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ABSTRACT

Improvement of dimensional stability and durability is wished for the use of wood as a building material. For the last decade, retification® has been industrially developed. It consists in a stabilization and preservation of wood by heat treatment.

The aim of this study is to find simple and fast methods to characterize heat treated beech. Non destructive testing is expected to be relevant to evaluate the level of treatment and the properties for the use of heat treated wood.

Six treatments were carried out in a pilot reactor. The parameters of the retification® stage (temperature and time) were studied.

For each treatment, the non destructive tests (free oscillations in the fundamental mode, colour and dry weight loss)were performed, and the properties for use (mechanical resistance and volumetric shrinkage) measured.

Lightness and dry weight loss seem to be suitable properties to characterize beech retification® when the time parameter is fixed. However, they are not suitable for other wood species, and for retification stages with a variable duration. Moreover, the correlation with the properties for use were plotted, but presented too large dispersion to be relevant.

After correction of moisture content, the longitudinal Young’s modulus of the material is slightly increased by each of the six treatments, but do not present any variation with changing parameters values. On the contrary, the mechanical resistance decreased with increasing temperature and time. Thus the dynamic Young’s modulus is not reliable to evaluate the treatment and to predict the loss of mechanical resistance.

The logarithmic decrement was not increased by any of the treatments, which is in opposition with the hypothesis that retification® generates cracks and microcracks in the material.

Effects of long time at low temperature have been investigated. From these experiments, properties of treated wood may be improved significantly by choosing appropriate values of the parameters.

INTRODUCTION

Improvement of dimensional stability and durability is wished for the use of wood as a building material. For the last decade, stabilization and preservation of wood by heat treatment have been industrially developed. Moreover, these heat treatments are more environmentally friendly than the chemical ones. Retification® is one of these heat treatments that stabilizes wood and improves its durability. However, their main drawback is the loss of mechanical resistance of the material that happens at high temperature.

Some tests involving rather complicated physical or chemical techniques have been developed for the evaluation of the treatment. The first aim of this study is to find simple and fast methods that allow to characterize the heat treatment of wood. Non destructive testing could easily reach these industrial requirements. But the considered properties have to vary significantly with the parameters of the retification stage (temperature and time), in spite of wood heterogeneity.

Six treatments have been carried out in a pilot reactor. The conduct was similar for all the treatments. The changing parameters were temperature (5 minutes at 200°C, 220°C, 240°C, and 260°C)) and time (600 minutes at 200°C and 60 minutes at 220°C).

Properties related to the use of wood, such as the modulus of rupture(MOR) and the volumetric shrinkage have been measured.

Three types of non destructive tests have been done. The measurement of CIE L* a* b* colour and dry weight loss were performed. Nevertheless, the efforts were focused on the free-free flexural vibrations test. Dynamic longitudinal Young's modulus (MOE) and logarithmic decrement were measured. These non destructive tests have been carried out before and after Retification®.

MATERIALS PREPARATION

The purpose of this work was to investigate the influence of the two main parameters of retification® on the material : temperature and time of exposure. Sis treatments were carried out in a pilot kiln, at the "Ecole Des Mines De Saint Etienne". This pilot kiln regulates by PID on the temperature of its atmosphere. The specimens were 850×85×25 millimetres beech beams, carefully cut in the longitudinal direction. Before heat treatment the specimens had been hold in a climatic chamber at 65% relative humidity 20°C. The moisture content (MC) of the specimens was 12%. Each batch was composed of sixteen specimens. The kiln is equipped with eight thermocouples that allow to follow the temperature reached by the wood specimens.

Table 1 summarises the treatments parameters. The atmosphere was composed of nitrogen gas. The increase in temperature matched the standard one, used for the retification® process (Weiland 2000).

Four treatments were performed with a fixed time of exposure of 5 minutes. As the wood transformation is known to begin around 200°C, we chose the following temperatures : 200°C, 220°C, 240°C and 260°C. In order to investigate the effect of long time exposure at low temperature of retification®, two treatments were carried out, at 200°C during 600 min and at 220°C during 60 min.

Table 1: Experimental parameters of the treatments

Treatment number	Temperature [°C]	Time [min]
1	200	5
2	220	5
3	240	5
4	260	5
5	220	60
6	200	600

For each treatment, non destructive tests (size, weight, colour and free vibrations) were performed before and after the treatment. After treatment, the specimens had been held until equilibrium in a climatic chamber at 65% relative humidity 20°C before being tested.
The physical properties (volumetric shrinkage, MOR, and MC) of untreated and treated wood were measured by destructive methods. For each measurement, 48 samples have been cut and tested.

