

# Optimization of Pyrolysis and Selected Physicochemical Properties of Groundnut Shells, Coffee and Rice Husks for Biochar Production

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## Abstract

Biochar is a solid biofuel that can be obtained from pyrolysis of lignocellulosic feedstocks. In this study, pyrolysis of agricultural wastes (groundnut shells, coffee and rice husks) and their physicochemical characterization was done. Parameters that influenced biochar yields were optimized using response surface methodology and Box-Behnken design. The results obtained showed that rice husks had the highest ash and total solids content of 22.94% and 89.54%, respectively. Coffee husks had the lowest ash and total solids (1.58% and 87.69%) but the highest cellulose content (40.45%). Groundnut shells and rice husks had mean cellulose contents of 28.28% and 20.81%. Moisture content was stable across all the biomass samples with 10.46%, 12.31% and 12.49% recorded for rice and coffee husks, and groundnut shells. Basing on the overall interactive effect of particle size, moisture and cellulose contents, the most optimal interaction that yielded the highest quantity of biochar was found to be at 0.36 mm, 10.18% and 31.51% for particle size, moisture and cellulose contents, respectively. In these interactions, cellulose levels corresponded with groundnut shells as the best biomass material for producing biochar. The study recommends the use of ground nut shells for pyrolysis to produce high yields of biochar for industrial and agricultural applications.

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**Keywords:** Agricultural biomass, energy resource, intermediate pyrolysis, optimization, industrial applications.

## 1. Introduction

Biomass is one of the most promising alternative energy sources because of its carbon neutrality and relative abundance [1, 2]. This is in part because biomass can be thermo-chemically converted to solid and liquid biofuels and gases such as methane, carbon monoxide and hydrogen. Biomass-based fuels offer clean and renewable energy sources that are environmentally friendly and could reduce reliance on non-renewable fossil fuels [3].

The availability of substantial amounts of biomass capable of yielding petroleum-like products presents a promising alternative to fossil fuels. Due to the non-renewable nature of fossil feedstocks, the production of high value fuel products from low value feedstock like heavy oil residues and biomass has garnered research interest in the past decades [4]. In this context, biomass from agricultural wastes presents a good opportunity for conversion to useful biofuel products [5]. Examples of agricultural wastes include groundnut shells,

cassava peels, palm oil wastes, jatropha seeds, rice, maize and coffee husks [6].

Biomass pyrolysis is a combustion process in the absence of oxygen to produce an oil-like liquid called bio-oil, a gas mixture containing mainly oxides of carbon, methane and lower gaseous hydrocarbons and biochar [7]. Among pyrolysis products, bio-oil is a complex mixture of organic compounds and can be used to produce bio-fuel by hydro-deoxygenation [8]. Moreover, bio-oil can be utilized as a source of valuable chemicals like phenols and biofuel [9]. Despite the major achievements in pyrolysis technologies, challenges still exist in the choice of the biomass for the best product yields at optimum conditions [8, 10].

Currently, optimization of process parameters is being done using dedicated proprietary software such as Design Expert® Stat Ease, Minitab®, COMSOL Multiphysics and MATLAB [11]. Design Expert® is preferred because it handles classic stages of screening for vital factors, locating ideal process settings for top performance, discover optimal product formulations as well as validation, which provides the

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flexibility to map complex tasks in a “simple” experimental design. On the other hand, response surface methodology is a set of advanced design of experiments (DOE) techniques used for optimization of processes. It refines the models after important factors have been established using screening designs or factorial designs, especially if curvature in the response surface is suspected [12]. Two main types of response surface designs can be appreciated namely: (i) Central Composite Design (can fit a full quadratic model, and is often used when the design plan calls for sequential experimentation because these designs can include information from a correctly planned factorial experiment); and (ii) Box-Behnken design (usually have fewer design points than central composite designs, thus, they are less expensive to run with the same number of factors [12]).

This study was undertaken to optimize the pyrolysis process and characterize biochar obtained from some underutilized agricultural waste biomass (groundnut shells, coffee and rice husks) in Uganda. These biomasses were previously shown to produce biosynthetic gas [13] while the biochar could be used for other purposes such as the manufacture of activated carbon, graphene, carbon nanoparticles and fibers or applied to agricultural fields for improved productivity.

## 2. Materials and methods

### 2.1. Sampling and physicochemical characterization of biomass

Samples of groundnut shells, coffee and rice husks were obtained from Ngora, Mbale and Busembatya districts of Eastern Uganda, respectively [13]. The samples were subjected to preliminary analyses i.e., determination of moisture, ash, volatile matter, total solids, extractives, lignin, hemicellulose, and cellulose contents.

Moisture content and total solids were respectively determined following the ASTM E871 and NREL/TP-510-42621 methods with slight modifications. The ash content was determined using ASTM D1102 and ASTM E1755-01 methods. Volatile matter was quantified following the ASTM E872 method, while extractives were determined using NREL LAP 2008 and TAPPI T 264 cm-07 methods.

The cellulose, hemicelluloses and lignin contents were determined using the methods described by the National Renewable Energy Laboratory (NREL) [14]. Briefly, hemicellulose content was determined using the methods described by the National Renewable Energy Laboratory (NREL) transferring 1.0 g of extracted biomass into a 250 ml Erlenmeyer flask followed by 150 ml of 0.5M sodium hydroxide solution. The mixture was boiled for 3.5 hours in

distilled water. It was then vacuum filtered after cooling and washed to neutral pH. The residue was dried to a constant weight at 105°C in an oven. The difference between the sample weight before and after treatment was the hemicellulose content (as %w/w of dry biomass). This was replicated five times for each biomass sample, and the hemicellulose content (%w/w) was calculated [14].

Lignin content was determined by weighing 0.3 g of dried and extracted biomass into glass test tubes and adding 3 mL of 72% sulphuric acid. The samples were kept at room temperature for 2 hours while carefully shaking at 30-minute intervals to allow for complete hydrolysis. After the initial hydrolysis, 84 mL of distilled water was added. The second step of hydrolysis was carried out in an autoclave for 1 hour at 121°C. The slurry was then cooled at room temperature. Hydrolysates were filtered through vacuum filter using a filtering crucible. The acid insoluble lignin was determined by drying the residues at 105°C and accounting for ash by incinerating the hydrolyzed samples at 575°C in a muffle furnace. The acid soluble lignin fraction was determined by measuring the absorbance of the acid hydrolyzed samples at 320 nm. The lignin content was calculated as the summation of acid insoluble lignin and acid soluble lignin. The cellulose content was calculated by difference, assuming that extractives, hemicellulose, lignin, ash, and cellulose were the only components of the entire biomass [15, 16].

