Dias Júnior, Ananias Francisco; Pereira Pirola, Lucas; Takeshita, Saly; Queiroz Lana,
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CERNE, vol. 22, núm. 4, 2016, pp. 423-430
Universidade Federal de Lavras
Lavras, Brasil

CERNE

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HIGROSCOPICITY OF CHARCOAL PRODUCED IN DIFFERENT TEMPERATURES

ABSTRACT: This research aimed to evaluate hygroscopicity of charcoal produced under four different final carbonization temperatures. For evaluation of hygroscopicity charcoal samples were conditioned in environments with controlled temperature and relative humidity, using saturated salt solutions. The final carbonization temperature significantly influenced the products yields and the properties of charcoal. The charcoal produced in the final temperature of 750 °C showed the highest adsorption capacity of water, indicated by the moisture content after conditioning, in the higher relative humidity environment. Correlations were observed between adsorbed moisture and the porosity of charcoal produced.

HIGROSCOPICIDADE DO CARVÃO VEGETAL PRODUZIDO EM DIFERENTES TEMPERATURAS

RESUMO: Este estudo teve por objetivo analisar a higroscopicidade do carvão vegetal produzido em quatro diferentes temperaturas finais de carbonização. Para avaliação da higroscopicidade as amostras de carvão vegetal foram acondicionadas em ambientes com temperatura e umidade relativa controladas, utilizando-se soluções salinas saturadas. Os rendimentos dos produtos da carbonização e as características do carvão vegetal foram significativamente influenciados pelas temperaturas finais de carbonização. O carvão vegetal produzido sob a temperatura final de 750 °C apresentou a maior capacidade de adsorção de água, indicada pela umidade após o acondicionamento. Verificaram-se correlações entre a umidade adsorvida e a porosidade do carvão vegetal produzido.

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INTRODUCTION

The production of charcoal in Brazil meets the demand of various sectors, especially the steel mill and metallurgy. Furthermore, charcoal is also used in the cement industry, pharmaceutical industry, cooking food (barbecue) and in soil additives to optimize fertilization (BRITO, 1990; BERGERON et al., 2013).

The wood carbonization process occurss in the presence of controlled amount of oxygen at a temperature above 300 °C. It may be influenced by factors such as residence time of the product in the reaction zones, final temperature, pressure and heating rate (BRITO, 1990; DERMIRBAS, 2004; DUBOC et al., 2007).

Therefore, the way the carbonization is driven interferes actively on the final quality of charcoal, such as density, strength, moisture content, fixed carbon content, ash content and volatile matters (OLIVEIRA et al., 2010; DIAS JÚNIOR et al., 2015a). According to Trugilho and Silva (2001), the final carbonization temperature is one of the variables most related to the final quality of charcoal. It directly influences the set of reactions that occurs during the carbonization process generating products with different physical and chemical characteristics (DERMIRBAS, 2004).

One of the inherent properties of charcoal is its high moisture absorption capacity, as a hygroscopic material. Its hygroscopicity can significantly influence other properties, e.g. mechanical resistance, which decreases due to expansion of steam when heated; an excessive release of smoke; a low ignition power; more energy consumption, retarding the ignition and lowering the combustibility (BRAHAN, 2002; DIAS JÚNIOR et al., 2015b.). The presence of significant amount of water can also increase spending during transportation, often based on the final weight of the product (SOMERVILLE; JAHANSHAHI, 2015). Moreover, the charcoal adsorption capacity is used to capture radioactive elements such as Radon (Rn) in India, Japan and some European countries (KARUNAKARA et al., 2015).

According to Luengo and Emmerich (1997), during the first hours after its production, charcoal can absorb from 4% to 16% of its weight in water, decreasing with time. The authors also point out that the charcoal hygroscopicity decreases with increasing end temperature. Andrade and Della Lucia (1995) suggest the correlation between hygroscopicity and high porosity of charcoal, given by the difference between its actual and its apparent densities. The same authors claim that the porosity of charcoal relates to the final carbonization temperature, and also regards the effect of wood density

which charcoal came from and the carbonization velocity (ANDRADE; DELLA LUCIA, 1995; ASSIS et al., 2016).

