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Optimizing the Carbonization Temperature of Water Hyacinth Biochar by Proximate Analysis Using Response Surface Methodology

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Abstract

Water hyacinth is a locally available biomass with the potential to be converted into biochar, serving as a renewable energy source. In this report, response surface methodology (RSM) was employed to optimize the carbonization temperature during the preparation of water hyacinth biochar. Water hyacinth was carbonized in a furnace at varied temperatures (400 °C, 500 °C, 600 °C) for 90 minutes. Characterization of the biochar derived from water hyacinth by proximate analysis was determined, including moisture content, ash content, volatile matter, fixed carbon, and calorific value. The results of the biochar before optimization indicated that moisture content and volatile matter decreased with increasing carbonization temperature, while ash content, fixed carbon, and calorific value increased. After optimization, the proximate analysis of the biochar was determined, with the optimal carbonization temperature found to be 533.54 °C. At this temperature, the optimal moisture content was 6.50%, ash content was 25.53%, volatile matter was 24.80%, and fixed carbon was 43,16%. These findings demonstrate the feasibility of using RSM to optimize the preparation conditions of water hyacinth biochar.

Introduction

Biomass is an important renewable energy source used for energy storage and fuel production [1], [2]. One abundant biomass resource is water hyacinth (*Pontederia crassipes*), a type of aquatic plant commonly found in rivers, lakes, or swamps [3]. Due to the uncontrolled and rapid growth of water hyacinth (approximately 3% per day), it is often considered a weed [4]. The swift expansion of water hyacinth can result in the obstruction of river or water surfaces, leading to disruptions in air transportation, decreased plankton productivity, river constriction, reduced light penetration, disturbances in agricultural land irrigation and drainage, and various other issues [5], [6]. Nonetheless, water hyacinth is abundant in cellulose, with roughly 25-57% cellulose content of its dry weight, primarily concentrated in its stem [7]. This characteristic renders it suitable for biochar production, serving as a renewable alternative energy source.

Biochar, characterized by its high carbon content, is generated through the carbonization process of biomass at elevated temperatures ranging from 300 to 600 °C in a closed reactor with limited or no oxygen present. This process involves the thermochemical transformation of biomass into biochar [7], [8]. For this reason, determining the optimal carbonization temperature in the biochar production process is important, considering that the loss of oxygen and hydrogen content occurs during carbonization due to drying and the release of volatile compounds. This ultimately improves the quality of the fuel in the resulting biochar product.

Previous studies have investigated the optimization of carbonization temperatures for biochar production from various biomass sources. Biochar produced from durian skin waste at a carbonization temperature of 450 °C demonstrated the most effective carbonization temperature, resulting in a carbon content of 77.87%, moisture content of 0.01%, ash content of 18.18%, and volatile matter content of 3.94% [9]. Biochar derived from rice husks synthesized at 650 °C exhibited an estimated optimal calorific value, fixed carbon content, moisture content, volatile matter content, and ash content of 5665 cal/g, 83.2%, 0.06%, 3.1%, and 6.86%, respectively [10]. Meanwhile, biochar from palm fiber that was synthesized 400 °C had a maximum combustion rate of 2.87 mg/min [11]. In general, carbonization temperature is an important parameter for determining the quality of biochar. Identifying the optimal carbonization temperature is essential to produce biochar with favorable physiochemical characteristics.

It is critical to use an experimental design to assess the effects of carbonization temperature on biochar preparation. Many researchers utilized response surface methodology (RSM) to investigate the interactions of two or more parameters [12]. Response surface methodology (RSM) encompasses a range of techniques used to identify the most favorable conditions for achieving optimal outcomes via experimental approaches [13]. The RSM offers a time-saving alternative and enhances result prediction accuracy. It is a great alternative for optimizing biochar preparation because it allows maximum information to be extracted with little experimentation [14].

Based on a preliminary study of the effect of carbonization temperature, which is expected to enhance the quality of biochar, there has been relatively limited research conducted to determine the optimal carbonization temperature for producing biochar from water hyacinth. Therefore, a study was carried out to determine the optimal carbonization temperature based on the proximate analysis of biochar derived from water hyacinth using RSM.

Experimental Method

Preparation of biochar

Figure 1 illustrates the preparation process of water hyacinth biochar. The stems of the water hyacinth are collected, cleaned with water, and dried in the sun for 24 hours. The dried water hyacinth is ground until it becomes a powder. The water hyacinth powder is then put into a furnace and heated at different carbonization temperatures of 400 °C, 500 °C, and 600 °C for 90 minutes. Finally, the sample is sieved with a mesh size of 200 to obtain the final product in the form of biochar from water hyacinth.