EXPERIMENTAL PROCEDURE

Evaluation of properties for use

There are three main properties for the use of wood : mechanical resistance, volumetric shrinkage, and fungus resistance. In this work, we focused on the physical properties : mechanical resistance (evaluated by static bending) and volumetric shrinkage.

Mechanical resistance

The bending strength of treated wood was measured using a four points bending device. 48 beams have been tested for each batch. The measurements were done following the French normative NF B 51-008. Normalised beams of dimension 20*20*360 mm underwent a force applied on the (LR) plane.

Volumetric shrinkage

This test is carried out on cubic samples of around 2cm edge size. The volume of a cube is measured in the water saturated state and in the anhydrous state. The volumetric shrinkage is then calculated by the formula :

$$S = 100 \times \frac{V_S - V_0}{V_S} \qquad (1)$$

where S is the volumetric shrinkage,
V_S is the volume of the water saturated sample,
V_0 is the volume of the dried sample.

Evaluation by non destructive testing

Non destructive evaluations can be done before and after the treatment. Consequently, they allow to get partly rid of the natural dispersion of wood, and to measure only the modifications due to the heat treatment.

Colour

The colour measurements were carried out using a MINOLTA Spectrophotometer CM-508i. The principle of colour measurement is inspired by the human vision. One colour is repaired in a three coordinate space. Thus, the device records three coordinates : L* (also called lightness), a*, and b*. They match the three following pairs of colour : from white (L*=100) to black (L*=0), from red (a=+60) to green (a*=-60) and from yellow (b*=+60) to blue (b*=-60). Thirty points have been recorded on each specimen, before and after the heat treatment.

Free oscillations

The vibrational properties were evaluated in the fundamental mode of free-free flexural vibrations. The impulse was given on the flat face of the specimens. The support were placed at the nodal location of the first vibration mode. A piezoelectric captor recorded the vibration of the sample at the end of the beam (Fig. 1). Since the length of the specimens was ten times superior to their thickness, we could do the computation of the dynamic Young's modulus following the elementary Euler-Bernouilli's theory. Two quantities could be measured : dynamic MOE and logarithmic decrement.

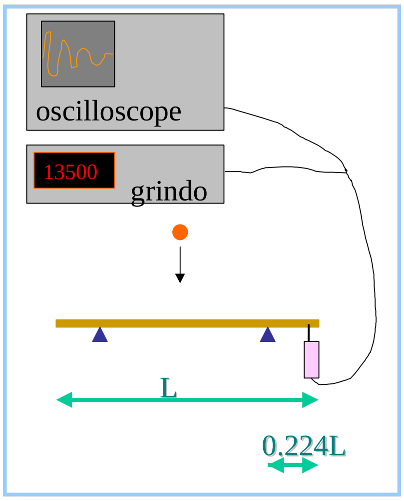

figure 1: scheme of the device used to measure free oscillations.

The dynamic Young's modulus can be calculated from the following Eq. 2. It is linked to the stiffness of the material measured by static bending (Pellerin 1965).

$$E = \frac{48\pi^2}{(kL)^4}\frac{ML^3}{ba^3}f_i^{\,2} \qquad (2)$$

Where E is the dynamic modulus of elasticity in the longitudinal direction, M is the beam weight, L its length, a its thickness, and b its width. f_i is the natural frequency of vibration in the i^{th} mode, and kL is the solution of Eq. 3 matching the i^{th} mode of vibration. This Eq. 3 is worth in the case of free-free vibrations.

$$1 - \cos(kL)\mathrm{ch}(kL) = 0 \qquad (3)$$

Since we are working in the first mode we have :

$$E = 0{,}9464\frac{ML^3}{ba^3}f_1^{\,2} \qquad (4)$$

or :

$$\frac{E}{\rho} = 0{,}9464\frac{L^4}{a^2}f_1^{\,2} \qquad (5)$$

where E/ρ is the specific dynamic MOE and ρ is the specific gravity of the specimen.
As the specimens were cut in the longitudinal direction, we measured the Young's modulus in the longitudinal direction.

