Effect of thermal treatment on physicochemical properties of Gympie messmate wood

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ABSTRACT

The aim of this study was to evaluate the chemical composition and the wettability of Gympie messmate (*Eucalyptus cloeziana*) thermally modified wood. To achieve this, Gympie messmate wood samples were thermally modified in an oven at 180–240 °C and in a combination of oven (180–240 °C) and autoclave (127 °C) for 4–5 h. Chemical composition were investigated by TAPPI standards, high-performance liquid chromatography, ATR-IR spectroscopy and thermogravimetric analysis (TGA), while wood wettability were evaluated by contact angle technique. The main results showed that increment of extractives was due to its condensation, while reduction most of the monomeric sugars was due to the thermal treatments at high temperatures. Hydrophobicity of thermally treated wood increased, in which treatments at 180 and 200 °C presented the best results in relation to the contact angle increment. The use of autoclave (pre-treatment) affects significantly only extractives and others sugars content. However, all the thermal treatments modified significantly the wettability and the chemical properties of Gympie messmate wood.

1. Introduction

Wood is a lignocellulosic material that has ample utilisation in different industrial sectors. Naturally, this material presents intrinsic properties that limit its use, such as hygroscopicity and anisotropy. However, along to the years, many wood treatment processes have been developed in order to aggregate value to this material. Specifically, the thermal rectification is an ecological process that avoids the use of toxic chemical products to preserve the wood and contributes to decrease the impacts of wood chemical treatment technologies on the environment.

The first reported experiments of wood exposition to high temperatures were performed in the Forest Products Laboratory in 1916 (Hill, 2006). The thermal modification of wood is the most advanced process commercially speaking, and it is performed between 180 and 260 °C. This treatment is mainly used to improve the dimensional stability and the decay resistance of wood (Hill, 2006). The level of modifications in the structure of wood depends on many factors such as temperature, time of exposure, the state of the specimens and the atmosphere of the treatment (Windeisen et al., 2007).

All the structural modifications of wood are related to its chemical composition. Cellulose and hemicellulose are polysaccharides that constitute the cell wall, whereas the lignin has an aromatic structure and presents itself as a "cement" of lignocellulosic fibers, providing rigidity to the material (Garcia et al., 2010). Cellulose is responsible for strength in wood fibers due to its high degree of polymerisation (Pandey, 1999).

Several studies have been developed (Pandey, 1999; Boonstra and Tjeerdsma, 2006; Brito et al., 2008; González-Peña et al., 2009) in order to understand the chemical behaviour after the thermal modification of wood. Lekounougou et al. (2009) reported that only significant chemical modifications increase wood durability.

The main modification of the chemical structure of wood after a thermal rectification occurs due to autocatalytic reactions of the cell-wall constituents (Tjeerdsma and Militz, 2005). Under heat, cellulose and lignin present a more slow degradation than the hemicelluloses. On the other hand, most extractives degrade or disappear under heat (Esteves and Pereira, 2009). At the same time, the degradation of hemicelluloses during thermal treatment leads to an increase of the crystallinity degree of cellulose (Cao et al., 2012).

Wood from Gympie messmate (*Eucalyptus cloeziana* F. Muell.) presents great durability (for both in-ground and above-ground applications) and mechanical strength, both rated class 1 (Harding et al., 2012). In this context, thermal treatment could be a worthy alternative to increase physical properties and, consequently,

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Table 1
Thermal treatments realised in the Gympie messmate wood.

Treatment	Temperature (°C)	Time (h)
1	20	–
2	180	4
3	127 + 180	1 + 4
4	200	4
5	127 + 200	1 + 4
6	220	4
7	127 + 220	1 + 4
8	240	4
9	127 + 240	1 + 4

the number of different utilisations of Gympie messmate wood could be increased. However, it is very important to understand its chemical behaviour in order to select the best combination and the treatment parameters.

Considering these facts, the present study aimed to evaluate the chemical composition and the wettability of Gympie messmate (*E. cloeziana*) wood thermally modified at different levels of temperature.

2. Materials and methods

2.1. Raw material

Six Gympie messmate (*E. cloeziana* F. Muell.) (17 years old) trees from homogeneous population were harvested in the north coast of Rio Grande do Sul state, in the south of Brazil (30°14′09.73″S, 50°19′55.07″W).

