

Patrícia DOS SANTOS, Silvia DA SILVA, Darci GATTO, Jalel LABIDI

FIRE RESISTANCE OF WOOD TREATED BY EMULSION FROM KRAFT LIGNIN

The use of wood for construction is not new, however, in some countries there are many resistances to its use, due mainly to the behaviour of wood during a fire. This study aimed to enhance the fire resistance of wood by treating the wood with Kraft lignin emulsion. An emulsion from Kraft lignin was used for the treatment of wood Pinus sp. by full cell methods. The specific mass, wettability and Brinell hardness were analysed. Fire resistances were determined (ignition, flame and ember time). The obtained results showed that the ignition times of treated wood, were higher than those of untreated wood. The emulsion hinders the ignition of samples in approximately 10 seconds. Conversely, the flame and ember time was significantly longer in wood treated, but there was a lower mass loss, compared to untreated wood. This shows that the treatment improves the behaviour of wood in relation to the ignition of fire and also increases the hardness of the wood. Wettability analysis was possible to observe that treatment reduces the rate of moisture uptake.

Keywords: fire retardancy of wood, Kraft lignin, ignition, flame and ember time

Introduction

The use of wood for construction is nothing new and many countries have a culture of building wooden houses. The wooden buildings provide a warm and comfortable addition. The use of wood in building has several advantages over other materials due to its low cost, excellent mechanical, thermal and acoustic properties, as well as providing a cosy atmosphere. The major factor considered in the utilization of wood in buildings, however, is its behaviour against fire [Tondi et al. 2012]. Because according to Zhang [2011], flammability is the biggest disadvantage of wood.

Fire resistance depends on the thickness of the charred layer of wood and the size of the residual section, which depends on the rate of carbonization of the wood species used [White 1995].

Patrícia SOARES BILHALVA DOS SANTOS[✉] (patricia.bilhalva@hotmail.com), Silvia FUENTES DA SILVA, (silviahfuentes@hotmail.com), Jalel LABIDI (jalel.labidi@ehu.eus), Chemical and Environmental Engineering Department, University of the Basque Country, San Sebastian, Spain, 20018; Darci GATTO, (darciatto@yahoo.com) Center Engineering, Federal University of Pelotas, Brazil, 96010-290

The pulp industry is the most important source of production of lignin, this waste is primarily used as fuel in furnaces to generate energy and the recovery of reactive inorganic used in the process. Lignin has a bio-protective activity [Dos Santos et al. 2012] and acts as one of the plant protectors against attack by microorganisms and pests [Quesada-Medina et al. 2010]. These properties could provide added value in the use of lignin.

The aim of this work was to study the fire resistance (ignition, flame and ember time) of wood after impregnation with emulsion of Kraft lignin using the full cell method. The specific mass, wettability and Brinell hardness of the resulting materials were analysed.

Materials and methods

Tests were performed on *Pinus* sp. sapwood (DBH height, 1.30 m) after sectioning each tree, the central plank was selected and cut according to the recommendation of the [ASTMD 5536:1994]. The samples were stored in a climatic chamber at 20°C and 65% of relative humidity until the equilibrium moisture was reached. The thickness of the log was then reduced from 8.0 to 6.0 cm through a plane and various test samples with different dimensions were cut.

Wood treatment was done with a preservative emulsion of Kraft Lignin (EKL), that was prepared with Kraft lignin (2 g/L) in the presence of NaOH (1%) and 97% of water was used. The treatment was carried out in a laboratory autoclave with 2 L capacity using the Full cell procedure. An initial vacuum of 1.0-1.2 bar for 30 min was applied, after the impregnation with the preservative solution, a pressure of 6.0 to 8.0 bars was applied for 60 min. Weight percent gains (WPG) of the samples due to the treatment with (EKL) were calculated by equation 1.

$$\text{WPG} = ((W_m - W_u) / W_u) \cdot 100 \quad (1)$$

where: W_m = dried weight of sample after treatment, W_u = dried weight of untreated samples.

The surface hardness (Brinell) was evaluated according to the standard test [EN 1534:2000], using Emco test automatic M4U075 equipment. The test was performed in the tangential surface of ten specimens ($80 \times 20 \times 10 \text{ mm}^3$) in three-points for each sample.

The short-term fire tests were done according the procedure described by Tondi et al. [2012]. The radial surfaces of the samples ($50 \times 25 \times 15 \text{ mm}^3$) were exposed to the flame of a Bunsen burner for 2 min, with distance at 7 cm between blue flame and wood surface. The ignition, flame and ember time were determined with a stopwatch and realized in quintuplicate for each treatment.

