0.48**
SO_3	**0.79**
Cl	**0.13**
L.O.I	**2.19**
Br	**0.004**
MnO	**0.529**
NiO	**0.008**
CuO	**0.013**
ZnO	**0.027**
PbO	**0.409**
SrO	**0.067**

3.2.1 Model Fitting and Statistical Analysis

The parameters of the process used are (time, temperature and concentration of NaOH) as independent variables which affect the yield of silica extraction. These parameters were investigated using Box-Behnken design (BBD) to optimize and evaluate their effects on the system response (yield). Table (3) shows the results of the experimental design of response surface methodology and the obtained yield at different conditions temperatures varying from (30 to 90℃), concentration from (1 to 3 N) and time from (1 to 3 h). The minimum silica yield was 7% at a temperature of 30℃, the concentration of 1 N and a time of 2 h while the maximum silica yield was 78% at a temperature of 90℃, the concentration of 3 N and a time of 2 h.

Several considerations were performed to choose the highest order polynomial that describes the relation between the response (yield) and the independent variables (time, temperature and concentration). It was found that the model is second order.

The polynomial quadratic model can be described as the following Eq (6):

$$Y = \beta 0 + \Sigma_{i=1}^{n} \beta i Xi + \Sigma_{j=1}^{n} \beta ii Xi^2 + \Sigma_{i=1}^{n-1} \Sigma_{i=1}^{n} \beta ij Xi Xj + \varepsilon \qquad (6)$$

Where Y represents the predicted response, β0 is the model intercept, and βi, βii, and βij are the regression coefficients for the linear, quadratic, and interactive effects of the model, respectively. Xi and Xj are the coded variables and ε is the error of the model [20].

Table 4 shows the results of ANOVA and the regression analysis. The F-value of the model is 27.71 while the p-value is less than 0.005. According to the results obtained, the low p-value and high F-value indicate the validity of the model. The reliability of the model is also investigated to confirm its validity of the model. The high value of coefficient R^2 (0.9727) and Adj-R^2 (0.9376) indicated that the model is related to the obtained experimental results and is suitable to predict the relation between different variables and the response (yield of silica).

Each p-value in the model term is an indication if it is significant or not. The model term is significant when p-value is less than 0.05 which indicates the effect of this variable on the response. In this case, A, B, C, BC, B^2 and C^2 are significant model terms.

The following Eq (7) express the quadratic polynomial order for silica yield:

$$Silica\ Yield\ (\%) = -83.5 + 14.575 * Time + 2.08167 * Temp + 22.325 * Conc + 0.141667 * Time * Temp - 0.5 * Time * Conc + 0.375 * Temp * Conc + -4.3 * Time^2 - 0.0200556 * Temp^2 - 7.3 * Conc^2 \qquad (7)$$

3.2.2 Optimization of Extracted Silica

The cube model determines the minimum and the maximum value of the three factors (time, temperature, and concentration of sodium hydroxide) that affecting on the yield produced which were respectively represented in A, B, and C. As shown in Fig (3) the minimum silica yield is obtained to be 1.2% at 1h, 30°C, and 1N while the maximum yield is obtained to be 82.7% at 3h, 90°C, and 3N. Five runs were performed at optimum conditions (3h, 90℃ and 1N) to assess the validity of these conditions. The mean percentage of silica yield was found 79.7% with a standard deviation equal to 2.6 which proves the validity of the suggested model. The calculated error percentage was found 3.63%.

Table 3: Silica Yield at Different Conditions

	Factor 1	**Factor 2**	**Factor 3**	**Response**
Run	A: Time (h)	B: Temperature (°C)	C:Concentration (N)	Yield (%)
1	2	30	3	14
2	2	90	3	78
3	1	60	1	29
4	1	60	3	59
5	2	60	2	62
6	3	30	2	17
7	2	60	2	62
8	2	90	1	26
9	2	60	2	54
10	1	30	2	8
11	2	60	2	52
12	3	90	2	69
13	2	60	2	53
14	3	60	3	60
15	1	90	2	43
16	3	60	1	32
17	2	30	1	7

3.2.3 Effect of Different Variables on Silica Yield

A contour plot indicates the minimum and maximum value and also indicates the effect of two factors on a certain response on a two-dimensional plot. In the contour statistical model graph, two factors change while the third factor remains constant. Changing the color from blue to red indicates that the yield increases. The bluer the color, the lower the yield, and the redder the color, the higher the yield under any condition. Each line shows the yield obtained at any condition corresponding to the two other parameters through the line. The contour plot is useful for establishing the response values and operating conditions as required.

