

## Original article

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# Wettability of welded wood-joints investigated by the Wilhelmy method: part 2. Effect of wollastonite additive

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**Abstract:** The effect of wollastonite on the wetting properties of welded Scots pine-joints was studied using the multicycle Wilhelmy plate method and by observation of the chemical composition of the welded joints. Welding pine with wollastonite for 5 s resulted in a decrease in the water uptake and the swelling, and an increase in the contact angle of the welded joint compared to welded wood without wollastonite. High-performance liquid chromatography and gas chromatography/mass spectrometry showed the presence of dehydration products such as furfural, 5-hydroxymethylfurfural, and levoglucosan in methanol extracts from welded joints of specimens welded with and without wollastonite. Phenols were also found by analysis using the Folin-Ciocalteu method and High-performance liquid chromatography. The importance of such compounds in relation to the wetting properties of the welded joint is discussed.

**Keywords:** apparent contact angle (CA); gas chromatography/mass spectrometry (GC/MS); high-performance liquid chromatography (HPLC); liquid sorption; pine; swelling; welding time.

## 1 Introduction

Wood welding is an innovative technology, which makes it possible to assemble wooden pieces without adhesives or metal fasteners. In general, welded woods have poor long-term durability because of the sensitivity to moisture of the welded joints. This behaviour can be related to insufficient attachment between the welded interface and the adjacent wood, and/or dimensional variations of the wood which strain the welded joint and lead to cracking in the joint (Vaziri et al. 2020a).

The resistance to water of welded wood can be improved through four main approaches:

- (1) Optimization of the welding parameters (welding time, welding pressure, etc.),
- (2) Optimization of parameters related to the geometry and structure of the wood (Belleville et al. 2016),
- (3) Dimensional stabilization of wood by thermal and chemical treatments such as acetylation and furfurylation (Pizzi et al. 2013), or
- (4) Surface coating with naturally derived additives (Amirou et al. 2017).

Most of the methods related to the third approach reduce the mechanical performance of the wood or contain components that may be of environmental concern, but the resistance of welded wood to water is a field that is still to a large extent unexplored, specially the moisture dynamics of the welded interface and the underlying mechanisms of water permeation in the welded joints. This study addresses the above-mentioned first and fourth approaches by pre-treating the weld interface of Scots pine with an environmentally compatible additive, wollastonite, and using two different welding times. The water sorption characteristics of the welded joints are studied through multicycle Wilhelmy plate experiments and chromatographic methods.

Vaziri et al. (2020b) showed that as a result of welding, the contact angles (CAs) of water on wood generally increased, and the uptake of water and the swelling

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decreased. Increasing the welding time from 4 to 5 s further pronounced these results for Scots pine. Further increasing the welding time causes charring, which reduces the mechanical properties at the welded joint. It is also known that a good bondline in welded wood requires a fine balance between peak temperature at the interface, welding time at high temperature, and charring (Abbasion et al. 2015; Belleville et al. 2013a). Vaziri et al. (2012) showed that an uneven distribution of heat in the weld interface might be one of the reasons behind the poor durability of the welded pine.

Wollastonite is the commercial name for calcium silicate ( $\text{CaSiO}_3$ ), which exists in a fibrous needle-shaped (acicular) form and is formed by contact metamorphosis of limestone. The chemical composition of wollastonite is mainly 48.3% calcium oxide ( $\text{CaO}$ ) and 51.7% silicon dioxide ( $\text{SiO}_2$ ). Traces of aluminium oxide ( $\text{Al}_2\text{O}_3$ ), magnesium ( $\text{Mg}$ ), and potassium ( $\text{K}$ ) may be found in this mineral compound (Maxim et al. 2014b). Wollastonite is widely used as a filler in polymers and cement to fabricate composites with improved mechanical properties. Although the absolute thermal conductivity of wollastonite is low, its value is relatively high compared to other materials such as wood or plastics; the addition of wollastonite can increase the thermal conductivity of the resulting composite (Karimi et al. 2013). In addition, the wollastonite particles can increase the friction and heat generation at the weld's interface. This, in turn, can increase the degradation of wood constituents, which can increase flow-induced material at the interfaces that leads to better adhesion at the joint. The natural and non-acidic properties of the wollastonite may lead to a modified wood product with little effect on humans or wildlife (Huuskonen et al. 1983; Maxim and McConnell 2005; Maxim et al. 2014a,b).

