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## EVALUATION OF THE PROPERTIES OF BIOCHAR OBTAINED FROM RICE HUSK FOR ITS APPLICATION IN AGRICULTURAL SOILS

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### Abstract

*The increasing world population boosted the demand for basic needs such as energy, water, and food. In turn, agricultural production has increased, generating a large amount of agro-industrial waste every year. Globally, Brazil is the seventh-largest rice producer, and the State of Rio Grande do Sul is responsible for 70% of the rice production in Brazil. Rice husks are composed of organic compounds (cellulose, hemicellulose, lignin, and extractives) and inorganic elements that include potassium, calcium, magnesium, and sulfur. These inorganic elements are essential nutrients in the soil. Since natural degradation is slow due to the aromaticity of lignin, rice husk (RH) waste accumulates and poses an environmental threat, causing air and water pollution. In this context, in order to return the biochar to the soil, the properties of rice husk and rice husk biochar were evaluated. Parameters such as pH, true density, bulk density, porosity, identification of the composition and concentration of the elements, analysis of the functional groups, total carbon (TC), and cation exchange capacity (CEC) were performed on RH biochar samples. These samples were produced in a laboratory-scale pyrolysis rig, under a controlled atmosphere of Nitrogen (N), using three peak temperatures (350 °C, 450 °C, and 550 °C) and three soak times (30, 60, and 120 min). Based on the results obtained in the characterization analyses of the biochar, as well as the yield values found, it is concluded that the best pyrolysis temperature for the production of biochar from rice husks, for application in soils, is 550 °C.*

**Keywords:** rice husk, agro-industrial waste, biochar, agricultural soils.

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## Introduction

Soil is a finite resource that is essential for the survival of humanity and the preservation of the planet's biodiversity. To meet the basic needs of a world population of 7 billion inhabitants, about 1.87 billion hectares of land are used for cultivation (USGS, 2018). Unfortunately, however, data show that, since the second decade of the 21st century, about 30% of the world's soil has been degraded (FAO, 2015) due to improper practices, which negatively affect their agricultural properties. Such degradations include increased compaction and erosion, loss of microorganisms and organic matter, and change in water holding capacity and pH.

However, agriculture needs soils with fundamental characteristics such as organic matter, some minerals, and capillary water (Brassley and Richard, 2016). Crops need fertile soil with a sufficient composition of nutrients, as well as favorable environmental conditions on the site. Macronutrients (C, H, O, N, P, K, S, Ca, and Mg) and micronutrients (B, Mn, Cu, Zn, Fe, Mo, and Cl), available in soils, are essential for crop growth and development (Merladete, 2022).

Thus, soil conditioners (Babla *et al.*, 2022) and substrates (which are materials that help to deal with some inadequate soil conditions) contribute to the nutritional dynamics of the growing place, in order to create the ideal conditions that satisfy the physical, chemical and biological needs of crops. (Schafer and Lerner, 2022). An example of a substrate that can be used for this purpose is 'biochar', a material that is produced by the carbonization of biomass.

Biochar is produced by the thermal decomposition of carbonaceous materials from different kinds of biomass, with limited oxygen and at relatively low temperatures. It is made up of high-carbon organic matter and inorganic components, which include minerals such as Ca, Mg, and K. Biomass carbonization has been used in agriculture to reduce the volume of waste and compromised land area. As a secondary objective, biochar is used as soil amendment for plant cultivation (Yong *et al.*, 2016).

Biochar differs from charcoal in that it is intentionally produced to improve soil adsorption properties, ion exchange capacity, low density (Oelbermann, Berruti and Lévesque, 2020). Many studies suggest that adding this material to the soil improves its sorbent capacity, avoiding the percolation of organic and inorganic pollutants due to the high surface area of biochar (Tong, 2014). Research conducted between 2000 and 2018 suggests increases in soil nutrient content and plant growth, but there is no data for long-term experiments.

According to the World Bioenergy Association (2017), the agricultural sector contributes at least 10% of the global supply of biomass waste, which is estimated between 3.6 to 17.2 billion tons of biomass waste per year (World Bioenergy Association, 2017). According to the United States Department of Agriculture - USDA (2018), the worldwide rice production in 2017-2018 was in the

range of 488.5 million tons (USDA, 2018). Brazil is the seventh-largest rice producer in the world and, according to the grain harvest bulletin of the National Supply Company (CONAB), it is expected to harvest about 10 million tons of rice in the 2022-2023 cropping season (National Supply Company, 2023).