### 2.2. Biomass preparation for pyrolysis

Three variables (particle size, moisture, and cellulose contents) were selected for optimization. Thus, biomass was separately ground and screened to three selected levels of particle size in the ranges of 3.35–2.36 mm, 2.36–1.00 mm and 0.300–0.425 mm using three sets of two different standard test sieves with the aim of obtaining average particle sizes of 2.855 mm, 1.68 mm, and 0.3625 mm, respectively. Cellulose content previously established for each biomass was used as a basis for variation in them.

The moisture content was varied by conditioning each sample to three different levels of moisture content chosen for optimization (high, mid, and low). These were (i) fresh samples, (ii) those subjected to half drying treatment, and (iii) bone-dry samples, respectively (**Table 1**). Each sample was then crushed into three different categories of particle sizes using a grinder and subsequently screened with each of the three pairs of standard test sieves. Thus, all the samples subjected to pyrolysis had various compositions used as a means of obtaining optimum quantities of pyrolysis yields. The time used for complete drying was halved to condition the moisture content, assuming uniformity in moisture loss during the drying process inside the convection oven.

**Table 1.** Design levels for the different factors optimized

| Factor            | Experimental level      | High                 | Mid                      | Low                |
|-------------------|-------------------------|----------------------|--------------------------|--------------------|
| Moisture content  | Actual value (%)        | 11.75                | 5.88                     | 0.00               |
|                   | Coded value             | 1.00                 | 0.00                     | -1.00              |
| Particle size     | Mean particle size (mm) | 2.86                 | 1.68                     | 0.36               |
|                   | Coded value             | 1.00                 | 0.00                     | -1.00              |
| Cellulose content | Average (%)             | 40.45 (coffee husks) | 28.28 (groundnut shells) | 20.81 (rice husks) |
|                   | Coded value             | 1.00                 | 0.00                     | -1.00              |

Using Design Expert® Stat Ease software (version 7.0.0, Stat-Ease Inc., USA), response surface methodology-Box Behnken design was chosen to determine the ideal process settings and to achieve optimal performance for the selected design parameters. Box Behnken design was chosen because it created designs with desirable statistical properties, and most importantly, it generated only a fraction of experimental runs required for its alternative 3-level factorial design, enabling to fit well within the time constraints of the entire data analysis process. All the factors used for response surface methodology were quantitative and continuous variables. Within the goal of optimizing the process, the objective was to find a desirable surface in the design space, Box-Behnken initial designs were used to fit three levels of the chosen factors as low, mid, and high, respectively. Because there are only three levels, the quadratic model was deemed appropriate and satisfactory for the initial design. Considering biochar and biosynthetic gas as responses, and the mean values of all variables selected, a total

number of 17 experimental runs were generated to fit the optimization (Tables 2 and 3).

### 2.3. Pyrolysis

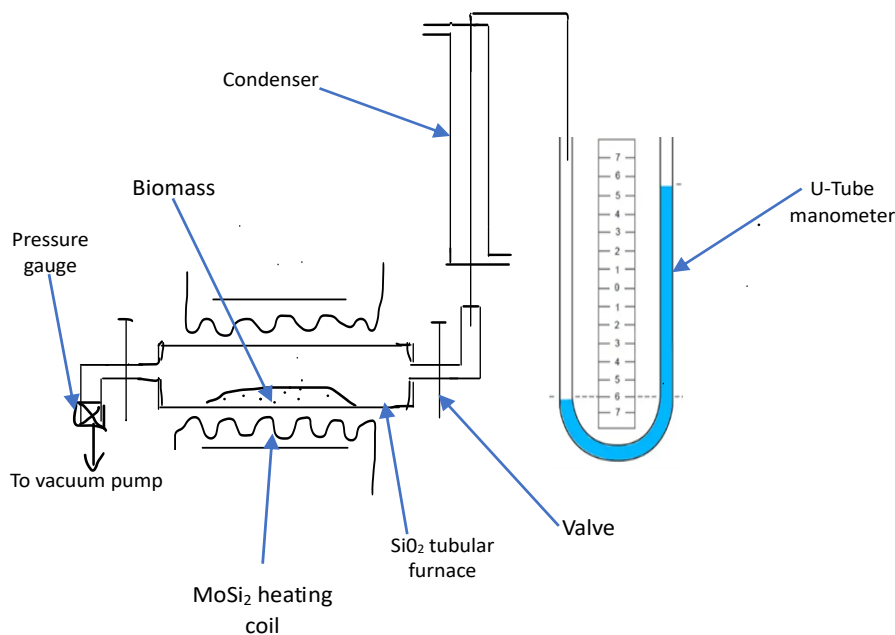
The method used for carrying out the pyrolysis was developed from the equipment operation manual for the tubular furnace pyrolyzer used in this study. Each of the experimental runs were conducted by feeding 4 g of the conditioned sample into the tubular furnace of the pyrolyzer shown in the Figure 1, and the air within the glass tubular furnace pumped out using a rotary vane vacuum pump before completely closing the furnace taps to ensure no further infiltration of atmospheric air occurs. The conditions kept constant throughout the pyrolysis were an average heating temperature rise of 1.5°C per second, with an average residence time of about 2.5 seconds for biosynthetic gas and 1 hour for biochar.

**Table 2.** Design parameters for optimization of groundnut shells, rice, and coffee husk biomass

| Factor | Parameter            | Type    | Low Actual | Mid Actual | High Actual | Low Coded | Mid Coded | High Coded |
|--------|----------------------|---------|------------|------------|-------------|-----------|-----------|------------|
| A      | Particle size (mm)   | Numeric | 0.36       | 1.68       | 2.86        | -1.00     | 0.00      | 1.00       |
| B      | Moisture content (%) | Numeric | 0.00       | 5.88       | 11.75       | -1.00     | 0.00      | 1.00       |
| C      | Cellulose (%)        | Numeric | 20.81      | 28.28      | 40.45       | -1        | 0         | 1          |