3.2.3.1 Effect of Extraction Time

Fig (4) shows the effect of varying time on silica yield at different concentrations of NaOH and temperature. it is indicated that increasing the extraction time causes an increase in the yield of silica. At the time of 1h, the maximum obtained silica yield was found 60%, while at the time of 3h the maximum obtained silica yield was about 80%. These results comply with that obtained by Park et al.[21], who found that increasing the extraction time above 3h, no significant change in silica yield.

Table 4: ANOVA for Regression Model of Silica Yield

	Sum of Squares	df	Mean Square	F-value	p-value	
Model	7890.43	9	876.71	27.71	0.0001	significant
A-Time	190.13	1	190.13	6.01	0.0440	
B-Temperature	3612.50	1	3612.50	114.19	< 0.0001	
C-Concentration	1711.13	1	1711.13	54.09	0.0002	
AB	72.25	1	72.25	2.28	0.1745	
AC	1.0000	1	1.0000	0.0316	0.8639	
BC	506.25	1	506.25	16.00	0.0052	
A^2	77.85	1	77.85	2.46	0.1607	
B^2	1371.80	1	1371.80	43.36	0.0003	
C^2	224.38	1	224.38	7.09	0.0323	
Residual	221.45	7	31.64			
Lack of Fit	122.25	3	40.75	1.64	0.3142	not significant
Pure Error	99.20	4	24.80			
Cor Total	8111.88	16				
R^2	0.9727					
Adj-R^2	0.9376					

3.2.3.2 Effect of Extraction Concentration of Sodium Hydroxide

From Fig (4), it is obvious that increasing the concentration of NaOH increases the yield of extracted silica at a minimum concentration of NaOH, the silica yield reached 30% while increasing the concentration causes increasing of yield to reach about 80%. This result complies with Ikram et al, Ajeel et. and Setyawan et al. The reason for this behaviour is due to increasing the amount of solvent used (NaOH) which increases the contact between rice straw ash and the solvent, this allows a higher distribution of solvent and the interaction between Na^+ ions and the negative ions of silicate that increases the formation of sodium silicate and therefor increases the yield of silica produced [22][23][13].

3.2.3.3 Effect of Extraction Temperature

The temperature has a great effect on silica yield. As seen in Fig. (4), at a minimum temperature of 30℃ there wasn't a significant change in the silica yield, the highest yield could be reached at a maximum concentration is 15%. This indicates that the effect of temperature is higher than the effects of concentration and time [24]. Increasing temperature causes a change in silica yield, at a temperature of 60℃ yield reaches 60% and increases with increasing temperature to reach 80% at 90℃. These results are similar to Yeon Park et al. [21] because silica has an

