

CONDENSED TANNINS EXTRACTED FROM EUCALYPTUS BARK WASTE

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Abstract

This study aimed to evaluate the quantity of condensed tannins in the waste of eucalyptus bark extracted according to two salt concentrations and two analysis methods (Stiasny number and ultraviolet method). Tannins were extracted using water and sodium sulfate (Na_2SO_4) and sodium carbonate (Na_2CO_3) solutions in different concentrations (0, 4, 6, 8 and 10%). The influence of the different concentrations of chemical compounds (Na_2SO_4 and Na_2CO_3) on yield and extract characteristics was evaluated: content of total solids, tannins yield, non-tannins yield, pH, polyphenols yield by Stiasny reaction, and content of reactive polyphenol by ultraviolet method. The results showed that the extraction with sodium carbonate (8%) provided higher amounts of tannins extraction, and sodium sulfate (4%) provided lower amounts. The influence of 8% of sodium carbonate on the eucalyptus bark characteristics in extraction provided the highest Stiasny number and content of tannins. This method also enabled a high extraction yield, indicating the importance of using this concentration of salt in the extraction process. The highest gravimetric tannins yield was 4% for sodium sulfate. Thus, eucalyptus bark may be used in tannins extraction since it is easily available and enables a good yield and quality of tannins.

Keywords: Polyphenols, sodium sulfate, sodium carbonate, sustainability.

Resumo

Taninos condensados extraídos de resíduos de casca de eucalipto. Este trabalho objetivou avaliar a quantidade de taninos condensados contidos nos resíduos de casca de eucalipto. Os taninos foram extraídos com água e soluções de sulfato de sódio (Na_2SO_4) e carbonato de sódio (Na_2CO_3) em diferentes concentrações (0, 4, 6, 8 e 10%). Avaliou-se a influência das diferentes concentrações dos sais no rendimento e características do extrato obtido: teor total de sólidos, rendimento de taninos, rendimento de não-taninos, pH, rendimento de polifenóis pela reação de Stiasny e conteúdo de polifenóis reativos por ultravioleta. Os resultados obtidos mostraram que a extração com carbonato de sódio (8%) proporcionou uma maior extração de taninos e a solução de sulfato de sódio (4%), as menores quantidades. A influência do carbonato de sódio a 8% nas características da casca de eucaliptos na extração proporcionou o maior número de Stiasny e conteúdo de taninos. Permitiu também obter alto rendimento de extração, indicando a importância de usar essa concentração de sal no processo de extração. Para o sulfato de sódio, o maior rendimento gravimétrico nos taninos foi de 4%. A casca de eucaliptos pode ser utilizada na extração de taninos por estar amplamente disponível e permitir um bom rendimento de tanino.

Palavras-chave: Polifenóis, sulfato de sódio, carbonato de sódio, sustentabilidade.

INTRODUCTION

Food and Agriculture Organization (FAO) predicts that the planet will have 9.1 billion people in 2050, who will consume three times more wood products. This situation will require an additional of 250 million hectares of forest area (FAO, 2017). In this context, people question how they can meet the growing demand for wood, reduce environmental impacts and global warming and mainly validate the commitments made in the Paris Agreement.

Thus, it is imperative to invest in sustainable alternatives that innovate in products and processes throughout the production chain, maximizing the use of wood and all of its waste. According to Brazilian Tree Industry (IBÁ, 2017), Brazil is the worldwide leader on forest productivity in the planted forest sector with approximately $36 \text{ m}^3 \text{ ha}^{-1} \text{ year}^{-1}$. Wood from planted forests supplies areas/industries of civil construction,

automobile, food, cosmetics and personal hygiene, electronics, pharmaceuticals, furniture, chemicals, and other sectors. For example, for the future, the replacement of carbon fibers in the composition of airplane wings is listed alongside the development of new technologies for forestry and wood technology.

In Brazil, eucalyptus plantations occupy 5.7 million hectares of the area planted with trees to supply the industrial demand in the forest sector (72.7% of total planted forest area) (IBÁ, 2017). The main product of interest on this species is the wood. Concurrently to the wood is the production of bark, which may correspond to 9 to 18% of the commercial volume of the trunk and may be influenced by genetic factors, as well as the type of bark.