**Table 3.** Experimental run sheet for actual factors

| Std | Run | Particle size (mm) | Moisture content (%) | Cellulose content (%) |
|-----|-----|--------------------|----------------------|-----------------------|
| 4   | 1   | 2.86               | 12.49                | 28.28                 |
| 6   | 2   | 2.86               | 5.23                 | 20.81                 |
| 1   | 3   | 0.36               | 0.00                 | 28.28                 |
| 14  | 4   | 1.68               | 6.24                 | 28.28                 |
| 15  | 5   | 1.68               | 6.24                 | 28.28                 |
| 7   | 6   | 0.36               | 6.16                 | 40.45                 |
| 16  | 7   | 1.68               | 6.24                 | 28.28                 |
| 5   | 8   | 0.36               | 5.23                 | 20.81                 |
| 17  | 9   | 1.68               | 6.24                 | 28.28                 |
| 2   | 10  | 2.86               | 6.24                 | 28.28                 |
| 8   | 11  | 2.86               | 6.16                 | 40.45                 |
| 9   | 12  | 1.68               | 0.00                 | 20.81                 |
| 12  | 13  | 1.68               | 12.31                | 40.45                 |
| 13  | 14  | 1.68               | 6.24                 | 28.28                 |
| 11  | 15  | 1.68               | 0.00                 | 40.45                 |
| 10  | 16  | 1.68               | 10.46                | 20.81                 |
| 3   | 17  | 0.36               | 12.49                | 28.28                 |



**Figure 1.** Schematic diagram of the pyrolysis process

The heating program was set to heat from around room temperature to 500°C and automatically stopping the heating, for all samples. The outlet tap was opened to allow the gaseous vapors produced to pass through an airtight water-cooled condenser with circulation temperatures of about 15°C so as to trap mostly heavier condensable vapors consisting of components like organic acids, phenolics and furans and directly connected to the U-tube manometer so as to measure the total volume of the non-condensable gases produced such as CO, CH<sub>4</sub>, CO<sub>2</sub>, H<sub>2</sub> and light hydrocarbons by volume displacement. After the furnace subsequently cooled all the biochar remaining was then weighed; the non-condensable gases were quantified at room temperature by displacement method using a U-tube manometer. All the responses were determined by taking direct measurements of volume and weight for non-condensable gas and biochar, respectively.

#### 2.4. Optimization of process variables to maximize biochar yields

Basing on the single factor effects of cellulose, moisture and particle size on biochar yields, the yield was set to be maximized at limits of 0.19 g to 1.54 g with a lower weight of 10 and the following goals: (i) for numerical optimization of both cellulose and moisture, they were set to be within the ranges of 25% to 35% and 10% to 12.5%, respectively, (ii) particle size was set to be minimized from 0.99 mm to 0.36 mm at an upper weight of 10, so as to allow for the use of experimentally feasible quantities (Table 4).

Table 4. Optimization constraints for high biochar yields

| Name                 | Goal        | Lower limit | Upper limit | Lower weight | Upper weight |
|----------------------|-------------|-------------|-------------|--------------|--------------|
| Particle size (mm)   | minimize    | 0.36        | 0.99        | 1            | 10           |
| Moisture content (%) | Is in range | 10          | 12.5        | 10           | 10           |
| Cellulose (%)        | Is in range | 25          | 35          | 1            | 1            |
| Biochar (g)          | Maximize    | 0.19        | 1.54        | 10           | 10           |

#### 2.5. Data analysis

The results obtained were analyzed using Microsoft Excel 2016 software, and the entire design of experiments and optimization was done using Design-Expert® Stat Ease (version 7.0.0 software, Stat-Ease Inc., USA). ANOVA, Sequential Model Sum of Squares: Type III was used to evaluate the model as well as perform lack of fit tests.

### 3. Results and Discussions

#### 3.1. Physicochemical properties of the biomasses

The results obtained for mean values of total solids, moisture, ash content and extractives reported on dry basis are shown in Figure 2. The data shows that rice husks had the highest level of total solids of 89.54 ± 0.18% while groundnut shells had the least amount. The values were, however, not significantly different (P<0.05). Volatile matter was highest in coffee husks and least in rice husks, but there were no significant differences among them (P<0.05). These results are in agreement with published literature (Table 5) on these biomasses [17, 18].

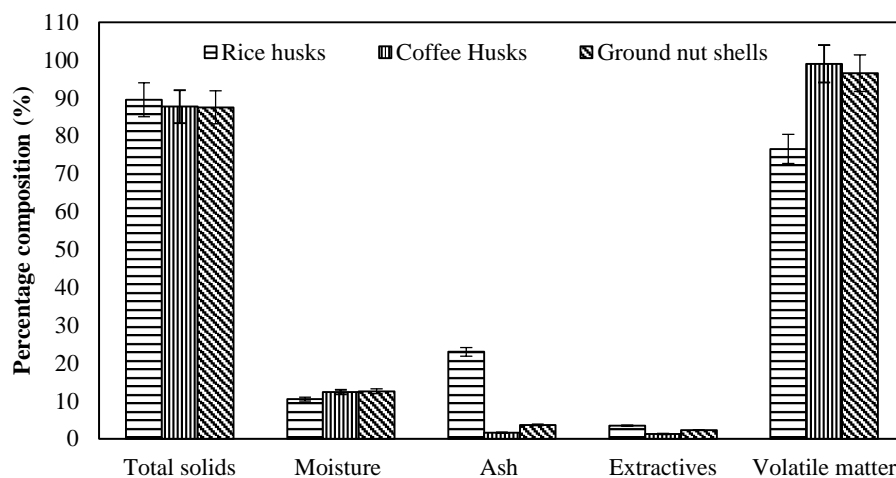


Figure 2. Physico-chemical properties of ground nut shells, rice, and coffee husks.

Table 5. Physicochemical properties of groundnut shells and rice husks available in literature

| Parameter                                   | Groundnut shells           | Rice husk        | Coffee husk        | References           |
|---|----------------------------|------------------|--------------------|----------------------|
| Moisture (%)                                | 7.98, 5.79, 7.40, 5.00     | 8.19, 8.68-10.44 | -                  | [19-23]              |
| Ash (%)                                     | 12.80, 4.11-4.41, 5.00     | 29.53, 22.60     | 7.75               | [20-22, 24, 25]      |
| Volatile matter (%)                         | 79.10                      | 61.68, 61.00     | -                  | [22, 25]             |
| Extractives                                 | -                          | -                | 47.38              | [24]                 |
| Lignocellulose                              | -                          | 74.10            | -                  | [26]                 |
| Cellulose                                   | 16.00, 33.80, 38.00, 48.00 | -                | 36.47, 35.4        | [21, 27-29]          |
| Lignin                                      | 9.00, 16.00                | 21.50            | 52.47, 23.2, 29.55 | [21, 24, 27, 29, 30] |
| Hemicellulose                               | 11.00, 36.00               | 33.8             | 55.78, 18.2        | [21, 27, 29, 30]     |
| Holocellulose (cellulose and hemicellulose) | -                          | -                | 15.32              | [24]                 |