Characterization of biochar

The biochar derived from water hyacinth was characterized through proximate analysis, which included assessments of moisture content, ash content, volatile matter, fixed carbon, and calorific value. To determine the moisture content, a fresh biochar sample was initially weighed and then dried in an oven at 105 °C for 24 hours, following ASTM D3173 standards. After drying, the sample was promptly weighed again to calculate the moisture content using the following formula:

$$\text{moisture content } (\%) = \left[\frac{initial\ weight - dry\ weight}{initial\ weight}\right] \times 100 \tag{1}$$

For ash content determination, a specific amount of dried biochar was weighed and placed in a muffle furnace, according to ASTM D3174. The furnace was heated to 750 °C and held at that temperature for 2 hours. Afterward, the sample was cooled in a desiccator, and the remaining residue, or ash, was weighed to determine the ash content with the following formula:

$$\text{ash content } (\%) = \left[\frac{weight\ of\ ash}{initial\ weight}\right] \times 100 \tag{2}$$

A specific amount of dried biochar was weighed and placed in a crucible with a lid for the measurement of volatile matter. The crucible was then heated in a muffle furnace at 950 °C for 7 minutes, in accordance with ASTM D3175. After cooling in a desiccator, the residue was weighed to determine the volatile matter content using the following formula:

$$\text{volatile matter } (\%) = \left[\frac{initial\ weight - weight\ of\ residue}{initial\ weight}\right] \times 100 \tag{3}$$

The fixed carbon content was calculated using the difference method by the following formula:

$$\text{fixed carbon } (\%) = 100 - [\%moisture\ content + \%ash\ content + \%volatile\ matter] \tag{4}$$

For the calorific value, the biochar sample was first dried at 105 °C for 24 hours to remove any moisture and then ground into a fine powder. A specific amount of the dried and ground

cleaned water hyacinth stems

water hyacinth stem drying process

dried water hyacinth stems that have been crushed

carbonization process of water hyacinth biochar at temperatures of 400 °C, 500 °C, and 600 °C

water hyacinth biocl

Figure 1. Synthesis procedure of water hyacinth biochar

biochar was weighed and placed in a bomb calorimeter, following ASTM D5865. After filling the calorimeter with oxygen, the sample was ignited, and the temperature change in the surrounding water was measured to determine the calorific value [15], [16], [17].

Proximate analysis using response surface methodology (RSM)

The experimental design for optimizing biochar production from water hyacinth was carried out using statistical analysis with response surface methodology (RSM). In the RSM method, a mathematical quadratic model is used to predict the optimal conditions for preparation and to understand the interaction between the preparation conditions. The RSM analysis involved measuring and analysing various factors, such as water content, ash content, volatile matter, and fixed carbon, to predict the optimal carbonization temperature and other preparation conditions. This approach allowed for the precise adjustment of variables to achieve the highest quality biochar. Ultimately, the quality of biochar from water hyacinth was evaluated based on the highest calorific value obtained.

Results and Discussion

Proximate analysis

The effect of carbonization temperature on the moisture content, ash content, volatile matter, fixed carbon, and calorific value of water hyacinth biochar is summarized in Table 1, which presents the proximate analysis of biochar produced at temperatures of 400 °C, 500 °C, and 600 °C. The moisture content is conducted to determine the hygroscopic properties of water hyacinth biochar, which is crucial in assessing the quality of the biochar produced. Moisture content refers to the amount of water present in a material [18], calculated by determining the ratio of water lost during the drying process compared to the initial weight of the biochar. As shown in Table 1, the rise of carbonization temperature resulted in a reduced moisture content of water hyacinth biochar. the highest moisture content was 8.03% at a carbonization temperature of 400 °C. At 500 °C, the moisture content decreased to 5.61% and the lowest moisture content was 4,98% at a carbonization temperature of 600 °C. Although the moisture content at 400 °C slightly exceeds the ASTM D-3302 standard limit of ≤6.2%, this may be due to the high initial moisture content of water hyacinth, an aquatic plant with a natural tendency to retain water. Additionally, variations in the drying and carbonization process, such as incomplete moisture removal during pre-drying or slight differences in heating rates, can influence the final moisture content in biochar.

