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# Response surface methodology for optimizing the higher heating value of biochar briquettes prepared by pyrolysis and densification of cottonseed shells (*Gossypium hirsutum)*

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## Abstract

This study aims to determine the optimal conditions for producing biochar briquettes from cottonseed shell residues (*Gossypium hirsutum*) generated by an industrial vegetable oil processing unit. A Box–Behnken experimental design with three factors—pyrolysis temperature, binder content, and compaction pressure—was employed to evaluate their combined effects on the Higher Heating Value (HHV) of the produced briquettes. Each factor was investigated at three coded levels (−1, 0, and +1). The results revealed that the combined pyrolysis and densification processes significantly enhanced the energy performance of the raw biomass. The HHV increased from 18.507 MJ·kg$^{-1}$ for the raw material to values ranging between 21.407 and 28.165 MJ·kg$^{-1}$ for the produced briquettes. These values exceed the minimum requirements for densified solid biofuels specified by the ISO 17225-1:2021 standard, indicating their suitability for domestic and industrial energy applications. The Response Surface Methodology (RSM) results indicated that the maximum HHV within the studied experimental domain was obtained at the coded levels −1, −1, and −1 for pyrolysis temperature, binder content, and compaction pressure, respectively. Under these conditions, the model predicted a maximum HHV of 28.290 MJ·kg$^{-1}$. The process is not only technically efficient but also economically viable, while complying with the principles of environmental sustainability. This study therefore highlights an innovative pathway for the valorization of this abundant agricultural residue, which has so far been utilized in industry in an unsustainable manner, and contributes to the diversification of renewable energy resources as well as to the promotion of the circular economy.

**Keywords:** Cottonseed shells; Biochar; Densification; Pyrolysis; RSM; HHV

## 1. Introduction

Energy demand in developing countries has significantly increased in recent years due to their rapid economic growth [1]. However, limited access to modern energy sources has led these countries to rely heavily on fossil fuels and traditional solid fuels such as wood and charcoal. This situation exerts considerable pressure on forest resources and leads to serious environmental problems, including deforestation and greenhouse gas emissions.To address these challenges, one of the most promising solutions is the energy valorization of agricultural residues, which can serve as a renewable, locally available, and low-cost alternative to conventional fuels. Several studies have shown that the

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densification of agricultural residues into biochar briquettes can significantly increase the density and higher heating value of the raw material, while also improving their storage, transportation, and utilization properties [2], [3]. In a country such as Benin, one of the major producers of cotton in Africa, with production reaching record levels of more than 600,000 tons of cottonseed in recent seasons [4], [5], cottonseed shells represent one of the most promising agricultural residues for such energy valorization. This lignocellulosic biomass is widely available but remains largely underutilized in local industries, and in many cases it is simply burned in open air. However, cottonseed shells possess a favorable lignocellulosic composition, giving them an energy potential comparable to other widely exploited biomasses such as rice husks, palm shells, and corn cobs [6], [7]. In practice, several factors may influence the energy performance of biochar briquettes, including the origin, growth conditions, and type of raw material, as well as pyrolysis temperature, binder type and proportion, and compaction pressure. To determine the optimal preparation conditions for such briquettes, researchers often employ statistical approaches such as Design of Experiments (DOE), particularly Response Surface Methodology (RSM). This methodology, widely reported in the scientific literature, allows the simultaneous optimization of multiple process parameters in order to maximize the higher heating value (HHV) of the produced briquettes [8]. Following a systematic review of the literature, no previous study has been identified combining the use of cottonseed shells, the production of biochar briquettes, and optimization using Response Surface Methodology, which gives the present study a novel contribution. Therefore, the main objective of this work is to develop and optimize a process for converting cottonseed shell residues into biochar briquettes with high energy potential, using a rigorous experimental design methodology.

## 2. Materials and methods

### 2.1. Biochar briquettes preparation

Cottonseed shells (Figure 1-a) used in this study were collected in July 2025 at the « Société des Huileries du Bénin (SHB) ». They were then sun-dried for seven days to reduce the initial moisture content and facilitate the carbonization process [9]. The shells were sorted to remove all foreign materials (stones, threads, wood fragments, etc.) and subsequently ground (Figure 1-b). The ground material was then sieved (Figure 1-c), and only particles with sizes smaller than 1 mm were retained for the subsequent steps.