Extracts of eucalyptus bark can replace phenol in the formulation of adhesives (VÁZQUEZ *et al.*, 2009). Tannins are polyphenolic compounds of high molecular weight and more reactive than phenol. Moreover, they have been highlighted as a source of raw material for replacing synthetic adhesives in manufacturing wood panels (FRIHART, 2005; MOSIEWICKI *et al.*, 2007). Different amounts of tannins in the wood (predominating in the bark) are observed in almost all taller plants (hardwoods and softwoods) (GUANGCHENG *et al.*, 1991; TRUGILHO *et al.*, 2003; VIEIRA *et al.*, 2011). In hardwoods, tannins can be obtained in satisfactory levels to produce adhesives. Trugilho *et al.* (2003) found condensable tannins content extracted in hot water in the proportion of 40.31% for barks of *Eucalyptus cloeziana* and 16.03% for *Eucalyptus paniculata*. Among timber species, the genus *Eucalyptus* has potential for tannins extraction, especially due to its bark volume generated by wood processing.

Tannins are classified according to their hydrolysable and condensed characteristics. Generally, hydrolysable characteristics do not have importance in manufacturing adhesives, unlike the condensates. By replacing synthetic resins, condensed tannins can be used for the production of wood adhesives due to their ability to precipitate with formaldehyde, thus forming a rigid polymer structure. In the process of tannins extraction, technical and economic feasibility is favored by the availability of bark as waste, as well as water use for industrial activity. However, it does not occur when using solvents to potentiate the extraction of tannins and may generate environmental harm. In addition, it can hinder the recycling of post-extraction waste (PIZZI, 1994).

Thus, the use of tannins extracted from eucalyptus bark from debarking of logs can be an alternative for many producers to increase their income source. In this perspective, tannins become a proposal for replacing or reducing the phenol formaldehyde in synthetic adhesives, which has excellent properties for use in wood but is dangerous to humans. Due to the favorable scenario of wide eucalyptus production in Brazil, targeting a technological use for the bark would be feasible, thus providing an economically and environmentally viable product. Therefore, this research aimed to evaluate the quantity of condensed tannins in the waste of eucalyptus bark, which was extracted by two salt concentrations using two analysis methods.

MATERIAL AND METHODS

Origin and processing of eucalyptus bark

The material used in this study was the eucalyptus bark obtained from log debarking, which was provided by a timber industry that processes wood from reforestation areas, located in the Southern region of the state of Espírito Santo, Southeast Brazil (20°44'3,08"S and 41°29'21,14"W). The company could not inform the age of the material, since the bark source originated from several rural producers.

The material was air-dried until it reached equilibrium moisture content between 12 and 15%. In order to standardize and accelerate the drying process, the material was periodically turned to avoid material loss. After air-drying, the bark was fragmented into particles, homogenized and pounded into a Wiley mill. The material obtained was classified by using sieves with fractions between 40 and 60 mesh (TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY – TAPPI T 257 om-92, 1992).

Tannins extraction

For the extraction of tannins from the bark, pure water and aqueous solutions of chemical reagents (sodium sulfate – Na₂SO₄ and sodium carbonate – Na₂CO₃) were used. Thus, nine compositions for extraction were formed (Figure 1).

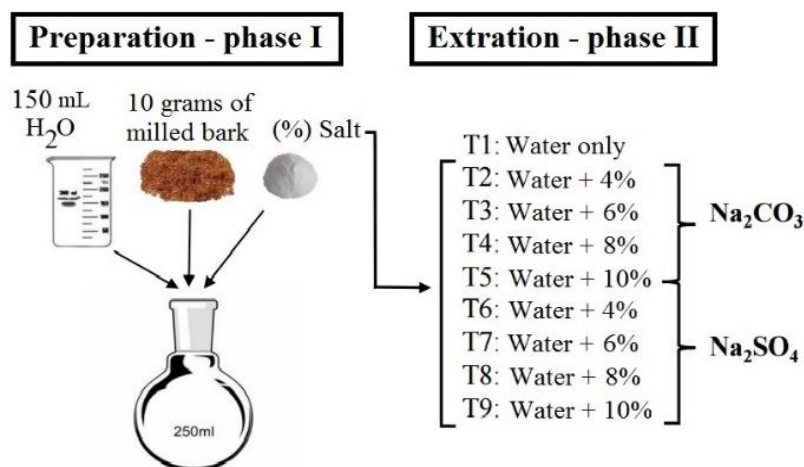


Figure 1. Compositions for extraction with the salts evaluated.

