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Bamboo leaves as an alternative source for silica in ceramics using Box Benhken design

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ABSTRACT

This study investigated the optimization of bamboo leaves as a source of silica for use in ceramics. Box Benhken design was used to study the interactions of three (3) variables namely: Temperature 400-700°C, Time: 2-7 h and Amount of bamboo leaves (BL) :2-6g. Optimal process level obtained was validated by confirmatory experiment. The silica obtained was characterized using X-Ray Diffractometer (XRD) and Scanning Electron Microscope (SEM). The result of the Box Benhken design generated 17 experimental runs. The optimal level observed were: Temperature: 600°C, Time: 4.50h; and BL: 5g. The result via the lowest Loss of Ignition and silica from the validated experiment were analysed using XRD and SEM. The result of the XRD showed a highly reactive amorphous nano silica was observed from the optimal process levels with high Specific surface area (915.33 m^2/g BET). Furthermore, result gotten from SEM showed agglomerated colloidal spheres of nano silica. It was observed that optimizing bamboo leaves is a viable process for converting a waste into a highly reactive silica for manufacture of ceramics.

Introduction

Bamboo leaves is a potential agro waste. It is a source of organic silica (bio-silica). The bamboo leaves ash has large silica content of about 75.90- 82.86% as reported by [1].

Some published studies/research on the production of silica from other agro-based waste include: rice husk [2]; Maize husk [3]; Corn cob, baggase and bamboo culm [4]. Furthermore, limited studies had been on the use of agricultural wastes like bamboo stem and bamboo leaf for silica production/extraction as a supplementary pozzolanic material to improve the various properties of concrete and ceramics, which can also reduce construction costs [5]). According to [6] rice husk ash was used for silica production in ceramics. [7,8], also reported that rice husk ash was used as a source for silica in glass ceramics production.

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Table 1
Process variables and levels

Variables	Units	Symbols	Coded levels		
			-1	0	+1
Temp	°C	A	400	550	700
Time	h	B	2	4.50	7
Amount BL	g	C	4	5	6

Table 2
Design Matrix for the Production of Ash from BL

Std	Block	Run	Factor 1 (°C)A	Factor 2(h)B	Factor 3 (g)C
7	Block 1	1	700	4.50	6
8	Block 1	2	400	4.50	4
3	Block 1	3	700	7.00	5
16	Block 1	4	550	7.00	4
2	Block 1	5	400	4.50	6
1	Block 1	6	700	2.00	5
14	Block 1	7	550	4.50	5
4	Block 1	8	700	4.50	4
10	Block 1	9	550	4.50	5
17	Block 1	10	550	2.00	4
13	Block 1	11	550	7.00	6
5	Block 1	12	550	2.00	6
15	Block 1	13	550	4.50	5
6	Block 1	14	400	7.00	5
11	Block 1	15	550	4.50	5
9	Block 1	16	400	2.00	5
12	Block 1	7	550	4.50	5

Table 3
Proximate Analysis

S/N	Proximate Analysis	RUN 1 (%)	RUN 2 (%)	RUN 3 (%)
1	Nitrogen	2.44	2.49	2.48
2	Crude protein	15.23	15.54	15.49
3	Ash	11.34	11.39	11.37
4	Crude fibre	32.37	33.23	33.34
5	Crude fat	1.11	1.13	1.12
6	Moisture	10.36	9.95	9.91
7	Volatile matter	55.6	55.59	55.60

Materials and Methods

Experimental Design

The Box Behnken Design was used to determine the effect of three variables: temperature, time and amount of BL on the amount of ash yield was ca. The advantage of Box Behnken design is that it reduce the number of experiments, which enables a less laborious and time-consuming optimization process for the research.

The Design method has been used to investigate the interactions between the variables and 17 runs were generated. The BL was subjected to calcination using the experimental runs. The effect of the three variables: Amount of Bamboo leaves, Temperature and Time were analysed. The ranges of the variables used were: Bamboo leaves: 4- 6 g, Temp 400-700 °C, and Time of calcination 2-7 h respectively. Table 10 shows the process variables and levels with corresponding values. Table 2 shows the matrix for the interactions of the three variables.

The approach of the study in Box Behnken Design involved the following steps:

i Design of an experiment using the Box Behnken Design.
ii Determination of the Process Variables. These are Bamboo leaves, Temp and Time of calcination.
iii Development of a Mathematical model
iv Prediction of the optimal process variables
v Experimental validation

Preparation of Bamboo Leaves (BL)

Bamboo leaves was collected from Teaching and Research Farm, Landmark Universtiy,Omu-Aran;Kswara State,Nigeria and then treated with1M HCl for 2 h to remove impurities.

Proximate composition

Proximate composition of the sample were determined according to [9]. However, all measurements were made in triplicate. Proximate analysis of the bamboo leaves was done to determine the amount of ash, crude protein, Nitrogen, Volatile matter, moisture content and crude fat respectively.