Table 1. Proximate analysis of water hyacinth biochar

Parameter	Carbonization temperature			Method
	400 °C	500 °C	600 °C	
Moisture content (%)	8.03	5.61	4.98	ASTM D3173
Ash content (%)	22.47	26.57	28.59	ASTM D3174
Volatile matter (%)	33.91	22.52	15.69	ASTM D3175
Fixed carbon (%)	35,59	45.30	50.74	-
Calorific value (cal/g)	2924.17	3237.20	3371.88	ASTM D5865

When the moisture content is low, biochar will easily ignite, and have higher calorific values [19]. The moisture content observed suggests that the biochar produced can easily ignite, produces less smoke during combustion, and higher calorific values are expected and are of high quality. Carbonization reduces the hydroxyl functional groups (-OH) in the biochar. By reducing these groups, the biochar will cause a reduction in the moisture content of carbonized material [19]. As the carbonization temperature increases, the biochar has less moisture content, which can be desirable for biochar to be used for fuel, as less moisture improves combustion efficiency.

Ash is the inorganic residue left after the combustion of biochar, consisting of non-combustible materials that lack carbon [20]. It primarily contains minerals that do not volatilize during the carbonization process. The ash content of water hyacinth biochar increases with increasing carbonization temperature (Table 1). The lowest ash content is 22.47% at a carbonization temperature of 400 °C, rising to 26.57% at 500 °C, and reaching the highest value of 28.59% at 600 °C. These results agreed with those obtained by Gezahegn et al. [21] who noted a rise in the amount of ash for water hyacinth biochar during a carbonization temperature rise. The increase in the ash content was due to the gradual accumulation of inorganic materials and the residues left from the combustion of organic matter [22]. However, the ash content values in this study exceed the ASTM D-3174 standard of 8.00% and are also higher than those reported in previous studies on water hyacinth biochar. Zhang et al. [23] observed an increase in ash content for water hyacinth biochar, ranging from 22.65% to 43.04%, as the carbonization temperature rose from 250 °C to 550 °C. Similarly, another study found that the ash content of water hyacinth biochar rose from 11,10% to 42,40% when the carbonization temperature increased from 250 °C to 500 °C. The increase in ash content with rising temperatures is likely due to the volatilization of the organic matrix at higher carbonization temperatures [24]. Additionally, the ash content in biochar largely depends on the composition of the feedstock [25]. In the case of water hyacinth biochar, the significant ash content could be attributed to the lower lignin content of the biomass, which is composed of 48% hemicellulose, 20%

cellulose, and only 3.5% lignin [26]. Biomass with high lignin content generally produces biochar rich in carbon, while low lignin content results in biochar with higher ash content [27]. Volatile matter refers to the part of biomass that vaporizes and combusts during thermal processes, resulting from the decomposition of materials [28]. Higher carbonization temperature resulted in a decreased content of volatile matter (Table 1). The result shows that the lowest volatile matter is 15.69% at a carbonization temperature of 600 °C, 22.52% at 500 °C, and the highest volatile matter content is 33.91% at 400 °C. A high volatile matter suggests that the biochar ignites easily, burns quickly, and produces longer flames but low heating values [19]. It has been reported by Chen et al. [29] that biochar stability is linked to its volatile matter, with lower volatile content indicating higher stability. The decrease in volatile matter is attributed to the gradual deposition of minerals and the volatilization of lignocellulosic materials as the temperature increases. Additionally, the thermal breakdown of organic compounds, accompanied by the release of CO_2, leads to a reduction in volatile content and a corresponding increase in ash content [30].