Figura 1. Composições utilizadas nas extrações com os sais estudados.

The percentage of salt was calculated in relation to the dry mass of the bark. Tannic substances contained in the bark were extracted by 150 mL of distilled water (bark:water ratio of 1:15) in a 250 mL round-bottomed flask. For this purpose, 10 g of bark (absolutely dry) and the established amount of salt in each composition (except for the composition with pure water) (Figure 1) were added. Then, the solution was subjected to boiling under reflux for 2 hours at a temperature of 50°C. Each sample was subjected to two extraction sequences in order to remove the maximum amount of extractives present.

The extract obtained was homogenized and filtered in a sintered glass crucible of porosity 2 in order to retain the thin waste of the bark. Then, it was concentrated to 150 mL by evaporating water through heating in a thermic manta. After the concentration was achieved, an aliquot of 50 mL was taken from each extract for the Stiasny number analysis.

Determination and characterization of polyphenols

Content of total solids of tannins and non-tannins and pH were determined by the extracts of tannins obtained from each composition. Tannin contents were obtained through Stiasny reaction and ultraviolet reactivity (VIEIRA *et al.*, 2011). Content of total solids of the bark resulted from the filtrate of each condition. The quantity of 25 mL allocated in Petri dishes was used for drying in an oven ($103 \pm 2^\circ\text{C}$) until constant mass. Then, the amount of total solids (in grams) in 25 mL of solution was obtained through the difference between the dry mass before and after drying in the oven, and the total percentage of content of solids was calculated considering the amount of particles (dry basis) and the initial volume used in the extraction (PAES *et al.*, 2013).

Polyphenol content was determined through Stiasny reaction, adapted as described by Guangcheng *et al.* (1991), and ultraviolet (UV) method. After each extraction, 4 mL of formaldehyde (37% w:w) and 1 mL of concentrated hydrochloric acid were added to 50 mL of crude extract in each aliquot to determinate the polyphenols by Stiasny reaction. Each combination was subjected to boiling under reflux for 30 minutes. Under these conditions, tannins formed insoluble complexes that were separated by simple filtration. For this case, a sintered glass crucible of porosity 2 was used. The material precipitated and retained on the filter was dried at $103 \pm 2^\circ\text{C}$ for 24 hours, and then the Stiasny number was calculated.

The reactivity of the solution obtained by the Stiasny method was then evaluated by the UV method. The aliquot was filtered through sintered glass of porosity 2 and the absorbance was obtained by using a spectrophotometer at 280 nm. The reactive polyphenols obtained on the filtrate of the aqueous extract (which did not receive hydrochloric acid or formaldehyde) were evaluated by reading the wavelength, considering the dilution of 1:90.

Content of non-tannins was obtained by the difference between content of total solids and content of condensed tannins obtained from each test sample.

Statistical analysis

Regression analysis was used with five replicates for the composition previously defined in order to evaluate the effect of salt in tannins extraction. Furthermore, a completely randomized design experiment was

performed to evaluate the effect of tannin extraction with different salts in the regression analysis with five replicates ($F, p < 0.05$).

RESULTS

Behavior of the content of total solids, tannins and non-tannins yield, and pH according to the concentration and type of salt used are illustrated in Figure 2.

