DOI: 10.5965/223811712212023123

Revista de Ciências Agroveterinárias 22 (1): 2023
Universidade do Estado de Santa Catarina

Determination of total extractives and cellulose content of Ipe wood submitted to thermal modification process

Determinação dos extrativos totais e teor de celulose da madeira de Ipê submetida ao processo de modificação térmica

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Submission: 19/04/2022 | Acceptance: 25/08/2022

ABSTRACT

The demand for products from wood has been expanding, because of this, there is a need for studies related to the quality and application of these materials. Thus, the present research aimed to evaluate the extractive and cellulose content of Ipê wood submitted to the thermal modification process. To carry out the study, four individuals of *Handroanthus chrysotrichus* (Mart. ex DC.) Mattos, 11 years old, were slaughtered. Subsequently, samples measuring 2.5 x 2.5 x 41 cm were prepared and placed in an oven with forced air circulation, at temperatures of 120, 150, 180 and 210 ºC, for a period of 4 h. Then, random portions of the specimens were sectioned and ground in a knife mill, and then, sieved in the 40/60 mesh fraction, to carry out the chemical tests of total extractives and cellulose content. The data were processed and submitted to the Tukey average test, using the R software. The results of the chemical properties studied indicated variations according to the temperatures, not showing a stabilization trend between the treatments applied. A relationship between the behavior of these properties and the other technological characteristics of the works available in the literature was observed. In general, further studies are recommended on the chemical properties of wood of this species associated with the application of high temperatures.

KEYWORDS: extractives content; cellulose content; thermal treatment; *Handroanthus chrysotrichus.*

RESUMO

A demanda de produtos oriundos da madeira vem se expandindo, por conta disso, há a necessidade de estudos relacionados à qualidade e aplicação desses materiais. Com isso, a presente pesquisa teve como objetivo avaliar o teor de extrativos e de celulose da madeira de Ipê submetida ao processo de modificação térmica. Para realização do estudo foram abatidos quatro indivíduos de *Handroanthus chrysotrichus* (Mart. ex DC.) Mattos, com 11 anos de idade. Posteriormente, confeccionaram-se amostras de 2,5 x 2,5 x 41 cm, as quais foram acondicionadas em estufa com circulação de ar forçada, nas temperaturas de 120, 150, 180 e 210 ºC, por um período de 4 h. Em seguida, porções aleatórias dos corpos de prova foram seccionadas e trituradas em moinho de facas, e então, peneiradas na fração de 40/60 mesh, para a realização dos ensaios químicos de teor de extrativos totais e teor de celulose. Os dados foram processados e submetidos ao teste de médias de Tukey, utilizando o software R. Os resultados das propriedades químicas estudadas indicaram variações de acordo com as temperaturas, não apresentando uma tendência de estabilização entre os tratamentos aplicados. Uma relação entre o comportamento dessas propriedades com as demais características tecnológicas dos trabalhos disponíveis na literatura foi observada. De modo geral, são recomendados mais estudos acerca das propriedades químicas da madeira dessa espécie associados a aplicação de altas temperaturas.

PALAVRAS-CHAVE: teor de extrativos; teor de celulose; tratamento térmico; *Handroanthus chrysotrichus*.

The use of wood as a source of raw material has been highlighted due to its wide application, such as in civil construction and furniture manufacturing. From this, to meet the consumer market is of fundamental importance the knowledge of its technological properties, as well as the variation when it is subjected to different conditions of its natural occurrence (FONTOURA et al. 2015, SANTOS et al. 2020).

To mitigate undesirable effects on products, techniques and transformation processes are often applied, such as thermal modification, which aims to conserve or improve wood's physical, chemical and mechanical characteristics (GALLIO et al. 2019, ZANUNCIO et al. 2014, ZAQUE et al. 2021).

Thermal modification is a method used since the middle of 2000, which aims to subject the material to temperatures below roasting, i.e., between 120 and 200 ºC (MODES et al. 2017). This procedure causes changes in the dimensional stability of the parts, in the mechanical properties, natural durability, staining and also in the chemical constituents (BATISTA 2019, MENEZES et al. 2014).

The intensity, temperature and duration of heat treatment cause some changes in the polymers that are constituents of wood, and these factors can alter its internal structure (ALMEIDA et al. 2015), such as: causing the generation of new extractives that can act as fungicides, chemical modification of cellulose, hemicelluloses, polysaccharides and lignin, and also promote the increase of hydrophobic properties of wood, reducing water absorption (JEBRANE et al. 2018, SILVA et al. 2008).

For *Handroanthus chrysotrichus wood* (ex Mart. DC.) Matto submitted to the thermal modification process, CANDATEN et al. (2020) and MANGINI et al. (2021) verified that the high temperature conferred a higher biological resistance to the wood of the species, but there was a reduction in mechanical resistance at 210 ºC; also, that the anatomical structure underwent considerable changes in treatments between 150 and 180 ºC. Based on the above, the aim of the present study was to evaluate the total extractive and cellulose contents of Ipe wood submitted to the thermal modification process.

To carry out the study, four individuals of *Handroanthus chrysotrichus*, aged 11 years, from a forest area belonging to the Federal University of Santa Maria, Frederico Westphalen campus (UFSM/FW), were slaughtered under geographic coordinates 27°22" S; 53°25" W, at 480 m altitude. From the extraction, 16 samples of 2.5 x 2.5 x 41 cm (length, width, and thickness, respectively) were made, randomly collected in the longitudinal and radial direction of the loggings, which were coupled in a greenhouse with forced air circulation, Marconi model MA035, at temperatures of 120, 150, 180 and 210 ºC, for a period of 4 h, thus conducting the heat treatment process in the material. The control samples used as controls had only air drying, observing stabilization with the moisture content of ambient equilibrium.