Characterization of silica

The ash with the lowest LOI from the results of Box Behnken design and the predicted optimal levels were characterised using Xray Diffractometer to determine its crystallinity. Specific area was characterized using BET Method and the morphology characterized using Scanning Electron Microscope (SEM) with EDX to determine its elemental composition.

Extraction of Silica from BL

The interactions from the 17 experimental runs generated were calcinated to obtain ash. Furthermore; 60 ml NaOH of 2M was added to 10 g of BLA and then boiled for 1h. Thereafter, solution was then filtered and washed. The product was aged for 20 h. Thereafter; the gel obtained was softly broken by adding 100 ml distilled water and centrifuged to make slurry.

It was later filtered and washed then discarded. The supernatant was dried at 60°C to obtain silica.

This methodology was adapted from [10].

Statistical Analysis

The experimental data were analyzed using Design Expert 6.0.8. software to obtain the Analysis of Variance (ANOVA). Both linear quadratic and linear interactive effects of the process variables on BLA and their respective significance evaluated by the ANOVA.

The p-value was used as a yardstick to measure the significance of the coefficients, and the values of $p \leq 0.05$ meant that the coefficient was significant. The experimental data were fitted to the second-order polynomial regression model and the suitability of the model evaluated by the coefficient of determination (R^2) was compared to the modified R^2value.

Results and Discussion

The proximate analysis of BL

The quantitative analysis of the constituents of BL is shown in Table 3.0.

The amount of silica observed in this research was higher than the amount reported by [11] with Sugarcane baggase: 1.73%, Cassava: 4.93; Maize stalk: 4.80**.**This made bamboo a better source for silica extraction.

Evaluation of Regression Model for BLA

The variables and the percentage of Ash yield was calculated using Box Behnken modelling technique. The yield of the BLA ash is as shown in Table 4. A polynomial quadratic regression equation of the form was fitted between the response (percent yield of ash) and the process variables. A polynomial quadratic regression equation of the form was fitted between the response (percent yield of ash). The regression equation obtained was:

$$\text{ash} = +83.88 + 1.60A + 2.28B + 1.10C + 0.35A^2 - 0.85B^2 + 0.13C^2 - 1.03AB - 0.84AC - 0.85BC$$

Where A: Temperature; B: Time; C- Amount of Bamboo leaves

Furthermore, low Loss of Ignition (LOI) is used to measure the homogeneity and pozzolanic activity of a material as reported by [12,13]. However, this confirmed that experiment run 10 had the lowest LOI of 3.19. This also confirmed the results of [14–18]; that maximum LOI of 6 wt % for Class C and F coal ash use in concrete and ceramics. The best process variables interactions from Table 4 is experimental run 10 with the lowest LOI which was confirmed by report of [16,19].

It was reported by [20] that synthesized mesoporous bioactive glass (BMG) made from RHA using solgel method was used in drug and bone regeneration. Moreover; [21,22] also reported production of nanaosilica from agricultural waste for ceramics, refractory glass, white ware, silica aerogel.

Table 4
Box Behnken Design for the Production of Ash from BL

Std	Run	Factor 1 (°C) A	Factor 2(h)B	Factor 3 Initial weight (g)C	Final Weight (g)	LOI	Ash (%)
7	1	700	4.50	6	0.78	5.22	86.970
8	2	400	4.50	4	0.80	3.20	80.070
3	3	600	7.00	5	0.69	4.31	86.220
16	4	550	7.00	4	0.60	3.40	84.900
2	5	400	4.50	6	0.94	5.06	84.400
1	6	700	2.00	5	0.87	4.05	82.660
14	7	550	4.50	5	0.90	4.10	82.000
4	8	700	4.50	4	0.56	3.44	86.000
10	9	550	4.50	5	0.74	4.27	85.300
17	10	550	2.00	4	0.81	3.19	79.680
13	11	550	7.00	6	0.90	5.10	84.930
5	12	550	2.00	6	1.01	4.99	83.120
15	13	550	4.50	5	0.82	4.18	83.600
6	14	400	7.00	5	0.69	4.31	86.140
11	15	550	4.50	5	0.68	4.32	84.460
9	16	400	2.00	5	1.08	3.92	78.480
12	7	600	4.50	5	0.78	84.02	84.020

LOI- Means Loss on Ignition

Table 5
ANOVA

Source Model	Sum of Squares 85.03	DF 9	Mean Square 9.45	F-Value 6.10	Prob > F 0.0132 significant
A	20.35	1	20.35	12.14	0.0085
B	41.63	1	41.63	26.87	0.0013
C	9.61	1	9.61	6.21	0.0415
A^2	0.52	1	0.52	0.33	0.5812
B^2	3.05	1	3.05	1.97	0.2031
C^2	0.08	1	0.08	0.05	0.8324
AB	4.20	1	4.20	2.71	0.1436
AC	2.82	1	2.82	1.82	0.2191
BC	2.91	1	2.91	1.88	0.2131
Residual		10.85	7	1.55	
Lack of Fit	4.86	3	1.62	1.08	0.4517 not significant
Pure error	5.99	4	1.50		
R-squared	0.8913				
Adj R-squared	0.7844				
Pred R-squared	0.8755				