The overall purpose of the present work is to gain information related to the wetting properties (water sorption, swelling, and apparent contact angle) of welded pine-joints modified with wollastonite. That information will be used to achieve a better understanding of the modification mechanism that also may enable us to tailor the welding process. A typical way to study the wettability of a wood surface is by measurement of the contact angle of a liquid in contact with the wood surface. One of the most widely used techniques for wettability measurements is the Wilhelmy method (Wilhelmy 1863) which is very simple to use and provides accurate, consistent, and reproducible data especially for rough, heterogeneous, and hygroscopic materials such as wood (Lander et al. 1993). An effort has also been made to analyse the chemical composition of welded wood with wollastonite using High-Performance Liquid Chromatography (HPLC) and Gas Chromatography

Mass Spectrometry (GC/MS). Combining wettability studies with chemical analysis provides valuable knowledge regarding the energetics and chemistry of the welded joint.

## 2 Materials and methods

The focus of the present work was to apply the Wilhelmy plate principle to study the effect of welding Scots pine with wollastonite on the wetting and water sorption properties of the welded joint. This study links wetting properties with surface chemistry to provides valuable knowledge regarding the mechanism by which wollastonite could influence the energetic and chemistry of wood surfaces that may guide us in modifying the permeability of welded woods.

### 2.1 Preparation of specimens

#### 2.1.1 Wettability

The wollastonite used in this study was a minimally refined powder that consisted of a mixture of various sizes of acicular particles (with a large proportion of the particle being approximately  $\sim 0.05\text{--}0.1$  mm in the longest direction). The wollastonite powder was sourced from the Svårtsång mine in Filipstad, Sweden. Wood specimens with planed surfaces and dimensions of  $20 \times 20 \times 230$  mm (radial [R]  $\times$  tangential [T]  $\times$  longitudinal [L]) were prepared from clear pieces of Scots pine (*Pinus sylvestris*. L) grown in northern Skellefteå, Sweden. The specimens were conditioned for two weeks at a temperature of  $20^\circ\text{C}$  and 65% relative humidity (RH) in an environmental chamber to 12% average moisture content (MC) and welded together in pairs to dimensions of  $20 \times 40 \times 230$  mm (longitudinal welding of a tangential face to a radial face) using a linear vibration welding machine, Branson model M-624 (Branson Ultraschall, Dietzenbach, Germany) with a frequency of 240 Hz and settings according to Table 1. The most influential welding parameters were selected based on the earlier studies, and the appropriate range of the

**Table 1:** The welding procedure. The initial pressure,  $p_{w1}$ , was applied during the first welding, time,  $t_{w1}$ ; followed by final pressure  $p_{w2}$ , during the second welding time,  $t_{w2}$ ; ending with the, holding pressure,  $p_h$ , during the holding time,  $t_h$ .

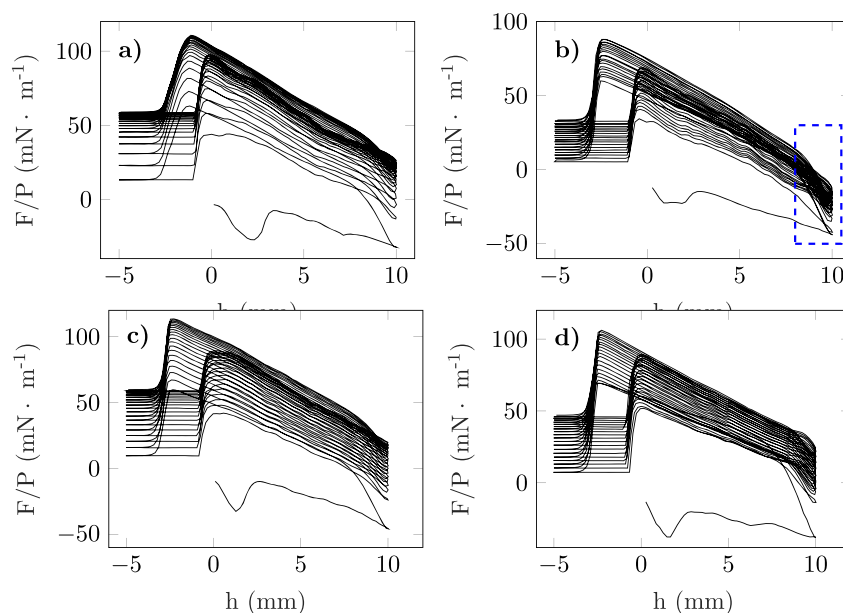
Specimens		$t_{w1}; t_{w2}; t_h$	$p_{w1}; p_{w2}; p_h$
Wollastonite	Control	(s)	(MPa)
W4	R4	2/2/10	1.3/1.7/2.7
W5	R5	2/3/10	1.3/1.7/2.7

parameters that could result in a maximum shear strength in the welded joints were defined by a screening test. For each testing group three replicates were prepared. The tangential-radial surface was chosen for a future study about influence of the end-grain direction on the distribution of wollastonite particles in the welded joint. One surface ( $20 \times 230$  mm) of each pair was manually evenly covered by 0.2 g of wollastonite powder just before welding them together.