The hygroscopic capacity is a relevant characteristic, mainly because of its influence on the properties and use of the charcoal, implying an increasing demand of studies on the product behavior correlated to its final use, and regarding to the exposure to different production and storage environments. For instance, once out of the production, the charcoal goes through several processes that range from cooling, sieving selection, packaging and transport (often in open trucks), until reaching the final destination. Under such circumstances, there are different environmental exposures with varied climatic conditions.

To supply the many applications of charcoal, this research analyzed the hygroscopicity of charcoal produced at four different final temperatures of carbonization. Moreover, the characterization induced the determination of gravimetric yields and of the properties of charcoal produced.

MATERIAL AND METHODS

The wood came from a 7-years old Eucalyptus urophylla x Eucalyptus grandis crop. The wood had the following average values: basic density = 0.416 g·cm^{-3}; extractives content = 5.79%; total lignin content = 25.80% (dry base); ash content = 0.15% and porosity = 65.71%.

Production and properties of charcoal

Samples of pre-dried wood at 103 ± 2 °C were carbonized in a metal cylinder inside a muffle furnace. The carbonization was carried out at final temperatures of 400 °C, 550 °C, 650 °C and 750 °C. The amount of wood used for each repetition was 200 ± 10 g, carbonized at a heating rate 1.67 °C·min^{-1}, remaining sixty minutes after achieving the stipulated final temperature (DIAS JÚNIOR et al., 2015a).

Several properties were determined as, gravimetric yields of charcoal, pyroligneous fluid and non-condensable gases (by difference) in relation to dry wood. The yield of fixed carbon (FCY) was calculated by multiplying the values of charcoal yield by the fixed carbon content, itself determined by charcoal immediate analysis according to NBR 8112 (ABNT, 1986).

The apparent density of charcoal was assessed according to the hydrostatic method describe at NBR 11941 (ABNT, 2003). The true density was appraised according to NBR 9165 (ABNT, 1985). The porosity was obtained from the true and apparent densities (Equation 1), where in: P = Porosity (%); AD = Apparent density of charcoal (g·cm^{-3}); TD = True density of charcoal (g·cm^{-3}).

$$P = \left(1 - \frac{AD}{TD}\right) \times 100 \qquad [1]$$

The Immediate analysis of charcoal was conducted according to the NBR 8112 (ABNT, 1986) for determination of the levels of volatile materials, ash and fixed carbon.

Hygroscopic packaging of charcoal

Samples of charcoal produced in each of the final carbonization temperatures (400 °C, 550 °C, 650 °C and 750 °C) were dried in an oven at 103 ± 2 °C until 0% moisture content. The hygroscopicity assay was carried out following the method described by Takeshita and Jankowsky (2015), ASTM E104-02 (ASTM, 2012) and ASTM D4933-99 (ASTM, 2010). The technique consists of samples packed inside plastic boxes with saturated salt solutions providing different relative humidities (RH).

The samples were studied within two situations. In a first case the analysis was driven under low RH-induced by use of silica gel, giving 33% RH and in the second under high RH provided by a solution of K2SO4 (potassium sulphate) saturated in Water, resulting in a 98% RH environment, both with controlled internal temperature of 22 °C. In order to maintain the internal testing conditions (RH and temperature), the plastic boxes were kept at 22 °C and 65% RH. Figure 1 details the performed test.

The monitoring of hygroscopicity was carried out by means of carbon control samples fixed by metal rods to the inner upper part of the housing (Figure 1D). Thereby made it possible to weigh the samples without opening the box, in order to avoid any humidity variation inside. The control samples were simultaneously lifted daily until reaching constant heft. Once the control samples stabilized, all the others had their masses measured.