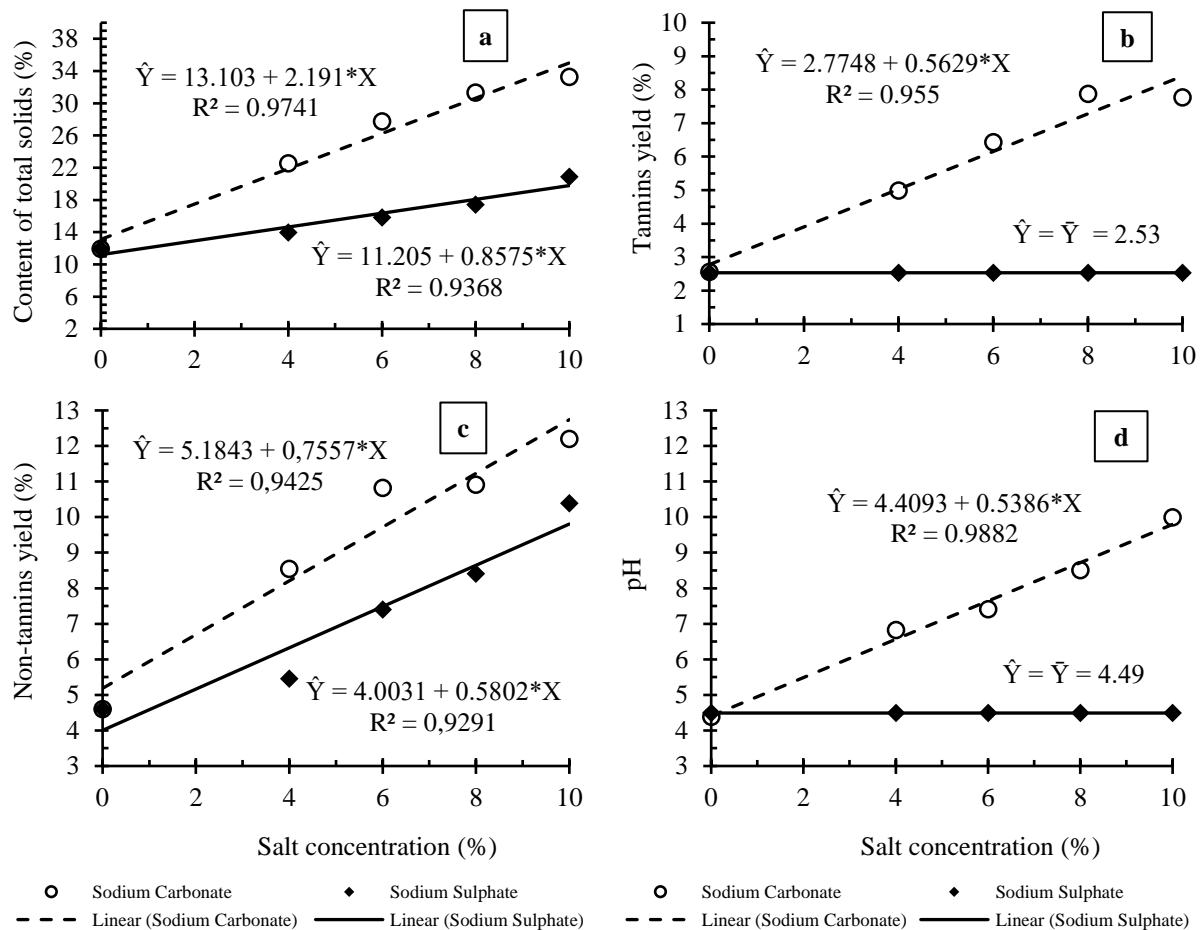


Figure 2. Content of total solids (a), tannins yield (b), non-tannins yield (c) and pH (d) in the tannic extracts obtained from sodium carbonate and sodium sulfate salts.

Figura 2. Teor de sólidos totais (a), teor de taninos (b), teor de não-taninos (c) e pH (d) nos extratos tânico obtidos a partir dos sais carbonato de sódio e sulfato de sódio.

Content of total solids is the first parameter used to indicate which composition supplies the highest yield of tannic extracts. There is a tendency to increase the content of solids as the salts concentration increases (Figure 2a), being the regression model significant. A more evident increase in the content of total solids is observed when sodium carbonate was used in the concentrations of 8 and 10% for the extraction of tannins. Regarding sodium sulfate, it occurred in the concentration of 10%.

Stiasny number and reactivity by ultraviolet exposure according to the concentration and type of salt used are shown in Figure 3.