The logarithmic decrement gives an evaluation of the internal friction of the material. It is linked to the volumetric viscous damping force, which depends on the presence of defects, of microcracks and on the MC. From the experimental point of view, we measure the amplitude of the Nth oscillation, and the amplitude of the (N+n)th oscillation and calculate the logarithmic decrement following Eq. 6 :

$$\delta = \frac{1}{n} \ln\left(\frac{A_{\max N}}{A_{\max N+n}} \right) \quad (6)$$

Since the MOE and the logarithmic decrement should depend on the MC of the specimens, we fixed the correction relationships. In this purpose, four specimens of untreated beam were conditioned in a climatic chamber at different relative humidity. Their moisture content varied from 0 to 20% and we plot the logarithmic decrement and the specific dynamic MOE versus their MC. For the specific dynamic MOE, linear regression gave an average slope of –0.255 GPa per percent of water. For the logarithmic decrement the linear regression gave an average slope of 9 10^{-4} per percent of water, but the data scattering was very large.

RESULTS AND DISCUSSION

Properties for use

Table 2 and Fig. 3 present the results of the tests.

The volumetric shrinkage decreases monotonically with increasing temperature and time of treatment. Indeed, the main effect of retification® is the destruction of the hemicelluloses (Weiland 2000), which are the most hydrophilic constituents of wood. Consequently, the wood is less sensitive to moisture.

Table 2: Properties for use of treated and untreated beech

Treatment number	**Volumetric shrinkage S [%]**	**MOR [Mpa]**
Untreated	15.9 (1.4)	94.8 (15.8)
1	15.0 (1.4)	100.4 (24.9)
2	14.0 (1.0)	97.9 (21.1)
3	11.7 (1.8)	74.3 (19.3)
4	8.3 (1.2)	59.6 (21.0)
5	11.6 (1.2)	84.6 (18.7)
6	10.6 (1.5)	74.0 (25.6)

The mechanical resistance decreases from 240°C. This decay matches a degradation of the material. Since the modulus of elasticity is also a mechanical property, we expect that it should vary similarly than the MOR.

Increasing time of treatment result also in decrease of the volumetric shrinkage and the MOR.

Effect of temperature of treatment

<u>Effect of the temperature on different properties</u>

The aim of this work is to find out properties easy to measure and that vary significantly with the level of treatment. For this purpose, a review of different properties of treated wood is presented in table 3. The colour (through the lightness L*) and the dry weight loss are the most influenced properties. They are already known to be relevant for the control of heat treatment of wood. Concerning the four treatment of five minutes, they both vary accurately with the temperature actually reached by the specimens (Fig. 2).

