

## Hydrothermal carbonization of rice husk for the production of hydrochar as a biofuel

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**Abstract:** Rice is a widely consumed food product worldwide, and it generates an agro-industrial waste product called rice husk, which currently lacks economic value and is poorly managed due to its difficult handling. Therefore, the objective of this work was to carry out the hydrothermal carbonization of rice husk to produce hydrochar as a biofuel using different reaction conditions. The hydrothermal carbonization of the rice husk was performed in a 1 L stainless steel reactor, model CF-1, using temperatures of 180, 190, and 200 °C and reaction times of 1, 2, and 3 h, respectively. The rice husk and the hydrochars were analyzed for pH, moisture content, total solids, volatile solids, ash content, electrical conductivity, elemental analysis, and higher heating value (HHV). The main results showed that the hydrochars had acidic pH values, moisture content below 10%, TS and VS values above 90% and 74%, respectively, high ash content and electrical conductivity, and HHV values greater than 15 MJ/kg. The thermochemical conversion of rice husks helps reduce the volume of a difficult-to-manage agro-industrial waste product and, in turn, provides a biofuel with a higher HHV than the initial biomass (14.34 MJ/kg). At the end of the study, it was observed that one hour was sufficient to improve the HHV compared to uncarbonized rice husks, due to the increase in C (> 38%) and low moisture (< 8%).

**Keywords:** rice husk, hydrothermal carbonization, hydrochar, biofuel

### Introduction

One of the most consumed agricultural products is rice, a cereal considered a staple food for approximately half the world's population (Nzereogu *et al.*, 2023). In Mexico, rice production during the 2022–2023 period was approximately 157,000 metric tons (SIAP, 2023a). Rice consumption yields two types of residues: straw and rice husk (RH). The husk is the outer layer of the rice grain (Bushra and Remya, 2020) and comprises 20% of the total grain weight (Naranjo *et al.*, 2023). It has a low bulk density, ranging from 96 to 140 kg/m<sup>3</sup> (Nzereogu *et al.*, 2023). RH is a type of lignocellulosic biomass composed primarily of cellulose (35%), hemicellulose (30%), and lignin (18%) (Nizamuddin *et al.*, 2018). Its physicochemical properties include moisture (4.5–10.8%), volatile matter (70.2–78.5%), ash (3.4–17%), and fixed carbon (3.4–19.8%) (Bushra and Remya, 2020). However, RH also has a high silica content (18–25%) of its total weight (Kordi *et al.*, 2023), which makes this residue difficult to manage and utilize in the same way as other agricultural residues.

As one of the alternatives for utilizing RH, hydrothermal carbonization (HTC) can be employed. This is a thermochemical process carried out in the presence of compressed water at temperatures ranging from 180 to 250 °C (Higgins *et al.*, 2020) and under autogenous pressures of around 4 MPa (Ighalo *et al.*, 2025). HTC is considered a sustainable process due to the operational energy savings and the elimination of sample pretreatment steps (Pauline and Joseph, 2020; Orzama-Hugo *et al.*, 2024; Selvaraj *et al.*, 2025). The main products resulting from the HTC are: 1) A carbon-rich solid phase known as hydrochar, 2) A liquid phase abundant in organic compounds referred to as process water (Ischia *et al.*, 2024), and 3) Synthesis gas, composed mainly of CO<sub>2</sub> and CO (Higgins *et al.*, 2020). Hydrochar has characteristics that make it attractive as a biofuel due to its higher heating value (HHV) and it has an HHV between 13 and 30 MJ/kg, depending on the initial energy content of the raw material (Romano *et al.*, 2023).