Subsequently, random parts of the specimens were sectioned and crushed in a knife mill, and then sieved at the fraction of 40/60 mesh, to perform chemical tests of total extractive content and cellulose content.

To determine the cellulose content in the samples, the technique was applied using nitric acid and ethanol (1:4), called direct method, as described by WASTOWSKI (2018). In the process of obtaining the total extractives, it was with the aid of Soxhlets connected in an extraction battery, using ethanol-toluene (1:2). All analyses were performed by treatment and in triplicate. The data were processed and submitted to the Tukey Means Test at 95% reliability using the R software.

The results obtained in relation to extractive contents indicated that this chemical property varied, not presenting a tendency of stabilization between the applied treatments. For the values of cellulose contents, it is noted that there were no statistical differences and there was also no disposition of behavior according to the increase in applied temperatures (Table 1).

For the extractive content, the highest value was evidenced at 150 ºC, not differing from the control and treatment at 210 ºC (Table 1). Still, although the results do not present significant differences, the percentage of extractives increases when compared to the control and the submission at 210 ºC. It also corroborates with the data obtained by SEVERO et al. (2016), which observed that when treating Hevea brasiliensis wood with a temperature of 220 ºC, for 2.5 hours, there was an increase in total extractive contents by 98.7%.

Furthermore, it is noticed that the submission of samples at 180 ºC presented lower values of extractive content than in the other treatments. This reduction can be explained by polymerizing these by-products with other cell wall polymers, which are fixed in wood (KAMDEM et al. 2002). In addition, this decrease may be associated with the characteristics of the species since it is classified as dicotyledonea, which is more sensitive to the thermal degradation process (PIERRE et al. 2011).

For the control samples, no studies were found with the species with chemical properties data in the literature, but for the same genus MEDEIROS NETO et al. (2012) observed 6.88% for total extractives and 63.85% for holocellulose content in *Handroanthus impertiginosus wood.* With results close to those observed

in the present study, OLIVEIRA et al. (2012) found values ranging from 50.26 to 51% for holocellulose of *Handroanthus vellosoi* wood in the longitudinal direction of the tree.

Table 1. Averages of percentages of total extractives and cellulose content of *Handroanthus chrysotrichus* wood subjected to thermal modification.

Treatments	Total Extractive Sums Content (%)	Cellulose Content (%)
Witness	21.5ab	53.7a
120 º C	14.1b	53.9a
150 º C	30.0a	50.9a
180 º C	15.8b	62.9a
210 º C	25.8a	51.7a

Where: lowercase letters next to means in the same column= Tukey's test at 5% probability of error. Coefficient of variation for total extractives 16.3% and cellulose content 28.1%.

In relation to the cellulose content, there was a slight decrease when compared to the percentages between the control and the treatment performed at 150 and 210 ºC. This is due to the temperature, in which the higher the roasting values the lower the holocellulose contents (PEREIRA et al. 2016). In addition, there was a decrease in the means for the test performed at 180 ºC (Table 1).

Similar results were also found by ZANUNCIO et al. (2014), MUSINGUZI et al. (2012) and BARNETOA et al. (2011), in which the decomposition of hemicellulose occurred from 200 °C forming alcohol/toluene soluble products, which increases the total extractive content (BRITO et al. 2008).

In the work of MANGINI et al. (2021) studying the wood of the same species in question (*Handroanthus chrysotrichus*) submitted to heat treatment, they verified degradation of the anatomical structure with emphasis on temperatures of 150 and 180 ºC, and discussed that in general, from mid-150 ºC, the changes in these wood properties are greater, which, consequently, can also influence other technological parameters. Furthermore, CANDATEN et al. (2020) also for thermally treated yellow Ipe, observed that a greater dimensional stability was conferred in wood treated with high temperature, however, there was a reduction in mechanical properties.

After thermal modification, the cellulose content may increase, however, this direction does not refer to the increase in the amount of the same, but to the high rate of degradation of hemicelluloses (HILL 2006). According to POLETTO et al. (2012) the rate of degradation of wood can reduce due to the crystallinity of cellulose, this result is the result of greater resistance to thermal degradation of the crystalline areas of cellulose when compared to the amorphous regions. The authors also state that higher extractive levels related to lower crystalline can stimulate the degradation process and reduce the thermal stability of wood.

These results corroborated well with those verified by BRITO et al. (2008) and SEVERO et al. (2012), in which the authors reported that, as a general trend, the relative proportion of lignin mass increased with the temperature rise and for cellulose, contents verified a simultaneous decrease. Thus, once the wood presents greater degradation of holocellulose, it is likely that there will be greater dimensional stability of this type of wood when thermally modified, due to the degradation of hemicelluloses, of the free hydroxyl groups in the amorphous region of cellulose and crosslinking of wood polymers during heat treatment (BRITO et al. 2008).

To conclude, the heat treatment in *Handroanthus chrysotrichus wood* at the studied temperatures did not present a behavior trend of the chemical properties of extractive and cellulose content. Further studies addressing the chemical properties of wood of this species combined with the thermal modification process are recommended to better understand the behavior of this type of material.

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