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Response surface methodology for silicon production from bamboo leaves

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Abstract. This study investigated the production of silicon from bamboo leaves to solve the challenge of silicon in solar market using Optimization approach. The effect of three independent variables of: temperature, time and amount of bamboo leaves were studied using Box Behnken design. The best process level observed from the Box Benhken Design and optimal predicted process were used to produce silica. It was then subjected to X-Ray Diffractometer to determine the most reactive silica. The most reactive silica observed was used to produce silicon. Silicon obtained was subjected to X-Ray Diffraction and Scanning Electron microscope. It was concluded that nanosilicon was produced which can be used as a solar cell component to solve the challenge presently in the solar market.
Keywords: Silica, Agricultural Residue, Central Composite Design, Optimization, Silica
Abbreviations
BL: Bamboo leaves
SEM : Scanning Electron Microscope
XRD: X-ray Diffractometer
LOI: Loss on Ignition

1. Introduction
Bamboo is a grass. In the true grass family Poaceae, bamboos are a group of woody perennial evergreen plants. It was the largest in the grass family with around 1000 species. They are found in diverse climates, from cold mountains to hot tropical regions [1]. Bamboos include some of the fastest-growing plants in the world due to a unique rhizome-dependent system [2]. Research had been on production of silica from agricultural wastes[3] such as: rice husk [4-5; Maize husk[6] ; Corn cob [7], baggase [8]; Wheat straw [9]. Furthermore [10], reported that silica content from bamboo leaf ash is the second largest after rice husk. It was reported in the literature that increasing demand for cheap, safe and nonconventional silica as raw materials in industries, and also the importance of having a pollution-free environment has led to worldwide attention on the research and applications of silica [11]. However; few studies had been on silica and silicon production from bamboo, but no work had been reported on via the use of bamboo leaves for silicon production using optimization approach. This study assessed silicon production from the bamboo leaves which is an agricultural residue.

2. Materials and Methods
2.1. Preparation of Bamboo Leaves (BL)
Bamboo leaves were treated for 2 hours with 1 M HCl to eliminate the impurities. The leaves were harvested from the Landmark University teaching and research farm.

2.2 Proximate composition
The proximate analysis was done according to AOAC [12]. Proximate analysis on the extract was conducted to determine the volume of nitrogen, ash, crude fat, volatile matter, crude protein and moisture content.

2.3 Experimental Design for silica production

The effect of three different variables (Temperature, Time, and amount of BL) on the amount of ash yield from Box Behnken design was determined. For this study, the design model used was Quadratic, and 17 runs were developed. The variables were: Temp 400-700 ·C, Time of calcination 2-7 h and Bamboo leaves: 4- 6 g, respectively. The variables and levels is shown in Table 1 while variables interaction is in Table 2.

The approach used Box Behnken design in this study:

i. Experimental design using Box Behnken Design approach to investigate interactions of the variable;

ii . The variables were defined. These are the Bamboo leaves, time and Temperature

iii. Obtaining the regression model expressing the relationship between the variables

iv. Optimal process variables' prediction

v. Validating the optimal variables.

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 of BL	g	C	4	5	6

Table 2: Design Matrix for the Production of Ash from BL

Std	Block	Run	Variable 1(·C) A	Variable 2(h) B	Variable 3 (g) C
7	Block1	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	2.00	4
10	Block 1	9	550	4.50	5
17	Block1	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	17	550	4.50	5

2.4. Preparation of Silica from BL

The interactions were conducted for obtaining ash from the 17 experimental runs produced. 60 ml of 2 M NaOH added to 10 g Ash Bamboo Leaves, boiled for 1h, and then filtered. Thereafter aged for 20 h, centrifuged and 100 ml of distilled water added. It was later filtered. It was then dried at 60·C to obtain silica as reported by [13].

2.5. Characterization of silica

X-Ray Diffractometer was used to determine the crystallinity of the ash with the lowest LOI observed from Box Behnken design and the ash from the validated experiment. Scanning Electron Microscope

(SEM) was used to determine the surface morphology while EDX to determine its elemental composition.