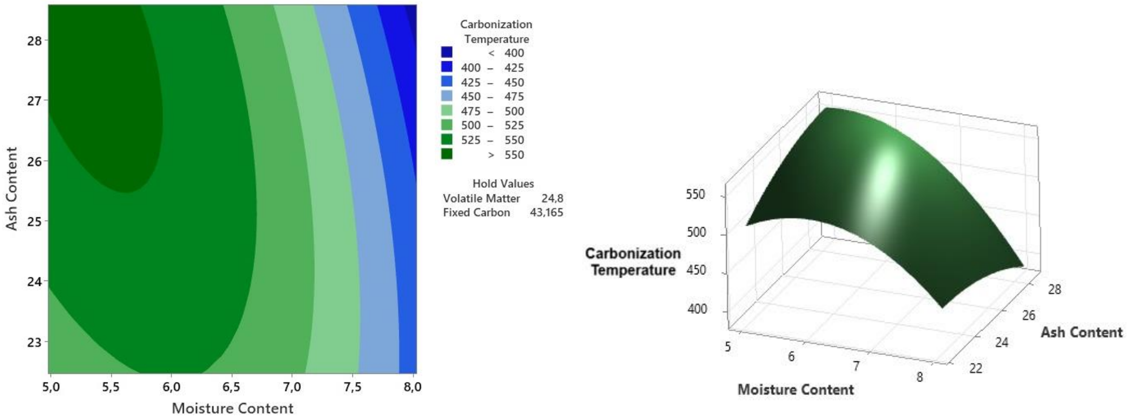
Fixed carbon refers to the solid, non-volatile carbon remaining in a material after all moisture, volatile matter, and ash content have been removed through carbonization. It is an important parameter in biochar, as it indicates the material's stability and energy content [31]. For biochar derived from water hyacinth, our results showed that the fixed carbon content increased with rising carbonization temperatures. Specifically, at a carbonization temperature of 400 °C, the fixed carbon content was 35.59%, which increased to 45.30% at 500 °C, and reached 50.74% at 600 °C. This trend can be attributed to the enhanced thermal decomposition at higher temperatures, where more volatile components are released, leaving a higher proportion of carbon in the biochar. The process, known as devolatilization, effectively concentrates the carbon content in the biochar as temperatures rise, contributing to its higher fixed carbon levels [32].

The calorific value indicates the amount of heat energy contained in a material. This study has found an increase in the calorific value of the water hyacinth biochar with increasing carbonization temperature (Table 1). In the current study, the calorific values of the water hyacinth biochar produced at carbonization temperatures 400 °C, 500 °C, and 600 °C were 2924.17 cal/g, 3237.20 cal/g, and 3371.88 cal/g, respectively. This finding is consistent with the work done by Altikat et al. [33], who noted in their research that the calorific values obtained at 400 °C were found to be lower compared to those at 600 °C. This suggests that higher temperatures more effectively promote the decomposition of biomass, resulting in products with higher energy.

Response Surface Methodology (RSM)

Effect of Carbonization Temperature on Moisture Content and Ash Content

Figure 2 shows the contour plot (2a) and surface plot (2b) of moisture content and ash content as affected by the carbonization temperature. In the contour plot (2a), the gradient from blue to green represents increasing carbonization temperatures from 400 °C to 550 °C. As shown, there is a notable reduction in moisture content as carbonization temperature rises. This trend indicates that higher carbonization temperatures enhance the removal of residual moisture



(a) (b)

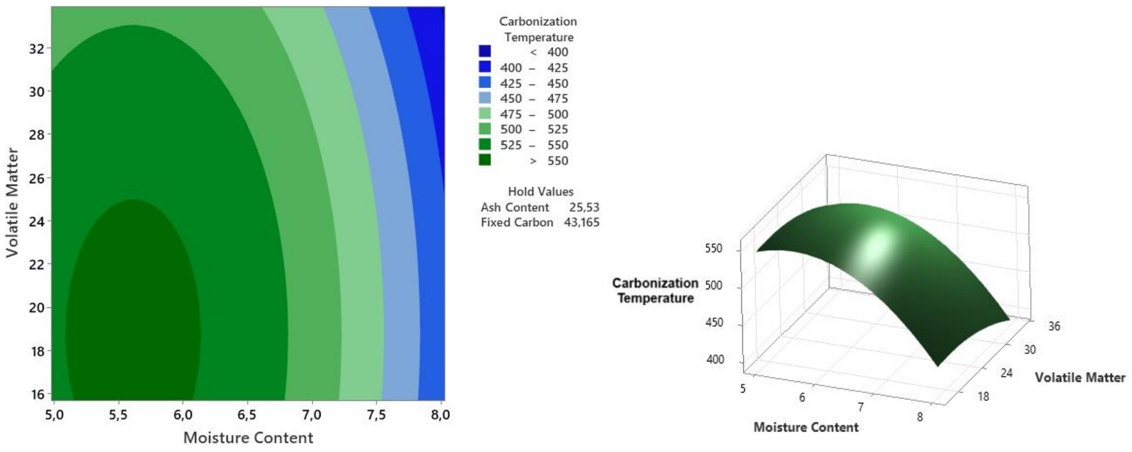
Figure 2. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on moisture content and ash content of water hyacinth biochar

from the biochar, consistent with the thermal decomposition process. The reduction in moisture content can be attributed to the initial stage of lignocellulose decomposition, as noted in previous studies [34], which indicate that moisture content is particularly sensitive to changes in pyrolysis temperature.