The pyrolysis temperature, binder proportion, and compaction pressure were subsequently optimized using a Box–Behnken experimental design, with each factor investigated at three levels (Table 1):

- −1 (corresponding to the lower level),
- 0 (the central level), and
- +1 (the upper level).

The central point (0, 0, 0) was repeated three times to ensure the reproducibility of the experimental results.

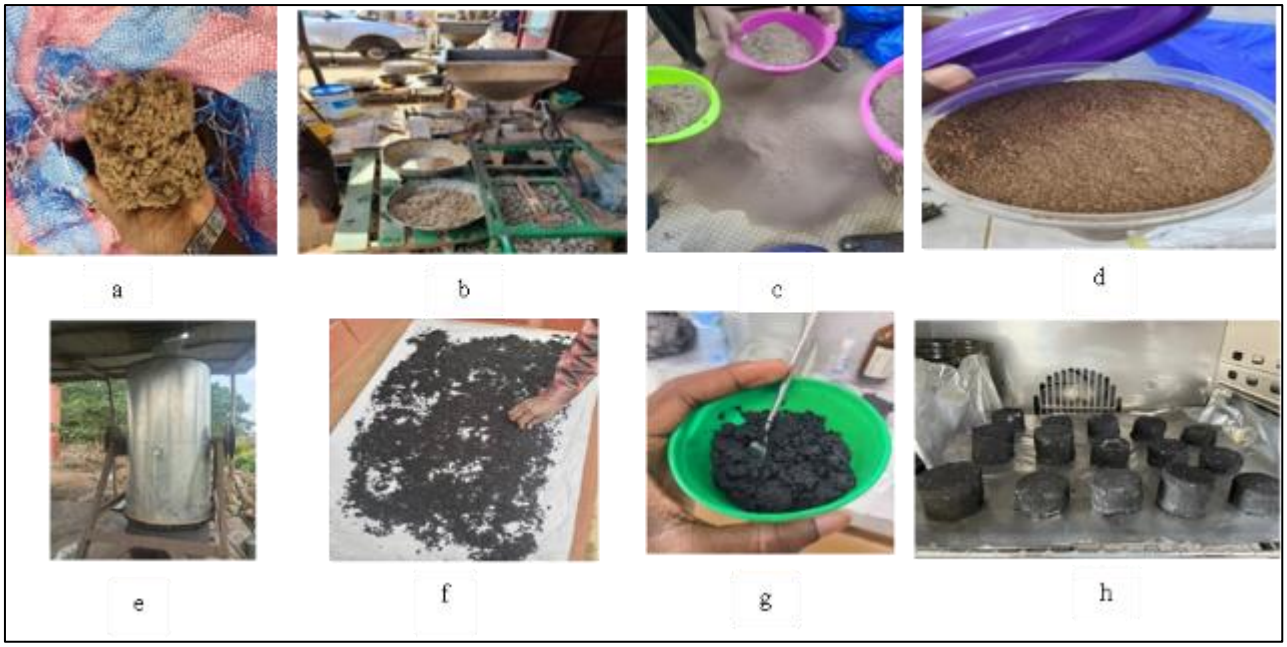



**Figure 1** Preparation of biochar briquettes:(a) Cottonseed shells; (b) Grinding of cottonseed shells; (c) Sieving of cottonseed shells; (d) Ground and sieved cottonseed shells;(e) Carbonization unit for pretreated cottonseed shells; (f) Cottonseed shell biochar; (g) Biochar–cassava starch mixture; (h) Biochar briquettes.