Data analysis

A completely randomized design with four final carbonization temperature (400 °C, 550 °C, 650 °C and 750 °C) and ten repetitions was carried out (except for the carbonization performed in three replicates). The data were subjected to the Kolmogorov-Smirnov test for the control of normality. An analysis of the variance through the Levene test was performed to test the hypothesis that the variances are equal to the analyzed category. Once these assumptions were confirmed, a linear Pearson correlation was applied on all the variables studied for charcoal. The result was then subjected to the t-test to evaluate the probability of difference between the sample categories.

An F test, regression analysis, was performed on the significant variables involving the carbonization temperature, the t-test was applied afterward to individually assay the R^2 coefficients. When necessary, the models have been adjusted to improve the values of the standard error and the coefficient of determination. All tests were done within a 5% tolerance range using Minitab 16® software.
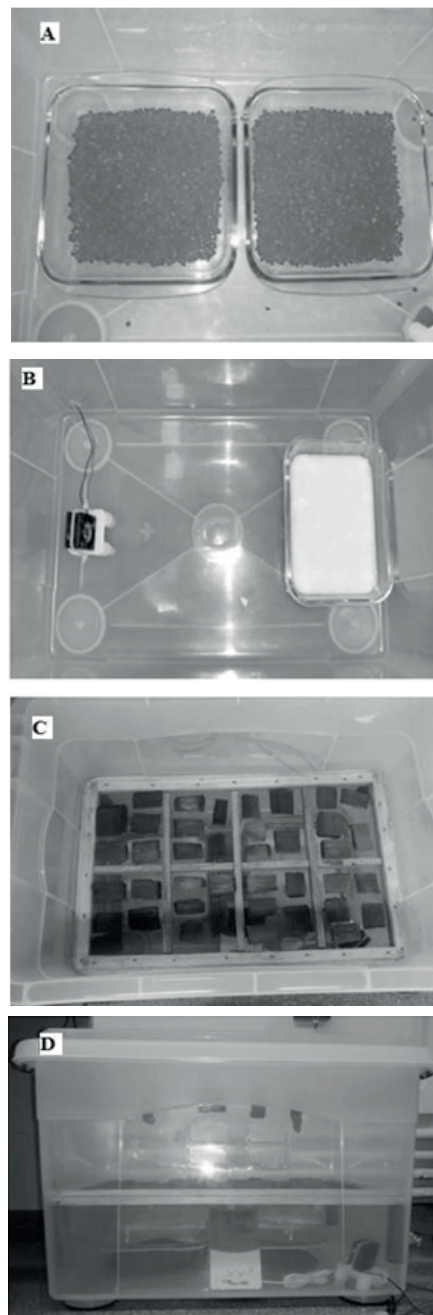

FIGURE 1 Plastic box with silica gel (A); plastic box with K_2SO_4 with a fan indoor air circulation (B); charcoal packaging (C) and set up test containing a saline solution, gauge of temperature and RH, fan and charcoal samples (D).

RESULTS

The results of yields, physical and prompt features and of charcoal according final temperature carbonization are shown in Table 1.

Based on the absolute average values (Table 1), we noticed the influence of the carbonization temperature on the yields and properties of charcoal. This is asserted observing the values of the correlations presented in Table 2.

Figure 2 shows the negative influence of the final carbonization temperature increase on the yield of charcoal, bulk density, volatile matter content and moisture content of conditioned charcoal in a 33% RH environment. On the contrary there was an increase in true density, porosity, fixed carbon content and in the humidity of acclimated charcoal at 98% RH due to the increase of the carbonization temperature.