Table 6
Optimal process Levels

Std	Run	Factor 1 (°C)A	Factor 2(h)B	Factor 3 (g)C	Amount of Ash	*.LOI
7	1	600	4.50	5	0.959	0.81

Result of Statistical Analysis

The interactive significance of the process variables was evaluated using ANOVA as shown in Table 5. The model adequacy was confirmed by the coefficient of determination (R^2), 0.8910 (89.1%) which is accurate. The value of the coefficient of determination reflects the percentage of the experimental results the model is able to explain it. The R-squared value: 0.8910 (89.10%); which is the ANOVA, shows that the model F-value of 189.06 observed suggests that the model is appropriate. Values of "Prob> F" less than 0.0500 model terms indicated are significant. In this case, the model terms A, B, C are important. The high value of the coefficient of determination (R^2=0.8913) further made a significant contribution to the significance and suitability of the established model. The high value of the coefficient of determination (R^2=0.8913) with the modified R^2 (0.7844) is in agreement and this has further contributed to the validity and adequacy of the developed model.

Surface Plots

Figure 1 showed the plot of Predicted Vs Actual while the Plot of Interraction between Temperature and Time is as shown in Figure 2. The result in Figure 3 showed that the selected quadratic model was adequate to predict the response variables for the experimental data. It was also observed that the result obtained showed that quadratic model was normally

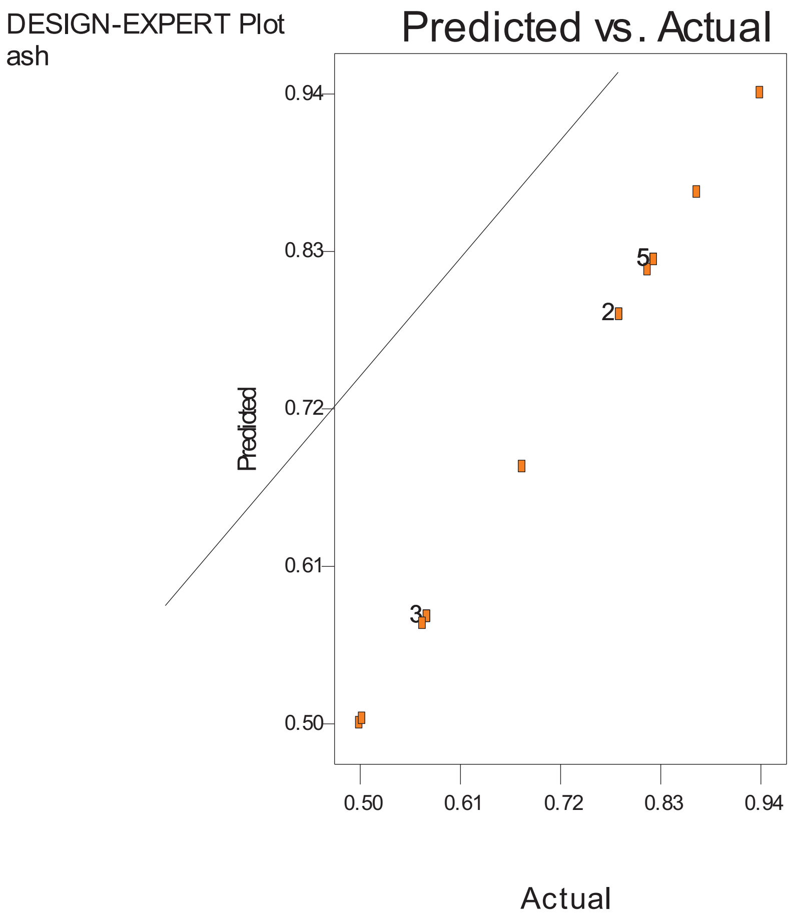


Fig. 1. Plot of Predicted Vs. Actual

Table 7
Data from Specific Surface Area of Brunauer-Emmett-Teller (BET)

SAMPLE: BOA Temperature (T/°C)	Specific Surface Area (m^2/g)	SAMPLE: Ao Temperature (T/°C)	Specific Surface Area (m^2/g)
100	670.50	100	915.33
200	677.30	200	900.21
300	680.80	300	927.30
400	670.50	400	910.10
500	670.70	500	912.20
600	667.95	600	816.42

distributed. Figure 4 showed the response of the interactions. The optimum prediction for Design Expert software is Temp: 600 °C, Time: 4.50 h, and BL: 5 g.