**Table 1** Experimental design for the Box–Behnken design

| N° | Pyrolysis temperature | Binder content | Compaction pressure |
|---|---|---|---|
| 1 | -1 | -1 | 0 |
| 2 | +1 | -1 | 0 |
| 3 | -1 | +1 | 0 |
| 4 | +1 | +1 | 0 |
| 5 | -1 | 0 | -1 |
| 6 | +1 | 0 | -1 |
| 7 | -1 | 0 | +1 |
| 8 | +1 | 0 | +1 |
| 9 | 0 | -1 | -1 |
| 10 | 0 | +1 | -1 |
| 11 | 0 | -1 | +1 |
| 12 | 0 | +1 | +1 |
| 13 | 0 | 0 | 0 |
| 14 | 0 | 0 | 0 |
| 15 | 0 | 0 | 0 |

## 2.2. Physicochemical characterization of cottonseed shells

### *2.2.1. CHNS/O elemental analysis*

The elemental determination of carbon (C) and sulfur (S) by complete combustion is widely recognized as a reliable and reproducible method for complex carbonaceous materials, which justifies its adoption in this study [10]. The ELEMENTRAC CS-i elemental analyzer was used to measure the concentrations of carbon and sulfur.

In the analyzer's induction furnace, the sample is melted under a pure oxygen atmosphere, causing sulfur to react to form sulfur dioxide ($SO_2$) and carbon to form a mixture of carbon monoxide (CO) and carbon dioxide ($CO_2$). Individual control of the oxygen supply during induction combustion is ensured by a lance that directs oxygen to the center of the crucible, thereby guaranteeing the complete oxidation of the carbon and sulfur contained in the sample (solid samples).

Sample masses ranging from 50 mg to 1000 mg are typically used for C/S analysis. The sample is weighed in a ceramic crucible, and accelerators such as tungsten are added. The geometry of the sample (wire, powder, pin, etc.) is not critical for reliable analysis.

The ceramic crucible is then placed on the pedestal, and the analysis is initiated using the ELEMENTS software. The software controls all subsequent steps, from combustion to data evaluation. Approximately 45 to 60 seconds after the start of the analysis, the measured carbon and sulfur concentrations become available for interpretation in the form of a report, and the oxygen content is determined by difference.

### *2.2.2. Lignocellulosic composition*

Lignin content

Approximately 1 g (accurately weighed) of the extracted sample was placed in a 100 mL beaker, then treated with 20 mL of 72% sulfuric acid, added dropwise under constant stirring using a small glass rod [11]. After complete dissolution, the reaction was allowed to proceed; the beaker was covered with a watch glass and kept at room temperature overnight.

The content was then quantitatively transferred into a 1 L round-bottom flask, diluted to 3% sulfuric acid, and heated under reflux for four hours. The lignin was filtered using ashless filter paper and washed with hot distilled water until neutrality. It was then determined gravimetrically and subsequently calcined at 850 °C for 45 minutes. The ash mass was subtracted to obtain the ash-free lignin percentage.

Hemicellulose content

The term alpha-cellulose refers to the fraction of cellulose insoluble in a 17.5% sodium hydroxide solution. The determination of alpha-cellulose was carried out according to the Zellcheming Merkblatt IV/29 A method.

Approximately m = 3 g (accurately weighed) of the sample (holocellulose) was placed in a 250 mL porcelain beaker. Then, 25 mL of 17.5% sodium hydroxide solution was added, and the sample was allowed to swell for 4 minutes (the time was measured precisely from the last drop). The material was then pressed with a glass rod for 3 minutes.

After pressing, an additional 25 mL of sodium hydroxide was added, and the mixture was thoroughly stirred until a homogeneous paste was obtained (approximately 1 minute). The beaker was then covered and allowed to stand for 35 minutes at 20 °C.

Subsequently, 100 mL of distilled water was added, and the material was quickly filtered under vacuum using a sintered glass funnel. The filtrate was then poured twice over the paste before washing with distilled water. After washing with distilled water until neutrality, 100 mL of 10% acetic acid was added dropwise, followed by distilled water [12].

Cellulose content

The cellulose content was determined using the following relationship:

$$\text{Cellulose (\%)} = \frac{mass\ of\ alpha-cellulose}{m} * 100$$

*2.2.3. Estimation of the higher heating value of cottonseed shells*

Several studies reported in the literature propose empirical equations for estimating the higher heating value (HHV) of biomass in the absence of a bomb calorimeter. Some of these equations are based on ultimate analysis results, others on proximate analysis, and still others on the cell wall (biochemical) composition of biomass [13], [14], [15], [16], [17].