The first log (3.15 m length) of each tree was cut. Then, samples of 16 mm × 16 mm × 250 mm (radial × tangential × longitudinal) in size were prepared, all of them with straight grain and made only of heartwood with absence of warps.

The wood samples were then placed in a climatic chamber at 20 °C and 65% of relative humidity to stabilise the equilibrium moisture, which was the initial condition for the thermal treatments.

2.2. Thermal treatments

Gympie messmate wood samples were thermally modified by eight distinct thermal treatments (Table 1).

Sixty wood samples were submitted to a pre-treatment in an autoclave steriliser (127 °C and 1.5 kgf cm^{-2}) for 1 h in order to simulate wet conditions and indirect steam. After this step, pre-treated samples were maintained in the climatic chamber to stabilise the equilibrium moisture. Thus, one hundred and twenty wood samples (pre-treated in autoclave and untreated) were treated in an oven without air-forced circulation at 180, 200, 220 and 240 °C for 4 h in order to simulate dry conditions. The heating rate of the oven was 2 °C/min. After the thermal treatments in the oven, the wood samples were then placed in a climatic chamber at 20 °C and 65% of relative humidity to stabilise the equilibrium moisture. Likewise, the control treatment (15 samples for reference) was maintained in the climatic chamber the whole time.

2.3. Chemical analysis

The chemical analyses were performed in control and thermally modified wood milled samples (40 mesh).

The ethanol–toluene extractives (TAPPI T204 om-97), hollocellulose (Wise et al., 1946), α-cellulose and hemicellulose (Rowell, 1983), and Klason lignin (TAPPI 222 om-98) were determined.

2.4. High-performance liquid chromatography (HPLC)

The content of main sugars (glucose, xylose, mannose, galactose and arabinose) present in wood was determined by high-performance liquid chromatography technique. For this, the sugar content in the liquid originated from Klason lignin tests (three samples in duplicate) was determined by a Jasco LC-Net II/ADC equipment with a photodiode array detector MD-2018Plus, refractive index detector RI-2031Plus and Rezex ROA Organic Acid H+ (8%) column.

Dissolution of 0.005 N H_2SO_4 with 100% of deionised and degassed water was used as a mobile phase. The conditions of the samples injection were 30 °C, 0.35 mL/min flow and volume of 40 μL.

2.5. ATR-IR spectroscopy analysis

The ATR-IR spectra of control and of thermally modified wood milled samples were measured in a Nicolet Nexus 570 equipment by direct transmittance at a resolution of 4 cm^{-1} for 32 scans in the range from 700 cm^{-1} to 4000 cm^{-1}. The alignment of the light equipment and the background spectra were collected before all the tests.

The representation of the spectra was determined by the average of 6 measurements for each treatment.

2.6. Thermogravimetric analysis (TGA)

The thermogravimetric analysis was carried out in order to observe the behaviour of control and thermally modified wood. The tests were done in Mettler Toledo equipment and the milled samples (between 5 and 10 mg) of each treatment were exposed to a temperature range between 25 and 800 °C at a constant heating rate of 10 °C/min under a constant inert atmosphere of N_2. The weight gain (TG) and the mass loss rate (DTG) were determined.

2.7. Wettability measurement

The wettability behaviour of control and thermally modified samples (16 mm × 16 mm × 250 mm) (stabilised at equilibrium moisture content) was measured by a Dataphysics 0CA goniometer (sessile droplet method).

The contact angle was determined through the disposition of a distilled water droplet (5 μL) in three distinct points of longitudinal radial and longitudinal tangential sections of each of five samples per treatment. The first measurement occurred after 5 s of the droplet contact with the sample surface. The other measurements were performed at 15 s intervals up to a total of 90 s, totalling seven measurements.

2.8. Data analysis

The collected data were assessed by statistical software OriginPro 8.6 using descriptive statistic and analysis of variance ($p < 0.05$) with a factorial design 2 × 4, use or not of pre-treatment (2 levels) and temperature of treatment (180, 200, 220 and 240 °C). When the null hypothesis was rejected, the average values were compared with Tukey Test at the level of significance of 5%.