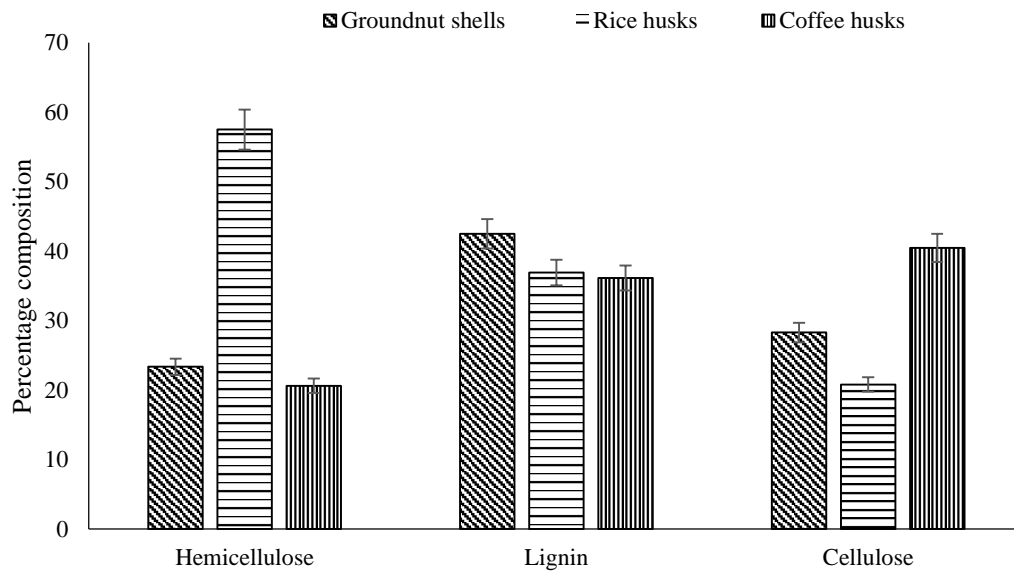
The biomasses were further assessed for their lignin, hemicellulose and cellulose contents, and the results are depicted in **Figure 3**. Rice husks had the highest mean hemicellulose content ( $57.49 \pm 2.15\%$ ), followed by that in groundnut shells ( $23.35 \pm 2.05\%$ ) and then coffee husks ( $20.62 \pm 2.02\%$ ). The hemicellulose content of rice husks in our study was nearly twice that in coffee husks. This could be attributed to the difference in the nature of these agricultural wastes, which is and the findings were in agreement with published literature [26, 29].

Furthermore, the highest amount of hemicellulose was found in rice husks, followed by groundnut shell, and then lastly coffee husk. The quantity determined in coffee husk was fairly in agreement with that reported by Collazo-Bigliardi et al. [27]. The amount of hemicellulose and cellulose found in

groundnut shell in the present study was in agreement with that reported by Gajula et al. [21]. The composition of lignin for rice husks was found to be almost equal to that of coffee husks, but highest for groundnut shells. The lignin and cellulose contents of coffee husks in the present study are in close agreement with 29.55% reported by Veiga et al. [24] and 43% by Franca et al. [30], respectively.

3.2. Results for pyrolysis of groundnut shells, coffee, and rice husks

The experimental results for 17 runs conducted are shown in Table 5. The highest biochar yield from experiment 6, run 2 as shown in **Table 6** may be attributed to high composition of ash in the rice husk (**Figure 2**).



**Figure 3.** Proportions of hemicellulose, lignin, and cellulose in biomass

**Table 6.** Actual design and the response for the pyrolysis of the studied biomasses

| Experiment no. | Run | A: Particle size | B: Moisture | C: Cellulose | Biochar yield |
|----------------|-----|------------------|-------------|--------------|---------------|
| 1              | 3   | 0.36             | 0.00        | 28.28        | 1.44          |
| 2              | 10  | 2.86             | 6.24        | 28.28        | 1.37          |
| 3              | 17  | 0.36             | 12.49       | 28.28        | 1.38          |
| 4              | 1   | 2.86             | 12.49       | 28.28        | 1.28          |
| 5              | 8   | 0.36             | 5.23        | 20.81        | 0.19          |
| 6              | 2   | 2.86             | 5.23        | 20.81        | 1.54          |
| 7              | 6   | 0.36             | 6.16        | 40.45        | 0.82          |
| 8              | 11  | 2.86             | 6.16        | 40.45        | 1.07          |
| 9              | 12  | 1.68             | 0.00        | 20.81        | 0.54          |
| 10             | 16  | 1.68             | 10.46       | 20.81        | 1.27          |
| 11             | 15  | 1.68             | 0.00        | 40.45        | 1.11          |
| 12             | 13  | 1.68             | 12.31       | 40.45        | 0.83          |
| 13             | 14  | 1.68             | 6.24        | 28.28        | 1.35          |
| 14             | 4   | 1.68             | 6.24        | 28.28        | 1.37          |
| 15             | 5   | 1.68             | 6.24        | 28.28        | 1.33          |
| 16             | 7   | 1.68             | 6.24        | 28.28        | 1.32          |
| 17             | 9   | 1.68             | 6.24        | 28.28        | 1.32          |

3.3. Analysis of variance for biochar yields

Analysis of variance (ANOVA) for biochar yield was conducted and results are as shown in Table 7. The Model F-value of 619.99 implied that the model was significant; there is only a 0.01% chance that a "Model F-Value" this large would occur due to noise. Values of "Prob > F" less than 0.05 indicated that model terms were also significant. In this case; A, B, C, AB, AC, BC, A<sup>2</sup>, C<sup>2</sup>, A<sup>2</sup>B, AC<sup>2</sup> were significant model terms. The value of R-squared signified a high correlation within the selected model terms. The "Lack of Fit F-value" of 0.44 implied the Lack of Fit was not significant relative to the absolute error. There is a 67.37% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit was very important because we wanted the model to

fit. Furthermore, the "Predicted R-Squared" of 0.9885 was in reasonable agreement with the "Adjusted R-Squared" of 0.9974 and the "Adequate (Adeq) Precision" ratio greater than 4 was desirable. Our ratio of 93.11 indicated an adequate signal. This model was therefore chosen for use in navigating the design space.