**Table 1.** Research by authors on the use of rice husk pyrolysis in different countries and applications.

Author	Country	Biochar application	Parameters pirólise
Zhang, Kaikaiet; Sun, Peng; Faye, Marie Christine As; Zhang, Yanrong. (2018)	China	Chlorobenzene degradation	Temperature of 550°C, heating rate 5 °C min <sup>-1</sup> , Flow of N 0.2 L m <sup>-1</sup> and residence time of 2h
Abbas, Qumber; Liu, Guijian; Yousaf, Balal; Ali, Muhammad Ubaid; Ullah, Habib; Munir, Mehr Ahmed Muitaba; Liu, Ruijia. (2018)	China	Investigation of the influence of various pyrolysis conditions and biomass particle sizes on products	Temperatures of 300, 400, 500, 600 and 700°C, heating rate 1-10 °C min <sup>-1</sup> , flow of N 0.002 - 0.2 L m <sup>-1</sup> and residence time of 1h
Connor, David O.; Peng, Tianyue; Li, Guanghe; Wang, Shuxiao; Duan, Lei; Mulder, Jan; Cornelissen, Gerard; Cheng, Zhenglin; Yang, Shengmao; Hou, Deyi. (2018)	China	Soil: Application of biochar and biochar modified with the addition of sulfur in mercury contaminated soil	Temperature of 550°C, heating rate 15 °C min <sup>-1</sup> , flow of N 0.2 L m <sup>-1</sup> and residence time of 2h
Li, Xiao Xiao; Chen, Xubing; Siwirska, Marta Weber; Cao, Junjun; Wang, Zhao Long (2018)	China	Root change based on sand and grass growth	Temperature of 400°C, heating rate 10°C min <sup>-1</sup> , unreported N flow and 5h residence time
Gunal, Elif; Erdem, Halil; Çelik, İsmail. (2018)	Turkey	Soil: Effects on silt water retention in slime and clay soils	Temperature of 500°C, heating rate 10°C min <sup>-1</sup> , unreported N flow and residence time from 4 to 6h
Cornelissen, Gerard; S, Jubaedah; Nurida, Neneng L; Hale, Sarah E; Martinsen, Vegard; Silvani, Ludovica; Mulder, Jan. (2018)	Norway	Soil: Reduced acidity and increased productivity	Temperature of 400 and 500°C, heating rate 26°C min <sup>-1</sup> , unreported N flow and 2h residence time
Oladele, S. O. O; Adeyamo, A. J.; Awodun, M.A.A. (2019)	Nigeria	Soil: Influence of rice husk biochar and inorganic fertilizer on soil nutrients	Temperature of 350°C, heating rate 1.5°C min <sup>-1</sup> , unreported N flow and residence time of ± 1h
Dunnigan, Lewis; Morton, Benjamin J.; Hall, Philip Anthony; Kwong, Chi Wai.(2018)	Australia	Evaluation of polycyclic aromatic hydrocarbon (HPAs) emissions	Temperature of 400, 500, 600, 700 and 800°C, heating rate 15°C min <sup>-1</sup> , Flow of N 0.25 L m <sup>-1</sup> and unreported residence time

Considering that 20 to 22% of the grain of rice is husk, a considerable amount of waste is generated (Pandey, 2010). Rice husk (RH) is the outer layer of the grain and is generated when rice is milled along the steps of grain processing, being essentially composed by silica, cellulose e hemicellulose between 55-60% and lignin 22% (Kaviyarasu *et al.*, 2016). In soils, lignin decomposes slowly because it has a complex chemical structure, making the natural process of decomposition of rice husk (RH) very slow. Therefore, it is common to see piles of rice husks exposed to the open air in rural areas (Kappler *et al.*, 2018).

It can also be considered that the use of rice husk biomass through pyrolysis has already been studied in different countries of the world in different applications, including the soil (as shown in Table 1). In this context, the purpose of the present research is reinforced, to study the feasibility of applying the biochar produced by the pyrolysis of rice husks for use as a soil conditioner or fertilizer.

The influence of pyrolysis parameters, as well as the available characterization methods, will be better explained in the following items.

#### *Influence of pyrolysis parameters on the characteristics of pyrolysis products*

Many authors have studied the influence of the operating conditions of a pyrolysis reactor (such as temperature) on the properties of biochar (Tan Zhongxin, 2018, Kim *et al.*, 2018). Tan *et al.* (2018) observed that the nitrogen retention rate, obtained by the tracer <sup>15</sup>N method, decreases from 45.23% to 20.09% in biochar as the pyrolysis temperature increases from 400 °C to 800 °C, due to the volatilization of organic material (Tan Zhongxin, 2018). Tomczyk, Sokołowska and Boguta, (2020) showed, through a literature review, the influence of pyrolysis parameters in relation to the increase in temperature and the decrease in the production of biochar or charcoal, caused by the increase in the volatile content.

Kim *et al.* (2018), on the other hand, reported the elemental analysis and pH variation in biochar produced from rice straw in different temperature ranges (Kim *et al.*, 2018). The authors verified pH of 8.2, 10.5, and 10.3 for pyrolysis temperatures of 300 °C, 550 °C, and 700 °C, respectively. And the elemental analysis showed that the carbon content ranged from 53.5% to 60.8%.