3. Results and discussion

3.1. Chemical analysis

According to Table 2, the holocellulose ($F = 67.57$, $p < 0.0001$) and lignin ($F = 13.70$, $p < 0.0001$) contents were significant only for the temperature of treatment factor. The extractives content was

Table 2
Summary of the factorial analysis of variance (ANOVA) for the chemical properties with pre-treatment and temperature as factors.

Variable	Source	SS	df	MS	F
Extractives	Pre-treatment (A)	3.76	1	3.76	16.33[*]
	Temperature (B)	56.59	4	14.14	61.35[*]
	A × B	1.14	4	0.28	1.25[ns]
	Residual	4.60	20	0.23	
	Total	66.10	29		
Holocellulose	Pre-treatment (A)	0.95	1	0.94	1.02[ns]
	Temperature (B)	250.77	4	62.69	67.57[*]
	A × B	4.70	4	1.17	1.26[ns]
	Residual	14.85	16	0.92	
	Total	269.20	25		
Lignin	Pre-treatment (A)	18.76	1	18.76	3.29[*]
	Temperature (B)	312.89	4	78.22	13.70[ns]
	A × B	8.89	4	2.22	0.39[ns]
	Residual	85.64	15	5.70	
	Total	433.40	24		

SS = sum of squares; df = degrees of freedom; MS = mean square; F = calculated value of F; ns = not significant.

[*] Significant at 5% of probability of error.

significant for the temperature of treatment ($F = 61.35$, $p < 0.0001$) and pre-treatment ($F = 16.33$, $p = 0.001$) factors. All the chemical analyses did not show significance for the interaction between pre-treatment (use of autoclave) and temperature of treatment.

Table 3 shows the chemical analyses of Gympie messmate thermally modified wood.

The extractives content increased significantly as a function of the temperature of treatment. The highest extractives value occurred in the thermal modified wood at 240 °C. In relation to the significant difference for the pre-treatment factor, extractives content with pre-treatment showed an average value (3.65%, SD = 1.31) significantly lower than the one for the treatment without pre-treatment (4.36%, SD = 1.65). Possibly, the use of autoclave as a pre-treatment caused loss of some extractives.

The increase observed in this study was due to the extractives condensation of thermal modified wood. Volatiles extractives and water are products that are condensed during the thermal rectification (Hill, 2006). A study with Flooded gum and Slash pine thermally modified wood (Brito et al., 2008) reported similar behaviour of the extractives content. Esteves et al. (2008) affirmed that most of the extractives disappeared and new compounds were formed due to the hemicellulose and lignin degradation.

The extractives and holocellulose contents of thermally modified wood showed a statistically significant reduction when compared to the control samples. Thus, it is possible to affirm that the hemicellulose content decreased due to the thermal modification, considering that component presents a lower degradation temperature than cellulose.

However, the reduction of the polymerisation degree (DP) of cellulose could be observed at 150 °C and in systems of treatments in the presence of air (Shafizadeh, 1984).

Statistical difference in the holocellulose contents, which remained between 53 and 54%, was not observed between the thermal treatments.

The insoluble lignin (Klason) content increased significantly as a function of the temperature of treatment. The same behaviour was showed in a study with the Scots pinewood (Boonstra and Tjeerdsma, 2006). The authors observed that lignin content increased due to the degradation of hemicellulose and cellulose. Another study also verified the same behaviour in Flooded gum wood thermally modified at 120 and 180 °C (Brito et al., 2008).

Moreover, in an interval of temperature (175–240 °C) similar to the one in the present study, the authors observed lignin condensation as a function of the temperature of treatment (Fengel and Wegener, 2003). The lignin is the wood compound which is most resistant to the thermal treatments, showing important levels of reduction only at temperatures higher than 300–400 °C.

3.2. High-performance liquid chromatography (HPLC)

According to Table 4, the glucose content was significant only for the temperature of treatment factor ($F = 39.09$; $p < 0.0001$), while the pre-treatment (use of autoclave) factor ($F = 0.009$; $p = 0.924$) and the interaction between the factors ($F = 2.37$; $p = 0.07$) were insignificant. The other sugars content was significant for the temperature of treatment factor ($F = 221.78$, $p < 0.0001$) and for the interaction between temperature of treatment and pre-treatment ($F = 55.46$, $p < 0.0001$). The pre-treatment (use of autoclave) factor was not significant ($F = 3.62$, $p = 0.064$).