In this study, the higher heating value of the cottonseed hull was estimated from the biochemical composition of the biomass using a weighted approach based on the average HHVs of cellulose (17.36 MJ/kg), hemicellulose (17.36 MJ/kg), lignin (24.42 MJ/kg), extractives (20 MJ/kg), and mineral matter (0 MJ/kg) [17].

$$\text{HHV} = \sum_{i=1}^{5}(x_i * HHV_i)$$

where $x_i$ represents the mass fractions of lignin, cellulose, hemicellulose, extractives, and mineral matter, respectively.

### 2.3. Higher heating value of the produced briquettes

The higher heating value (HHV) of the briquettes was determined using a Parr bomb calorimeter (Figure 2) following the procedure described by [18], [19] to ensure the reproducibility of the results.

First, the calorimeter was calibrated, which consists of determining its energy equivalent (the sum of the heat capacities of the calorimeter components) for a given temperature rise. Benzoic acid was used as the calibration standard for the bomb calorimeter. Since its heat of combustion is known, multiplying this value by its mass makes it possible to determine the energy equivalent of the calorimeter.

Subsequently, briquette samples of known masses were introduced into the calorimeter [20], and the corresponding temperature rise was recorded for each experiment. The product of this temperature rise and the energy equivalent of the calorimeter gives the amount of heat released, and the ratio of this heat quantity to the initial mass of the sample provides the higher heating value of the briquette sample.

**Figure 2** Parr bomb calorimeter

## 3. Results and discussion

### 3.1. Physicochemical characterization of cottonseed shells

*3.1.1. CHNS/O elemental analysis*

The results of the ultimate analysis of cottonseed shells is presented in Table 2

**Table 2** Ultimate analysis

| Eléments chimiques | %C | %H | %N | %S | % O |
|---|---|---|---|---|---|
| Pourcentage | 81.34±0.02 | 6.07±0.02 | 1.4±0.01 | 0.18±0.004 | 11.01±0.02 |

These results indicate a high carbon content (81.34 ± 0.02%), followed by hydrogen (6.07 ± 0.02%), nitrogen (1.4 ± 0.01%), and sulfur (0.18 ± 0.01%).

This high proportion of carbon reflects a significant energy potential, which is favorable for the production of biochar or briquettes with high calorific value [21]. It should also be noted that the obtained carbon value is higher than those usually reported in the literature for raw, non-carbonized biomass. This can be explained by the fact that the analysis performed using the ELEMENTRAC CS-i analyzer is based on the direct quantification of carbon and sulfur through combustion, while oxygen is determined by difference.

This approach, which is suitable for carbonaceous materials, may lead to an overestimation of the carbon fraction in lignocellulosic biomasses that are rich in oxygen.

The low sulfur and nitrogen contents also indicate cleaner combustion, with reduced emissions of pollutants such as nitrogen oxides (NOx) and sulfur oxides (SOx). These values are consistent with the indicative composition limits established in the European standards (EN 14961-1:2010) [22], which aim to minimize pollutant emissions during solid fuel combustion.

Therefore, cottonseed shells constitute a lignocellulosic biomass well suited for energy valorization through pyrolysis.

*3.1.2. Cell wall composition*

The cell wall composition of cottonseed shells, presented in Table 3, is as follows.

**Table 3** Cell wall composition

| Parameters | Percentage (%) |
|---|---|
| Lignin | 22.07±0.13 |
| Cellulose | 49.64±0.18 |
| Hemicellulose | 19.86±0.06 |
| Extractives | 5.26±0.01 |
| Mineral matter | 3.17±0.04 |

The analysis of Table 3 reveals lignin, cellulose, and hemicellulose contents of 22.07, 49.64, and 19.86, respectively. This composition is typical of biomasses suitable for biochar production, which may contain 40 to 60% cellulose, 15 to 30% hemicellulose, and 10 to 25% lignin [23]. Lignocellulosic biomass is mainly composed of cellulose and hemicellulose because these components decompose more easily than lignin. The more recalcitrant lignin promotes the formation of solid carbonaceous compounds after pyrolysis. This composition may vary depending on the species, growth conditions, and environment [24].

Furthermore, a moderate ash content of 3.17% is observed, which is consistent with the requirements for biomass intended for the production of combustible biochar. A high volatile matter content is expected since holocelluloses mainly decompose into volatile compounds [25].