Recently, several applications of HCT using RH have been carried out under different operating conditions, among which the following stand out: 1) Nizamuddin *et al.* (2018) performed the HTC process with microwaves at 220 °C for 30 min. The hydrochar yield was 35.9% with a HHV of 16.10 MJ/kg, used as an adsorbent, carbon sequestrant, and soil remediator for agriculture. 2) Danso-Boateng *et al.* (2020), evaluated the adsorption capacity of the hydrochar obtained at 200 °C for 20 h, observing that after the process there is an increase in C and a decrease in O, going from 53.7% to 74.1% C. 3) Hossain *et al.* (2020) evaluated the properties of hydrochar as a fuel and adsorbent, obtaining hydrochar at 180 °C for 20 min of reaction with a HHV between 20.27 and 19.02 MJ/kg. 4) Yang *et al.* (2021) carried out a co-HTC with RH and coal from the Zhundong region at 200 °C for 2 h and the hydrochar had a composition of 98.9% SiO<sub>2</sub> in the

ash. 5) Li *et al.* (2021) added metal chlorides to the HTC feed water to evaluate the improvement of carbonization, and this addition directly affected the hydrochar yields from 28.97% to 47.10%.

Based on the previous analysis, it was identified that there is a need to evaluate the hydrothermal carbonization of rice husk at low and intermediate operational temperatures during moderate times. In this regard, the novelty of this work lies in performing the HCT at 180, 190, and 200 °C for 1, 2, and 3 hours, seeking a cost-effective process for obtaining hydrochar as a biofuel. Therefore, the objective of this work is to carry out the hydrothermal carbonization of rice husks for the production of hydrochar as a biofuel.

## Materials and Methods

The materials and methods used during this investigation are described below.

### *Collection and characterization of rice husk*

Rice husk (RH), provided by a food processing plant in Orizaba, Veracruz, Mexico, were used as raw material and this plant generates approximately 10,000 metric tons per month of rice husk. Usually, the processed RH consists of domestic rice plus rice imported from countries such as India, Thailand, Vietnam, and Pakistan (SIAP, 2023b). The RH were transported and stored in the Environmental Engineering Laboratory I of the Technological Institute of Orizaba for its analysis. As part of the study, a physicochemical characterization was performed in triplicate using the following methods: pH was determined by TAPPI T252 om-90; moisture and total solids content were determined by gravimetric method 2540 B; and volatile solids and ashes content were determined by gravimetric method 2540 E. Additionally, electrical conductivity (EC) was determined using a conductivity meter (HANNA® Instruments, HI98130, USA). Elemental analysis was also performed using an analyzer (Perkin Elmer, PE2400, USA) to determine carbon, hydrogen, nitrogen, and sulfur content. Oxygen content was determined by difference using CHNS and ashes.

### *Application of the hydrothermal carbonization of the rice husk*

Because the rice husk had a low moisture content, it was ground and hydrated at a mass ratio of 1:3 rice husk:water. After hydration, the moisture content of the rice husk was  $75.58 \pm 2.40\%$ , that represents the minimum moisture content (70%) for the hydrothermal carbonization process according to Pagés-Díaz and Huiliñir (2020). The hydrothermal carbonization was carried out in a 1 L high-pressure stainless steel reactor (Toption Instruments, CF-1, China), as shown in Figure 1. The reactor were operated at 180, 190 and 200 °C, with different reaction times 1, 2, and 3 hours, respectively.

### *Determination of the higher heating value*

From the elemental analysis and the ashes content, the higher heating value (HHV) was determined using the Equation 1, employed by Parikh and Chinniwala (2002):

$$HHV = 0.3491 * C + 1.1783 * H + 0.1005 * S - 0.1034 * O - 0.015 * N - 0.0211 * Ash \quad (1)$$

where: C = Carbon (%), H = Hydrogen (%), S = Sulfur (%), O = Oxygen (%), N = Nitrogen (%) and Ash = (%). All percentages were expressed by weight. HHV values are expressed in MJ/kg.

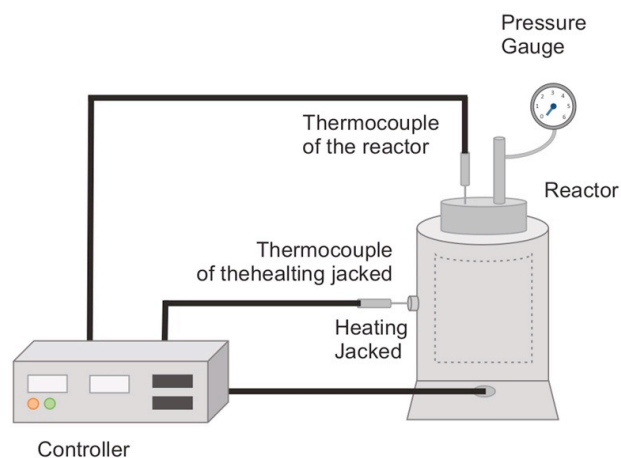


Figure 1. Hydrothermal carbonization reactor for obtaining hydrochar from rice husk