The surface plot (2b) further highlights the relationship between carbonization temperature, moisture content, and ash content. As the carbonization temperature increases, not only does the moisture content decrease, but the ash content tends to increase slightly, particularly at temperatures above 500 °C. This may be due to the higher thermal stability of inorganic components in the biochar, which remain as ash after the volatile components are driven off [29]. The 3D surface plot provides a clear visual of how these parameters are interrelated, demonstrating that optimizing the carbonization temperature can control the moisture and ash content in water hyacinth biochar.

Effect of Carbonization Temperature on Moisture Content and Volatile Matter

Figure 3 shows the contour plot (3a) and surface plot (3b) of moisture content and volatile matter affected by the carbonization temperature. In the contour plot (3a), the gradient from blue to green represents increasing carbonization temperatures from 400 °C to 550 °C. From the result, it shown that the carbonization temperature increases, the surface moisture content and volatile matter will decrease. As carbonization temperature rises, moisture is the first to be eliminated, followed by the gradual breakdown and release of volatile compounds. This relationship indicates that at higher temperatures, water hyacinth biochar will have both lower moisture content and reduced volatile matter. The same thing is also shown in paper [35] that carbonization temperatures between 400°C and 700°C cause moisture, hemicellulose, and



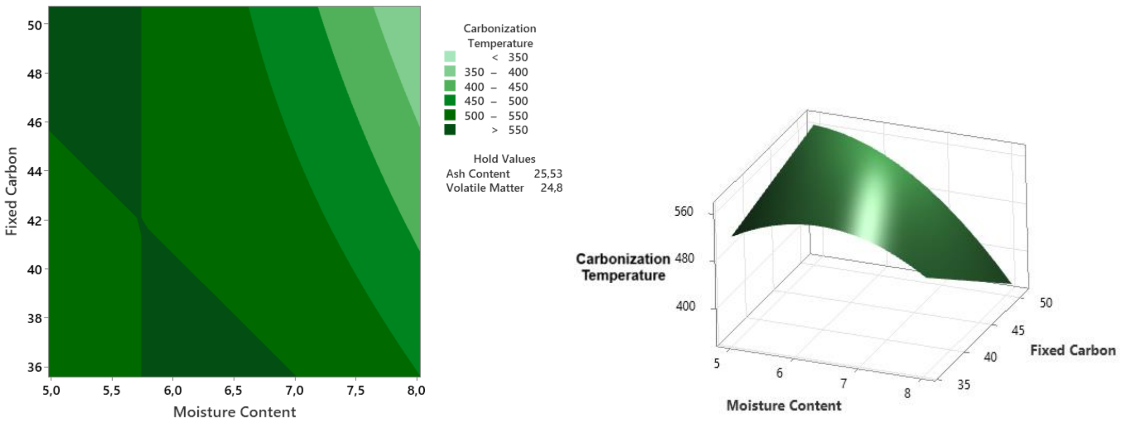
(a) (b)

Figure 3. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on moisture content and volatile matter of water hyacinth biochar

cellulose to be fully decomposed and volatilized. Therefore, the carbonization temperature increases, the surface moisture content and volatile matter will decrease.

Effect of Carbonization Temperature on Moisture Content and Fixed Carbon

Figure 4 shows the contour plot (4a) and surface plot (4b) of moisture content and fixed carbon affected by the carbonization temperature. The light green colour on the plot indicates a lower carbonization temperature (< 350 °C), while the darker green colour indicates a higher carbonization temperature (> 550 °C). Based on this result, increasing the carbonization temperature leads to a decrease in moisture content and an increase in surface fixed carbon. During the initial stages of carbonization, moisture is the first component to be released from the biomass. This stage primarily occurs at lower temperatures (typically below 200°C). As the



(a) (b)

Figure 4. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on moisture content and fixed carbon of water hyacinth biochar