Experimental Model Validation

However, the experiment was carried out according to the optimum conditions in order to validate the predicted optimum values.of Analysis

Results of the BET Surfae Area

The BET,Pore volume, pore size distribution and Pore diameter are as shown in Tables 7-10 respectively. The surface area obtainned was more than the amount reported by [20–22], but confirmed result by [23]; respectively. Pore volume confirmed what was reported by [21,24] but higher than 5.60 - 9.60nm as obtianed by [25]**.** BET, Pore length, pore size distribution and pore diameter are shown in Tables 7-10, respectively. The silica was obtained from the ash after the leaves had been subjected to calciantion.The surface area obtained was more than the amount recorded by [20–22], but the result

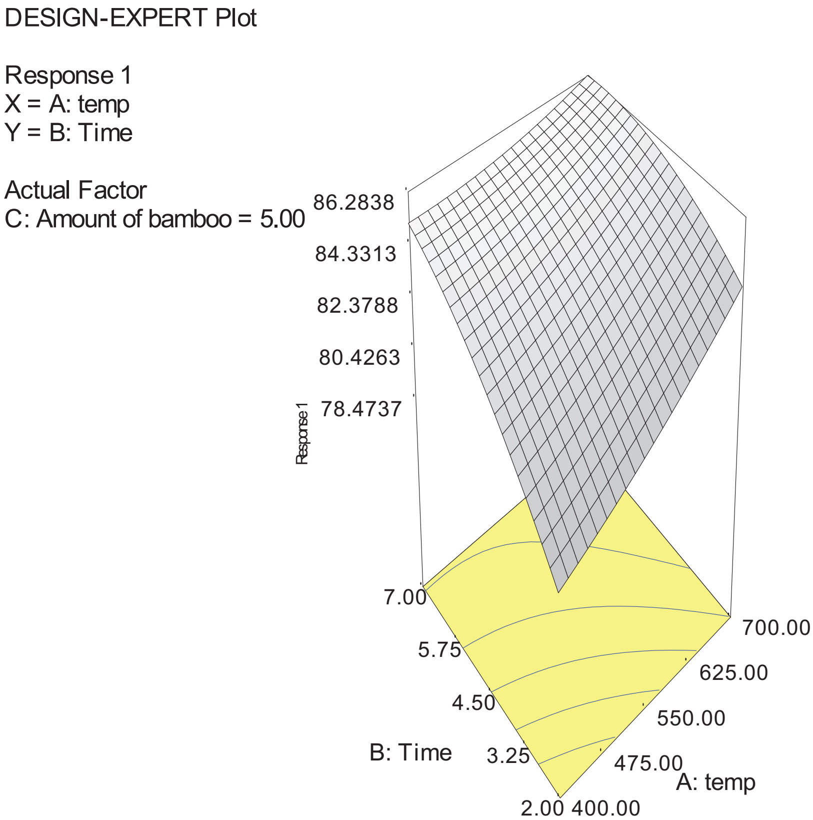


Fig 2. Plot of interraction between Temp and Time

Table 8
Pore Volume

SAMPLE: Ao Relative Pressure (P/P_0 X 10^{-2})	Volume $(cc/g)^3$	SAMPLE: BOA Relative Pressure (P/P_0 X 10^{-2})	Volume $(cc/g)^3$
3.20	12.30	3.12	9.35
4.52	14.80	4.50	15.2
6.00	16.90	6.50	22.6
6.90	25.30	6.90	30.5
7.50	36.50	7.50	45.2
8.20	55.90	7.90	58.4

Table 9
Pore Size Distribution

SAMPLE: Ao Seive Mesh (micron)	Pore Size Distribution	SAMPLE: BOA Seive Mesh (micron)	Pore Size Distribution
<20um	13.65	<20um	25.80
20-40um	5.45	20-40um	20.22
40-100um	51.00	40-100um	20.22
100-350um	28.40	100-350um	32.95
350um	1.50	350um	0.78
Total	100	Total	99.97

was verified by[24]; Pore volume confirmed what was reported by [24,21] but higher than 5.60-9.60 nm as reported by [25]. Figures 5-7 showed the graphs of BET Surface Area, Pore Volume AO(Silica obtained through Optimal Predicted Process) and BOA(Silica with Lowest LOI from the results of experimental Design BOA). The average pore diameter results confirmed what was reported by [26] that the silica was highly reactive and can be used as a good pozzolanic.This report showed that