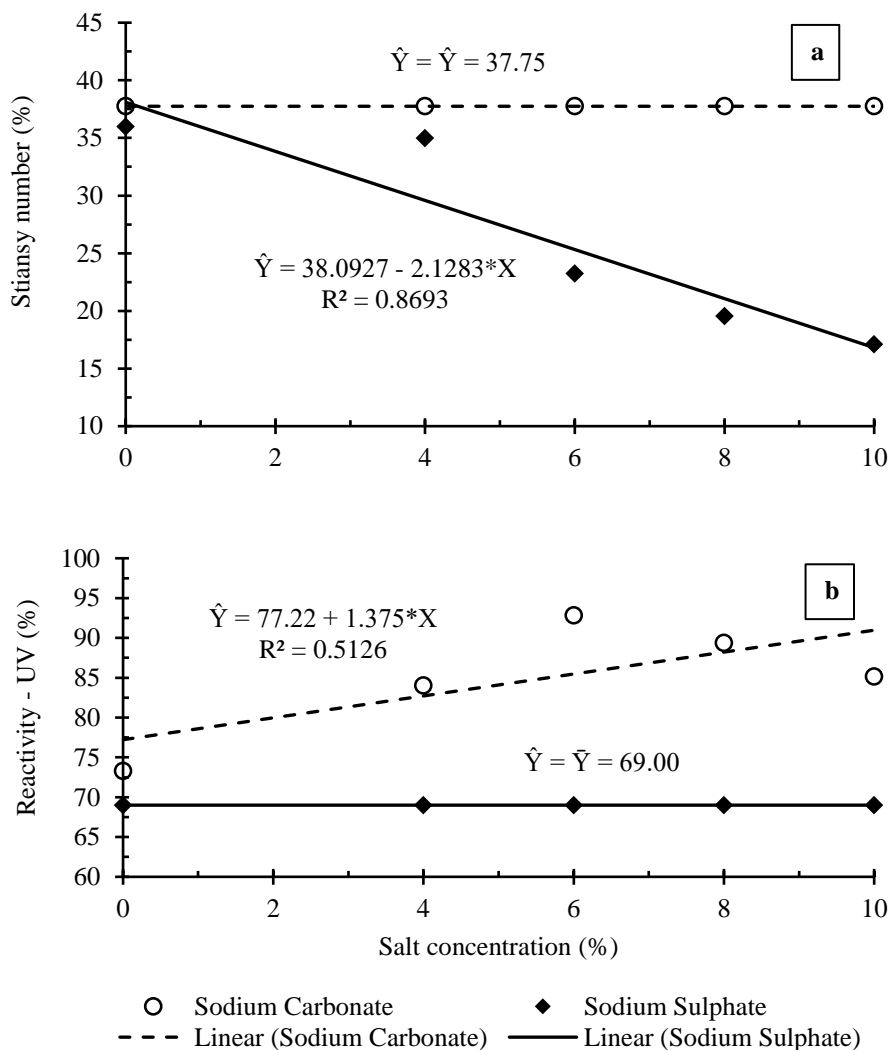


Figure 3 Stiasny number (a) and ultraviolet reactivity – UV (b) in the tannic extracts obtained from sodium carbonate and sodium sulfate salts.

Figura 3. Número de Stiasny (a) e reatividade por ultravioleta (b) nos extratos tânicos obtidos a partir dos sais carbonato de sódio e sulfato de sódio.

The total extract content of substances that reacted with formaldehyde in acidic medium to sodium carbonate (Stiasny number) was not significant. Thus, the presence of sodium carbonate in the extractive solution maintained the pH in the acidic range (Figure 2d).

DISCUSSION

The content of total solids is a desired characteristic, considering that tannins are part of the extracts. Moreover, by increasing its concentration, there is a consequent increase in tannins content. Vieira *et al.* (2011) observed a similar tendency in the extraction of tannins from the bark of *Pinus oocarpa* with the addition of 5% of sodium carbonate, which increased the extraction of tannins. However, the concentration of 8% (Figure 2b) was chosen since it promoted lesser spent on reagents and had better characteristics in relation to the concentration of 10%.