DISCUSSION

Gravimetric yields and charcoal analysis

The charcoal yield decreased as the final carbonization temperature increased and the highest average was at the final temperature of 400 °C (Figure 2A). The pyroligneous liquid yield at the same temperature had the lowest value (Table 1). The carbonization temperature rising causes an expanding of condensable and uncondensable gas emission, wherein, among the condensable gases, themore volatile are the vapors one which contribute to increasing the amount of pyrolineous liquid (KELYOUNSSI; HALIM, 2014). The the pyroligneous liquid augmentation related to the carbonization temperature rise was also observed by Vieira et al. (2012).

Taking into account the non-condensable gases emission calculated from coal and liquid outputs, the highest averages were detected for the highest final carbonization temperatures of 650 °C and 750 °C.

Thus, the higher the temperature, the greater the amount of carbonized material degraded, leading to higher gas discharges (DERMIRBAS, 2004). This is supported by the observation that the lowest carbon yield as well as the highest pyroligneous and non-condensable gas yields occurred during carbonization at a final temperature of 750 °C (Table 1). Fixed carbon yields obtained are consistent with those observed in the literature. They stands around 27% for *Eucalyptus* charcoal, regardless of carbonization temperature, stating the existence of an inverse relationship between the gravimetric efficiency of coal and the fixed carbon content (GOMES; OLIVEIRA, 1980; SOARES et al., 2014).

The analysis presented in Table 1 and Figure 2B demonstrate that the apparent density of charcoal decreases as the temperature increases, ranging from 0.346 g·cm⁻³ at 400 °C to 0.268 g·cm⁻³ at 750 °C. This

TABLE 1 Gravimetric yields, physical and prompt properties of charcoal.

Variables	Final carbonization temperature			
	400 °C	550 °C	650 °C	750 °C
GY (%)	37.54 ± 0.46	32.71 ± 0.56	30.22 ± 0.32	28.07 ± 0.13
PL (%)	41.43 ± 0.90	45.60 ± 1.99	47.39 ± 0.44	44.16 ± 1.51
NCG (%)	21.03 ± 1.36	21.68 ± 1.43	22.37 ± 0.47	27.75 ± 1.37
FCY (%)	25.68 ± 1.32	24.39 ± 0.80	25.97 ± 0.78	26.02 ± 1.11
AD (g·cm⁻³)	0.346 ± 0.02	0.307 ± 0.01	0.292 ± 0.01	0.268 ± 0.02
TD (g·cm⁻³)	1.053 ± 0.05	1.058 ± 0.02	1.105 ± 0.02	1.127 ± 0.01
P (%)	66.74 ± 2.05	70.93 ± 0.41	73.57 ± 0.31	76.23 ± 0.17
VM (%)	38.91 ± 0.34	25.16 ± 0.10	14.42 ± 0.11	7.06 ± 0.15
FC (%)	60.82 ± 0.37	74.46 ± 0.11	85.74 ± 0.37	92.61 ± 0.21
AS (%)	0.28 ± 0.03	0.39 ± 0.01	0.49 ± 0.02	0.33 ± 0.04
H$_{RH\ 33\%}$ (%)	1.57 ± 0.07	1.19 ± 0.09	0.80 ± 0.04	1.03 ± 0.03
H$_{RH\ 98\%}$ (%)	6.36 ± 0.07	6.61 ± 0.01	7.03 ± 0.05	8.93 ± 0.08

Wherein: GY = charcoal gravimetric yield; PL = pyroligneous liquid yield; NCG = yield of non-condensable gases; FCY = fixed carbon yield; AD = apparent density; TD = true density; P = porosity; VM = volatile matters content; FC = fixed carbon content; AS = ashes content; H$_{RH\ 33\%}$ = humidity in a 33% relative humidity environment; H$_{RH\ 98\%}$ = humidity in a 98% relative humidity environment. (±) = mean standard error.

TABLE 2 Correlation matrix of the variables studied.