## Results and Discussion

### Characterization of the rice husk and the hydrochars

The following are presented the results of the physicochemical characterization of the RH and the hydrochars in the Table 1. Based on the pH measurement, the rice husk showed a value close to neutral, similar to 6.5 reported by Nwajiaku *et al.* (2018), which is a typical pH value for RH. However, when comparing the moisture content of the RH, a higher value was obtained in contrast to 7.5 of Hossain *et al.* (2020) and 9.3 of Steven *et al.* (2021). Although the moisture percentage of the RH analyzed in this study was  $10.30 \pm 0.09\%$ , it was found to be insufficient for the biomass to undergo the hydrothermal carbonization process, as this process requires at least 70% moisture according to Pagés-Díaz and Huiliñir (2020). Therefore, it was necessary to hydrate the RH as explained in the previous section. Furthermore, the  $78.71 \pm 0.19\%$  of VS content indicates the availability of organic matter present in the sample for carbonization. Regarding ash content, the value of  $21.29 \pm 0.19\%$  is higher than that reported by Nwajiaku *et al.* (2018) with 11.5% and Steven *et al.* (2021) with 17.6%. It is important to mention that, according to Steven *et al.* (2021), between 83.6 and 93.5% of the ash content corresponds to silica present in the RH. The presence of silica in the animal feed makes this type of waste difficult to manage. The limitations due to its high silica concentration are: 1) animals cannot digest it, and 2) it is highly resistant to burning (Ojeda-Rodríguez *et al.*, 2024). On the other hand, the electrical conductivity was  $373.67 \pm 8.08 \mu\text{S/cm}$ , a value close to  $330 \mu\text{S/cm}$  reported by Nwajiaku *et al.* (2018).

Regarding the behavior of the hydrochars, their pH values are below 4, as shown in Table 1, indicating acidic behavior due to the increased temperature and the reaction time of the HCT process. These values are consistent with Masoumi *et al.* (2021) findings that hydrochars have an acidic pH. It is noteworthy that the moisture content of the hydrochars is lower than that of the RH, because after the hydrothermal carbonization process, the process water was removed from the hydrocarbons by evaporation over two to three hours at  $105^\circ\text{C}$ . A moisture content of less than 10% is a key criterion for evaluating the HHV of a biofuel. The moisture content (%) of the hydrochars ranged from  $5.43 \pm 0.04$  to  $8.28 \pm 0.19$ , as shown in Table 1. The percentages of TS, VS and ash are proportional to the moisture content. It can be observed in the Table 1 that all hydrochars presented values above 90% for TS, obtaining ranges of  $74.44 \pm 0.16$  to  $75.36 \pm 0.05$  for VS (%) and  $24.64 \pm 0.05$  to  $25.56 \pm 0.16$  for ash (%). By comparing with the literature, Yang *et al.* (2021), carbonized RH at  $200^\circ\text{C}$  and 2, obtaining an ash content of 21.99%; while Nakason *et al.* (2017), obtained hydrochar at  $200^\circ\text{C}$  during 1, 2 and 3 h, reporting 20.9, 21.2 and 21.6% ash, respectively. Electrical conductivity shows an increasing trend with increasing temperature and time. This increase is due to the structural arrangement of the carbon atoms conferred by the HTC; at higher temperatures, the hydrochar structure becomes even more defined, allowing for improved electron flow (Selvaraj *et al.*, 2025). This is further supported by the amount of carbon obtained in the elemental analysis, as well as the conversion to a solid through the production of primary carbon (Ischia *et al.*, 2024).