2.6. Silicon Production from Silica Extracted

Silica produced from the best silica via the predicted optimal level was compared with the silica of lowest LOI. The best from XRD and SEM was used for silicon production. It was subjected to magnesiothermic reduction process. The silica obtained was mixed with MgCl in ratio of: 1:4; the product was subjected to calcination at 650 ◦C for 2 h.

2.7. Surface Characterization of Silicon Produced

The silicon obtained were subjected to XRD to determine its crystallinity and phase with nickel-filtered Cu Kα radiation. Furthermore; the surface characterization was done on the silicon produced to determine the surface morphology via SEM while EXD was used to determine its chemical composition.

3. Results and Discussion

3.1. Result of Proximate Analysis

The result of the Proximate Analysis with presence of Ash (11.34%) as shown in Table 3 confirmed report by [6].

Table 3: Proximate Analysis of the Bamboo Leaves

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	55.6	

3.2. Discussion of Result on Box Behnken Design for the Production of BLA

It was reported that Loss on Ignition (LOI) test is used method for estimating the amount of unburnt carbon left in the ash [14]. The best process best experimental run from the variable's interaction was experimental run 10 in Table 4. The yield of the BL ash is as shown in Table 4.

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

Std	Block	Run	Variable 1(°C)	Variable 2(h)B	Variable 3	Intial weght(g) C	LOI	Ash(%)
7	Block1	1	700	4.50	6	0.782	5.218	86.97
8	Block 1	2	400	4.50	4	0.797	3.203	80.07
3	Block 1	3	700	7.00	5	0.689	4.311	86.22
16	Block 1	4	550	7.00	4	0.603	3.397	84.90
2	Block 1	5	400	4.50	6	0.936	5.064	84.40
1	Block 1	6	700	2.00	5	0.869	4.046	82.66
14	Block 1	7	550	4.50	5	0.900	4.100	82.00
4	Block 1	8	700	2.00	4	0.560	3.440	86.00
10	Block 1	9	550	4.50	5	0.735	4.265	85.30
17	Block1	10	550	2.00	4	0.813	3.187	79.68
13	Block 1	11	550	7.00	6	0.904	5.096	84.93
5	Block 1	12	550	2.00	6	1.013	4.987	83.12
15	Block 1	13	550	4.50	5	0.820	4.180	83.60
6	Block 1	14	400	7.00	5	0.693	4.307	86.14
11	Block 1	15	550	4.50	5	0.676	4.324	84.46
9	Block 1	16	400	2.00	5	1.076	3.924	78.48
12	Block 1	17	550	4.50	5	0.779	4.221	84.02

3.3. Evaluation of Regression Model for BLA

The ANOVA result showed in Table 5 that the Model F-value of 6.10; implied the model was significant.Values of "Prob>F" less than 0.0500 indicated model terms were also significant. The ANOVA result showed that A, B and C are significant models. The significant and adequacy of the established model were further collaborated by the high value of coefficient of determination (R^2=0.8913) with which is in close agreement with the adjusted R^2 (0.7844). This indicated that the model was suitable for use in the experiment.

Table 5: ANOVA

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	85.03	9	9.45	6.10	0.0132	significant
A	20.35	1	20.35	13.14	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	0.075	1	0.075	0.048	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 Sig
Pure Error	5.99	4	1.50			
R-Squared	0.8913					
Adj R-Squared	0.7844		Pred R-Squared	0.8755		

The model in terms of the coded values of the process a parameter is given by:
Final Equation in Terms of Coded Factors:

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
Optimal Process Levels of: 600 °C, Time :4.50 h and amount of bamboo leave:5 g was observed from the experimental design as shown in Table 6.