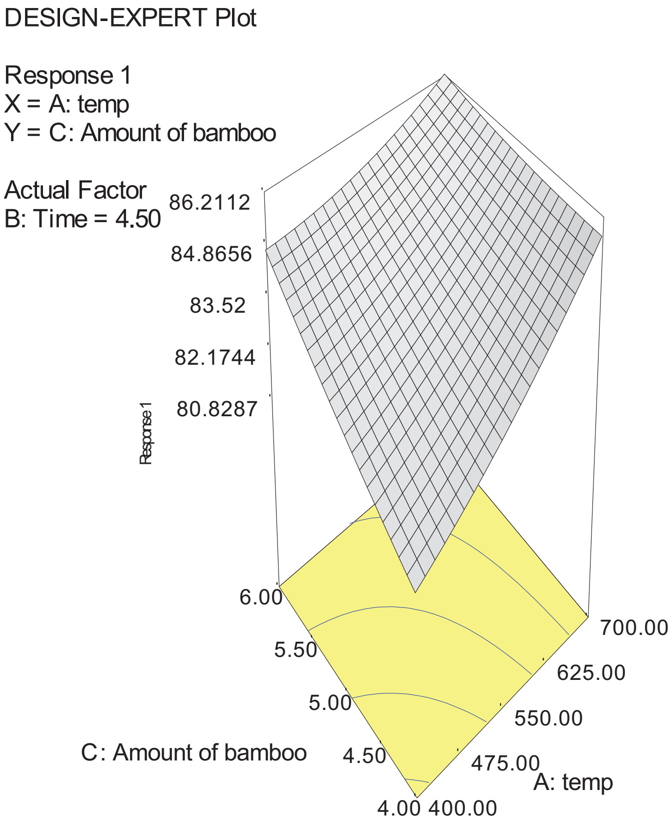


Fig 3. .Plot of Interraction between Temp and BL

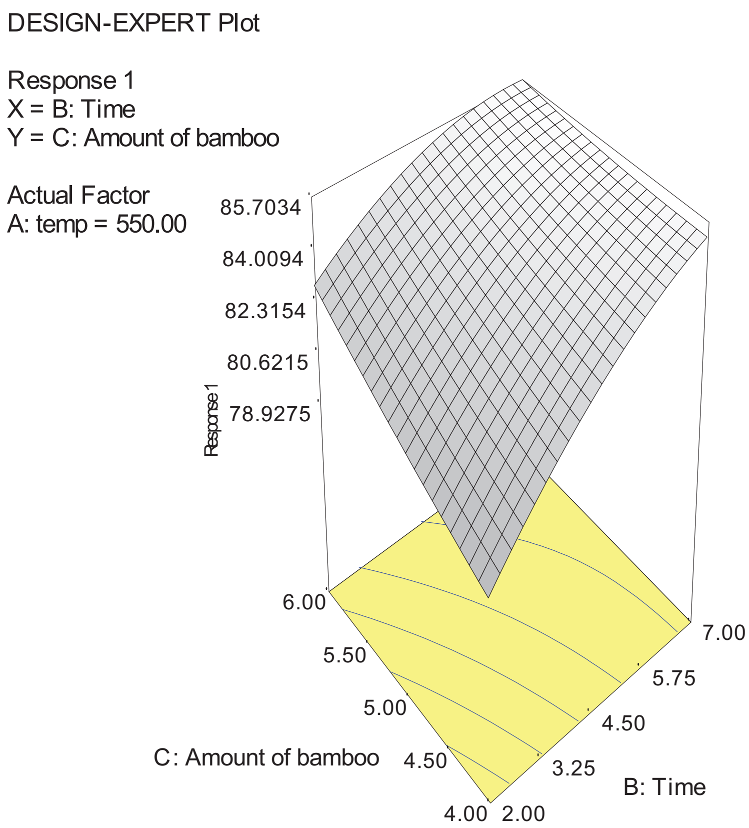
Table 10
Pore Diameter (nm)

SAMPLE: Ao Relative Pressure (P/P_0 X 10)	Diameter (nm)	SAMPLE: BOA Relative Pressure (P/P_0 X 10^{-2})	Diameter (nm)
3.00	5.50	3.00	7.12
4.00	5.90	4.00	7.18
6.00	6.10	6.00	7.40
6.00	6.60	6.00	7.60
7.00	7.50	7.00	7.80
8.00	7.80	8.00	7.80

OA has the highest surface area corresponding to [27–29] which indicated that amorphous structure, high specific surface area, high pore volume and high reactivity were very significant for the determination of highly reactive silica.

XRD and SEM Results

The structure and phase of silica as determined by XRD is shown in Figures 8–9. It shows that the Silica obtianed from OA and BO are reactive amorphous silica. Silica fume or micro-silica is the most commonly used mineral admixture in high strength concrete [31] which confirmed the silica from OA (Fig 10). The silica structure and process as calculated by XRD are shown in Figures 8–9. It has been shown that the silica extracted from OA and BO are reactive amorphous silica. Silica smoke or micro silica is the most commonly used mineral mixture in high-strength concrete [30] confirmed by OA silica. Figure 10 was observed to be the best silica in the SEM result due to its spherical (colloid) morphology. It was noted that not all amorphous silica was a highly active source of silica, but that the reactivity depends solely on the surface structure and morphology as analysed with the SEM. Morphology was shown in Figures 10–11, while elemental composition via EDX is shown in Figures 12–13, respectively.