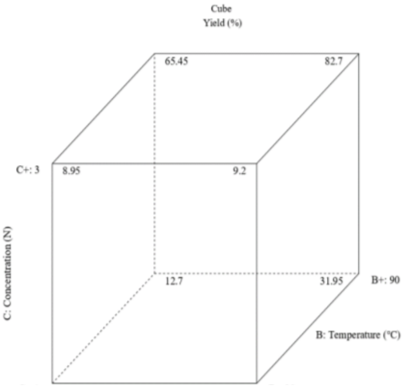
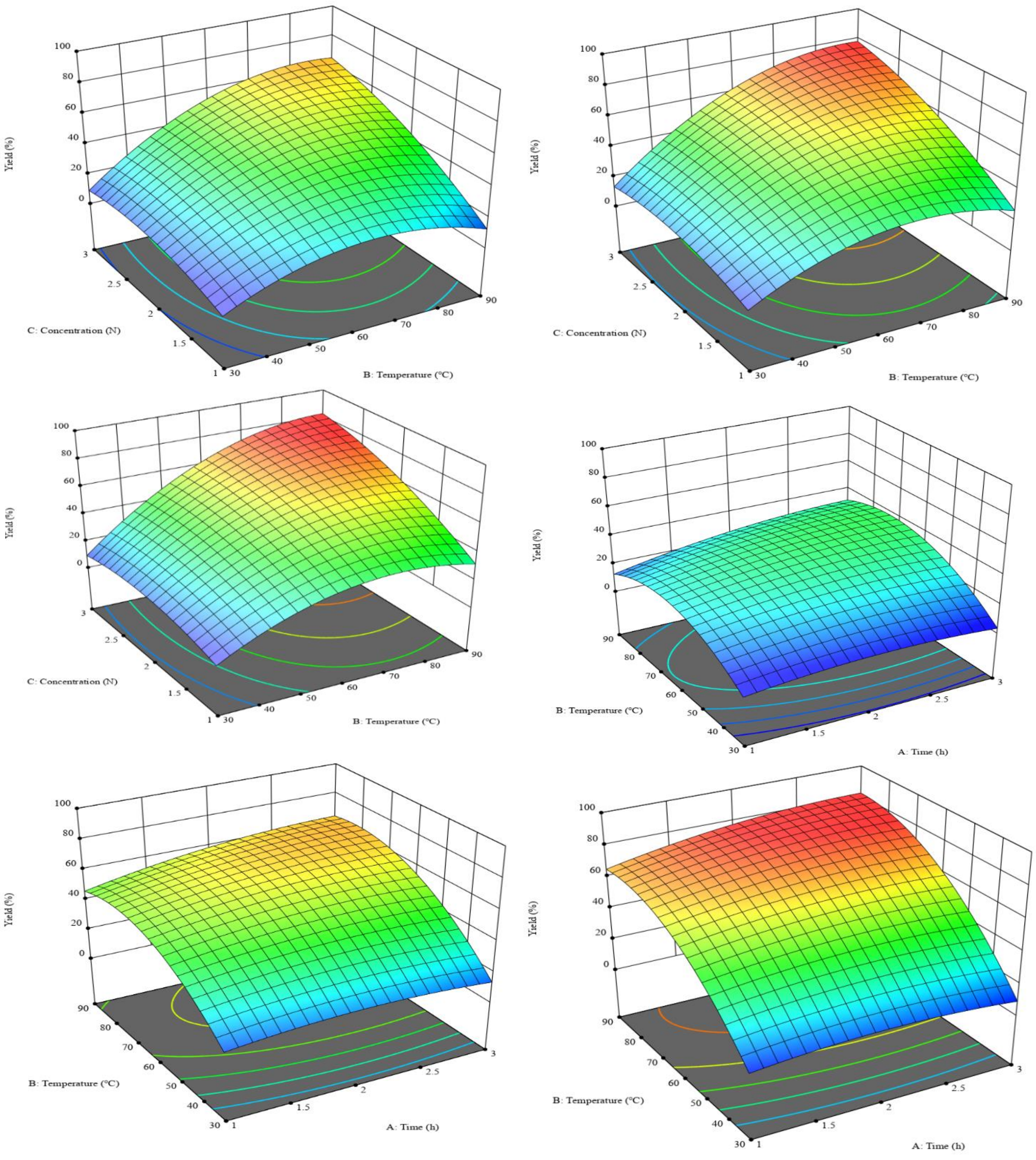


Fig. 3: Cube Model of Extracted Silica Yield

acidic nature which produces a higher yield when reacting with sodium hydroxide solution.



