Research Article

Optimization of Microwave-Assisted Artificial Coalification for Maximization of Sawdust Hydrochar Production using Response Surface Methodology

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Abstract

Sawdust hydrochar was synthesized through microwave-assisted artificial coalification. The process temperature (180 – 220 °C), residence time (20 – 60 min) and biomass to water ratio (1:10 –1:20) were optimized within the given range for maximum hydrochar yield by applying a statistical technique of response surface methodology. The study revealed that process temperature and residence time had a significant effect on hydrochar yield with a higher F-value and lower p-value (<0.05). The maximum yield and higher heating value at the optimum process conditions of 183.4 °C, 20.5 min and 1:10 BWR were 78.9 % and 20.1 MJ/kg.

Keywords: Sawdust; Hydrochar; Microwave Synthesis; Artificial Coalification, RSM

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Introduction

Bio-carbon materials produced from lignocellulosic resources are a sustainable and long-term replacement for conventional fossil fuels [1], [2]. The low quality of biomass feedstock, which included low heating value and density with high oxygen and water content, resulted in a number of problems, limiting the use of direct utilization of lignocellulosic biomass [3]. Sawdust is an industrial by-product of wood machining operations which is lignocellulosic in nature with abundant availability. The structural polymers cellulose, hemicellulose and lignin are the main components of sawdust. Material processing is difficult because of the complex structure of the biomass. Management and conversion of sawdust into a useful product is gaining a lot of attention these days [4]. Sawdust can be carbonized in a subcritical water environment with higher carbon efficiency compared to pyrolysis. Artificial coalification (AC) is the process in which feedstocks can be effectively transformed into carbon-rich material known as hydrochar under mild temperature (180 – 250 °C) and auto-generated pressure (10 – 40 bar) under a compressed water environment [5]. Chemical-rich liquid and a lesser quantity of gases (< 5 %) are also the by-products of the AC process [6], [7]. In terms of the heating process, microwave-assisted heating differs significantly by the heating principle. Microwave is high-frequency electromagnetic radiation (300 MHz–300 GHz) [8]. This study aims to showcase sawdust as a potential feedstock for hydrochar synthesis which can be used as a solid fuel.

2. Materials and methods

2.1. Feedstock collection and size reduction

Sawdust was collected from nearby sawmills at Coimbatore. Sawdust was dried in a solar tunnel dryer. The dried material was sieved to 0.2 mm size for the uniform size distribution of particles and stored in an air-tight container for hydrochar synthesis.

2.2. Hydrochar synthesis

The hydrochar was synthesized in a microwave digestion system having a 2.45 GHz frequency and 1900 W capacity (Milestone Ethos Easy). Sawdust mixed with distilled water in a pre-determined ratio was put in the digestion vessel. The vessel was tightly sealed and placed inside the digestion system. After the process, the slurry was filtered using Whatman filter paper (No.3. Ashless) and the solid residue was dried in a hot air oven at 105 °C for 24 h. The yield of hydrochar was calculated using Eqn. 1.

$$Hydrochar\ yield\ (\%) = \frac{Mass\ of\ hydrochar}{Mass\ of\ sawdust}\ X\ 100 \quad (1)$$

2.3. Optimization of AC process

The efficiency of the AC process and hydrochar yield were mainly affected by three process parameters namely, process temperature, residence time and solid to liquid ratio. The effect of the abovementioned parameters on hydrochar yield was studied and optimized for maximum hydrochar yield using the Response surface methodology (RSM). The statistical optimization was carried out using Design Expert 13 Software. Since the three independent variables were equally spaced, Box-Behnken Design (BBD) was employed. The effect of three independent variables including process temperature, residence time and biomass to water ratio was evaluated and optimized in the range of 180 to 220 °C, 20 to 60 min and 1:10 to 1:20 respectively. Totally 15 experimental run with three center point replications were developed for the given levels.

2.4 Characterization of sawdust and hydrochar

The proximate analysis of sawdust was analyzed as per ASTM procedure to determine moisture (D3173), volatile matter (D3175) and ash content (D3174). The fixed carbon content of sawdust was calculated by subtracting the volatile and ash content from a total of 100. The Higher Heating Value (HHV) was determined using a digital bomb calorimeter (C200, IKA, USA).