The operating conditions adopted for the reactor configuration in this work were based on the values obtained in exhaustive research on the most frequent operating conditions used in benchtop pyrolysis reactors. The literature shows that the temperature range for obtaining biochar from several types of biomass, including rice husk, varies from 300 °C to 800 °C (Zang *et al.*, 2018; Abbas *et al.*, 2018; Connor *et al.*, 2018; Li, Xiao *et al.*, 2018; Günal, Elif *et al.*, 2018; Cornelissen *et al.*, 2018; Oladele *et al.*, 2019; Dunnigan, Lewis *et al.*, 2018).

### Standard methods of biochar characterization for application in the soil in Brazil and worldwide

There is still no worldwide unified standardization concerning biochar characterization methodology. However, some of the methods used are:

- European Biochar Certificate: European non-profit foundation based in the Netherlands. The standardized methods for biochar characterization follow the DIN standard (Deutsches Institut für Normung).
- International Biochar Initiative: International Organization with approximately 400 paying members from 34 countries, located in the United States. Some of the analyses standardized by them for biochar characterization follow the American Society for Testing and Materials (ASTM) standard.

In Brazil, there is no specific standard for biochar. However, some normative instructions can serve as a basis to guide production standards of this material, being:

- Normative Instruction SDA-MAPA 35 (MAPA, 2006), which determines necessary tolerances and criteria for soil conditioners and fertilizers and shows some possible paths to be followed for application in the soil.
- Normative Instruction - MAPA 53 (MAPA, 2013), which establishes the minimum requirements for organic and mineral fertilizers produced in Brazil.
- In addition to these normative instructions, there is the Manual of Official Analytical Methods of Fertilizers and Correctives prepared by the Ministry of Agriculture, Livestock, and Supply of 2017, which describes all the necessary methods and analyses for a product to be considered a soil fertilizer.

In this context, this study aims to: evaluate the feasibility of converting rice husk biomass into a carbonaceous material suitable for use as a soil conditioner or fertilizer; compare characteristics of the biochar obtained with a commercial soil conditioner expanded perlite; contribute to a closed life cycle for RH biomass, responding to environmental, social, and economic aspects of sustainable development (Kappler *et al.*, 2018; Souza *et al.*, 2018; Souza *et al.*, 2019).

### **Materials and methods**

The materials used in this work and their origin are presented in Figure 1.

Table 2 summarizes the parameters measured in the rice husk, biochar and perlite samples, as well as the methods or instruments used to carry out the analyses.

Sample:	Sample:	Sample:
Rice husk	Biochar	Expanded perlite
		
Origin:	Origin:	Origin:
Rice industry, city of Santo Antônio da Patrulha, RS.	Produced through carbonization, in an inert atmosphere in the Laboratory of the University of Vale do Rio dos Sinos	Vitreous volcanic rock. (Predominant composition in silica. Marketed as an organomineral fertilizer for the physical improvement of agricultural soil )

**Figure 1.** Samples of rice husk, carbonized RH, and commercial expanded perlite.

**Table 2.** Description of which techniques were used in samples of in natura rice husks, biochar and perlite.

Parameter/Technic	Husks	Biochar	Perlite	Method/ Instrument
Apparent density	✓	✓	✓	Teixeira <i>et al.</i> (2017)
True density	✓	✓	✓	Pycnometer (helium gas) model AccuPyc II 1340
Porosity	✓	✓	✓	Raji, 1987
X-Ray Fluorescence - XRF	✓	✓	✓	Espectrometer, by energy , mark EDX 720 HS
Superficial Area	✓	✓	✓	Brunauer-Emmett-Teller
Hydrogen potential	✗	✓	✓	IN SDA n° 17 (BRASIL,2007)
Total Carbon	✓	✓	✓	Equipment LECO SC 144-DR – burning temperature 1300°C
Analysis of Funcional Groups	✓	✓	✓	Infrared Spectroscopy with Fourier Transform and Attenuated Total Reflectance
Cationic Exchange Capacity	✗	✓	✓	IN SDA n° 17 (BRASIL,2007)
Fixed carbon yield	✗	✓	✗	Protásio <i>et al.</i> (2014)

### Biomass used: rice husk - preparation and characteristics

Rice husks are generated during the peeling stage in the grain processing. About 20% of the gross grain mass becomes a non-hazardous and non-inert Class IIA waste according to the ABNT, 2004 classification.

In the laboratory, the RH sample was washed with deionized water to remove dust particles and dried at 105°C for 24 hours to remove moisture (Zhang, Kaikai *et al.*, 2018; Abbas, Qumber *et al.*, 2018).