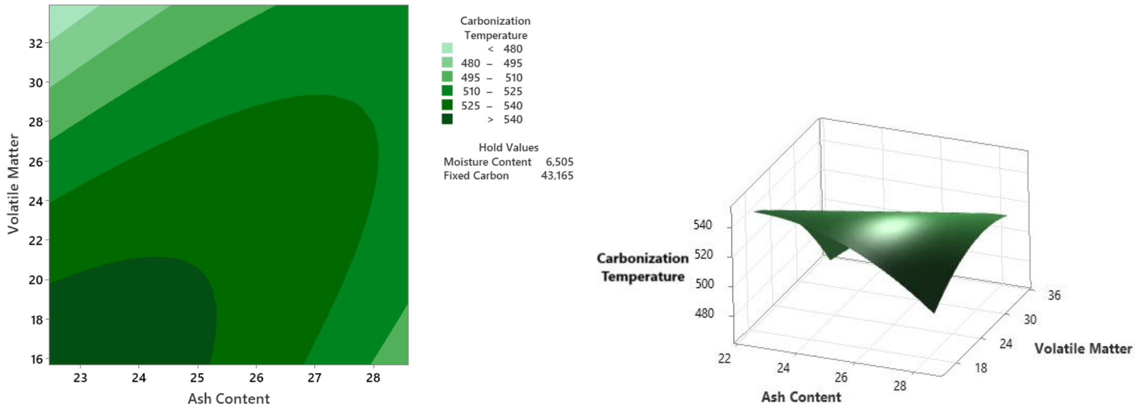
temperature rises, more water evaporates, and the biochar's moisture content decreases. As the carbonization process continues and temperatures reach higher levels, the biomass undergoes further decomposition, breaking down volatile organic compounds like hemicellulose, cellulose, and part of the lignin. These volatile components convert to gases and are released from the material, leaving behind a higher proportion of fixed carbon and ash content in the biochar [35].

Effect of Carbonization Temperature on Ash Content and Volatile Matter

Figure 5 shows the contour plot (5a) and surface plot (5b) of ash content and volatile matter affected by the carbonization temperature. The light green colour on the plot indicates a lower carbonization temperature (< 480 °C), while the darker green colour indicates a higher carbonization temperature (> 540 °C). Based on this result, increasing the carbonization temperature leads to an increase in ash content and a decrease in surface volatile matter. In paper [36], it was reported that the effect of temperature on ash content involves a complex interaction of thermal decomposition, volatilization, mineral transformation, and selective retention of certain components

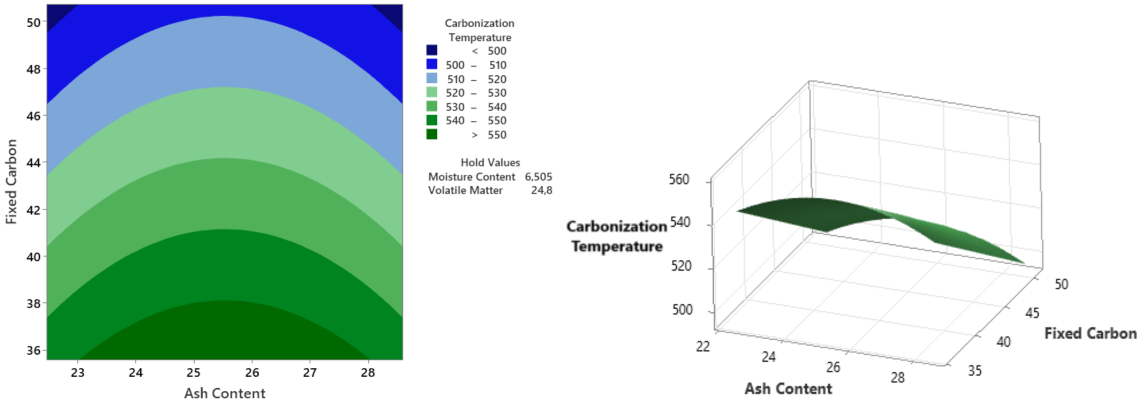
Effect of Carbonization Temperature on Ash Content and Fixed Carbon

Figure 6 shows the contour plot (6a) and surface plot (6b) of ash content and fixed carbon affected by the carbonization temperature. In the contour plot (6a), the gradient from blue to blue represents increasing carbonization temperatures from 500 °C to 550 °C. Based on this result, increasing the carbonization temperature leads to an increase in ash content and fixed carbon. At elevated temperatures, minerals in the biomass undergo structural transformations that can increase the stability and retention of these minerals as ash. This could mean that the



(a) (b)

Figure 5. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on ash content and volatile matter of water hyacinth biochar