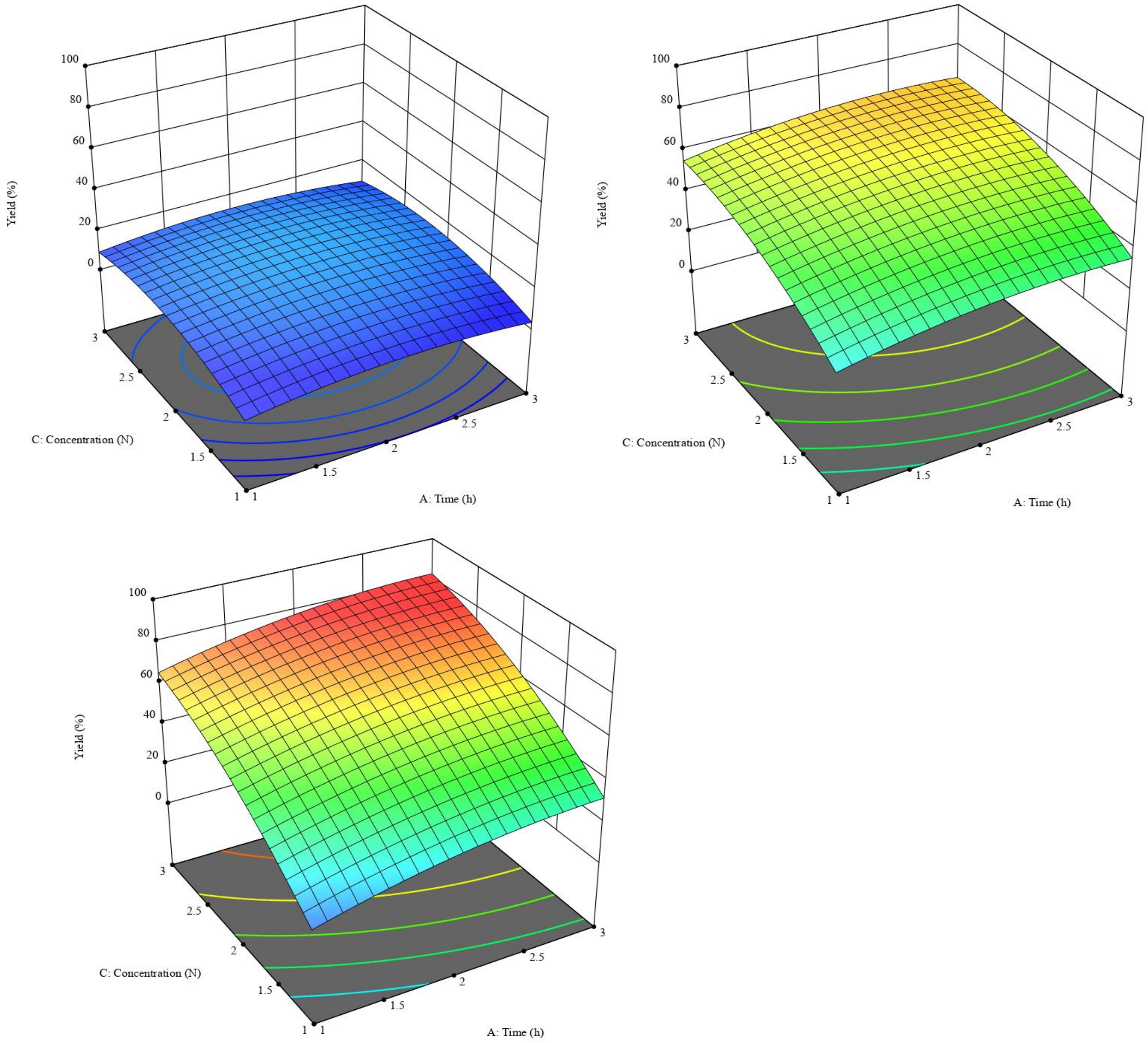


Figure 4: 3D Plot of Response Surface for Effect of Different Variables (Time, Temperature and Concentration of NaOH) on Silica Yield

3.3 Characteristics of Extracted Silica

3.3.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis of Extracted Silica

The extracted silica was confirmed using FTIR and was compared to commercial silica to indicate the similarity between their functional groups and confirm its purity as shown in Fig (5). The spectrum ranged from 4000 to 500 cm^{-1}, The absorption bands at 3457.86 and 1644.42 cm^{-1} were due to the hydroxyl group (H–O–H) stretching and bending modes of the adsorbed water, respectively. The vibrations peaks at 1089.79, 794.5, and 464.5 cm^{-1} indicate the presence of the asymmetric, symmetric, and bending modes of SiO_2 respectively [25][13]

3.3.2 Mineralogical Analysis (XRD)

The X-Ray Diffraction pattern of extracted silica was illustrated in Fig (6). A typical amorphous silica structure was observed and the absence of any ordered crystalline structure indicated a relatively high disordered structure of silica. The high sharp peak at 2θ = 32° could represent impurities, mainly Na, as confirmed by the chemical analysis in the next section. These results were similar to the silica pattern produced by Setyawan et al.[13], Biswas et al. [26], Music et al. [27]and Ying et al.[28].