The characterization was carried out at the Materials Characterization and Valuation Laboratory (LCVMat) at the University of UNISINOS. Fresh rice husks were analyzed for: the specific surface area, determined by nitrogen adsorption using the Brunauer-Emmett-Teller - BET method; true density with Pycnometer (helium gas) model AccuPyc II 1340; analysis of functional groups by Fourier Transform Infrared Spectroscopy and Attenuated Total Reflectance (FTIR-ATR). Porosity was measured by the method of Raji (1983) which uses the apparent density (measured pycnometer) and the true density (measured by pycnometer), according to Equation 1.

$$n = 1 - \left( \frac{\text{apparent density}}{\text{true density}} \right) \times 100 \quad \text{Equation (1)}$$

### Production of biochar

The pyrolysis experiments of RH were carried out in a horizontal quartz reactor, heated by an electric furnace, in an inert atmosphere. The nitrogen flow was 0.2 L m<sup>-1</sup> (Güenal, Elif *et al.*, 2018; Cornelissen *et al.*, 2018; Oladele *et al.*, 2019) and the heating rate 10 °C min<sup>-1</sup> (Abbas, Qumber *et al.*, 2018; Li, Xiao *et al.*, 2018; Güenal, Elif *et al.*, 2018; Grutzmacher, Priscila *et al.*, 2018; Wagas, M. *et al.*, 2018). The selected pyrolysis temperatures were 350 °C, 450 °C, and 550 °C (Zhang, Kaikai *et al.*, 2018; Abbas, Qumber *et al.*, 2018; Connor *et al.*, 2018; Li, Xiao *et al.*, 2018; Güenal, Elif *et al.*, 2018; Cornelissen *et al.*, 2018; Oladele *et al.*, 2019; Dunnigan, *et al.*, 2018).

Approximately 25g of biomass sample was placed into the reactor in a 410 stainless-steel crucible. A rotameter controlled the flux of Nitrogen at 100 cm<sup>3</sup> min<sup>-1</sup>. The pyrolysis temperature was measured by a K-type thermocouple and controlled by a PLC model NOVUS N1200. The stainless-steel crucible had an orifice so that the thermocouple could be placed inside the biomass sample. The pyrolysis was carried out in two ways for each temperature and each residence time, resulting in 18 tests. The pyrolysis gases passed through impingers filled with cotton (used as a filter) and placed in a thermal box with cold water. The condensable oil was trapped into the impingers, and the permanent gas was released into the atmosphere.

Slow pyrolysis was carried out to obtain the carbonization of the biomass and transformation into biochar. Slow pyrolysis is characterized by slower heating rates to ensure that volatiles are released with sufficient time to recombine with the biochar, maximizing yield and producing a carbon-dense biochar. (FOONG *et al.*, 2020; AL-RUMAIHI *et al.*, 2022).

#### Evaluation of the yield of pyrolysis products

The yield of the biochar samples obtained from RH biomass was calculated to evaluate the influence of soaking time. The results were obtained according to Protásio *et al.* (2014), using Equation 2.

$$\text{Yield (\%wt)} = \left( \frac{\text{mass of biochar}}{\text{mass of biomass}} \right) \times 100 \quad \text{Equation (2)}$$

Analysis of Variance (ANOVA) was applied to statistically analyze the results of the yields obtained. Therefore, the Tukey test with a confidence interval of 95% was used to identify significant inequalities within each treatment found in the ANOVA, in which statistical differences  $P \leq 0.05$  were considered.

#### Characterization of biochar and expanded perlite

The biochar samples and the expanded perlite were analyzed for specific surface area (determined by Nitrogen adsorption using the Brunauer-Emmett-Teller - BET method), true density (determined using a helium gas Pycnometer model AccuPyc II 1340), analysis of functional groups (by Fourier Transform Infrared Spectroscopy and Attenuated Total Reflectance FTIR-ATR).

Cation exchange capacity (CEC) and the pH were determined by the Normative Instruction n° 17 of the Agricultural Defense Secretariat - SDA of the Ministry of Agriculture, Livestock, and Supply – MAPA, 2007.

The apparent density was performed according to Teixeira *et al.* (2017). This method consists of obtaining the mass by weighing the sample on a dry basis in a cylinder of known internal volume. Porosity was calculated according to Raij (1983).

The characterization was carried out at the Materials Characterization and Valuation Laboratory (LCVMat) at the University of UNISINOS.

## Results

The results obtained from the methodology used are presented as follows.

### Analysis of the yield

Table 3 presents the pyrolysis yields at different immersion times, followed by the Tukey test results. The results at the temperature of 350 °C were the most satisfactory in relation to the other temperatures, staying between 48%, with a time of 30 minutes, and 47%, with 60 and 120 minutes of immersion. At the temperature of 450 °C, the yield of the carbonizations of 30, 60, and 120 minutes was maintained at an average of approximately 40%, having a decrease of 1% with the maximum immersion time. At 550 °C, the highest yield observed was with an immersion time of 30 minutes, with an approximate drop of 1% for the time of 60 Minutes and 2% in 120 minutes. It is possible to observe, regarding the yield, that there is no statistically significant difference ( $P \leq 0.05$ ) in the immersion times between 30 and 120 minutes by the Tukey test. On the other hand, the yield results suggest that, as the pyrolysis temperature increases, the yield decreases. These results agree with studies carried out by other authors about biomass pyrolysis (Dunnigan, Lewis *et al.*, 2018; Usman, *et al.*, 2015; Muñoz, *et al.* 2017; Kieling, 2016). The biochar samples selected for further characterization are the samples obtained at 30 minutes of soaking time. The criteria for this decision were the higher biochar yield and the lower consumption of electricity than the other treatments.