There was no statistical transformation required for biochar as indicated in the software evaluation and the cubic model resulted in different combinations of terms as shown in the final model equation (1).

Final equation in terms of coded factors was as follows;  
 Biochar = 1.34 - 0.12A + 0.11B + 0.036C + 0.067AB - 0.28AC - 0.25BC - 0.04A<sup>2</sup> - 0.40C<sup>2</sup> - 0.075A<sup>2</sup>B + 0.52AC<sup>2</sup> (1)  
 Where; A - Particle size, B - Moisture content, C - Cellulose.

Table 7. Analysis of variance for biochar (Partial sum of squares - Type III)

| Source           | Sum of Squares | Df            | Mean Square           | F Value          | P-value (Prob> F)    | Significance          |
|------------------|----------------|---------------|-----------------------|------------------|----------------------|-----------------------|
| Model            | 2.01           | 10            | 0.20                  | 619.99           | < 0.0001             | significant           |
| A-Particle size  | 0.06           | 1             | 0.06                  | 169.98           | < 0.0001             | significant           |
| B-Moisture       | 0.05           | 1             | 0.05                  | 155.82           | < 0.0001             | significant           |
| C-Cellulose      | 0.01           | 1             | 0.01                  | 32.36            | 0.0013               | significant           |
| AB               | 0.02           | 1             | 0.02                  | 56.10            | 0.0003               | significant           |
| AC               | 0.30           | 1             | 0.30                  | 931.08           | < 0.0001             | significant           |
| BC               | 0.26           | 1             | 0.26                  | 784.96           | < 0.0001             | significant           |
| A <sup>2</sup>   | 0.01           | 1             | 0.0069                | 21.21            | 0.0037               | significant           |
| C <sup>2</sup>   | 0.66           | 1             | 0.66                  | 2031.73          | < 0.0001             | significant           |
| A <sup>2</sup> B | 0.01           | 1             | 0.01                  | 34.63            | 0.0011               | significant           |
| AC <sup>2</sup>  | 0.54           | 1             | 0.54                  | 1648.60          | < 0.0001             | significant           |
| Residual         | 0.0019         | 6             | 0.00033               | -                | -                    | -                     |
| Lack of Fit      | 0.00035        | 2             | 0.00018               | 0.44             | 0.6737               | not significant       |
| Pure Error       | 0.0016         | 4             | 0.0004                | -                | -                    | -                     |
| Cor Total        | 2.02           | 16            | -                     | -                | -                    | -                     |
| <b>Std. Dev.</b> | <b>Mean</b>    | <b>C.V. %</b> | <b>Adeq Precision</b> | <b>R-Squared</b> | <b>Adj R-Squared</b> | <b>Pred R-Squared</b> |
| 0.02             | 1.13           | 1.59          | 93.11                 | 0.9990           | 0.9974               | 0.9885                |

Table 8. Coefficient estimates for the selected design terms

| Factor           | Coefficient Estimate | df   | Standard Error | 95% CI Low | 95% CI High |
|------------------|----------------------|------|----------------|------------|-------------|
| Intercept        | 1.34                 | 1.00 | 0.01           | 1.32       | 1.35        |
| A-Particle size  | -0.12                | 1.00 | 0.01           | -0.14      | -0.10       |
| B-Moisture       | 0.11                 | 1.00 | 0.01           | 0.09       | 0.13        |
| C-Cellulose      | 0.04                 | 1.00 | 0.01           | 0.02       | 0.05        |
| AB               | 0.07                 | 1.00 | 0.01           | 0.05       | 0.09        |
| AC               | -0.28                | 1.00 | 0.01           | -0.30      | -0.25       |
| BC               | -0.25                | 1.00 | 0.01           | -0.27      | -0.23       |
| A <sup>2</sup>   | -0.04                | 1.00 | 0.01           | -0.06      | -0.02       |
| C <sup>2</sup>   | -0.40                | 1.00 | 0.01           | -0.42      | -0.37       |
| A <sup>2</sup> B | -0.08                | 1.00 | 0.01           | -0.11      | -0.04       |
| AC <sup>2</sup>  | 0.52                 | 1.00 | 0.01           | 0.49       | 0.55        |

### 3.4. Model graphs for biochar

The single factor effects of the investigated factors affecting biochar yield of the different biomasses were studied. For particle size, its increase led to a reduction in the yield of biochar up to the midpoint (Figure 4). After that, the yields started to increase with an increase in particle size up to the highest level used in this study (2.86 mm). This observation is in complete agreement with that of Demirbas[25]. Increase in biochar yields beyond 1.61 mm particle size has been previously observed[31]. This could be attributed to larger particles that restrict the rate of disintegration, resulting in the increased scope of secondary biochar forming reactions [32]. Hence, larger particle sizes are good for obtaining more biochar yields.

On the other hand, increasing moisture favors the generation of biochar at the pyrolysis conditions employed in

this study (Figure 5). Moisture lowers biomass ignition temperature as energy is dissipated via the latent heat of vaporization of water, thus restricting biosynthetic gas formation due to less depolymerization with increasing moisture and allowing more biochar yields. This agrees with Demirbas[25] who reported high solid product yields for moisture ranges of 10–50% wet basis.

For cellulose content of the biomass feedstocks, biochar yields were observed to be highest at mid-level quantities of cellulose (Figure 6). This observation is consistent with previous studies on the effect of cellulose on biochar yields[33] which were shown to be directly proportional to each other. This may be the case because a large amount of cellulose contributes to the overall carbon content in biochar after thermal degradation[24, 34].

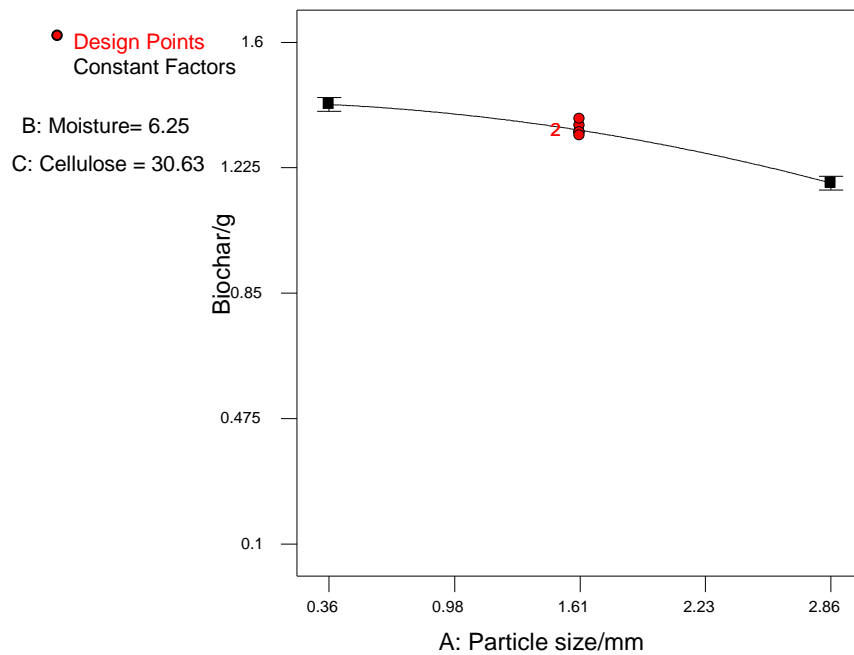


Figure 4. Effect of particle size on biochar.