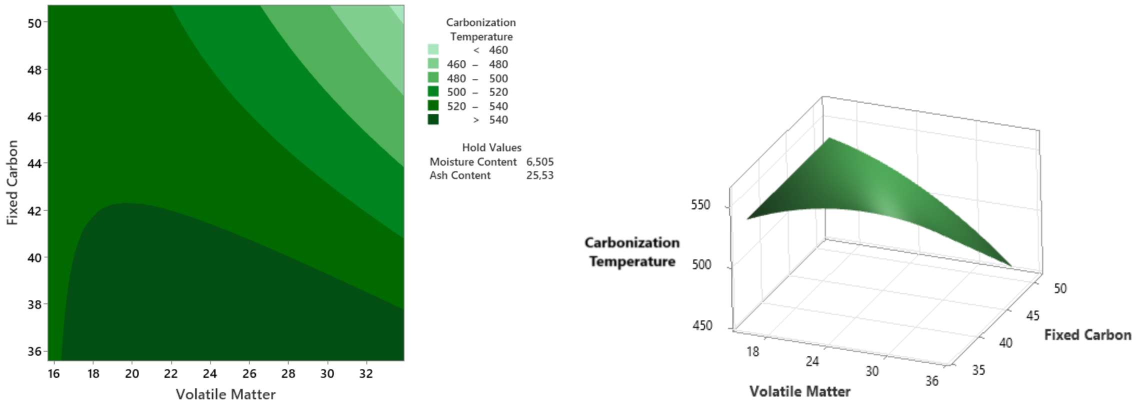
(b) (b)

Figure 6. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on ash content and fixed carbon of water hyacinth biochar

biomass retains more of its mineral content, contributing to higher ash content, especially if these minerals undergo sintering or fusing that prevents volatilization [37].

Effect of Carbonization Temperature on Volatile Matter and Fixed Carbon

Figure 7 shows the contour plot (7a) and surface plot (7b) of volatile matter and fixed carbon affected by the carbonization temperature. The light green colour on the plot indicates a lower carbonization temperature (< 460 °C), while the darker green colour indicates a higher carbonization temperature (> 540 °C). Based on this result, increasing the carbonization temperature leads to a decrease in volatile matter and an increase in surface fixed carbon. During carbonization, increasing the temperature drives off more volatile matter. As a result,



(c) (b)

Figure 7. (a) Contour plot and (b) surface plot of the effect of carbonization temperature on volatile matter and fixed carbon of water hyacinth biochar

the remaining material becomes richer in fixed carbon. At higher carbonization temperatures, volatile compounds are further reduced, concentrating the fixed carbon even more [38].

Optimization Carbonization temperature

Optimization of the carbonization temperature for water hyacinth biochar was conducted by plotting optimization using a surface methodology (RSM). The results of optimization using RSM produce a mathematical model that shows the relationship between measurement variables and the carbonization temperature, which is expressed in the following equation:

$$Y = -2828 + 565X_1 + 81X_2 - 1.6X_3 + 33.8X_4 - 21.5{X_1}^2 - 1.33{X_2}^2 - 0.151{X_3}^2 \\ -5.36X_1X_2 - 4.33X_1X_4 + 0.90X_2X_3 - 0.362X_3X_4 \quad (5)$$

where:

Y = Carbonization temperature (°C)

X_1 = Moisture content (%)

X_2 = Ash content (%)

X_3 = Volatile matter (%)

X_4 = Fixed carbon (%)

In this model, each variable and its interactions are included to predict the optimal carbonization temperature (Y) based on changes in these characteristics. Positive coefficients (e.g., for moisture content, ash content, and fixed carbon) suggest that as these variables increase, so does the temperature needed for optimal carbonization, while negative coefficients (such as for the squared terms) indicate diminishing returns or stabilizing effects at higher values. The interaction terms (e.g., X_1X_2, X_1X_4) reveal how combinations of these

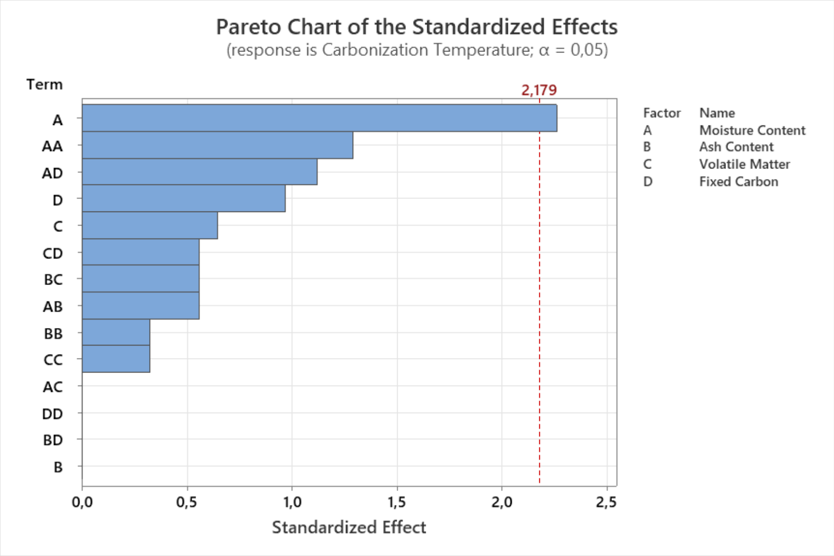


Figure 8. Graphical pareto analysis of process parameter effect on responses.