**Table 3.** Statistical analysis of pyrolysis yields at different immersion times.

	Yelds (%)		
	Immersion Time (30 min)		
	Biochar 350 °C	Biochar 450 °C	Biochar 550 °C
1 <sup>st</sup> sample	48.71	40.03	40.42
2 <sup>nd</sup> sample	48.07	40.32	37.32
Average	48.39 b	40.17 a	38.87 a
	Immersion Time (60 min)		
	Biochar 350 °C	Biochar 450 °C	Biochar 550 °C
1 <sup>st</sup> sample	47.49	40.36	37.41
2 <sup>nd</sup> sample	47.19	40.14	37.72
Average	47.34 b	40.25 a	37.56 a
	Immersion Time (120 min)		
	Biochar 350 °C	Biochar 450 °C	Biochar 550 °C
1 <sup>st</sup> sample	46.72	39.28	37.28
2 <sup>nd</sup> sample	47.08	39.17	37.36
Average	46.90 b	39.23 a	37.32 a

\* Average followed by the same letter do not differ statistically from each other, by Tukey's test ( $P \leq 0.05$ )

Characterization of rice husks, biochar samples, and expanded perlite

Table 4 summarizes the comparative results between rice husks, expanded perlite, and rice husks biochar produced at 350 °C (B350 °C), 450 °C (B450 °C), and 550 °C (B550 °C) with a soaking time of 30 minutes for all temperatures.

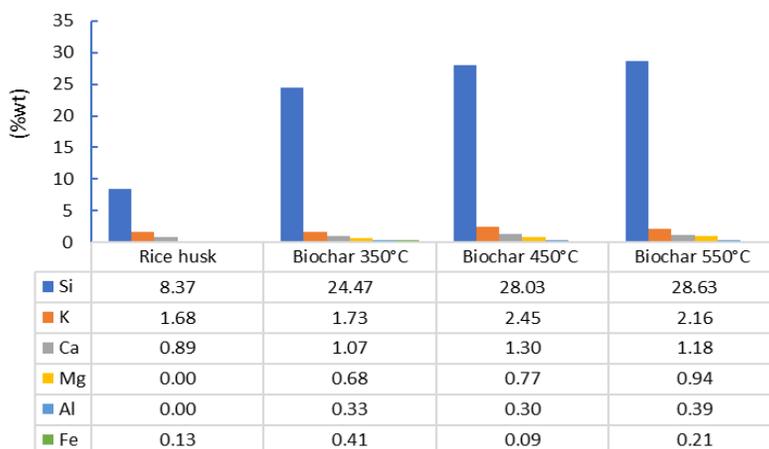
Expanded perlite is commercialized in Brazil as an organomineral fertilizer, resulting from the combination of mineral and organic fertilizers (MAPA, 2005). Organomineral fertilizers are used to improve the physical properties of the soil, such as aeration, and increase the interaction of minerals with plants. Thus, expanded perlite was used in this research as a source of comparison between its properties and that of the produced biochars.

The porosity of expanded perlite is 92%, close to that of the biochar samples, which averaged at 94%. This porosity of perlite justifies the fact that it is marketed as a soil conditioner with an aeration effect. Both the alkaline pH of perlite and biochar are following what is specified in the Chemical Products Safety Information Sheet (FISPq) and demonstrate that the results of porosity of biochar samples are favorable for the same application as perlite.

**Table 4.** Results of characterization analyses.

Parameter	Units	Rice Husk	B350 °C	B450 °C	B550 °C	Expanded perlite
Surface area	m <sup>2</sup> g <sup>-1</sup>	0.700	5.544	20.720	163.525	1.053
pH	-	-	6.4	7.7	8.7	7.4
CEC	mmol kg <sup>-1</sup>	-	43	47	49	63
True density	g cm <sup>-3</sup>	1.293	1.420	1.478	1.542	1.292
Apparent density	g cm <sup>-3</sup>	0.091	0.077	0.089	0.089	0.107
Porosity	%	-	95	94	94	92
Total carbon	%	27.974	44.706	52.242	53.562	0.063

The semi-quantitative elemental chemical composition of the rice husk and biochar samples is shown in Figure 2. The results were presented in elementary form with no estimation concerning detectable and non-detectable elements, because the limits are given in an elementary form and not in oxides, according to the Ministry of Agriculture, Livestock, and Supply (MAPA) normatization. Results are expressed as a percentage of mass. As presented, silicon (Si) content is the main inorganic element in the samples. Fernandes *et al.* (2018), Kieling (2016), and Calheiro *et al.* (2016) also report Si as one of the main components of rice husk, being a topic of study in several areas (GAI, Xiapu *et al.*, 2014). Furthermore, with the increase in the pyrolysis temperature, there was an increase in the concentration of inorganic elements — and, consequently, Si — in mass percentage.