Contact angles (CA) were measured by the sessile droplet method ($5 \mu\text{L}$ distilled water) in all samples at equilibrium moisture content (EMC). The first measurement of CA was made after 5 seconds (time zero). Subsequent CA,

measurements were repeated at intervals of 30 seconds up to 120 seconds, totalling six CA measurements per sample and in five samples for each treatment

Results and discussion

The basic characteristics of aqueous preservative emulsion of Kraft Lignin (EKL), analysed in this work were pH 6.8, density $0.97 \text{ g}\cdot\text{ml}^{-1}$ and black colour. Samples presented a weight percentage gain (10.5%) after 24 hours with the utilized treatment.

In table 1 the results of Brinell hardness is shown, where the treated wood showed a higher hardness with ($13.44 \text{ N}\cdot\text{mm}^{-2}$) compared to the untreated wood ($5.70 \text{ N}\cdot\text{mm}^{-2}$). According to Fejfer et al. [2014], the basic density is a standard procedure in the assessment of the degree of the degradation of wood.

Table 1. Weight percent gains (WPG) and hardness (Brinell) after treatment of wood *Pinus* sp. with EKL

Samples	WPG (%)	Density 12% ($\text{kg}\cdot\text{m}^{-3}$)	Brinell hardness (HB) [N/mm^2]
Untreated	–	453.8 (7.2) ^a	5.70 (1.38) ^a
Treated	10.5 (0.15)	476.6 (8.4) ^b	13.44 (1.73) ^b

The values in parentheses are SD. Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey test.

It is known that the hardness varies with the material density, but in this study, it was not possible to observe the increase in hardness due to the variation of the density, because the density increment values were relatively small compared to an increase in hardness, which showed an increase of approximately 135% in *Pinus* wood treatment, compared with untreated wood.

The behaviour against fire is an important factor for the use of wood in the construction of buildings. The short time fire exposure test simulates the ignition process, whilst the weight loss test after longer exposure, provides better information about the strength of a wood species [Tondi et al. 2012].

The ignition time shown in table 2 was significantly different for both treated timber (26.6 s) and untreated (13.2 s). The EKL emulsion retarded the ignition by over 10 seconds compared to untreated wood.

Moreover, the flame was significantly longer in the treated wood (172 s) and with longer glowing time (714 s). Nevertheless, the treated wood presented lower weight loss (26.9%) compared to the untreated wood (33.5%) which had smaller fire time and ember (137.7 and 335.20 s respectively). These results show that treatment improves the performance of the timber in relation to

ignition time and mass loss when the wood is exposed to direct heat for 2 min. According to Li et al. [2014], a fire retardant is seen as any substance that by physical or chemical action inhibits combustion.

Table 2. Short time fire test and ignition time of the three species studied

Samples	Ignition time (s)	Flame time (s)	Ember time (s)	Weight loss (%)
Untreated	13.2 (1.6) ^a	137.7 (18.0) ^a	335.2 (31.3) ^a	33.5 (1.4) ^b
Treated	26.6 (0.9) ^b	172.2 (10.2) ^b	714.2 (24.0) ^b	26.9 (1.3) ^a

The values in parentheses are SD. Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey test.

The fire resistance depends on the thickness of the wood carbonized layer and the size of the residual section, which for optimal use of wood species with respect to fire resistance depends on the charring rate [White 1995].

The differences in the wetting characteristics of wood treated and untreated by the sessile droplet method are presented in figure 1.

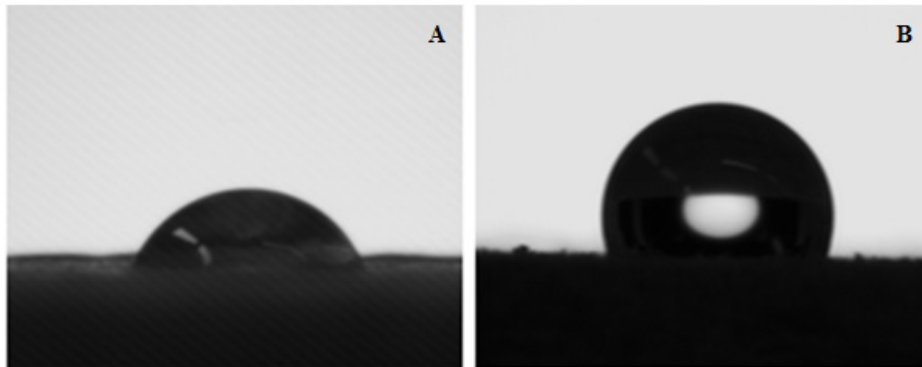


Fig. 1. The image droplet water in wood surface. A: untreated wood (time = zero), B: treated wood with EKL (time = zero)

In figure 2, it is possible to observe that the treatment reduces the rate of moisture uptake, but over time the wood absorbs a little of the water droplet reducing contact angle. The treated wood samples became more hydrophobic, that means that the treatment with the emulsion of Kraft lignin by full cell method is also more effective regarding the contact angles (CA). The effect that could have the decrease of the hygroscopicity in the dimensional stability of wood, however, was not studied.