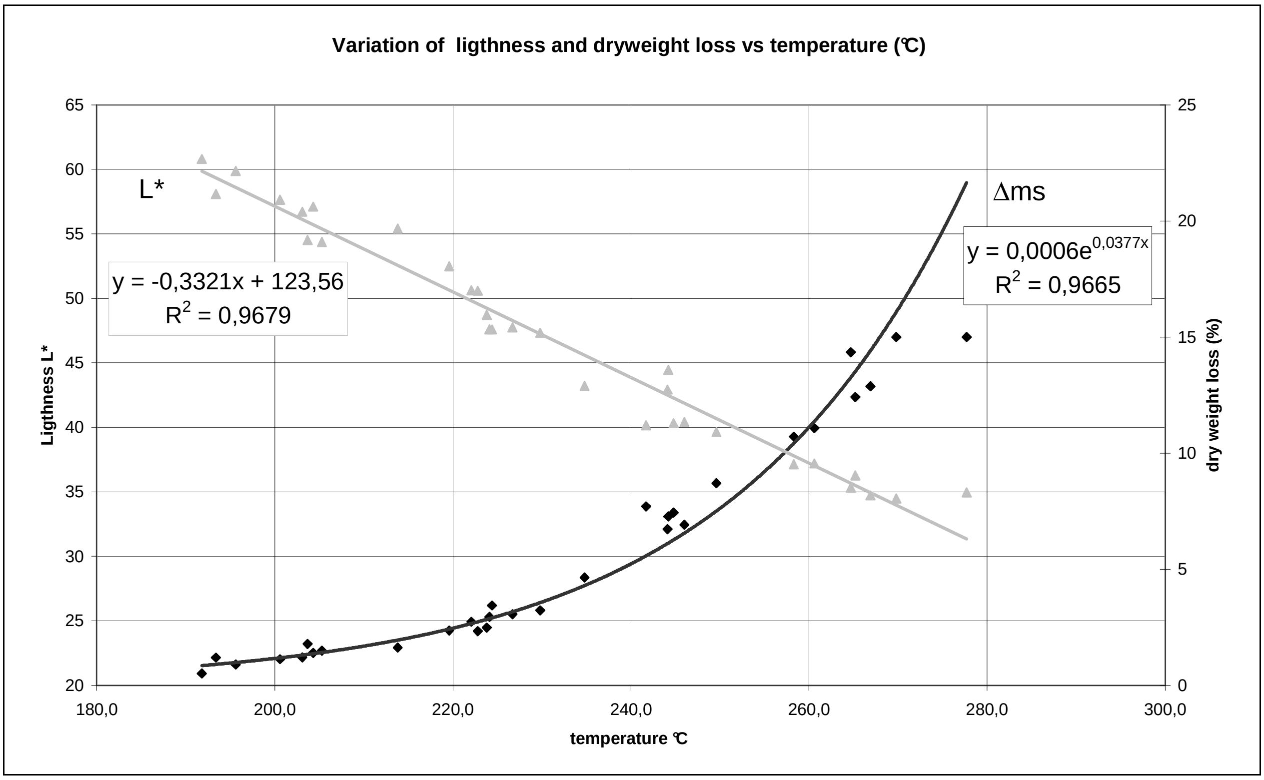

Figure 2: dry weight loss and lightness plot against the temperature reached by each beam of treatment 1, 2,3 and4.

Consequently, once the time of exposure is fixed, dry weight loss and lightness can be used to evaluate the retification® temperature.
However, they can not be used to evaluate both temperature and time of retification®. Indeed, treatments 5 and 6 result approximately in the same lightness and in the same dry weight loss as treatment 3, but they involve longer time.

Nevertheless, the dry weight loss should be used carefully. In this work it varies well with temperature, because beech are broad lived tree and do not contain resin. The weight loss of softwood is known to be unreliable as it depends on their resin content.
The use of colour also may not be relevant with all species of tree. Indeed, beech wood has a very homogeneous colour, which is not the case for all wood species.

Table 3: Results of non destructive tests

Treatment number	**Dry weight loss Δms [%]**	**Equilibrium MC [%]**	**Colour : lightness L***	**Colour : a***	**Colour : b***
untreated	-	12.0 (0.3)	70.7 (3.5)	7.7 (0.7)	18.6 (1.2)
1	1.3 (0.3)	7.0 (0.8)	56.5 (2.2)	7.4 (0.6)	17.3 (0.6)
2	2.7 (0.7)	5.3 (0.5)	49.6 (2.9)	8.1 (0.4)	17.2 (0.9)
3	6.5 (1.7)	4.1 (0.7)	41.9 (2.9)	7.8 (0.5)	14.5 (1.4)
4	12.8 (1.9)	3.7 (0.3)	36.2 (2.1)	6.3 (0.7)	11.3 (1.6)
5	6.9 (1.6)	4.2 (0.5)	42.6 (2.3)	7.5 (0.3)	14.6 (1.2)
6	7.8 (0.8)	4.9 (0.5)	41.7 (1.9)	8.5 (0.5)	16.0 (1.2)

Moreover, correlation between the properties for use and lightness and dry weight loss were plotted, but presented too large dispersion to qualify the retification®. That is why, we proposed to evaluate the relevance of the free oscillations method.

Vibrational properties

Table 4: Vibrational properties of heat treated beech

Treatment number	Ratio : ((Ed/ρ)treated)/ ((Ed/ρ)untreated)	Ratio : (δtreated)/ (δuntreated)
Natural	1 (0)	1 (0)
1	1.14 (0.06)	0.98 (0.10)
2	1.13 (0.07)	0.86 (0.07)
3	1.16 (0.02)	0.76 (0.09)
4	1.14 (0.02)	0.80 (0.07)
5	1.14 (0.02)	0.64 (0.09)
6	1.13 (0.02)	0.85 (0.08)

There are two observations concerning the specific dynamic Young's modulus (Table 4):
- it increases from natural to treated state,
- there is no obvious variation following the parameters.

To conclude, it is not possible to evaluate the treatment with this property.