Table 6: Optimal process Levels

Std of ash	Run LOI	Variable 1(°C)		Variable 2(h)	Variable 3(g)		Amount
7	1	600	4.50	5	0.959	0.81	

3.4. Characteristics of silica

The results of the XRD are shown in Fig 1 and 2. However Fig 2 is a better reactive silica wince it has 2 = 23.4. Furthermore; The Surface morphology result from SEM is as shown in Fig 3 and Fig 4; confirmed the report of [15-16]. Fig 4, showed spherical and an agglomerated form of the silica obtained from the optimal levels. The pattern is amorphous nano scaled silica which confirmed result of [16-18]. This showed that Fig 4 has better surface morphology (colloidal).
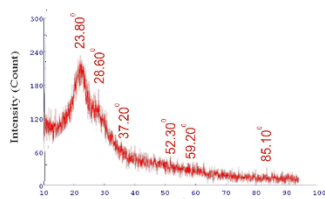
Fig 1:XRD of Ash with Lowest LOI
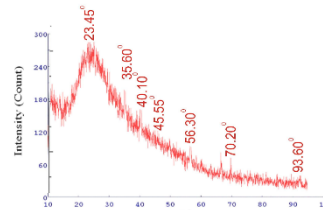
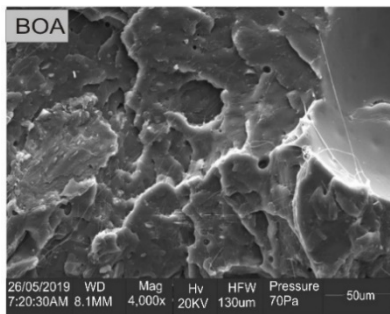Fig 2:XRD of Silica obtianed via optimal predicted levels
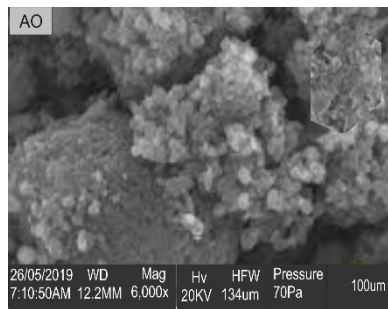

Fig 3: SEM of silica with lowest LOI

Fig 4: SEM of silica obtained at optimal levels

3.5. Characterisation of Silicon

The XRD Diffraction of the silicon produced is shown in Fig 5. The SEM of the silicon produced is shown in Fig 6 which showed nano silicon with spherical particle shape while the EDX is shown in Fig   7.

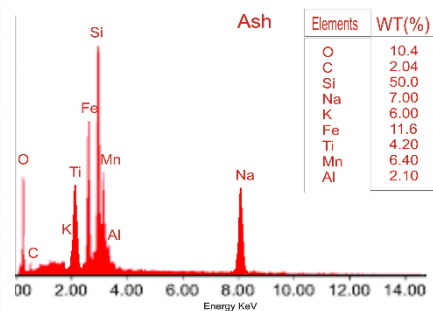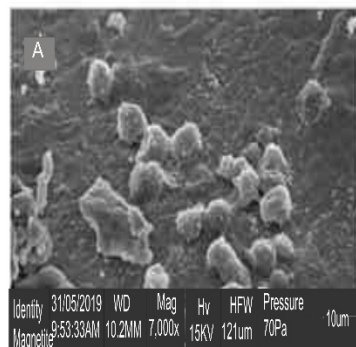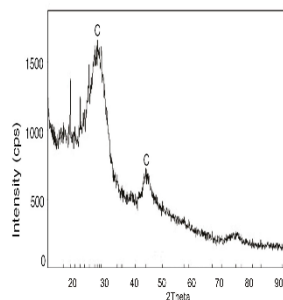| Ash Elements	WT(%)
O	10.4
C	2.04
Si	50.0
Na	7.00
K	6.00
Fe	11.6
Ti	4.20
Mn	6.40
Al	2.10

Fig 5: XRD of silicon produced Fig 6: SEM of Silicon produced   Fig 7: EDX of silicon produced

4. Conclusion

The Optimal Process Levels of: 600 °C, Time :4.50 h and amount of bamboo leave:5 g was observed from the experimental design. Amorphous silica was extracted from bamboo leaves through chemical extraction method. The silica extracted was used for the production of silicon. Optimization approach had been highly successful for the production of silicon from bamboo leaves. The XRD and SEM results obtained showed that the silicon was highly amorphous with spherical and agglomerated shape. It can be concluded that the nano silicon produced can be used in making solar cells.

Acknowledgement

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