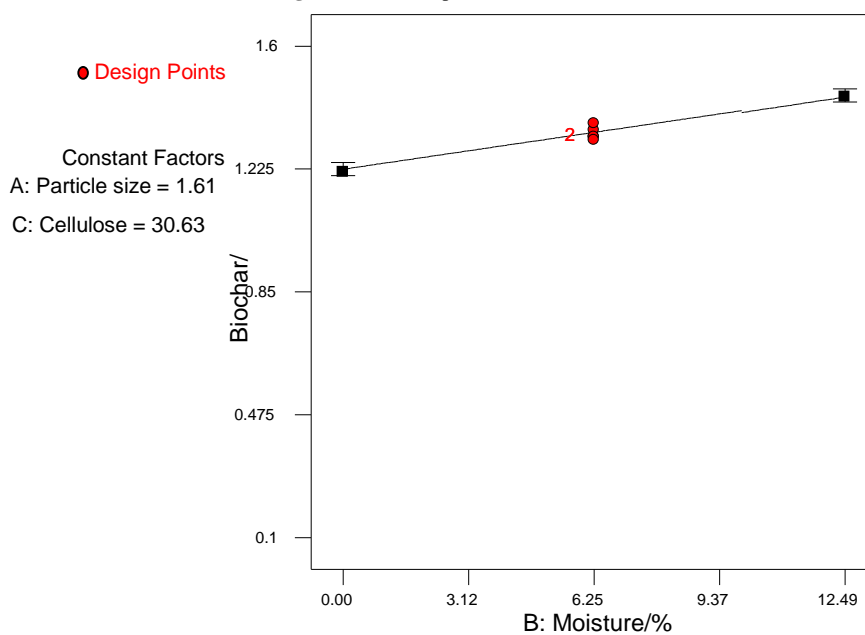


Figure 5. Effect of moisture content on biochar yields of the studied biomasses.

In addition to the foregoing plots, response surface plots were generated for interactive effects between the variables on biochar yields. The interactive effect of moisture content and particle size on biochar yield (**Figure 7**) unveiled that biochar yield at low particle size and high moisture content is low. However, it increased as particle size increases and with decreasing moisture content. The peak yield for biochar is realized at a low level of moisture content and a high level of particle size that lies between 2.23 mm to 2.86 mm. The interactive effects of cellulose content and particle size (**Figure 8**) indicated that biochar yield increases with increase in cellulose content and decrease in particle size,

attaining a maximum at particle size in the range of 0.99 mm to 0.36 mm and cellulose content at 30.40%. This shows that high biochar yields are favored by increase in cellulose and decrease in particle size, which is in consonance with previous studies [25, 33].

For cellulose and moisture contents (**Figure 9**) biochar yield increased with a decrease in cellulose content and increase in moisture content, resulting in a maximum output at cellulose contents in the range of 30.4% to 25.6% against high moisture content of 9% to 12%. This implies that biochar yield is more favored by increasing moisture content at a medium level of cellulose[33, 34].