The simple linear regression between tannins content and sodium carbonate concentration was significant. The absence of a behavior pattern in relation to the increase in salt concentration with the content of extracted tannin may be expressed statistically. Thus, the content of tannin extracted by using sodium sulfate corresponds to the average content of tannin obtained (2.5%). The model generated in the regression does not correspond the

criteria for choosing the highest concentration of tannins for extraction. Thus, the best choice of salt concentration for extraction in large quantities may be represented by the average of the values. The solution with the concentration of 4% was then chosen since it provided smaller amounts of non-tannins and higher Stiasny number.

The yield of tannins extraction with carbonate and sodium sulfates in different concentrations was considered satisfactory (Figure 2b). Considering tannins yield and that 7.9% of tannins are extracted with 8% of sodium carbonate, it is estimated that 12.706 kg of bark would be necessary to obtain 1 kg of tannin. For a lower concentration of sodium sulfate, a lower tannins yield is observed. Considering the choice of 4% of salt for a yield of 2.9% to 1 kg, 34.364 kg of bark would be necessary for the extraction of 1 kg of tannin.

Miranda *et al.* (2016) also observed high concentrations of extractives for the bark of *Eucalyptus Sideroxylon*, whereas Teodoro and Lelis (2003) found lower levels of extractives when performing extraction of tannins from *Eucalyptus pellita*, ranging from 9.1 to 11% with sodium sulfite and urea reagents. Comparing the values obtained by the studies, it is noteworthy that the differences between the contents of total solids may be attributed to the difference of species used.

The yield of tannin bark extraction was lower when compared to the values found by Vázquez *et al.* (2009) for *Eucalyptus globulus* (6.8 to 18.9%), having better extraction results with 10% of NaOH and lower water use. Hoong *et al.* (2009) also showed relatively high tannins yields for *Acacia mangium* with aqueous sulphite-carbonate, producing yields of 13.8% in water, 17.1% in 1% of sulfite and 18.9% in 2% of sodium sulfite.

The addition of salts during tannin extraction intends to increase the amount of extracted tannins. However, this is not the only parameter used during the analysis, considering that the salt provides not only tannins extraction, but also other phenolic substances contained in the bark. Therefore, the quantities of non-tannins need to be low.

The extracting solution with no salt showed a lower content of non-tannins (about 4.6%) (Figure 2c). However, sodium carbonate and sodium sulfate were added into the extractor solution as there was an increased extraction of non-tannins. It consequently showed that the salts are responsible for most hydrolysis and extraction of larger amounts of non-tannins (starches and sugars). The content of non-tannin of the extraction solution is close to the ones obtained by Vital *et al.* (2004) and Vieira *et al.* (2011) for *Eucalyptus grandis*, *Eucalyptus pellita* and *Pinus oocarpa* with the increase of sodium sulfite concentration on extracting solution. An increase in the content of non-tannins in the respective species was verified.

Carneiro *et al.* (2007) found the value of 10.3% of non-tannins in *Anadenanthera peregrina* extracted at 100°C with 6% of sodium sulfite. Vieira *et al.* (2011) also observed that as the sodium sulfite concentration was increased in the solution of the bark of *Pinus oocarpa*, there was no increase in the content of non-tannins. Tannins extracts are better with lower content of non-tannins and therefore higher Stiasny number, which are desirable characteristics for subsequent wood bonding and cardboard manufacture (BERTAUD *et al.*, 2012).

The addition of sodium carbonate in the extraction process increased the pH value of the extracts (Figure 2d), which indicates a significant difference on the variations among the results. Extracts in the composition with pure water presented the lowest pH values, distinguishing the acid nature of tannins in aqueous extracts. When higher concentrations of sodium carbonate were added, the pH value increased, and the extract presented an alkaline character. In this regard, according to Panamgama (2007), alkaline condition adhesives become very viscous, thereby making it impossible to use it as adhesive (pH 10.5). Similarly, Vázquez *et al.* (2009) reported that elevated pH increases the extraction yield since non-tannins values also increase, which reduces the quality of the tannins extract. However, by using sodium sulfate in the extractor solution, there was no significant difference of pH among the different concentrations, keeping the solution in acidic range.