*3.1.3. Higher Heating Value (HHV)*

The HHV of cottonseed hulls estimated using the biochemical composition approach is 18.507 $MJ \cdot kg^{-1}$. This value falls well within the range commonly reported in the literature (17 to 22 $MJ \cdot kg^{-1}$) for dry lignocellulosic biomass. It is even located in the upper part of this range, which further supports the suitability of this biomass for energy valorization [17]. This may be explained by the high fixed carbon content and the low ash content observed.

### 3.2. Produced Briquettes as Industrial Fuels

The formulated briquettes are characterized by higher heating values ranging from 21.407 $kJ \cdot g^{-1}$ to 28.165 $kJ \cdot g^{-1}$. These values are generally comparable to those reported in the literature for other fuels (Table 4).

These calorific values are not only higher than those of several commonly used fuels but are also comparable to those of high-quality charcoal. The obtained values are all higher than the minimum thresholds generally required for densified solid fuels according to ISO 17225-1:2021 [26]. These HHV levels are comparable to those of commercially accepted products used in industrial and domestic solid combustion applications. The high energy density observed in

the formulated briquettes may result from the production process, particularly compression, which promotes more uniform and efficient combustion (with fewer thermal losses due to moisture or porous structure) compared with lighter or poorly compacted briquettes.

Thus, these briquettes can be recommended as alternatives to coal and wood for industrial use, particularly in local oil mills that require thermal energy for their boilers [27]. Moreover, [28] showed that these briquettes can be used to produce heat and steam in agro-industrial facilities without requiring major modifications to existing equipment.

**Table 4** Comparison of the higher heating values of some fuels.

| Numbers | Fuels | HHV (MJ.$Kg^{-1}$) | References |
|---|---|---|---|
| 1 | Charcoal particle–based briquette | 24.9 | [29] |
| 2 | Rice biochar briquettes | 39.72 | [2] |
| 3 | Biochar briquettes from sorghum stalks and peanut shells | 20.08 to 24.36 | [30] |
| 4 | Paper, sawdust, and charcoal briquettes | 32.345 to 34.482 | [31] |
| 5 | Human waste–derived char briquettes | 25.60 | [32] |
| 6 | Cocoa shell briquettes | 17.941 | [33] |
| 7 | Sawdust briquettes | 22.623 to 24.719 | [34] |
| 8 | Dry wood | 17 to 21 | [17] |
| 9 | Charcoal briquette | 29 to 33 | [17] |
| 10 | Corn cob briquettes | 23.50 | [35] |
| 11 | Banana stem briquettes | 17.51 | [35] |
| 12 | Palm kernel shell briquettes | 18.72 | [36] |
| 13 | Cottonseed shell biochar briquettes | 21.407 to 28.165 | This study |

### 3.3. Influence of Carbonization Temperature, Binder Amount, and Compaction Pressure on Higher Heating Value

*3.3.1. Regression Equation in Uncoded Units*

The study of the influence of factors such as carbonization temperature, binder amount, and compaction pressure using the response surface methodology allowed the development of a mathematical equation relating the higher heating value (HHV) to the three studied factors. The corresponding model is expressed as follows:

HHV = 24.09 – 0.136 Temperature – 0.646 Binder amount + 0.436 Compaction pressure + 0.96 Temperature*Temperature + 0.39 Binder amount*Binder amount + 1.02 Compaction pressure*Compaction pressure – 0.11Temperature*Binder amount + 0.16 Temperature*Compaction pressure + 1.43 Binder amount*Compaction pressure

The coefficient of determination is 0.49, a moderate but acceptable value, which can be explained by the presence of other factors (such as the elemental composition of the briquettes and their moisture content) that have a greater impact on the HHV than those considered in this study [37].

The negative coefficients for temperature (-0.136) and binder amount (-0.646) indicate that an increase in these two parameters within the experimental range tends to decrease the HHV. Conversely, the positive coefficient for compaction pressure indicates that a moderate increase in this parameter could slightly improve the HHV. The positive quadratic coefficients (0.96, 0.39, 1.02) suggest the existence of an optimum within the experimental domain.

*3.3.2. Analysis of Pareto, Contour, and Surface Plots*

Figures 3, 4, and 5 present the Pareto diagrams, response surface plots, and contour plots, respectively.