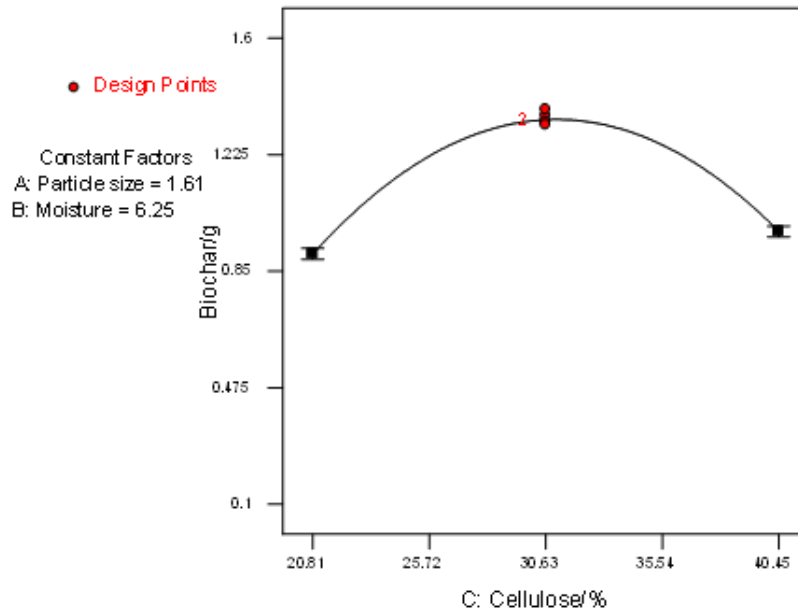


Figure 6. Effect of cellulose content on biochar yields

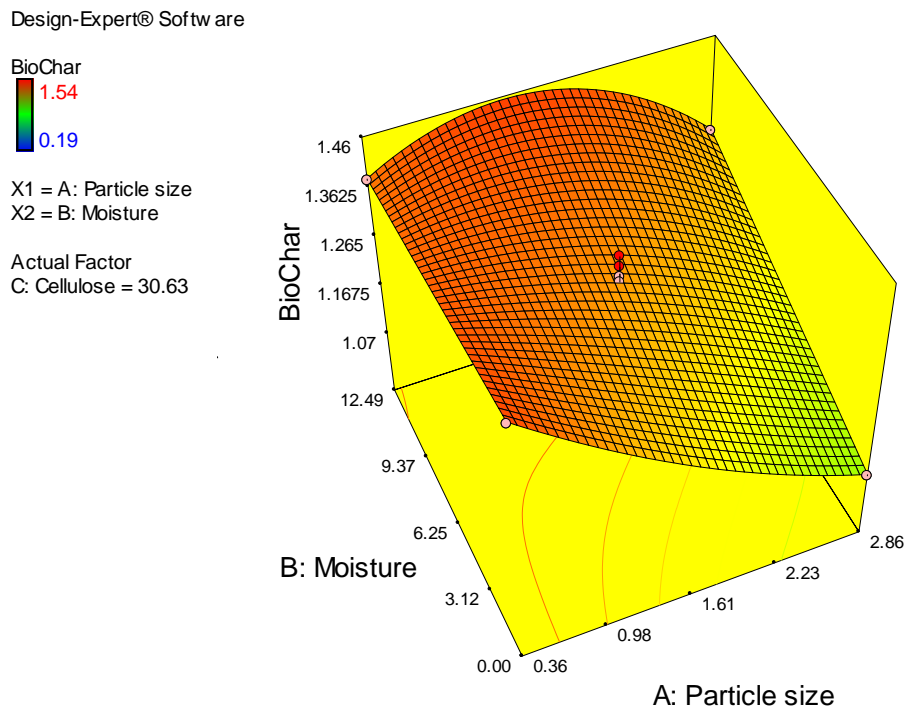
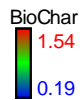


Figure 7. Response surface of moisture content and particle size against biochar yield

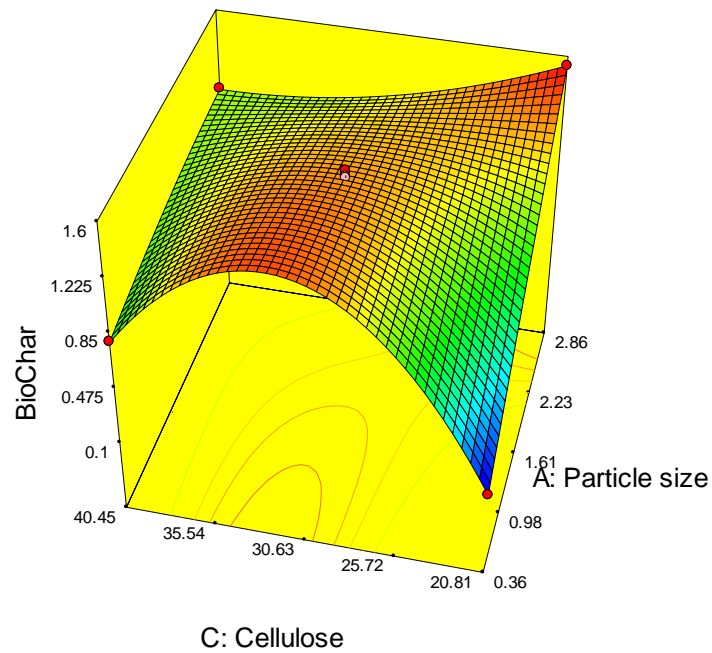


Design-Expert® Software



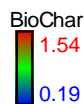
X1 = A: Particle size  
X2 = C: Cellulose

Actual Factor  
B: Moisture = 6.25



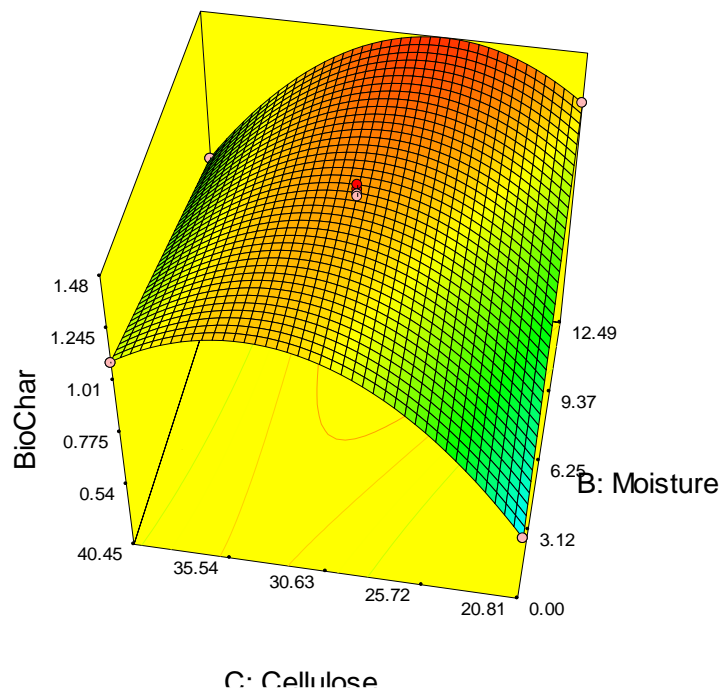
**Figure 8.** Response surface of cellulose and particle size against biochar yields.

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X1 = B: Moisture  
X2 = C: Cellulose

Actual Factor  
A: Particle size = 1.61



**Figure 9.** Response surface of cellulose and moisture content on biochar yields

**3.4.1. Optimal solutions generated**

As can be seen in Table 9, a total of 15 solutions were generated for maximizing biochar, and optimization solutions were selected with the most optimal set of conditions that gave the highest biochar yield and satisfies all the goals set for biochar.

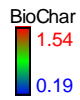
The optimal interactive effects were further explored. Figure 10 shows that the most optimal effect of moisture content and particle size against biochar yield was at 2.86 mm

and 0% moisture content. On the other hand, the most optimal interactive effect between cellulose content and particle size for the highest biochar yield was at 2.86 mm and 40%, respectively (Figure 11). In the context of cellulose and moisture content, the optimal biochar yield was at a high cellulose content (40%) and moisture content of 0% (Figure 12).