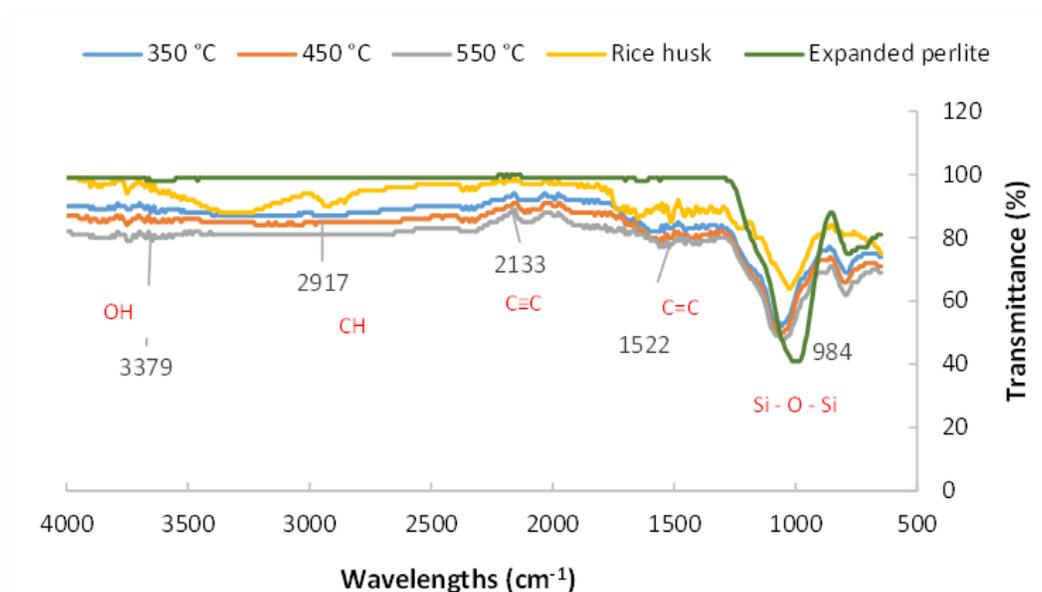
**Figure 2.** Elements found through the analysis of semiquantitative FRX in rice husk and biochar samples.

Because the FTIR-ATR is a qualitative analysis, it was only possible to identify the existing functional groups in biomass, biochar, and perlite samples through their spectra. The spectra of the biochar and expanded perlite samples are very similar, especially in the fingerprint region, between the wavelengths from 1500 to 500  $\text{cm}^{-1}$ , where the peak pattern usually varies more from one compound to another. (CAREY, 2011). In both, in this region, there is an intense vibrational stretching in the band between 1060 and 1000  $\text{cm}^{-1}$ , followed by another place-intensity stretching, between 1100 and 800  $\text{cm}^{-1}$ , characteristic of the absorption of the Si-O-Si bond (Weller, 2015; Zhao *et al.*, 2018; Zhang *et al.*, 2018).

The biochar samples show a low intensity stretching in the region between 3650 and 3200  $\text{cm}^{-1}$  associated with the absorption of the O-H (WELLER, 2015; CAREY, 2011) hydroxyl group, followed by the C-H in the bands between 3200 and 2840  $\text{cm}^{-1}$ , which suggest the presence of alcohols. The absorption of  $\text{C}\equiv\text{C}$  in the low-intensity stretching between 2260 and 2100  $\text{cm}^{-1}$  is identified in the biochar produced at a temperature of 450 °C and in the expanded perlite, which was not possible to observe in the other samples, this fact may be associated with the Alkynes group, which has a symmetrical bond, with identical or very similar groups, not absorbed in the infrared (Pavia, 2013).

The presence of the aromatic ring in the biochar samples can be identified between the bands of 1600 and 1450  $\text{cm}^{-1}$  with the presence of the C=C group (DIAS *et al.*, 2016; LIU *et al.*, 2013). The finding of the presence of aromatic rings, of structural origin of lignin, even after the process of thermochemical transformation of in natura rice husks into biochar, reinforces its relevance as a conditioner in cultivable soils, and the greater the degree of aromaticity. Thus, the recalcitrance of labile carbon in soils will be higher, due to the higher degree of aromaticity (TRAZZI *et al.*, 2018),

which in turn allows for greater stability in the quality of the soil-plant system (NUNES and Rezende, 2015).



**Figure 3.** Spectral profile of biochar, raw RH, and expanded perlite samples.

## Discussion

In this section, discussions are held on the results obtained.