Specimens for wetting were prepared as described. Wilhelmy plate is a thin, generally rectangular plate which is vertically immersed in the test liquid along one of its larger dimensions. To make such plates entirely of welded material, the welded joints of the specimens should be opened to get access to their welded area. The welded joints of the specimens were opened with a steel chisel (Figure 1c), carefully cleaned with hexane before each use. The welded areas were band sawn to 0.5 mm thick strips. Two chips with dimensions of  $20 \times 10 \times 0.5$  mm were cut from one of the strips with a scalpel carefully cleaned with hexane. The chips were glued together on their rear sides using a waterproof glue (Contact A3, Bostik) to make two-sided specimens with dimensions of  $20 \times 10 \times 1$  mm. The cross sections of the specimens were end-sealed with synthetic alkyd/polyurethane varnish to avoid liquid sorption through these sides of the specimens. Specimens with dimensions of  $20 \times 10 \times 1$  mm were also cut from non-welded wood adjacent the welded areas to be used as reference.

## 2.2 Wetting experiment

The experiment was performed based on the Wilhelmy method as is described thoroughly by using a Sigma 70 tensiometer from KSV Instruments with water and heptane as a non-swelling liquid. The specimens were dried at room temperature,  $22.5 \pm 0.5$  °C and  $35 \pm 5\%$  RH, to 7% MC and weighed immediately before the wettability measurement. Each specimen was mounted in the tensiometer and partially immersed in ultra-pure water at a speed of  $12 \text{ mm}\cdot\text{min}^{-1}$ . After 50 s (corresponding to a depth of immersion of 10 mm), the specimen was withdrawn at the same speed, until the water was detached from the wet solid surface (5 mm above the surface), and thereafter, with no pause, immersed again. Changes in the force as a function of depth of immersion were recorded graphically for 20 cycles in water. Thereafter the specimen was immersed and withdrawn for two cycles in a non-swelling liquid (heptane) to determine its perimeter after water absorption. The liquids used were distilled water (surface tension and density of  $72.0 \pm 0.2 \text{ mN}\cdot\text{m}^{-1}$  and  $0.997 \text{ g}\cdot\text{cm}^{-3}$ , at 25 °C) and heptane (surface tension and density of  $21.3 \text{ mN}\cdot\text{m}^{-1}$  and  $0.397 \text{ g}\cdot\text{cm}^{-3}$ , at 25 °C). The measurements were performed at about 22 °C and 50% RH. Three independent replicates were tested, and fresh liquid substrate was used for each specimen. The dynamic force measurements were made as described in Vaziri et al. (ibid.).



**Figure 1:** Typical Wilhelmy curves while the specimen were immersed and withdrawn from the water for 20 cycles. The instrument detected the force normalized with the sample perimeter ( $F/P$ ) as a function of immersion depth ( $h$ ). The lower and upper parts of each cycle represent the advancing and receding curves, respectively. (a) Welded sample with wollastonite for 4 s (W4), (b) Welded sample with wollastonite for 5 s (W5), (c) Welded sample without wollastonite for 4 s (R4), and (d) Welded sample without wollastonite for 5 s (R5). The marked areas in (b) shows dipping at the right-hand side of the curves.

### 2.2.1 High-Performance Liquid Chromatography (HPLC)

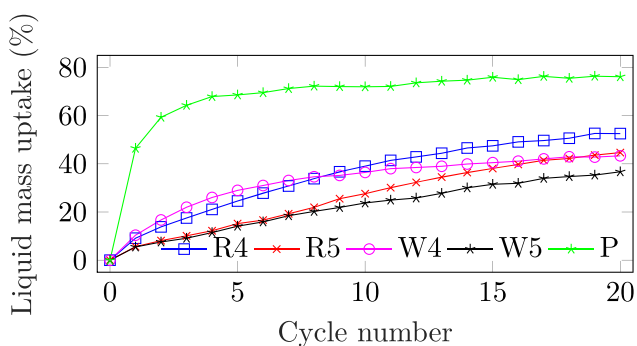
Hundred milligram welded wood was manually scraped from the welded joint of each specimen using a razor blade cleaned with hexane before each use. The welded material was mixed with 3 ml of methanol over night. The filtered extract (0.2  $\mu\text{m}$ ) was diluted and analysed with the HPLC equipment LC-2030, Shimadzu, Japan with column: Kinetex 5  $\mu\text{m}$  EVO C18 100  $\text{\AA}$ , LC column 150  $\times$  4.6 mm, detector: PDA 254 nm, and an oven of 25–85  $^{\circ}\text{C}$ . For each test group three replicates, a total of 12 specimens, were tested. Different gradients were investigated in order to optimise the chromatographic separation (Table 2). The eluent consists of two solvents, solvent A (0.1% formic acid) and solvent B (acetonitrile). The substances were identified by UV-spectra.