**Table 4.** Optimal solutions generated for biochar production from the different biomass.

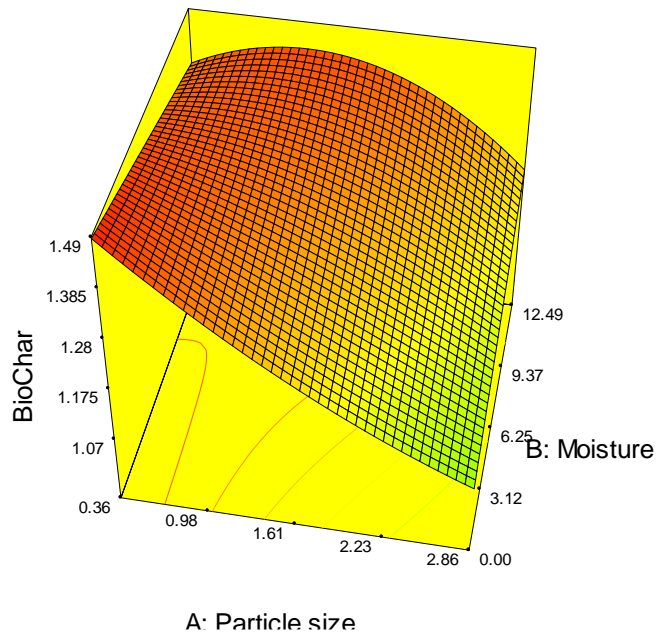
| Exp't no. | Particle size (mm) | Moisture (%) | Cellulose (%) | Biochar yield (g) | Desirability |
|-----------|--------------------|--------------|---------------|-------------------|--------------|
| 1         | 0.36               | 10.18        | 31.51         | 1.40              | 0.50         |
| 2         | 0.36               | 10.46        | 31.31         | 1.39              | 0.50         |
| 3         | 0.36               | 10.77        | 31.22         | 1.39              | 0.50         |
| 4         | 0.36               | 11.24        | 31.32         | 1.39              | 0.50         |
| 5         | 0.36               | 11.68        | 31.1          | 1.39              | 0.50         |
| 6         | 0.36               | 11.9         | 31.13         | 1.39              | 0.50         |
| 7         | 0.36               | 12.02        | 30.94         | 1.39              | 0.50         |
| 8         | 0.36               | 12.49        | 31.18         | 1.38              | 0.50         |
| 9         | 0.36               | 12.49        | 32.33         | 1.37              | 0.49         |
| 10        | 0.36               | 12.49        | 32.68         | 1.36              | 0.49         |
| 11        | 0.36               | 12.49        | 33.18         | 1.34              | 0.49         |
| 12        | 0.36               | 12.49        | 28.64         | 1.33              | 0.49         |
| 13        | 0.36               | 12.49        | 28.32         | 1.32              | 0.48         |
| 14        | 0.36               | 10           | 27.28         | 1.24              | 0.47         |
| 15        | 0.36               | 10           | 26.55         | 1.17              | 0.45         |

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X1 = A: Particle size  
X2 = B: Moisture

Actual Factor  
C: Cellulose = 31.51



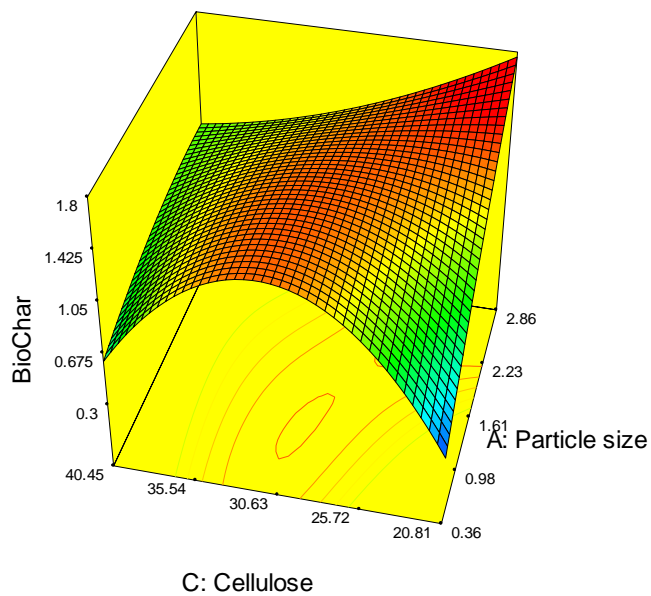
**Figure 10.** Response surface of particle size and moisture against biochar

Design-Expert® Software



X1 = A: Particle size  
X2 = C: Cellulose

Actual Factor  
B: Moisture = 10.18



**Figure 11.** Response surface of cellulose and particle size against biochar

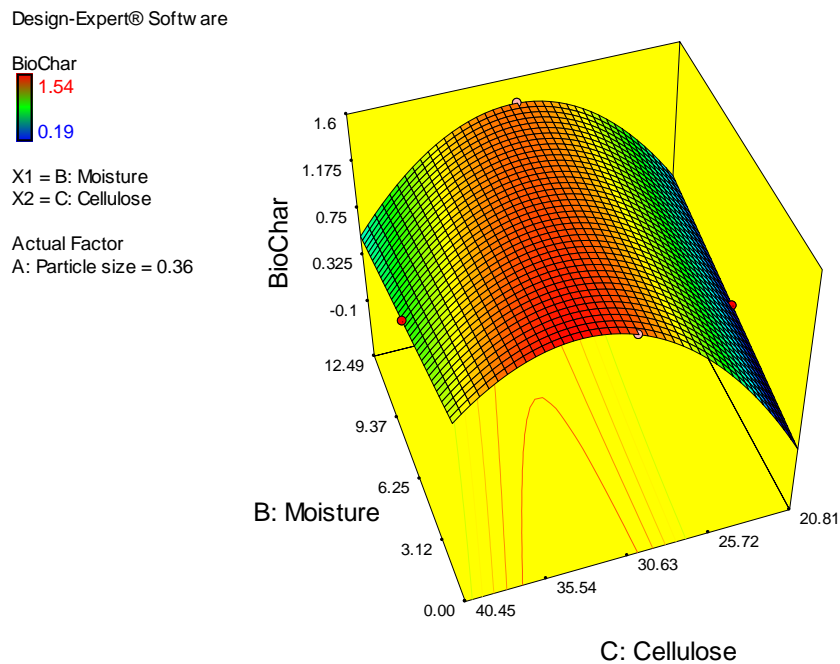


Figure 12. Response surface of cellulose and moisture content against biochar

#### 4. Conclusions

This study showed that of the three biomasses used, rice husks have the highest ash content and the most suitable for optimization studies that seek to improve on biochar yields via pyrolysis. Basing on the overall interactive effect of particle size, moisture, and cellulose content to maximize the yields of biochar, the most optimal interaction that resulted in the highest quantities of biochar was found to be at levels of 0.36 mm, 10.2% and 35.51%, respectively. In all interactions, cellulose content indicated that groundnut shells are the best biomass material for producing biochar. Other than cellulose content, other intrinsic characteristics of biomass such as bulk density and fixed carbon content can be investigated for their collective impact on biochar yields as well as how they can be suitably optimized to ease the scaling up of the pyrolysis process for commercial production of chemical feedstocks and biofuels.

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#### Declaration of conflicting interests

The authors declare no conflicting interests.

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