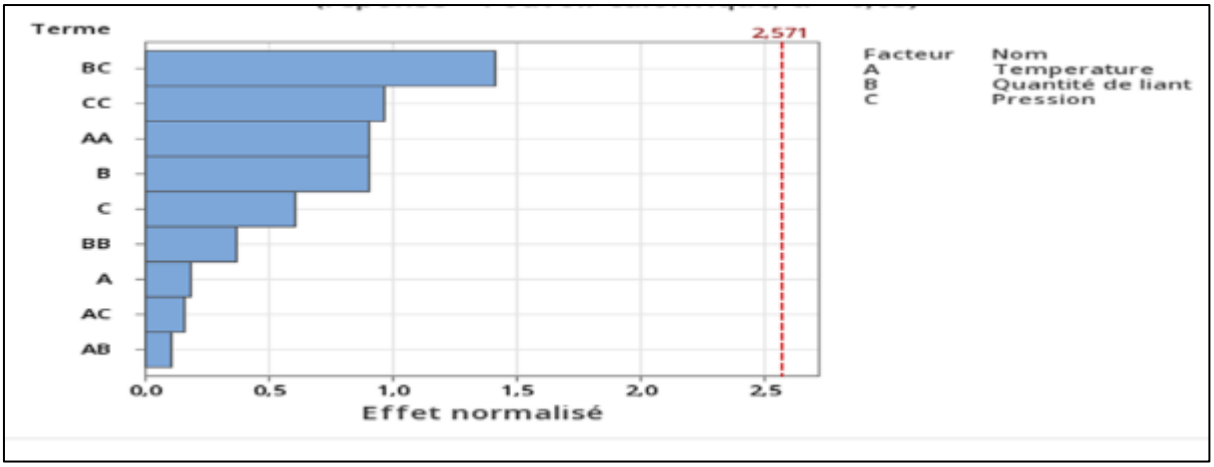
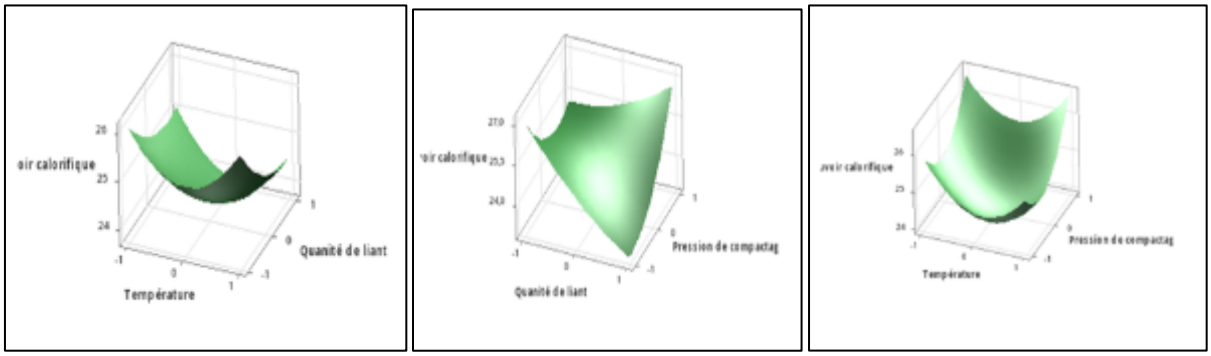
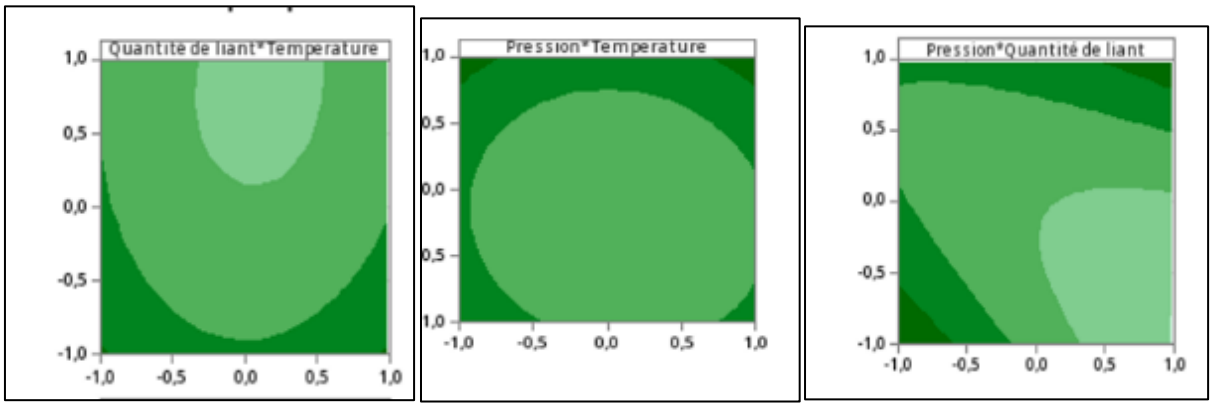