3. Results and Discussion

The results of the optimization study to maximize the sawdust hydrochar are discussed below.

3.1. Optimization study

The process and conditions (Factor 1, Factor 2 and Factor 3) and the respective responses are given in Table 1. The yield of hydrochar was in the range of 48.34 to 73.01 %. The maximum yield was obtained for lower severity conditions (180 °C, 20 min and 1:15 BWR) and the minimum yield was obtained for higher severity conditions (220 °C, 60 min and 1:15 BWR). The model developed in terms of coded factors is given by Eqn. 2.

$$\text{Yield (\%)} = 49.81 - 1.12\,A^{*} - 2.34\,B^{*} - 0.66\,C + 2.01\,AB^{*} + 4.35\,BC + 0.12\,AC^{*} + 8.37\,A^{2*} + 8.99\,B^{2*} + 5.74\,C^{2*} \quad (2)$$

Where, A – Process temperature in °C, B – Residence time in minutes and C – Biomass to water ratio. The significance of the developed model was studied by an analysis of variance (ANOVA) test. The R^2 value of the developed model was 0.92. The process temperature and residence time had a significant effect on hydrochar yield with $p < 0.5$. The effect of biomass to water ratio and combined effect of time and biomass to water ratio was found to be insignificant ($p > 0.05$). The parameters had a significant effect on hydrochar yield was denoted by an asterisk symbol in the coded equation. The lack of fit was found to be insignificant with a p-value of 0.71. The optimized conditions for maximum hydrochar synthesis were found to be 183.4 °C, 20.5 min and 1:10 BWR.

Table 1. Experimental values for developed RSM – BBD model

Run	Factor 1 A: Temp (°C)	Factor 2 B: Time (min)	Factor 3 C: BWR[a]	Response 2 SDHC[b] Yield (%)
1	200	20	1:10	68.15
2	200	40	1:15	48.25
3	180	20	1:15	73.01
4	180	40	1:20	60.17
5	220	20	1:15	66.04
6	220	40	1:20	67.35
7	200	60	1:10	63.25
8	220	60	1:15	45.34
9	200	40	1:15	50.16
10	220	40	1:10	58.95
11	200	60	1:20	61.17
12	200	20	1:20	65.58
13	200	40	1:15	51.01
14	180	60	1:15	64.28
15	180	40	1:10	69.17

[a]BWR – Biomass to water ratio
[b]SDHC – Sawdust hydrochar

With the increase in process temperature, residence time and biomass to water ratio, the degree of decomposition of sawdust was enhanced [9]. The breaking of hydrogen bonding present in the hemicellulose and cellulose polymers was initiated at temperatures higher than 180 °C, which further improved the conversion of polysaccharides to monosaccharides like glucose [10]. The microwave irradiation accelerated the degradation of sawdust by increasing the temperature and pressure within the process, which aided in the rupture of lignin and cell walls due to hydrolysis and dehydration reactions [11]. The enhanced decomposition of feedstock at higher severity conditions was the reason behind the reduction in hydrochar yield.

The three-dimensional response surface plots to interpret the combined effect of process temperature and time (Fig. 1) and temperature and BWR (Fig. 2) which had a significant effect on hydrochar yield are shown below.

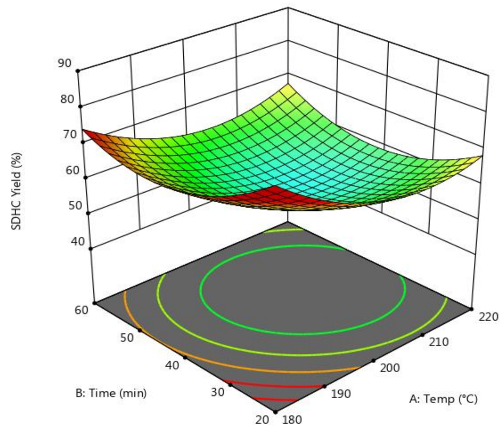

Fig. 1. Surface plot for Interactive effect of temperature and time on hydrochar yield.