Variable	Temperature	GY	PL	NCG	FCY	AD	TD	P	VM	FC	AS	H$_{RH33}$
Temperature	1											
GY	-1.00*	1										
PL	0.59	-0.65	1									
NCG	0.84*	-0.79*	0.05	1								
FCY	0.36	-0.29	-0.10	0.46	1							
AD	-1.00*	1.00*	-0.60	-0.83*	-0.27	1						
TD	0.93*	-0.90*	0.41	0.85*	0.67	-0.89*	1					
P	1.00*	-1.00*	0.59	0.83*	0.34	-1.00*	0.93*	1				
VM	-1.00*	1.00*	-0.62	-0.81*	-0.37	0.99*	-0.93*	-1,00*	1			
FC	1,00*	-1.00*	0.63	0.80*	0.38	-0.99*	0.94*	1.00*	-1.00*	1		
AS	0.44	-0.51	0.97*	-0.12	-0.01	-0.44	0.33	0.45	-0.49	0.50	1	
H$_{RH33}$	-0.84*	0.87*	-0.91*	-0.41	-0.26	0.83*	-0.76*	-0.84*	0.87*	-0.87*	-0.86*	1
H$_{RH98}$	0.86*	-0.81*	0.10	1.00*	0.49	-0.85*	0.88*	0.86*	-0.84*	0.83*	-0.06	-0.46

Wherein: GY = charcoal gravimetric yield (%); PL = pyroligneous liquid yield (%); NCG = yield of non-condensable gases (%); FCY = fixed carbon yield (%); AD = apparent density (g cm-3); TD = true density (g cm-3); P = porosity (%); VM = volatile matters content (%); FC = fixed carbon content (%); AS = ashes content (%); HRH 33% = humidity in a 33% relative humidity environment (%); HRH 98% = humidity in a 98% relative humidity environment (%).*Significant values at 5% significance.

wood thermal degradation increasing emerges from the final temperature rising. Howbeit this observation Trugilho and Silva (2001) report that the apparent density of charcoal decreases to the exact temperature of 700 °C, as the mass loss caused by the release of volatiles is greater than the volume reduction of charcoal. Nevertheless from 700 °C, the charcoal density tends to increase as the mass loss is lower than the shrinkage of the charcoal, phenomenon coming out above all because of the volatilization of H$_2$, which has low molecular weight (TRUGILHO; SILVA, 2001). In any case, this trend was not observed in this research.

The true density gain is noticed as the final carbonization temperature increases (Figure 2C). This might be explained by the formation of graphite crystals (graphitization) provoked from changes in carbon
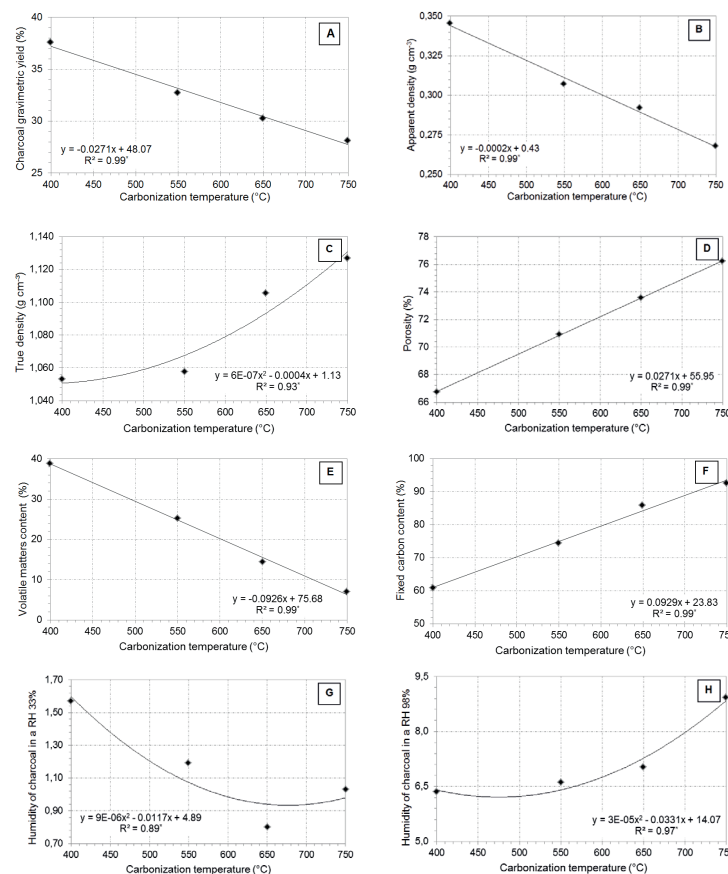
FIGURE 2 Adjusted models of analyzed variables in relation to final carbonization temperature. *Significant values at a 5% significance.