The logarithmic decrement decreases with increasing temperature of treatment from 200°C to 240°C. But the scattering of the data is so large that it is again impossible to qualify the heat treatment with this property.
By destruction of the hemicellulose, heat treatment makes the wood loose its affinity for water. As a consequence, viscous damping is reduced and the logarithmic decrement decreases. Moreover, the stress strain curves of the high temperature batch (treatment 3 and 4) have the shape of brittle material. The heat treated wood behaves more like brittle material progressively with increasing values of the parameters.

During retification®, two phenomenon may influence the vibrational properties : loss of MC (that increase the MOE and decrease the logarithmic decrement), and lignocellulosic material modification. In order to investigate the influence of the material modification on the vibrational properties alone, it was necessary to calculate and to eliminate the influence of the MC.

Correction of the vibrational properties with MC

We know that the equilibrium MC of the treated wood decreases with increasing temperature and time of treatment (Table 3). Consequently, we had to do the correction of the vibrational properties values according to the variation in moisture content. Linear regression of the specific dynamic MOE of untreated wood vs. MC between 0% and 20% has been plot. For each batch, one could then compute the specific dynamic MOE of untreated wood at the same moisture content as the equilibrium moisture content of the treated wood ((Ed/ρ)untreatedcor).
The ratio ((Ed/ρ)treated)/((Ed/ρ)untreatedcor) so calculated (Table 5) allow to compare the natural and treated wood at the same moisture content.

Once the correction is carried out, one observe that MOE of wood is slightly enhanced by each of the treatments. We can conclude from this, that the modification of material result in an increase of the Young's modulus. Phenomenon involved in this modification are discussed. As well as before correction, the scattering of the increase is large, which forbids to make a difference between treatments of different parameters.

The same correction in MC was done for the logarithmic decrement. Considering the values obtained and the scattering of the data, one couldn't notice obvious difference between the decrement of anhydrous wood and the decrement of treated wood.

Table 5: corrected vibrational properties of heat treated beech

Treatment number	**Ratio : ((Ed/ρ)treated)/ ((Ed/ρ)untreated cor)**	**Ratio : (δtreated)/ (δuntreated cor)**
1	1.07 (0.06)	1.12 (0.13)
2	1.04 (0.02)	1.00 (0.10)
3	1.04 (0.02)	0.90 (0.12)
4	1.02 (0.01)	1.00 (0.10)
5	1.04 (0.02)	0.77 (0.12)
6	1.04 (0.01)	1.03 (0.11)

We can conclude from this, that the measurement of the vibrational properties in the fundamental mode is not relevant to make an evaluation of the retification® of beech beams.

Mechanical and vibrational properties

Another point concerns the mechanical resistance of the treated wood. The bending strength decreases significantly from 240°C (Table 4). During the heat treatment, physical and chemical transformations of the material lead to a loss of mechanical resistance. In the spite of this, the specific Young's modulus is not modified from 200°C until 260°C. Thus, the material degradation causes reduction of mechanical resistance, but no variation of the modulus of elasticity.

The longitudinal MOE of a beam depend strongly to the microfibril angle of the S2 layer, and depend less strongly on the crystallinity of cellulose and stiffness of the amorphous matrix (Ono, Norimoto 1983). These three elements of the wood structure could be slightly influenced by the heat treatment and result in this slight increase of the young's modulus.

The mechanical strength of wood is more linked to the presence of cracks, and their opportunity of initiation and propagation. Thus, the loss of mechanical resistance may be attributed to a higher number of cracks and microcracks in the treated material, or/and to a decrease of the energy necessary for a crack to initiate and propagate in the material.

With microscopic investigation of the microstructure, no obvious damage is visible on heat treated wood (Avat 1993). Moreover, since the logarithmic decrement is decreased by heat treatment (or at least not increased once the moisture correction is done), the hypothesis of a higher number of microcracks in the treated beech should be rejected. Indeed, the presence of more microcracks should lead to a higher internal friction, and thus to an increase of the logarithmic decrement.

As a conclusion, more investigation is necessary to find out the influence of the retification® on the structure and microstructure of the wood, and to do the link with the macroscopic properties as mechanical resistance and vibrational properties.

Effect of long time treatment

The last point of the study was the effect of long time treatment. Treatments number 5 and 6 were performed (Fig.3) and (Table 2). One of the benefit of long time treatment at low temperature is that the conduct of the process is easier. Indeed, no exothermic effect is expected and the temperature homogeneity is better in the pilot kiln. Moreover, a lot of reaction is involved during retification® (Bohnke 1993, Weiland 2000). By choosing low temperature and long time treatment, one intend to improve reactions that decrease volumetric shrinkage, and to slow reactions that decrease the MOR.