Table 1. Physicochemical characterization of the rice husk and the hydrochars obtained under different operational conditions

		pH	Moisture (%)	TS (%)	VS (%)	Ash (%)	EC ( $\mu\text{S/cm}$ )
Rice husk		$6.79 \pm 0.01$	$10.30 \pm 0.09$	$89.70 \pm 0.09$	$78.71 \pm 0.19$	$21.29 \pm 0.19$	$373.67 \pm 8.08$
180 °C	1 h	$3.88 \pm 0.01$	$7.49 \pm 0.20$	$92.51 \pm 0.20$	$75.04 \pm 0.38$	$24.96 \pm 0.38$	$559.67 \pm 7.02$
	2 h	$3.69 \pm 0.00$	$7.89 \pm 0.54$	$92.11 \pm 0.54$	$75.03 \pm 0.22$	$24.97 \pm 0.22$	$729.67 \pm 2.52$
	3 h	$3.85 \pm 0.04$	$6.91 \pm 0.22$	$93.09 \pm 0.22$	$75.36 \pm 0.05$	$24.64 \pm 0.05$	$781.00 \pm 6.56$
190 °C	1 h	$3.69 \pm 0.03$	$6.17 \pm 0.01$	$93.83 \pm 0.01$	$75.06 \pm 0.07$	$24.94 \pm 0.07$	$678.33 \pm 16.01$
	2 h	$3.73 \pm 0.10$	$5.43 \pm 0.04$	$94.57 \pm 0.04$	$74.94 \pm 0.09$	$25.06 \pm 0.09$	$703.00 \pm 6.93$
	3 h	$3.68 \pm 0.01$	$6.55 \pm 0.12$	$93.45 \pm 0.12$	$75.08 \pm 0.16$	$24.92 \pm 0.16$	$789.33 \pm 41.86$
200 °C	1 h	$3.86 \pm 0.02$	$6.05 \pm 0.05$	$93.95 \pm 0.05$	$74.82 \pm 0.12$	$25.18 \pm 0.12$	$792.67 \pm 6.81$
	2 h	$3.84 \pm 0.02$	$5.78 \pm 0.05$	$94.22 \pm 0.05$	$74.44 \pm 0.16$	$25.56 \pm 0.16$	$766.67 \pm 12.90$
	3 h	$3.65 \pm 0.02$	$8.28 \pm 0.19$	$91.72 \pm 0.19$	$74.74 \pm 0.20$	$25.26 \pm 0.20$	$663.67 \pm 8.33$

### Elemental analysis and HHV evaluation

Regarding the elemental analysis of the RH, the C content was  $36.70 \pm 0.42\%$ , which contrasts with the value reported by Hossain *et al.* (2020), which was 25.9%. The percentages of HNS are similar to those found by Hossain *et al.* (2020), that were 4.7, 0.6 and 0.3%, respectively. However, Steven *et al.* (2021) mention a similar C content of 36.60%, but higher percentages of H (5.83%) and N (3.31%). The presence of CHON in the RH, especially C, is highly favorable for hydrochar, as it is a solid residue rich in C. The reason why the properties of the RH evaluated in this research differ from those reported in the literature can be attributed to the fact that the processed rice in this case study, and therefore its waste, consists of domestic rice plus rice imported from countries such as India, Thailand, Vietnam, and Pakistan (SIAP, 2023b); these are cultivated under different environmental conditions, including soil type, climate of each growing region, and planting and harvesting seasons.

Table 2. Results of the elemental analysis and the higher heating value of the rice husk and the hydrochar obtained under different operational conditions