Table 5 presents the results of high-performance liquid chromatography (HPLC) analyses for the sugars content in the Gympie messmate thermally modified wood.

The glucose content showed a small increase as a function of the temperature of treatment. Repellin and Guyonnet (2005) also observed a discreet increasing of glucose content as a function of the temperature and the time for the European Beech and Maritime Pine thermally modified wood by Rectification® process.

Table 3
Chemical characterisation of Gympie messmate thermally modified wood.

Temperature (°C)	Extractives (%)	Holocellulose (%)	Lignin (%)
Control (20)	1.59 ± 0.30a	62.26 ± 0.37a	31.75 ± 2.12a
180	3.82 ± 0.47b	53.38 ± 0.87b	36,88 ± 1.75b
200	4.15 ± 0.88bc	53.90 ± 0.96b	38.91 ± 2.18b
220	4.69 ± 0.58c	54.15 ± 1.00b	41.49 ± 3.09b
240	5.77 ± 0.69d	53.35 ± 1.34b	40.60 ± 2.69b

Averages values in the same column (for the temperature of treatment) followed by the same letter are not statistically different at level of 5% by the Tukey test.

Table 4

Summary of the factorial analysis of variance (ANOVA) for the monomeric sugars with pre-treatment and temperature as factors.

Variable	Source	SS	df	MS	F
Glucose	Pre-treatment (A)	0.65	1	0.65	0.02[ns]
	Temperature (B)	1517.67	4	379.42	13.54[*]
	A × B	143.01	4	35.75	1.28[ns]
	Residual	1288.8	46	28.01	
	Total	2917.72	55		
Other sugars	Pre-treatment (A)	0.77	1	0.77	3.62[ns]
	Temperature (B)	190.19	4	47.55	221.78[*]
	A × B	47.56	4	11.89	55.46[*]
	Residual	8.79	41	0.21	
	Total	277.90	50		

SS = sum of squares; df = degrees of freedom; MS = mean square; F = calculated value of F; ns = not significant.

[*] Significant at 5% of probability of error.

Table 5

Monomeric sugars present in the Gympie messmate control and thermally modified wood samples.

Variable	Pre-treatment (°C kgf cm⁻²)	Treatment (°C)				
		Control (20)	180	200	220	240
Glucose	–	23.13 ± 1.67a	30.57 ± 1.37b	31.23 ± 1.53b	39.66 ± 1.60c	34.67 ± 1.87bc
	127–1.5					
Other sugars[*]	–	8.54 ± 0.11a	10.47 ± 0.39bB	7.32 ± 0.19cA	3.80 ± 0.44dB	4.69 ± 0.04eB
	127–1.5	8.54 ± 0.11ab	8.80 ± 0.45aA	7.93 ± 0.73abA	7.62 ± 0.91bA	3.2 ± 0.38cA

[*] Values corresponding to the content of manose, galactose and xylose due to all the peaks were superposed; Averages values in the same row (for the temperature of treatment) followed by the same minuscule letter and in the same column (for each pre-treatment) followed by the same uppercase letter are not statistically different at level of 5% by the Tukey test.

The obtained results are in agreement with other studies (Brito et al., 2008; Repellin and Guyonnet, 2005; Mburu et al., 2008) showing that the other sugars identified in the treated samples (mannose, galactose and xylose) presented a decrease due to the thermal treatments in comparison to the control samples. However, it was not possible to identify the presence of arabinose because the amount of this sugar in hardwoods is small, and it could have been degraded either due to the thermal treatments or because of the use of sulphuric acid for the insoluble lignin extraction.

The decreasing of other sugars content was more visible in the treatments performed at 220 °C for the treatments without use of autoclave (pre-treatment) and only at 240 °C for the treatments with pre-treatment in autoclave.

The distinct behaviour (inversely proportional) of the glucose and the other sugars content identified in the thermally modified wood occurred due to the action of the thermal treatments in the chemical structure of the material. Units of β-D-glucose are present in the structure of cellulose (Sjöström, 1993), which had a less degradation than the hemicellulose after the thermal treatments. On the other hand, the hemicelluloses are formed by xylose, galactose, mannose and arabinose (Rowell, 2005), sugars that presented a significant reduction as a function of the temperature of the treatment in this study.