structure during charcoal production (SOMERVILLE; JAHANSHAHI, 2015; BROWN et al., 2006). Besides the porosity, characteristic related to the hygroscopicity of coal, is positively correlated with the final carbonization temperature (Figure 2D), this same behavior was observed by Brown et al. (2006), Yu et al. (2006) and Bergeron et al. (2013).

The carbonization at high temperatures has enabled the unclogging pores by volatilization of the residual tar, increasing the porous area of the charcoal (BROWN et al., 2006). The porosity increased due to the reduction of the apparent density of charcoal, since the true density is relatively independent of carbonization (SOMERVILLE; JAHANSHAHI, 2015). The charcoal produced at the final temperature of 750 °C presented smaller amounts of volatile matters (Figure 2E), due to their higher disposal at higher temperatures. Thus, one can notice higher fixed carbon content occurred in these charcoal samples (Figure 2F) caused by the increase of the final temperature of carbonization, already well known in the literature (WENZEL 1970; VALENTE et al., 1985 DERMIRBAS, 2004; OLIVEIRA et al., 2010).

Ash content ranged from 0.28% at 400 °C to 0.49% at 650 °C. In spite of the very low values, Trugilho et al. (2005) states that this feature is function of several factors. Indeed, ashes are non thermal degradable mineral fraction of the source material. Thus, this decrease might be explained by the ignition of a mineral component in these temperature ranges. In the case of *Eucalyptus* wood, this fraction is generally less than 1% and almost negligible in its charcoal. There is a trend to the increase of the ash content with the elevation of carbonization temperature, once there might be further degradation and reduction of other constituents of the wood, proportionally increasing the concentration of minerals.

Charcoal hygroscopicity

The moisture absorbed by the charcoals showed significant correlation values with the final carbonization temperature (Table 2). In the environment of lower relative humidity (33% RH) the carbonization conducted at 400 °C and 650 °C, the temperatures have practically reduced of half the humidity, from 1.57% to 0.80% (Table 1). Albeit of low magnitude, the values should be considered significant and influenced by the final carbonization temperature, demonstrated in Figure 2G.

The negative relation noted in the samples conditioned at 33% relative humidity (Figure 2G) may be associated to an environment insufficient to provide the adsorption of moisture by the charcoal and thus, prevailed

its own moisture loss. For best results, specific studies should be conducted addressing minor intervals of RH.

On the other hand, the charcoal samples placed in the environment with high RH showed an increase in humidity by the heightened final carbonization temperature (Figure 2H), with the highest average detected for charcoal produced at 750 °C (Table 1). This was the reason why the charcoal produced in this temperature showed higher moisture related to higher porosity presenting a larger area available for water condensation inside the pores (Table 1).

The difference between the moisture adsorbed by the charcoal packed in two environments can be attributed to more water molecules present in the humid atmosphere (RH = 98%) and a greater ease of inducing the porous region of the charcoal. This means that the more humid the environment, the greater the adsorption of moisture by charcoal.

These two different moisture conditions represent different possible situations of charcoal storage where high relative humidity is not ideal. The moisture adsorption for the samples produced at 400 °C, 550 °C and 650 °C packaged in high RH humidity environment obtained was about 7%, recommended for use in combustion, for example (DIAS JÚNIOR et al., 2015b). Thus, in practice, these results show that moisture charcoal can withstand high RH.