### Influence of different temperatures (350 °C, 450 °C, and 550 °C) on the properties of the biochar

The values for the surface area (SA) of biochar samples produced at temperatures of 350 °C and 450 °C were, respectively, 5.5442 m<sup>2</sup> g<sup>-1</sup> and 20.7202 m<sup>2</sup> g<sup>-1</sup>, and are similar to the results presented by Zhao *et al.* (2018), who found 20.25 m<sup>2</sup> g<sup>-1</sup> in RH biochar samples at a temperature of 700 °C. The biochar samples produced at the pyrolysis temperature of 550 °C presented the highest SA among the samples (163.5246 m<sup>2</sup> g<sup>-1</sup>). This difference in SA of RH biochar samples between one temperature and another was also identified by Qin *et al.* (2017) at temperatures of 300 °C, 400 °C, 500 °C, and 600 °C, with surface areas of 0.99 m<sup>2</sup> g<sup>-1</sup>, 1.89 m<sup>2</sup> g<sup>-1</sup>, 13.38 m<sup>2</sup> g<sup>-1</sup>, and 152.4 m<sup>2</sup> g<sup>-1</sup>, respectively. These authors attributed the increase in surface area at higher temperatures to a higher volatilization rate and, consequently, loss of organic compounds (Kappler *et al.*, 2018; Crombie *et al.*, 2013). The same behavior was observed between biochar samples and other types of biomasses, such as apple tree branches, which, at pyrolysis

temperatures of 400 °C, 500 °C, and 600 °C, presented SA values of 11.9 m<sup>2</sup> g<sup>-1</sup>, 58.6 m<sup>2</sup> g<sup>-1</sup>, and 208.69 m<sup>2</sup> g<sup>-1</sup>, respectively (Jindo, K. *et al.*, 2014).

The results obtained in the analysis of biochar samples indicate that, for the studied temperature range (350 °C to 550 °C), the increase in pyrolysis temperature is proportional to the increase in:

- Surface area (Abbas, Qumber *et al.*, 2018; Quin *et al.*, 2017; Xiaofeng *et al.*, 2017);
- CTC and pH (Kim *et al.*, 2018, Quin *et al.*, 2017; Xiaofeng *et al.*, 2017);
- True density and carbon content (Kim *et al.*, 2018; Quin *et al.*, 2017; Xiaofeng *et al.*, 2017).

#### Potential applications of RH biochar on agricultural soils

The total organic carbon content of the three biochar samples was three times above the minimum threshold specified by MAPA, suggesting the feasibility of its application in soils, contributing to carbon fixation through its relatively slow process of biodegradation, which results in its mobilization (Menegale *et al.*, 2015).

The spectra of the biochars show, qualitatively, smaller transmittance peaks in the bands of 1600 and 1450 cm<sup>-1</sup> than the spectrum of rice husk, suggesting that the aromatic rings present in rice husk increased after pyrolysis. In addition to its higher degree of aromaticity, it also has higher labile carbon recalcitrance (organic compounds are more easily mineralized by microorganisms) in the soil (Trazzi, *et al.*, 2018), which in turn allows for higher stability in the quality of the soil-plant system (Nunes, 2005; Rezende *et al.*, 2011).

Sustainable socioenvironmental and economic development depends on the availability of material and energy resources (Tukker *et al.*, 2013; Wiedmann *et al.*, 2013). The traditional linear model has limited the environmental component to theoretic models (Heshmati *et al.*, 2013; Szita *et al.*, 2017) that accept the generation of waste and emissions as inevitable due to the entropy of processes, considering these externalities and causing material and energy losses. This model is unsustainable, and the closed cycle models may present alternatives to obtain economic and socioenvironmental gains simultaneously (Winkler *et al.*, 2011).

Therefore, it is plausible to assume that the results found for the organic carbon content, the presence of aromatic rings, and the presence of macronutrients and micronutrients in the biochar samples strengthen the importance of returning these materials to the soil. In this way, in addition to increasing the return of nutrients to the soil, it also captures and stores carbon. The stability of carbon in biochar produced from different types of biomass has been studied by different authors (Rosas *et al.*, 2021; Raya-Moreno *et al.*, 2017; Xu *et al.*, 2018; Grutzmacher *et al.*, 2018). Its application in the sustainable management of soil and plants in the perspective of circular economy (De Corato *et al.*, 2021) has also been studied. As an example, Kappler *et al.* (2018) conducted a study with green coconut shells. The authors state that by ensuring a closed cycle,

as proposed, there is a reduction in the use of fertilizers and pesticides, and, thus, a reduction of global warming. As "the value chain is expanded by making industries be created, materials be regenerated, and energy to be recovered, building this closed cycle in the production chain."

