Study of the wettability and color modification of torrefied wood

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1 Introduction

The presented work integrates a larger study about the stability of the properties conferred by the heat treatment of wood. In this article are presented the first results of this study, the evolution of the mass, the wettability and the color of heat-treated wood in function of the temperature treatment. Many works have already been done on the evolution of mass and color (Acharya et al. 2012; Herrera-Díaz et al. 2019; Sivrikaya et al. 2019; Xu et al. 2019). We have chosen to work on two European wood species, commonly used in industry (beech and fir). The process is deliberately carried out beyond the normal heat treatment temperature (230 °C) to go into the area of low wood pyrolysis (300 °C) in order to be able to observe the temperature influence. The results which are presented here are in good agreement with the literature, except the work on the wettability, the results are different from what can be found in the literature (Hakkou et al. 2005; Pétrissans et al. 2003). Using the Wilhelmy method, to characterize the wettability, which seems to us to be the most suitable, unlike the very used method of the sessile drop; we observe a strong evolution of the advancing contact angle. There is also a strong correlation with the thermal transformation parameters, here the temperature. The evolution is almost linear. The receding contact angle remains constant and equal to zero. The hydrophilic wood becomes hydrophobic after heat treatment. More the wood is treated, more the wood becomes hydrophobic. Obtaining an advancing contact angle = 90°, completely prevents the sorption of water in the wood by capillarity. The wood is protected from liquid water and thus preserved from certain degradation agents. This point is interesting, but deserves to be confirmed over time, depending on the use that is made. This will be the subject of the overall study.

2 Material and methods

2.1 Wood heat treatment

We have chosen to work on heartwood with two European wood species, commonly used in industry. The wood species are: Beech (*Fagus Sylvatica*) and Fir (*Abies Alba*). The size of the sample heat treated is: 140mm*60mm*20mm (L*R*T). Wood heat treatment is realized under different temperature :120, 140, 160, 180, 190, 200, 210, 220, 230, 240, 250, 275, 300℃. To perform the heat treatment, we used a reactor, placed in a controlled oven. The atmosphere is inert, using nitrogen.

Treatment process is realized as following drying of the samples for 48h at 103℃, still mass stabilization, then we measured the weight (M_1). The heat treatment is done from the room temperature to the target temperature, by an increasing of 2°C/min, the temperature constant during 120min and cooled to the room temperature without control of the decreasing of the temperature. After the heat treatment the weight is measured (M_2), and the mass loss (ML) calculated:

$$ML = (M_1 - M_2)/M_1 \qquad \text{Equation 1}$$

2.2 Wettability of wood

The advancing contact angles of beech and fir heat-treated at different temperatures were tested by the Wilhelmy method (Wålinder et al. 2001) with Krüss machine. We intercepted small samples along the grain from the large specimens (140mm*60mm*20mm) before and after heat treatment for testing, and the specimens heat-treated at each temperature were tested 10 times. The length of the test is 24mm (consistent with the direction of L), the width is 1mm (consistent with the direction of R), the immersion depth is 10mm (consistent with the direction of T), and the immersion speed is 5mm/min. The final contact angle (advancing and receding) values is the average of 10 valid tests.

Figure 1 is a schematic diagram of the Wilhelmy method. Immersing a sample plate (width l) of wood into a probe liquid (water) along an immersion depth (h) and pulling it out allows the calculation of advancing and receding contact angles (θa, θr).

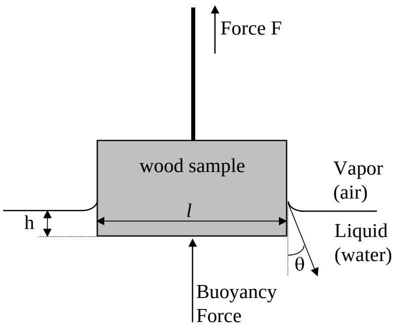


Figure 1. The Wilhelmy method

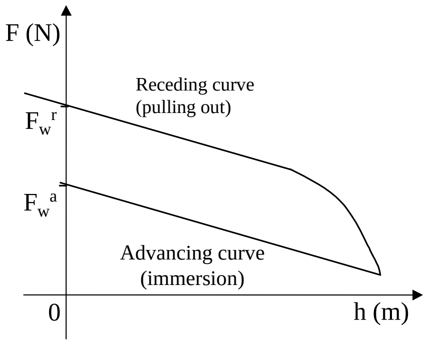


Figure 2. Forces recorded by the tensiometer as a function of immersion depth (h)

Figure 2 shows the force recorded by tensiometer as a function of immersion depth (h). The wetting forces (F, F_w^r; F_w^a) applied on the plate and the immersion depth is measured during the test cycle. For the value of the forces the dynamic contact angles (θa, θr) can be calculated according to equation: $F_w = \cos\theta \, \gamma \, P$ Equation 2

where γ is the liquid surface tension and P is the sample perimeter.

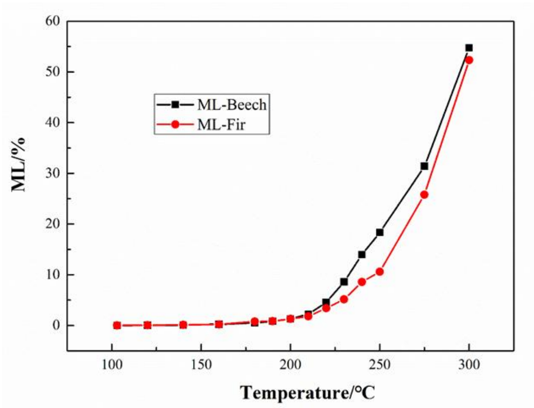
2.3 Wood color

Color change of the heat-treated samples was measured by a Chroma Meter CR-410 spectrophotometer according to the CIELab system using three replicates. Based on the L*, a*, b* color coordinate system, L* represents the black-and-white axis; for black, L* = 0 and for white, L* = 100; a* represents red-green color based on the positive and negative axes and b * represents yellow-blue color (positive value to yellow, negative value to blue). There are two test pieces at each heat treatment temperature. The front and the back sides of the heat-treated samples of each collecting point were photographed. Therefore, the average value of 8 points is taken as the final value.