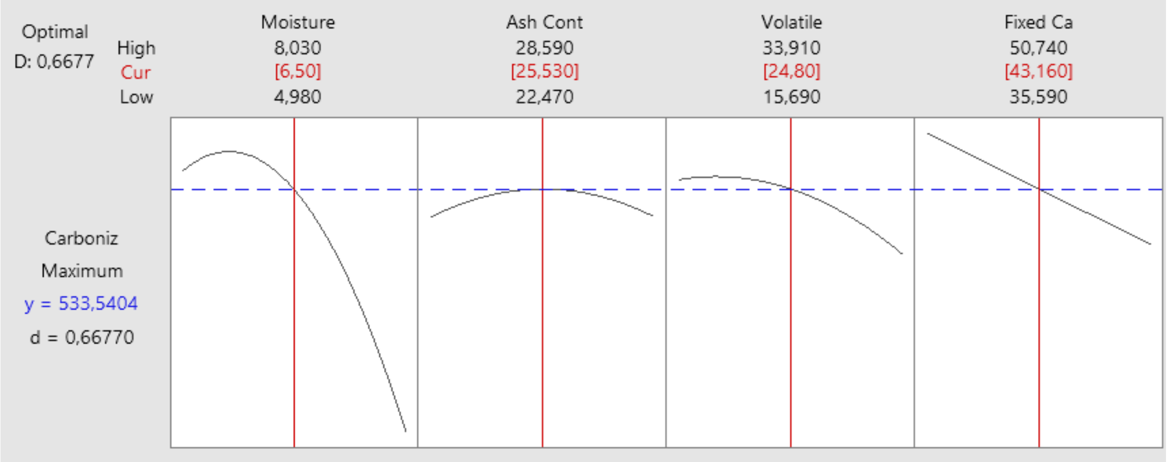


Figure 9. Optimization plot of carbonization temperature from water hyacinth biochar.

factors affect the temperature, highlighting the complex interplay between biomass composition and carbonization conditions. This model is crucial for identifying the precise temperature needed to optimize biochar properties and maximize carbonization efficiency.

The contributions of interaction effects were also investigated using the Pareto charts, shown in Figure 8. The Pareto chart highlights that the influence of contributing factors on specific outcomes is not evenly distributed. By identifying and focusing on the most significant factors first, the time required to achieve the desired results can be reduced, along with minimizing effort and costs [39]. In the case of determining the optimum carbonization temperature for biochar, using a Pareto chart allows for the identification of key factors, such as temperature, that have the greatest impact on biochar properties, enabling a more efficient optimization process. Figure 8 indicates that moisture content significantly impacts the carbonization temperature optimization of the water hyacinth biochar.

The optimum conditions were determined by analysing the entire response to variations in carbonization temperature using a profiling plot. Figure 9 illustrates the optimization graph for the carbonization temperature of water hyacinth biochar, showing that the optimum conditions differ for each response. The determination of optimum conditions involves balancing all responses. As depicted in Figure 9, the optimal values are 6.50% for moisture content, 25.53% for ash content, 24.80% for volatile matter, and 43.16% for fixed carbon. These values were obtained at an optimal carbonization temperature of 533.54 °C.

Conclusion

Based on the results, the carbonization temperatures have an important effect on the proximate analysis of water hyacinth biochar. Studies show that as carbonization temperature rises, biochar ash content, fixed carbon, and calorific value increase, while moisture content and volatile matter decrease. Optimization of the carbonization temperature for water hyacinth biochar using response surface methodology (RSM) indicated a moisture content of 6.50%, an optimum ash content of 25.53%, an optimum volatile matter of 24.80%, and an optimum fixed carbon of 43.16% at an optimum carbonization temperature of 533.54 °C. This study highlights the importance of optimizing carbonization temperature to enhance water hyacinth biochar's quality and calorific value. Adjusting the carbonization process parameters can significantly impact the biochar's characteristics, making it a more efficient and sustainable fuel source.

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