From the plot, it was found that maximum hydrochar yield was obtained at lower process temperature and residence time. The results was in agreement with a study conducted by [12] for paddy straw hydrochar synthesis**.**

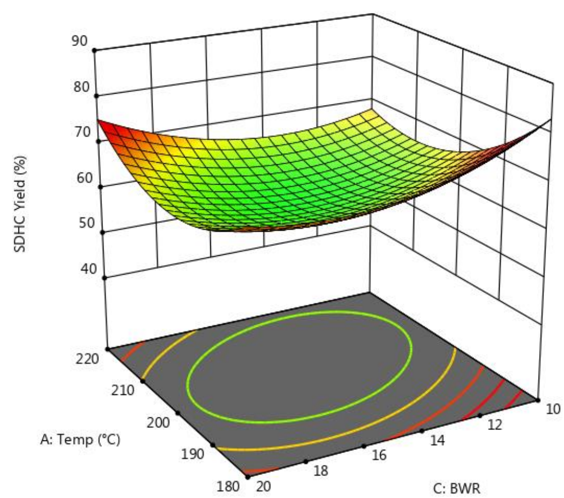

Fig. 2. Surface plot for Interactive effect of temperature and BWR on hydrochar yield.

The plot showed that the hydrochar yield was found to be maximum at lower process temperature and biomass to water ratio. A similar trend was observed for corn stalk hydrochar synthesis through microwave-assisted artificial coalification**.**

3.2 Characterization of hydrochar

Sawdust possessed higher volatile matter (77.6 %) and lower fixed carbon content (21.1 %). The yield and characterization of sawdust hydrochar produced at optimized condition are shown in Table 2. The volatile matter of sawdust hydrochar showed a reducing trend from 77.6 to 74.9 % at lower severity conditions. Due to hydrolysis, dehydration and decarboxylation reactions, the volatile compounds were escaped from the biomass matrix under the compressed water environment, which may be the reason behind the volatile matter reduction. Leaching of inorganic materials into the process liquid due to the respective temperature and pressure may be the reason for the ash content reduction from 1.3 to 1.2 %. The decreasing trend of volatile matter and ash content was in agreement with a previous study carried out for willow wood [13].

Table 2. Proximate properties and energy content of hydrochar produced at optimized conditions

Material	Hydrochar yield (%)	Volatile matter (%)	Ash content (%)	Fixed carbon (%)	HHV (MJ/kg)
Sawdust	-	77.6	1.3	21.1	19.7
SDHC	78.9	74.9	1.2	23.9	20.1

The energy value of hydrochar increased from 19.7 to 20.1 MJ/kg. This increasing trend was also found for rapeseed husk [14]. Enhanced degradation of sawdust was initiated by the synergetic effect of process temperature and pressure through different process reactions which may be the reason for improved energy content. Table 3 shows the yield and HHV of different hydrochar produced through microwave assisted artificial coalification and comparison with common forms of coal namely lignite and peak. The HHV of sawdust hydrochar with maximum yield was evident from the comparison with different studies [12-15]. Hydrochar produced through artificial coalification can be used as a solid fuel.

Table 3. HHV of hydrochar produced from various feedstocks

Material	Yield (%)	HHV (MJ/kg)	Reference
Sawdust	-	19.7	This study
Sawdust hydrochar	78.9	20.1	This study
Rice straw hydrochar	57.9	17.6	[12]
Rapeseed husk hydrochar	54.5	21.6	[14]
Lignite coal	-	16.9	[15]
Peat	-	15.4	[15]

Conclusion

A microwave mode of heating was applied to synthesize hydrochar from sawdust through artificial coalification. The process temperature, residence time and biomass to water ratio were optimized using RSM – BBD model to maximize the hydrochar yield. The maximum yield (78.9 %) was obtained at an optimum process temperature of 183.4 °C, the residence time of 20.5 min and biomass to water ratio of 1:10 BWR with HHV of 20.1 MJ/kg. The study reveals that microwave-assisted artificial coalification is an energy-efficient process to synthesize hydrochar with lesser residence time which can be used as a solid fuel.

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