Treatment 5 and 6 result both in a decrease of the volumetric shrinkage and in a loss of the mechanical resistance. It seems to be difficult to get reduction of volumetric shrinkage without reduction of the bending strength. In the spite of this general observation, there is no evidence that these two effects are caused by the same reactions ; and we may find an optimum of the parameters values, where the mechanical loss would be reduced.

Concerning the volumetric shrinkage, a one hour retification stage at 220°C (batch number 5) allowed to reach the same level of stabilisation as a five minutes treatment at 240°C (batch number 3). The mechanical resistance seem to be slightly less reduced, but the dispersion of MOR is large. Nevertheless, the difference on average MOR between number 3 and 5 is statistically significant at risk 2%.

Since this difference is statistically significant, we expect that an optimum of the parameters exists to get the best compromise for the use properties. Moreover, comparing treatment 6 and 3, the volumetric shrinkage is lower for treatment 6 with approximately the same value of MOR.

According to these observations, reactions that improve the reduction of volumetric shrinkage should prevail at low temperature (until 220°C) with regard to reactions that cause mechanical degradation. Though, more investigation is necessary to find out what reaction occurs at a given temperature.

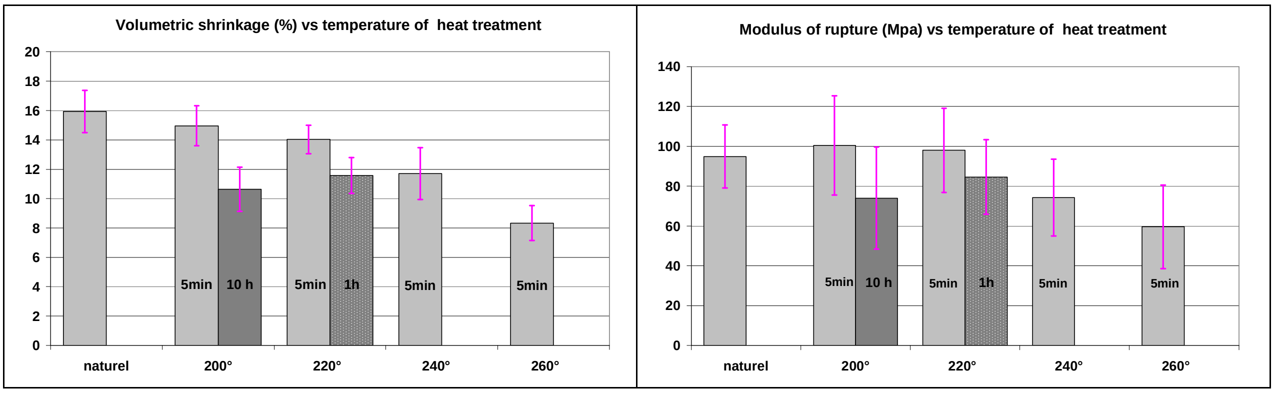

Figure 3 : properties for use of retified® beech.

CONCLUSION

Six treatments have been carried out in a pilot reactor. The parameters of the retification® stage (temperature and time) have been studied.

For each treatment, the non destructive tests (free oscillations in the fundamental mode, colour and dry weight loss) were performed, and the properties for use (mechanical resistance and volumetric shrinkage) measured.

Lightness and dry weight loss seem to be suitable properties to characterize beech retification® when the time parameter is fixed. However, they are not suitable for other wood species, and for retification stages with a variable duration. Moreover, the correlation with the properties for use were plotted, but presented too large dispersion to be relevant.

After correction of moisture content, the longitudinal Young's modulus of the material is slightly increased by each of the six treatments, but do not present any variation with changing parameters values. On the contrary, the mechanical resistance decreased with increasing temperature and time. Thus the dynamic Young's modulus is not reliable to evaluate the treatment and to predict the loss of mechanical resistance.

The logarithmic decrement presented a large dispersion, which does not allow to qualify retification® with this property. After moisture correction, it was not increased by any of the treatments. This observation is in opposition with the hypothesis that retification® generates cracks and microcracks in the material.

Effects of long time at low temperature have been investigated. From these experiments, it results that significant improvement of the wood properties may be expected by choosing appropriate values of the parameters.

More investigation is required to find out what reaction prevails according to the temperature, and to understand better the mechanisms that cause the loss of mechanical resistance.

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