3 Results and discussion

3.1 Mass loss

Figure 3 is a graph of the mass loss trend of beech and fir at different temperatures. It can be seen from the figure that under the same conditions (Candelier et al. 2013), as the heat treatment temperature increases, the mass loss of the two types of wood shows an upward trend. Between 100-210°C, the mass loss does not change significantly. Between 210-300°C, the value of mass loss rises sharply. Compared to beech, the mass loss of fir is slightly lower, because the thermosensibility of the hemicelluloses of the hardwood is superior than the softwood, this phenomenon is consistent with the result of scholar Čermák P (Čermák et al. 2021).



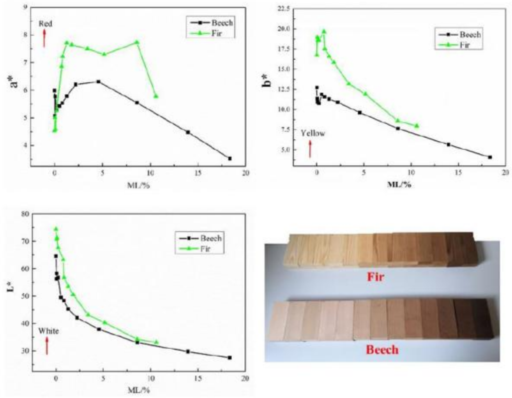


Figure 3. The mass loss of beech and fir at different temperatures

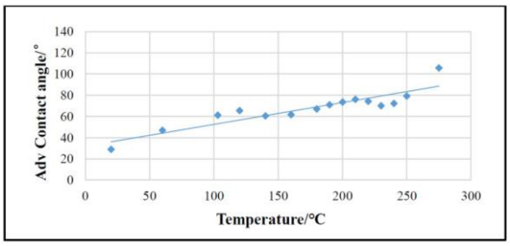
Figure 4. Color changes of beech and fir specimens under different mass loss

3.2 Wood color (before and after heat treatment)

Figure 4 shows the color changes of beech and fir specimens under different mass loss. It can be seen from the figure that as the heat treatment temperature continues to increase, the color of the beech and fir specimens will become darker and darker, beech is obviously darker than fir. The color of these two woods before heat treatment is white or yellow. As the heat treatment temperature increases, the degree of white or yellow becomes lower and lower. This change is more obvious in beech than fir.

3.3 Wettability of Beech and Fir

Figure 5 and Figure 6 are the advancing contact angles of beech and fir heat-treated at different temperatures, tested by the Wilhelmy method. All the measurement of the receding contact angle are equal to zero, that means that before and after the treatment the wood cannot be dewetted by the water, this result is in good accordance with the literature (Hakkou et al. 2005; Moghaddam et al. 2016). It can be seen from the figure that the advancing contact angles of beech and fir both show a trend of increasing first, then flattening, and finally increasing. The contact angle of the beech wood specimen increased from room temperature to 103°C, tended to a constant state at 103°C-160°C, and continued to increase at 160°C-275°C. The contact angle of fir specimens increases from room temperature to 60°C, tends to be constant at 60°C-120°C, and continues to increase at 120°C-300°C.



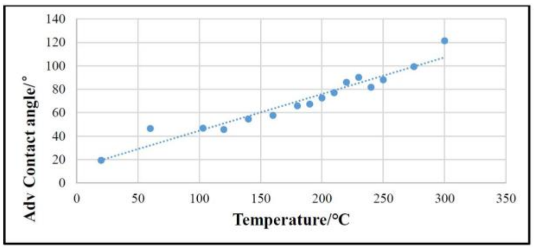


Figure 5. Advancing contact angle of Beech

Figure 6. Advancing contact angle of Fir

In these conditions we can observe the great dependance between the wettability (advancing contact angle) and the temperature of the treatment. The wood has a hydrophilic character before the treatment and became hydrophobic after. Upper than 90°, due to the physical law (Jurin law) the capillary rising is equal to zero, that means that the water cannot be sorbed by the wood. This observation is very important for the preservation of the wood. Protected to the water, his durability increase.

4. Conclusion

The results obtained on mass loss and color are in good agreement with the literature. The wood losses its mass as the temperature increases. The evolution is stronger for the beech than for the fir. This phenomenon is well known, it is due to the thermosensitivity of the hemicelluloses of the hardwood (beech) which is greater than that of the softwood (fir). Regarding the color, the increasing of the treatment temperature, the wood became darker and then black. This color change is generated by the thermodegradation of biopolymers. Concerning the wettability, we observe a strong evolution of the advancing contact angle. There is also a strong correlation with the thermal transformation parameters, here the temperature. The evolution is almost linear. The receding contact angle remains constant and equal to zero. The hydrophilic wood becomes hydrophobic after heat treatment. The higher treatment temperature, the more hydrophobic will become. Obtaining an advancing contact angle = 90°, completely prevents the sorption of water in the wood by capillarity. The wood is protected from liquid water and thus preserved from certain degradation agents. This point is interesting, but deserves to be confirmed over time, depending on the use that is made. This will be the subject of the overall study.

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