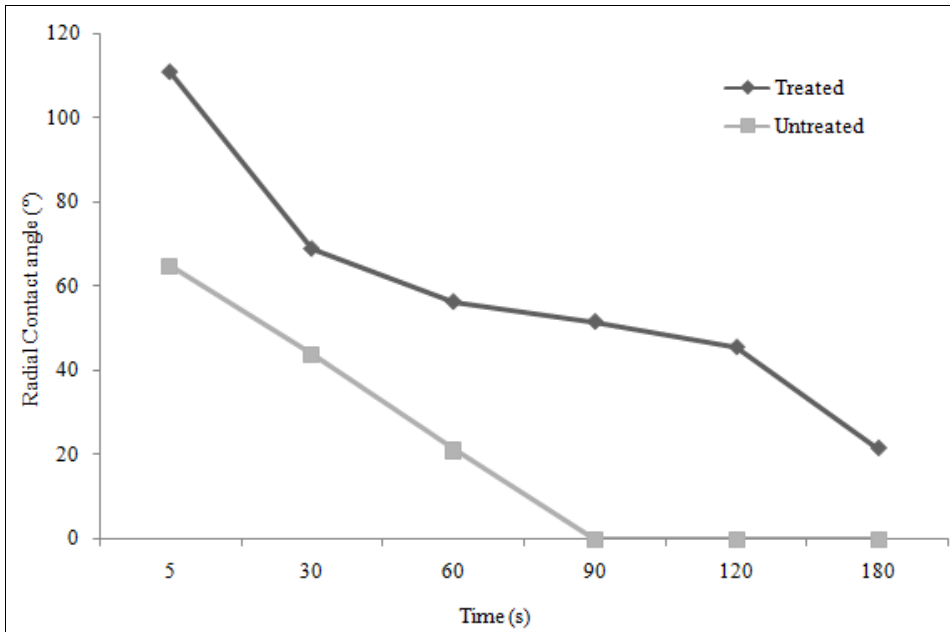


Fig. 2. Values of radial contact angle (°) in wood treated compared with untreated wood

Conclusions

The impregnation with EKL by the full cell method improved the performance of wood in relation to ignition of fire and the hardness of the wood, when compared with untreated wood. Furthermore, it improved the hydrophobic properties of wood species *Pinus* sp.

It is extremely important to perform other types of analyses (such resistance to leaching and evaporation, decay resistance) in future work to verify the possible applications and the limitations of using EKL.

References

- Dos Santos P.S., Garcia A., Cademartori P.H., Gatto D.A., Labidi J. [2012]: Study of the use of organosolv lignin as bio-preservative of wood. In: IUFRO Conference Division 5 Forest Products: IRG/WP 12-30603
- Fejfer M., Zborowska M., Adamek O., Dzieduszynska D., Kittel P., Petera-Zganiacz J., Twardy J. [2014]: Properties and dimensional stability of 12 500-year-old subfossil pine wood. *Drewno* 57 [193]: 81-95
- Li L., Hu H., Guo C. [2014]: The synergistic effect of boric acid and ammonium polyphosphate on the thermal degradation and flammability of pine-needles. *Drewno* 57 [193]: 38-49

- Quesada-Medina J., López-Cremades F.J., Olivares-Carrillo P.** [2010]: Organosolv extraction of lignin from hydrolyzed almond shells and application of the δ -value theory. *Bioresource Technology* 101 [21]: 8252-8260
- Tondi G., Wieland S., Wimmer T., Thevenon M.F., Pizzi A., Petutschnigg A.** [2012]: Tannin-boron preservatives for wood buildings: mechanical and fire properties. *European Journal of Wood and Wood Products* 70 [5]: 689-696
- Zhang H.** [2011]: Chapter 9 – Wood. In: *Building Materials in Civil Engineering*: 1st ed. Elsevier: 238-252, 423.
- White R.H.** [1995]: Analytical methods for determining fire resistance of timber members. *The SFPE handbook of fire protection engineering*. 2nd ed. Quincy, MA: National Fire Protection

List of standards

- ASTM D 5536:1994** Standard method of Sampling Forest Trees for Determination of Clear Wood Properties
- EN 1534:2011** Wood Flooring – Determination of resistance to indentation – Test method

Acknowledgements

The authors would like to give thanks to CNPq – CAPES Brazil (PhD scholarship (246018/2012-7 and 207252/2014-9), and project (014/2012), the Department of Education, and the Spanish Ministry of Economy and Competitiveness (CTQ2013-41246-R), the Federal University of Pelotas for financially supporting this work.

Submission date: 23.07.2015

Online publication date: 26.09.2016