**Figure 3** Pareto Diagram




*a* *b* *c*

**Figure 4** HHV Surface Plots – a) Temperature-Binder Amount, b) Binder Amount-Compaction Pressure, c) Temperature-Compaction Pressure




*a* *b* *c*

**Figure 5** Contour Plots – a) Binder Amount-Temperature, b) Pressure-Temperature, c) Pressure-Binder Amount

The combined analysis of the Pareto diagram (Figure 3), response surfaces (Figure 4), and contour plots (Figure 5) shows that the HHV is mainly influenced by nonlinear relationships and combined effects among the studied operational parameters, namely temperature, binder amount, and compaction pressure. The absence of statistically significant effects at the 5% level suggests that no single factor dominates HHV behavior within the experimental domain considered [38, 39]. This outcome is common in response surface methodology studies, where factors interact in a complex manner and quadratic effects play a significant role [40].

The surface and contour plots highlight marked curvature in the responses, reflecting the notable influence of quadratic terms for temperature and pressure. This behavior indicates the presence of intermediate optimal values beyond which HHV decreases, likely due to structural changes in the material. Indeed, several studies have shown that excessively high temperatures or pressures can degrade the carbon structure or cause over-densification, reducing the fixed carbon fraction and, consequently, the HHV [41].

*3.3.3. Maximization of Higher Heating Value*

The optimization of HHV was performed with the objective of maximizing it. The optimal solution was obtained under the following conditions:

Temperature: -1

Binder Amount: -1

Compaction Pressure: -1

The optimal HHV is then 28.29 $MJ \cdot kg^{-1}$, a value very close to the maximum experimentally obtained HHV (28.165 $MJ \cdot kg^{-1}$).

The fact that the maximum HHV is obtained at the minimum levels of the three factors suggests that:

Excessive temperature may cause losses of energy-rich compounds or unfavorable changes in the carbon structure;

Excessive binder amount dilutes the carbon-rich combustible fraction, leading to a decrease in HHV [42];

Excessive compaction pressure may lead to over-densification, limiting the porosity necessary for efficient combustion.

Economically, the optimal preparation conditions are particularly interesting because they imply the lowest carbonization temperature (less energy required for carbonization), lowest binder amount (lower formulation costs and higher carbon-rich fraction) [43], and lowest compaction pressure (less energy input and the possibility of using a simple manual press), while still providing an HHV comparable to that of high-quality commercial charcoals.

## 4. Conclusion

In this study, a new valorization route for cottonseed hull residues was proposed. This agro-industrial residue exhibited low nitrogen (1.4 ± 0.01%) and sulfur (0.18 ± 0.01%) contents, ensuring clean combustion with low pollutant emissions. After pyrolysis and densification, the raw biomass saw its HHV increase from 18.507 $MJ \cdot kg^{-1}$ to values ranging between 21.407 and 28.165 $MJ \cdot kg^{-1}$. Within the experimental conditions, the predicted maximum HHV is 28.290 $MJ \cdot kg^{-1}$, a value highly competitive with other commercial fuels intended for industrial and household use. This maximum HHV is achieved at the combination (-1, -1, -1) for carbonization temperature, binder amount, and compaction pressure, ensuring low production costs. Therefore, this study proposes a locally implementable, technically reliable, economically attractive, and environmentally friendly solution to help meet the energy needs of local industries and households.

## Compliance with ethical standards

*Disclosure of conflict of interest*

No conflict of interest to be disclosed.

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