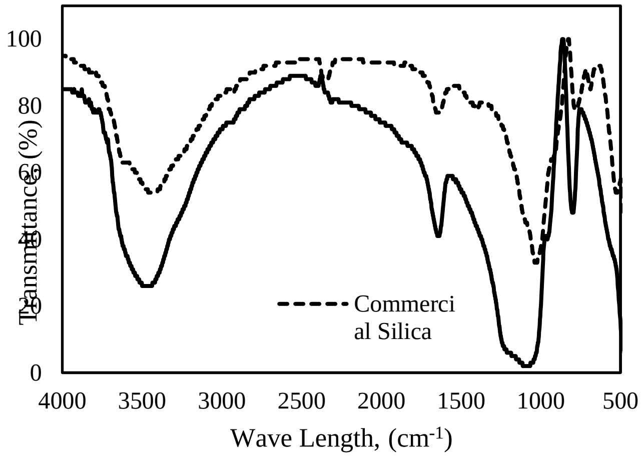


Fig. 5: FTIR of Extracted and Commercial Silica

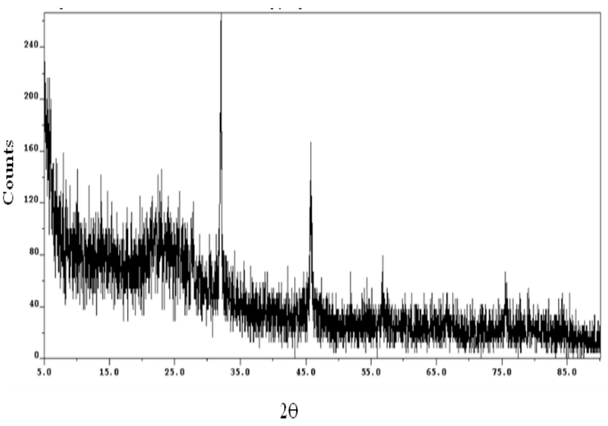


Fig. 6: XRD Pattern of Extracted Silica

3.3.3 Chemical Analysis of Silica (XRF)

There are different constituents present in the extracted silica. The chemical analysis shows the purity of extracted silica as seen in Table (5) which is compared with commercial silica. The results indicated that the purity of extracted silica is nearly the same as commercial silica, while the composition of sodium oxide in extracted silica is more than that found in the commercial. To increase the purity of silica, several steps of washing the precipitated silica to remove any impurities.

Table 5: Chemical Analysis of Extracted and Commercial Silica

Main Constituents	Extracted Silica (wt%)	Commercial Silica (wt%) [29]
SiO_2	94.844	95.37
TiO_2	0.0106	–
Al_2O_3	0.5103	0.63
$Fe_2O_3^{tot}$	0.5103	0.06
MgO	0.0318	0.06
CaO	0.0956	0.02
Na_2O	3.465	2.47
K_2O	0.1594	0.01
P_2O_5	0.0318	–
SO_3	0.0212	–
Cl	0.2551	0.07
MnO	0.0425	–
PbO	0.0127	–
ZnO	0.0042	–
ZrO_2	–	0.04

3.3.4 SEM Analysis

SEM micrograph of extracted silica showed agglomeration appearance of silica shown in Fig (7). The results indicated that silica has a non-uniform particle size and was widely distributed. The results are in agreement with silica produced by Imoisili et al. [30] and Sompech et al. [31].

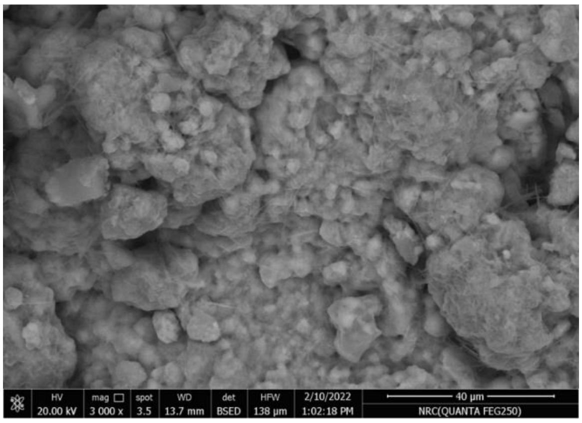


Fig. 7: SEM of Extracted Silica

4. Conclusion

High purity amorphous silica was produced from rice straw ash using an alkaline extraction method. Optimization of the main parameters was studied using response surface methodology which is temperature, time, and concentration of NaOH. The results indicated that extraction of silica is more sensitive to temperature change than time and increasing the concentration of sodium hydroxide increases the silica yield. A maximum silica yield of 83% is obtained at optimum conditions of 90℃, 3 N and extraction time of 3h. The results showed that the model is valid with a high value of coefficient R^2 (0.9727) and Adj-R^2 (0.9376) indicating that the model is related to the obtained experimental results. The extracted silica was characterized using XRF, XRD, FTIR and SEM.

Conflict of Interest

Regarding the publishing of this work, the authors affirm that there are no conflicts of interest.

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