Luengo and Emmerich (1997) found that hygroscopicity decreased as the carbonization temperature increased. According to the authors, charcoal, as soon as it is produced, has the ability to absorb moisture varies from 4% to 16%, ranging from 21% for temperature of 150 °C, 7% for temperature of 250 °C, 6% for charcoal obtained at 350 °C, 4% for charcoal prepared at 450 °C and only 2% for charcoal obtained at 1500 °C. According to the authors, , as soon as it is produced, the charcoal has the ability to absorb moisture varies from 4% to 16%, ranging from 21% at 150 °C, 7% at 250 °C, 6% for charcoal obtained at 350 °C, 4% for charcoal prepared at 450 °C and to only 2% for charcoal obtained at 1500 °C. Charcoals conditioned in low humidity environment had the same behavior. However, the results obtained in high humidity environment in this study are consistent with the statements of Andrade and Della Lucia (1995), which mentioned that the hygroscopicity directly relates to the porosity of charcoal (Figure 3).

The obtained results indicate that the moisture of samples conditioned in the atmosphere with 98% of RH showed behavior inversely proportional to the
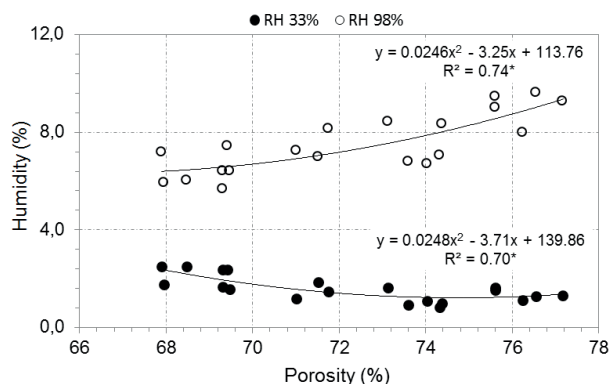
FIGURE 3 Relationship between porosity and humidity of charcoal conditioned in two RH environments. *Significant values at 5% tolerance.

apparent density (Table 2). Somerville and Jahanshahi (2015) had also noticed this fact, wherein the increase of density reduces the number of pores and, therefore, reduces the hygroscopic capacity of charcoal. The higher the porosity the higher will be the surface area of the charcoal, providing more contact to water molecules in the environment and their adsorption (BERGERON et al., 2013; KELYOUNSSI; HALIM, 2014). Furthermore, Yu et al. (2006) emphasized that even when the porosity is similar, the pores size influences the adsorption of soluble compounds by charcoal, wherein the micropores have a high power of capillarity and appear more in charcoals produced under elevated carbonization temperatures. This can explain, for example, the behavior between the porosity and the humidity of charcoal conditioned at 33% RH. It is possible that the pore diameters of these samples were smaller than the samples conditioned at 98% relative humidity.

Yu et al. (2006) discussed the adsorption of herbicidal agents by soils with and without charcoal incorporation, produced at 450 °C and 850 °C. The results showed better adsorption for the soils that received charcoal produced under the highest final temperature of carbonization.

The charcoal produced at elevated temperatures showed higher porosity and higher adsorption capacity, in the 98% of RH environment. This demonstrates the possibility of the hygroscopicity adjustment, depending on the desired characteristics of charcoal and its use. Moreover, further studies are necessary regarding to chemical composition and covering a greater range of environmental conditions.

CONCLUSIONS

The final carbonization temperature affected significantly the gravimetric yields and the properties of charcoal.

The final carbonization temperature directly influenced the porosity, which besides influenced the hygroscopicity of charcoal that had different behaviors depending on the environments condition.

The hygroscopic conditioning allowed the assertion of a correlation between porosity and moisture adsorbed by charcoal.

ACKNOWLEDGMENTS

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