The slight increase in the glucose content is directly related to the significant reduction of mannose, galactose and xylose content, and, consequently, to the hemicellulose content of the wood. According to Weiland et al. (1998), thermal treatments at 200–260 °C cause high degradation in the hemicellulose content and a great release of acetic acid.

3.3. ATR-IR spectroscopy analysis

The ATR-IR spectra of Gympie messmate thermally modified wood are shown in Fig. 1, whereas the typical bands found are shown in Table 6.

Peak changes were similar for the treatments only in an oven and in a combination (autoclave and oven). In general, compared to the control samples, the chemical structure of Gympie messmate thermally modified wood samples was changed.

The peaks between 1700 and 1750 cm⁻¹ refer to C=O, are typical in unconjugated ketone and carboxyl acids present in lignin and hemicelluloses (Colom et al., 2003).

The peak intensity at 1731 cm⁻¹ was reduced as a function of the temperature, mainly after the treatment at 200 °C and 127 + 200 °C. This reduction in relation to the control samples was around 50%.

The intensity variation of the band at 1731 cm⁻¹ provides much information about the chemical reactions in the wood during the thermal treatment. Moreover, ester and carboxyl groups are the most abundant functional groups in hemicellulose (Chow, 1971; Marchessault, 1962; Timmel, 1965).

The peaks at 1592 and 1504 cm⁻¹ refer to aromatic skeletal in the lignin and they presented an intensity reduction with the increase in the temperature, mainly after the treatments at 240 °C and 127 + 240 °C. A reduction of lower intensity due to increasing
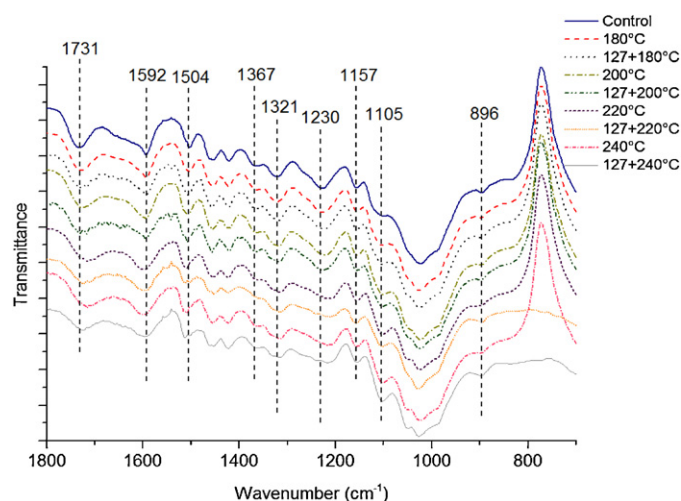

Fig. 1. ATR-IR spectra of Gympie messmate control and thermally modified wood.

Table 6
Typical bands assignment of ATR-IR of Gympie messmate thermally modified wood.

Frequency	Assignment	Reference
1731	C=O stretching in xylan; C=O stretching of acetyl or carboxylic acid	1; 2; 3; 4
1592	Benzene ring stretching in lignin	1; 3
1504	Benzene ring stretching in lignin; C=C stretching of the aromatic ring in lignin	1; 3; 5
1367	CH_2 bending in cellulose and hemicellulose	1; 4
1321	CH_2 wagging vibration in cellulose	1; 5
1230	C—O stretching vibration in xylan	1; 4
1157	C—O—C asymmetric band in cellulose and hemicellulose	1
1105	OH association in hemicellulose and cellulose	1
896	C1 group frequency in cellulose and hemicellulose	1; 3; 4; 5

1 = Kuo et al. (1988); 2 = Chow (1971); 3 = Fackler et al. (2010); 4 = Hesse and Meier (2000); 5 = Colom et al. (2003).

temperature was verified in the peak at 1367 cm^{-1} for O—H and C—O stretching in hemicellulose.

On the other hand, the band at 1321 cm^{-1} did not show significant difference between the control and treated samples. This peak is attributed to the C—H vibrations in cellulose. Typical of xylan, the peak at 1230 cm^{-1} in the treated samples was lower than in the control samples. In general, the peak at 1157 cm^{-1} (asymmetric C—O stretching in cellulose) was constant with the increase in the temperature of treatment.