ig 4. Plot of Time and BL

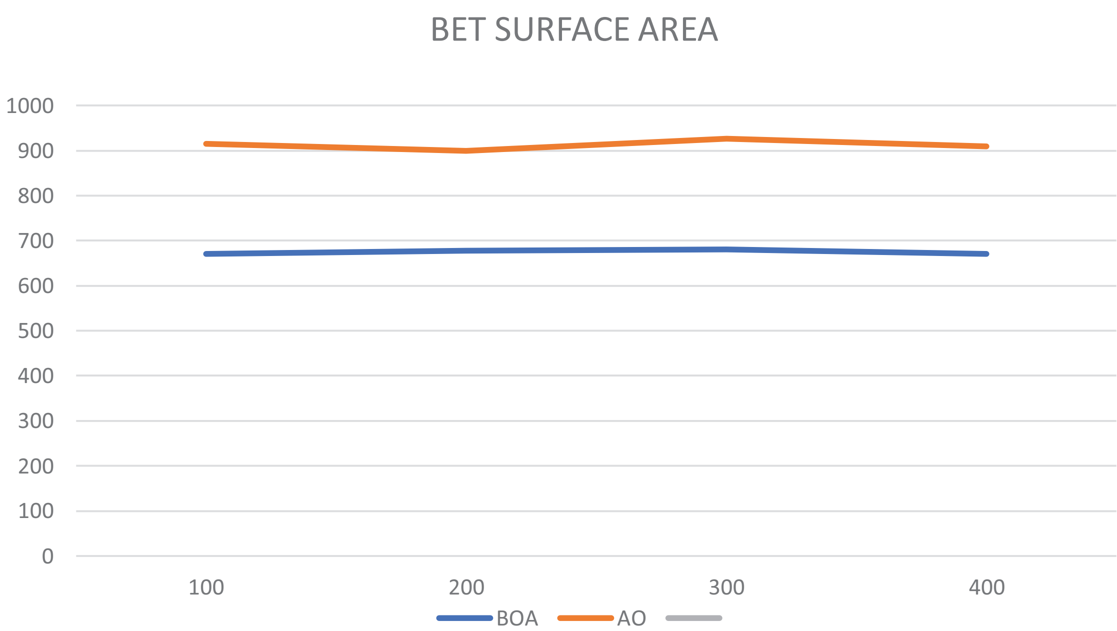


Fig 5. BET of BOA and AO

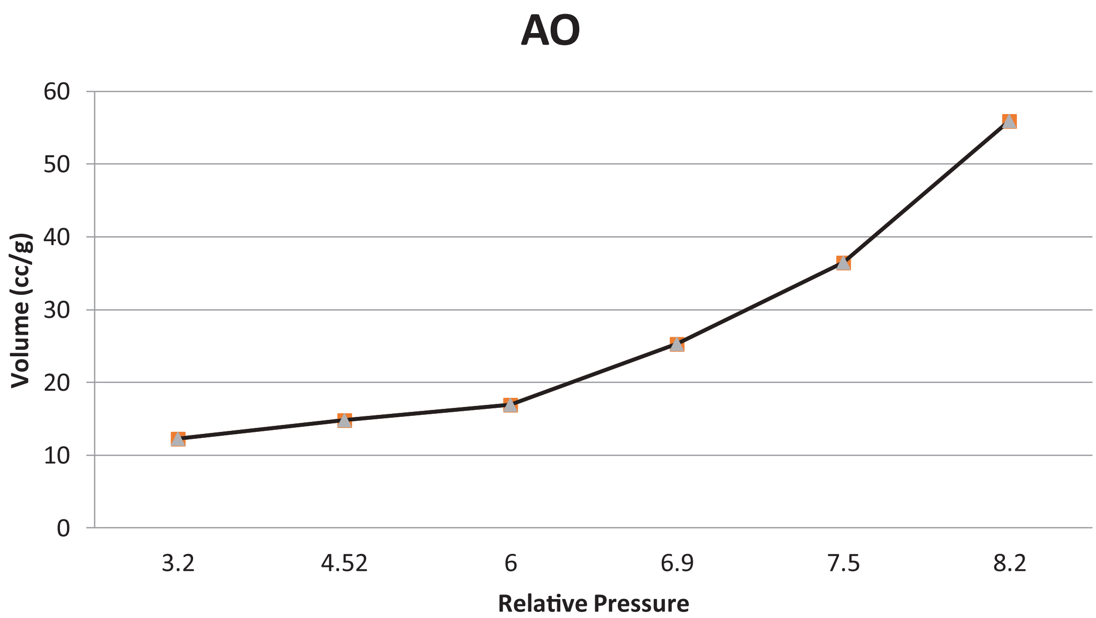


Fig 6. Plot of Pore volume AO

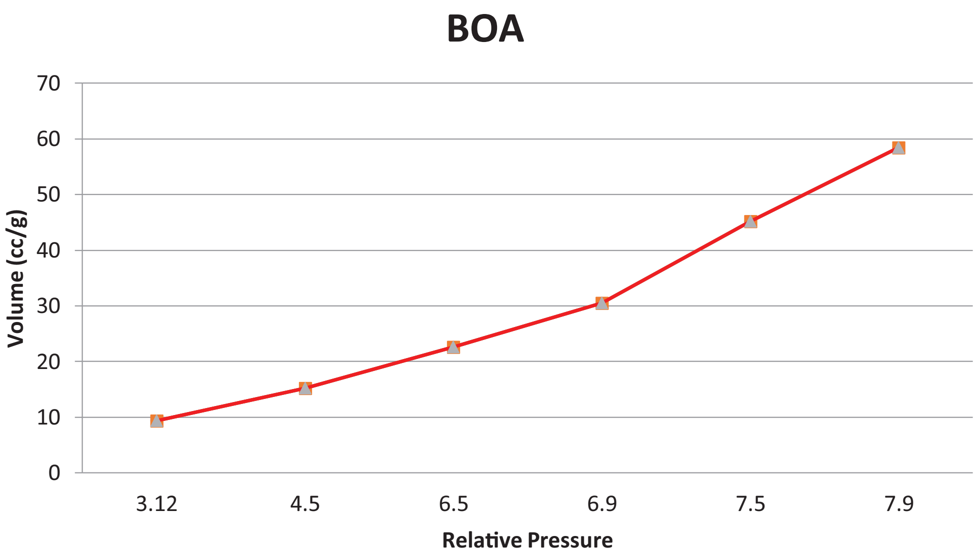


Fig 7. Pore Volume of BOA

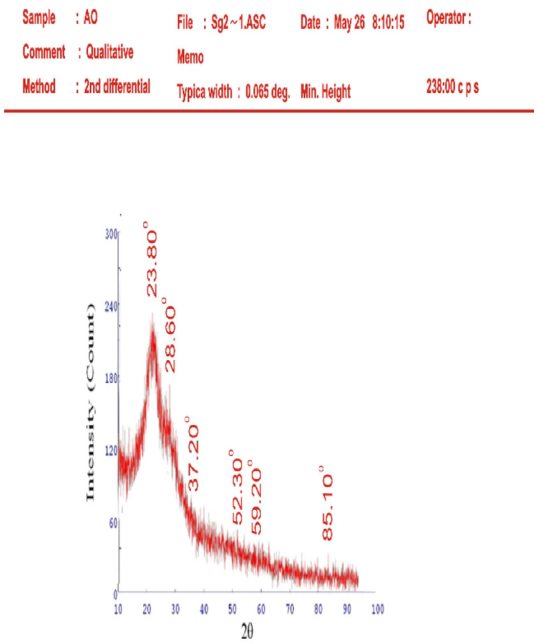


Fig 8. :XRD of AO

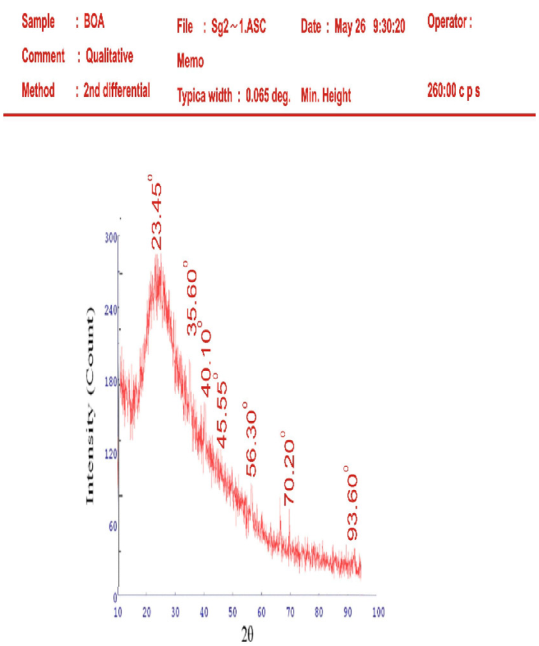


Fig 9. :XRD of BOA