The pH found in the biochar samples was alkaline, suggesting that its application as a conditioner in acidic soils could intervene beneficially in the pH-soil relationship, increasing its potential ((Singh *et al.*, 2018; Amoakwah, *et al.*, 2017; Pandit, N. Raj *et al.*, 2018; Kappler *et al.*, 2018; Cornelissen *et al.*, 2018).

The elements Mg, Ca, and K are present in the RH biochar samples. These elements are considered essential macronutrients for soils and may improve CEC properties (Teixeira *et al.*, 2017), suggesting an increase in the fertility of soils with the addition of RH biochar. Moreover, Si is the main element identified in the semiquantitative FRX analysis. Though it is not called a soil nutrient, it is beneficial to plants (Molina Junior, 2017). The intensive and successive cultivation leads to a decrease in the concentration of Si in the soil. Consequently, it may affect the productivity of some crops (Menegale *et al.*, 2015). Silica deficiency in plants makes them more susceptible to tipping and fungal infections (Taiz *et al.*, 2017). Although it is not considered a nutrient for the soil, silicon is classified as beneficial for plants (Molina Jr., 2017) and can provide greater rigidity to plant cells, in addition to controlling pests and increasing the productivity and/or quality of crops. (Cassel *et al.*, 2021). According to Bakhat *et al.* (2018), soluble Si in the cytosol releases metabolic pathways that cause the production of jasmonic acid, increasing plant defenses against insects, fungi and bacteria. Intensive and successive cultivation leads to a decrease in the concentration of Si in the soil, reflecting on the productivity of some crops (MENEGALE *et al.*, 2015). Silicon deficiency in plants makes them more susceptible to damping-off and fungal infections (Taiz *et al.*, 2017). The Brazilian Ministry of Agriculture, Livestock and Supply does not specify a limit on the silicon content for soil conditioners. It is important to note that although the elements Ca, K, Mg, Fe and Si are present in the biochar, the availability of these elements to plants will depend on factors such as hydrogenic potential and cation exchange capacity of the place. The process occurs with the absorption of soil compounds through the roots and incorporation into organic compounds, which are essential for growth. Cations taken up by plant cells form complexes with organic compounds, in which the cation becomes bound to the complex by non-covalent bonds. Non-covalent bonds, which are defined by electrostatic and coordinated valence bonds. Therefore, plants are able to assimilate cationic macronutrients such as potassium, magnesium and calcium. (Taíz *et al.*, 2017).

Rice husk biochar samples presented low CEC values when compared to the findings of other authors. The disagreement between the CEC values found in this study and those of others exposes the divergences in the methods applied by different authors. In Brazil, there are divergences in studies that used the same normative instruction to analyze the CEC. Normative Instruction n°17 of MAPA (2007) establishes 5 grams of sample for 2 grams of activated carbon, as well as the use of calcium acetate for substrates or soil conditioners.

Regarding total organic carbon content (TOC), MAPA does not establish a minimum threshold for soil conditioners, but it does for fertilizers (IN n° 25). For instance, the lowest TOC is 8% for organometal fertilizers and 15% for mixed organic fertilizers and compost, while the minimum sulfur content is 1% for mixed organic fertilizers and composts. Thus, three of the biochar samples produced in this study meet the requirements to be used as organic fertilizers in terms of carbon content but would not meet the minimum sulfur content. Perlite did not meet either of the two criteria.

It is relevant to point out that the porosity, although not within the thresholds established by the MAPA, is a substantial property for the quality of arable soils. In soils with limited aeration, plant growth is affected by metabolic processes, water absorption, and nutrients. Low porosity values cause O<sub>2</sub> deficiency, which interferes with the organic matter decomposition process, causing some microorganisms to start using the anaerobic form to survive, reducing the decomposition of some substances, which can cause toxic effects to plants (Molina Junior, 2017). Because of its porosity, when biochar is added to soils, it has the potential to increase, in the long term, their water holding capacity (Trazzi *et al.*, 2018) and contribute to the reduction of nitrous oxide emissions from the soil (Petter *et al.*, 2012).

### Conclusions

Regarding the characterization of carbonized rice husks, it has been noted that the granular porosity, bulk density, true density, specific surface area, and cation exchange capacity have raised as the pyrolysis temperature increased, suggesting that the best pyrolysis temperature to produce biochar from rice husks, aiming for soil application, is 550 °C.

The properties of RH biochar, reinforced by the theoretical framework studied, suggest that its application in agricultural soils to improve fertility is interesting.

The similarity in the spectral profile of the samples of expanded perlite and biochar highlights the potential use of biochar, produced by pyrolysis of rice husks, as a soil-improving agent, mainly as an aerating agent as well as for raising the pH.

From the perspective of the potential characteristics analyzed in the case of biochar, the conclusion is that it falls within the concept of soil improver, as a product that promotes the improvement of physico-chemical or biological activity of the soil, which can recover degraded or nutritionally unbalanced soils, in accordance with approved standards for specifications and guarantees of soil improvement for agriculture.

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