At 1105 cm^{-1}, a peak representative of the stretching of glucose rings increased as a function of the temperature of treatment. The peak of low intensity at 896 cm^{-1} tended to disappear with the increase in the temperature of treatment due to the domain of β-glycosidic stretching of the sugars.

3.4. Thermogravimetric analysis (TGA)

Fig. 2 shows the thermograms (TG) and its derivatives thermograms (DTG) of Gympie messmate thermally modified wood.

In general, all the thermograms were similar, without significant differences. Four regions were characterised and four peaks were observed.

A peak in the first region (between 0 and 125 °C), which shows the moisture loss of the samples, was observed. The second region (225–320 °C) presented another peak, mainly in the control, 180 and 127 + 180 °C samples. This peak referred to the hemicellulose degradation, whose maximum value was around 290 °C. According to Yang et al. (2007), the highest weight loss of hemicellulose was at 268 °C.

However, the treatments at 200, 127 + 200, 220, 127 + 220, 240 and 127 + 240 °C did not present a similar peak to the control, 180 and 127 + 180 °C samples. Therefore, it is possible to affirm that thermal treatments at high temperatures (above 200 °C) presented a more intense degradation of hemicellulose than the other treatments.

The cellulose degradation started in the same band as hemicellulose degradation. The band of highest intensity and total degradation of cellulose was between 310 and 400 °C, with the maximum peak at approximately 350 °C for all the samples. According to White and Dietenberger (2001), the cellulose degradation occurs between 300 and 500 °C.

After 400 °C, a similar peak with low intensity referred to the lignin was observed for all the samples. The lignin is the wood component with the highest resistance to high temperatures. Yang et al. (2007) affirmed that the lignin degradation is slow and occurs from 100 °C to 900 °C with low weight loss (<0.14 wt.%/°C).

However, the lignin degradation is more intense and occurs fully after 400 °C. Therefore, the thermal treatments employed in the present study did not modify the lignin content of the Gympie messmate wood samples significantly.

Fig. 3 shows the weight loss of each treatment for the hemicellulose (225–320 °C) and cellulose (310–400 °C) bands.

The lignin weight loss was not characterised due to the absence of significant peak and to the fact that the degradation had occurred along the increase in temperature.

The highest weight loss of hemicellulose occurred in the control, 180 and 127 + 180 °C samples. The lowest values were found in the samples treated at 240, 220, 127 + 240 and 127 + 220 °C, respectively. Moreover, the weight loss of control samples was similar to the samples treated at 180 and 127 + 180 °C, whereas only after treatment at 200 °C and 127 + 200 °C the significance of weight loss was lower.

The highest weight loss in control, 180 and 127 + 180 °C samples is due to the high hemicellulose content in the wood structure. Therefore, hemicellulose components with low resistance at high
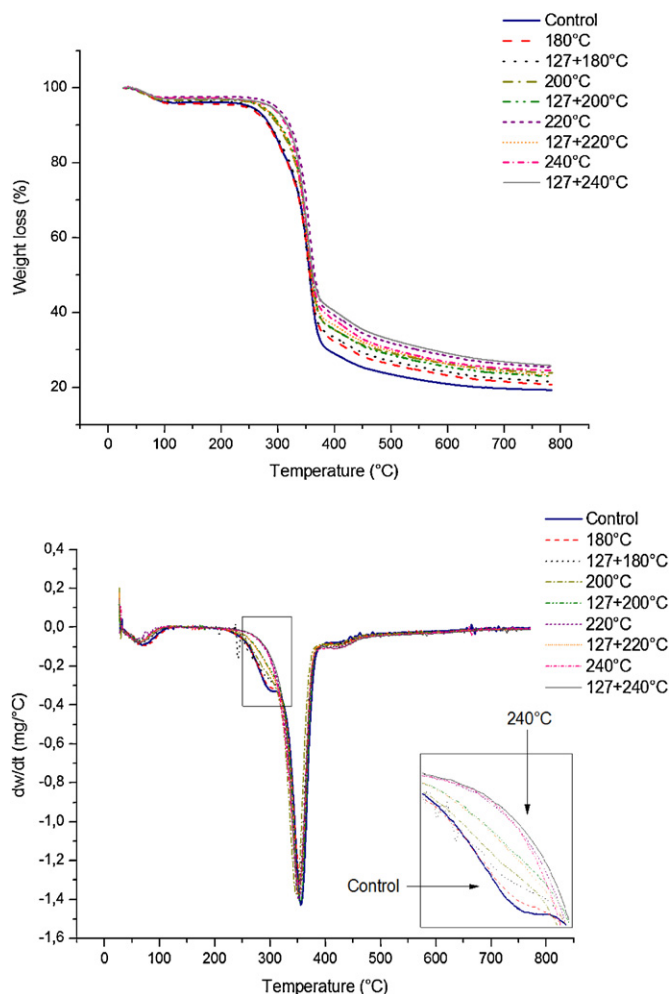