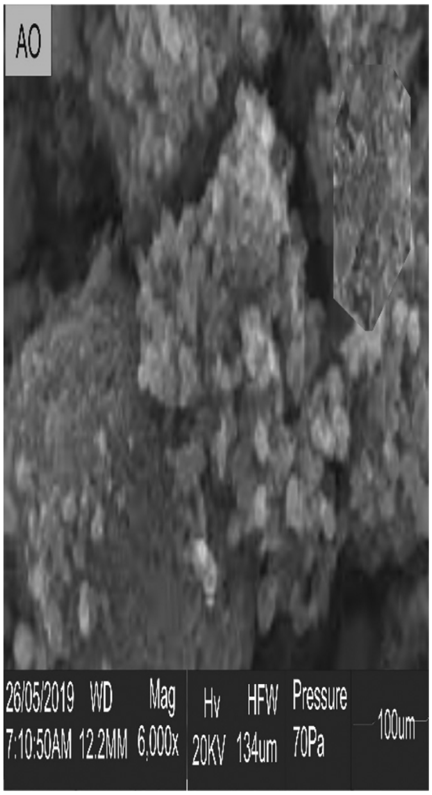


Fig 10. SEM of AO

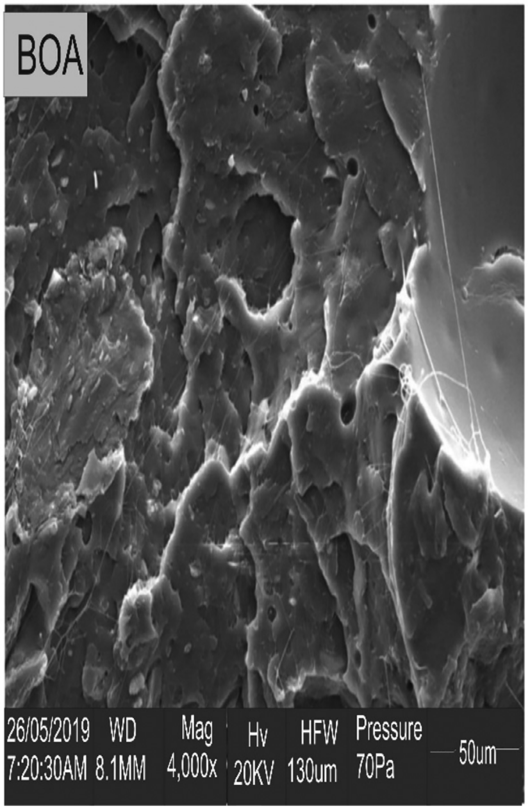


Fig 11. SEM of BOA

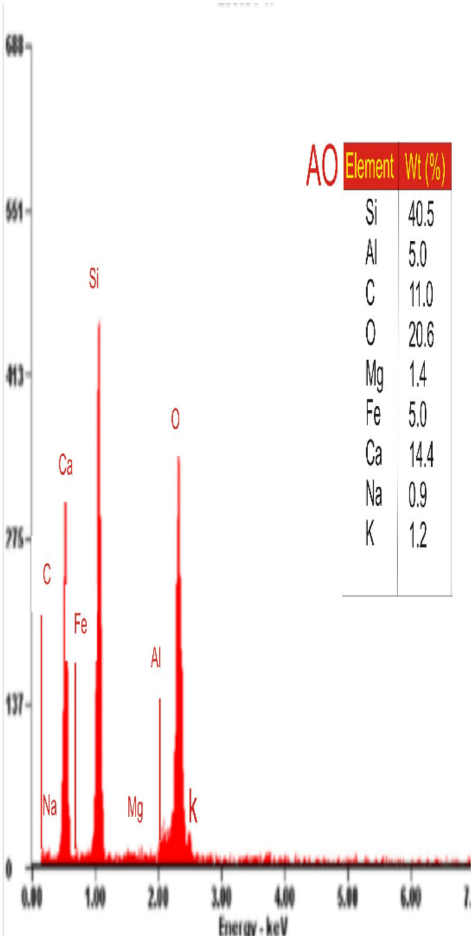


Fig. 12. EDX of OA

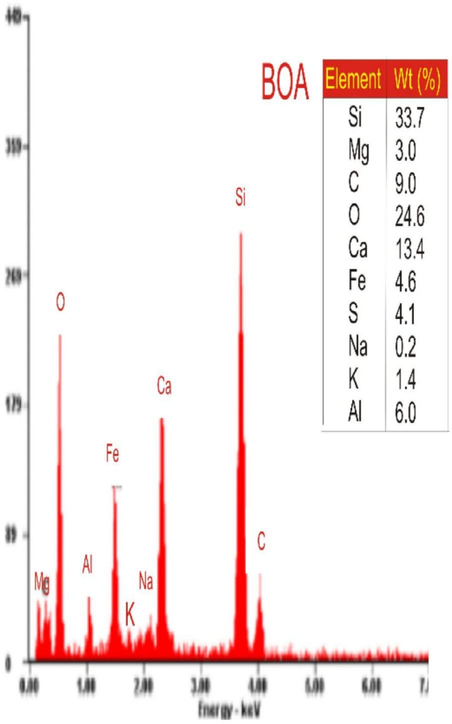


Fig. 13. EDX of BOA

Conclusion

It can be inferred that the optimization of bamboo leaves for silica production was viable. The optimum process levels predicted were temp:600°C, time:4.50 h and amount of bamboo leaves:5 g, respectively, which gave a highly reactive nanoscale amorphous silica with a high specific surface area (915.33 m2/g BET), which can be used in ceramics.

Declaration of Competing Interest

None.

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