It is noteworthy that the condensation reaction between tannins and formaldehyde only occurs in pH acidic range, thus providing the resin cure (VIEIRA *et al.*, 2011). The reaction speed between formaldehyde and tannin is accelerated as the pH values are reduced in the extractor solution with sodium sulfate (VIEIRA *et al.*, 2011). In order to not degrade the wood fibers, adhesive pH limit must not exceed the range of 2.5 to 11.0 (IWAKIRI *et al.*, 2005). Therefore, the pH of the solutions must be within the limit for subsequent adhesives formulation that joint the wood and do not affect its structural composition.

The Stiasny number in the extraction with sodium sulfate was similar to that obtained by Paes *et al.* (2013) when evaluating the content of tannins from the bark of *Anadenanthera colubrina* var. *cebil*. Increasing the concentration of salts reduced the Stiasny number and increased the content of non-tannins for sodium hydroxide, sodium sulfite and hydroxide + sulfite solutions. This is inversely related to the content of non-tannic substances. When the Stiasny number is reduced, it results in producing adhesives with problems in viscosity and resistance to glue line (PIZZI, 1994; VITAL *et al.*, 2004).

In addition, the Stiasny number in the extracts containing carbonate was higher than the values found by Vázquez *et al.* (2009) for the bark of *Eucalyptus globulus* with different salts for extraction (NaOH, Na₂SO₃ and Na₂CO₃). They obtained high values for extraction with water and extract containing 2.5% of Na₂SO₃.

Antwi-Boasiako and Animapauh (2012) also observed that tannins extraction with distilled water was more efficient than with 1% of NaOH. They obtained a Stiasny number of 88.5% for *Tetrapleura tetraptera* and 74.6% for *Funtumia elastica*. Moreover, Vital *et al.* (2004) used sulfite in *Eucalyptus grandis* and *E. pellita* and observed an increase in the Stiasny number as they increased the salt concentration.

The Stiasny number in extraction with sodium sulfate decreased as the salt concentration in the extractor solution increased. The highest value (35%) occurred for the concentration of 4% and the lowest (17.1%) for the concentration of 10%. Therefore, the addition of sulfate in the solution promotes a decrease in the Stiasny number, meaning lower reactivity between tannins and formaldehyde. This behavior damages the adhesive formulation performance.

The polyphenols quantification by ultraviolet absorption (UV) is considered more efficient than by the Stiasny method, since the first also considers the polyphenols that react with formaldehyde that does not precipitate in acid medium. In the simple linear regression between salt concentrations and UV reactivity (Figure 3b), it was noticed that the regression model was significant for sodium carbonate. However, the difference on sodium sulfate was not significant among the different salt concentrations.

The content of tannins obtained by UV reactivity was higher than that found by the Stiasny number. This difference can be explained by the reaction of some polyphenols with formaldehyde that is not condensed by the acid reaction (Figure 3b), making its quantification impossible. Thus, more efficient UV reactivity may be considered for the quantification of tannins levels in the solution. UV reactivity values increased in extracts that contained sodium carbonate as the salt concentration also increased, promoting higher reactivity of extracted polyphenols.

Close values were found by Teodoro and Lelis (2003) when verifying UV reactivity of extracts from the bark of *Eucalyptus pellita*. They found values above 60%, increased reactivity to added chemical products (2% Na₂SO₃), and lower water reactivity. Similarly, Bertaud *et al.* (2012) obtained about 66% to 90% of phenolic tannins quantified by UV (280 nm) for the extracts obtained using urea and sulfate for *Picea abies*, *Pinus pinaster*, *Pinus halepensis*, *Abies alba*, and *Eucalyptus globulus*.

CONCLUSION

- The concentration of 8% of sodium carbonate influenced the extraction of eucalyptus bark and provided the highest Stiasny number (higher precipitation of flavanols in acid medium) and tannins. In addition, a high extraction yield was achieved, indicating the importance of using this salt concentration in the extraction process.
- The highest gravimetric tannins yield was achieved with the concentration of 4% of sodium sulfate.
- The content of condensed tannins quantified by the UV reactivity method was higher in relation to the Stiasny number, regardless of the salt used and considering the polyphenols that reacted with the formaldehyde with no precipitation in acid medium.
- Eucalyptus bark may be used in tannin extraction since it is widely available and produces satisfactory yield and quality.

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