Fig. 2. Thermograms (TG) and derivative thermograms (DTG) of behaviour of Gympie messmate thermally modified wood. (A) TG and (B) DTG.
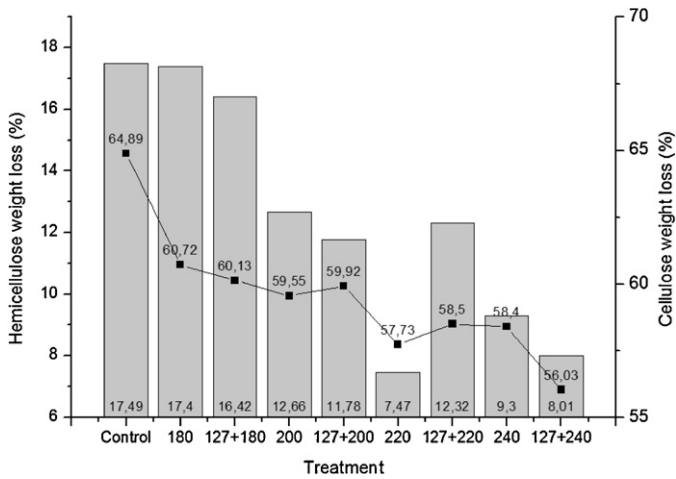
Fig. 3. Percentage of weight loss in the hemicellulose (column) and cellulose (line) peaks of Gympie messmate thermally modified wood.

temperatures are not degradable during the thermal treatments, allowing its identification through thermogravimetric analysis.

In the same way, the weight loss behaviour of cellulose was similar to the hemicellulose. However, the intensity of weight loss in cellulose is lower than the one verified in the hemicellulose.

3.5. Wettability

Fig. 4 shows the results of wettability of Gympie messmate thermally modified wood.

The Gympie messmate thermally modified wood presented a higher contact angle than the control samples in both anatomical sections (longitudinal radial and longitudinal tangential). Consequently, the thermal modified wood showed highest rates of hydrophobicity. In general, the contact angle values remained constant as a function of the time, except for control samples, in which a

reduction of the contact angle was observed because of the natural hydrophobicity rates of wood.

This behaviour was expected because the thermal treatment reduces the interaction of wood with many liquids, mainly water. The repellence rates to water are highest in the thermal modified wood.

Therefore, all the thermal treatments modified the wettability of the Gympie messmate wood, in which the contact angle of longitudinal tangential section showed slightly higher values than the longitudinal radial section for the seven points of measurement.

The treatments at 180 and 200 °C presented the best results of wettability reduction in the thermal modified wood (Fig. 4). On the other hand, the thermal treatments at 220 and 240 °C, even with values higher than the ones in control samples, presented a contact angle reduction when compared to the treatments at 180 and 200 °C due to the lower increments. Moreover, small superficial cracks were observed in the samples treated at 220 and 240 °C. This degradation is possibly influenced by the deposition of drop water in the surface of the wood sample and, consequently, lower contact angles than the treatments at 180 and 200 °C were observed. However, such an intense formation of superficial cracks was not identified in the treatments at 127 + 220 and 127 + 240 °C.

Considering the process used, the wettability behaviour showed differences compared with other studies (Unsal et al., 2011; Hakkou et al., 2005; Kocaefe et al., 2008). Nevertheless, Metsä-Kortelainen and Viitanen (2012), studying *Pinus sylvestris* and *Picea abies* thermally modified wood, observed the same behaviour as the present study. The authors concluded that the thermal treatment at high temperatures (230 °C) reduced the wood wettability, implying the influence of the wood components (hemicellulose, for example) and the extractives migration to the material surface.