		C (%)	H (%)	N (%)	S (%)	O (%)	HHV (MJ/kg)
Rice husk		$6.79 \pm 0.01$	$10.30 \pm 0.09$	$89.70 \pm 0.09$	$78.71 \pm 0.19$	$21.29 \pm 0.19$	$373.67 \pm 8.08$
180 °C	1 h	$39.14 \pm 0.94$	$5.25 \pm 0.05$	$0.57 \pm 0.02$	0	$30.08 \pm 0.97$	$16.21 \pm 0.50$
	2 h	$38.59 \pm 0.04$	$5.09 \pm 0.04$	$0.59 \pm 0.03$	0	$30.75 \pm 0.04$	$15.76 \pm 0.04$
	3 h	$39.39 \pm 0.46$	$4.69 \pm 0.06$	$0.58 \pm 0.00$	0	$28.69 \pm 0.52$	$15.75 \pm 0.29$
190 °C	1 h	$40.53 \pm 0.63$	$4.77 \pm 0.46$	$0.45 \pm 0.26$	0	$29.31 \pm 0.84$	$16.21 \pm 0.86$
	2 h	$39.71 \pm 0.57$	$4.92 \pm 0.09$	$0.25 \pm 0.01$	0	$30.05 \pm 0.65$	$16.03 \pm 0.38$
	3 h	$39.50 \pm 0.65$	$4.71 \pm 0.07$	$0.30 \pm 0.02$	0	$30.56 \pm 0.71$	$15.65 \pm 0.39$
200 °C	1 h	$39.41 \pm 0.24$	$4.77 \pm 0.03$	$0.30 \pm 0.00$	0	$30.33 \pm 0.28$	$15.71 \pm 0.15$
	2 h	$40.67 \pm 0.29$	$4.79 \pm 0.02$	$0.23 \pm 0.00$	0	$28.75 \pm 0.28$	$16.33 \pm 0.10$
	3 h	$41.00 \pm 0.41$	$4.71 \pm 0.03$	$0.26 \pm 0.02$	0	$28.77 \pm 0.40$	$16.36 \pm 0.14$

For the elemental analysis of the hydrochars, the carbon content ranged from  $39.14 \pm 0.94$  to  $41.00 \pm 0.41\%$  under the different operating conditions, as shown in Table 2. Yang *et al.* (2021) reported a very similar value of 41.56%, while Nizamuddin *et al.* (2018) and Hossain *et al.* (2020) found 47.2% carbon. Table 2 also shows a trend of decreasing H<sub>2</sub>O and O<sub>2</sub> concentrations as temperature and reaction time increase, due to the dehydration and decarboxylation reactions responsible for these decreases (Masoumi *et al.*, 2021; Yang *et al.*, 2021). The increase in C percentages and the decrease in H and O percentages, as a result of the HTC, are due to the decomposition of the RH; that is, as H and O are eliminated as H<sub>2</sub>O and CO<sub>2</sub>, C concentrates in the hydrochar. Finally, the N concentration was very low compared to CHO, while S was absent.

Evaluating the HHV, Table 2 shows that the RH was  $14.34 \pm 0.09$  MJ/kg, which is the reference value for analyzing the trend of using higher HTC severities to obtain hydrochar with HHV values higher than the initial biomass. The highest HHV value was  $16.36 \pm 0.14$  MJ/kg for hydrochar obtained at 200 °C for 3 h. Comparing these results with other authors, Nizamuddin *et al.* (2018) reported 16.10 MJ/kg for hydrochar obtained at 220 °C for 30 minutes, while Hossain *et al.* (2020) found a value of 20.27 MJ/kg at 180 °C for 20 minutes, but with a very low ash content, equivalent to 9.4%. The similarities or differences in the results of this research compared to other authors can also be attributed to the region where the rice was cultivated, the agricultural practices used for rice cultivation, and the physicochemical composition of the RH as a component of the hydrochars. Finally, the hydrochars processed by HTC from the RH can be used for heat and electricity generation through combustion, replacing fuels such as charcoal.

### Conclusions

The purpose of this work was to perform the hydrothermal carbonization of rice husk from a food processing plant to produce hydrochars as a biofuel using different operating conditions for hydrothermal carbonization. It is important to

highlight that rice husk is a residue with low moisture content, which had to be hydrated for the HCT, but possess valuable characteristics, including their carbon content, thus allowing for the utilization of a biomass that currently lacks an adequate management plan. Characterization of the hydrochar revealed that it is an acidic product with a high ash content, attributed to the amount of silica inherent in rice husks. HHV of the hydrochars values ranged from 15.71 to 16.36 MJ/kg, indicating that it is possible to improve the HHV of rice husk through HTC. The hydrothermal carbonization process proved to reduce the volume of rice husks and yield a biofuel such as hydrochar.

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