Variations of the contact angle occur due to conformational changes of the cellulose molecules and to the lignin plasticisation effect in different water contents (Alén et al., 2002; Weiland and Guyonnet, 2003; Pétrissans et al., 2005; Mburu et al., 2008). Moreover, these modifications in wettability are related to wood surface inactivation. According to Nussbaum (1999), surface inactivation
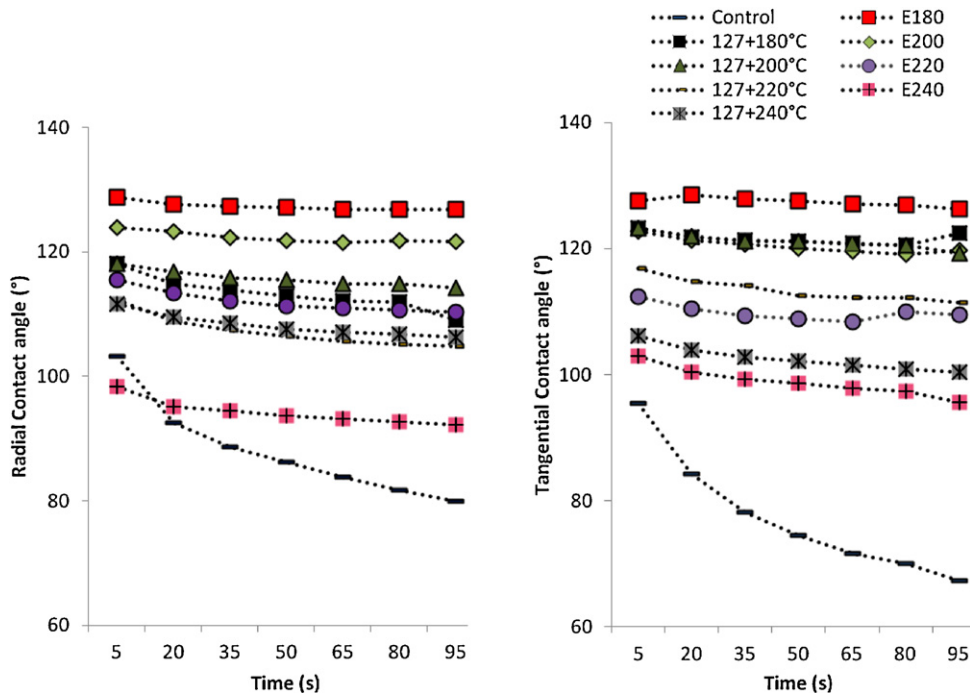

Fig. 4. Contact angle behaviour as a function of the time to the Gympie messmate thermally modified wood in longitudinal radial and longitudinal tangential sections.

induces a decrease in surface energy and, consequently, a poorer liquid wettability.

This phenomenon of wood surface inactivation is influenced by several mechanisms of attractive forces reduction on wood surface, such as surface molecular reorientation, micropore closure, removal of surface hydroxyl bonding sites, surface oxidation and migration of extractives to the wood surface (Christiansen, 1991), the latter having been observed in this study.

The contact angle characterisation is difficult because wood is a heterogeneous and porous material, which directly influences the contact of the liquid with the surface material.

4. Conclusions

In general, the thermal treatment modified the wettability and the chemical properties of Gympie messmate wood significantly. The samples thermally treated at 220 and 240 °C showed the highest wood degradation rates. The increase in the extractives content occurred due to the condensation of these compounds, whereas the increase in lignin was related to the holocellulose degradation. The monomeric sugars decreased as a function of the temperature of treatment, except in the case of the glucose content. In the infrared spectroscopy analysis, the thermal treatments at 220 and 240 °C presented the highest modifications in the typical bands analysed. Likewise, the thermogravimetric analysis showed a reduction in the hemicellulose peak, which was confirmed with the weight loss percentage. The thermally modified samples were more hydrophobic than the control samples. The thermal treatments at 180 and 200 °C presented the best results in relation to the contact angle increment. The use of autoclave as a pre-treatment had a significant effect only on the extractives and other sugars contents of Gympie messmate thermally modified wood.

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