### 2.2.2 Gas chromatography mass spectrometry (GC/MS)

The filtered extract of welded wood (0.2  $\mu\text{m}$ ) used for HPLC analysis was mixed with the desired amount of methyl-D-xylopyranoside (3 mg in 1 ml water) and evaporated to dryness on a petri dish. The residue was trimethylsilylated (TMS) in dry dimethylsulfoxide (DMSO) using hexamethyldisilazane-trimethylsilyl chloride 2:1 and analysed using gas chromatograph mass spectrometry as described by Karlsson et al. (2012). Data from gas

**Table 2:** Eluent gradients. Gradient represents share of solvent B, the remainder of 100% is solvent A.

Time (min)	0.01	7	15	20	25	28	30	35
Gradient (%)	5	5	25	20	95	95	5	5



**Figure 2:** Average water uptake expressed in % of the specimen's weight vs. cycle number. R4 denotes pine welded for 4 s without wollastonite, R5 pine welded for 5 s without wollastonite, W4 pine welded for 4 s with wollastonite, W5 pine welded for 5 s with wollastonite, and P non-welded pine.

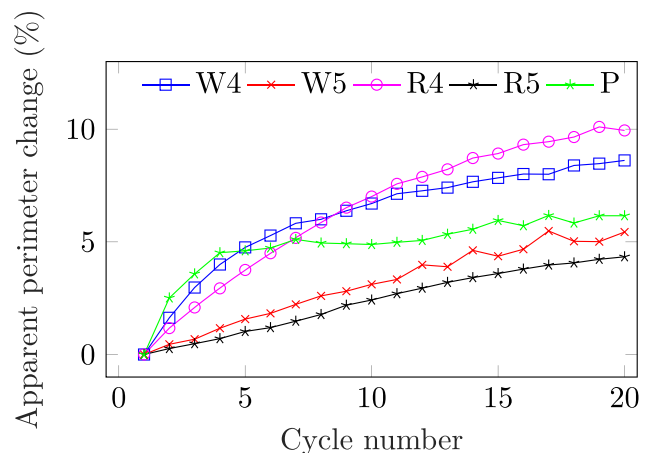
chromatography–mass spectrometry (GC-MS) of trimethylsilyl (TMS) derivatives of dried extracts from welded joints was analysed using the database of National Institute of Standards and Technology (NIST).

## 3 Results and discussion

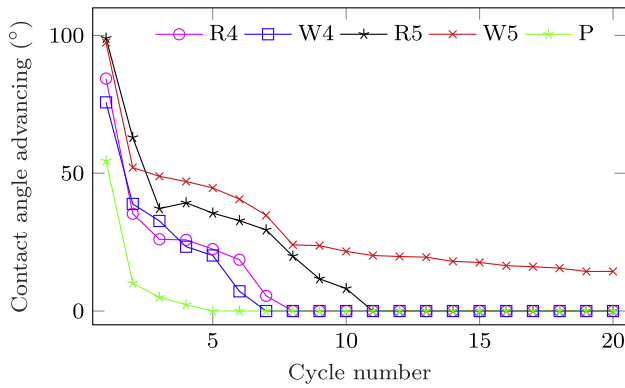
### 3.1 Wetting

Figure 1 shows typical multicycle Wilhelmy curves for specimens welded together with (W4, W5) and without wollastonite (controls: R4, R5). Due to the chemical heterogeneity and high roughness of the surface of specimens welded with wollastonite (Figure 1a,b), these curves had a less regular shape than the controls (Figure 1c,d). Furthermore, after a couple of cycles the right-hand end of the plots “dipped”, especially for W5 (Figure 1b), indicating that the specimens welded for a longer time were less affected by the water sorption (less hydrophilic). The dynamic force measurements (advancing force  $F_A$ , receding force  $F_R$ , and final force  $F_f$ ) were made as described.

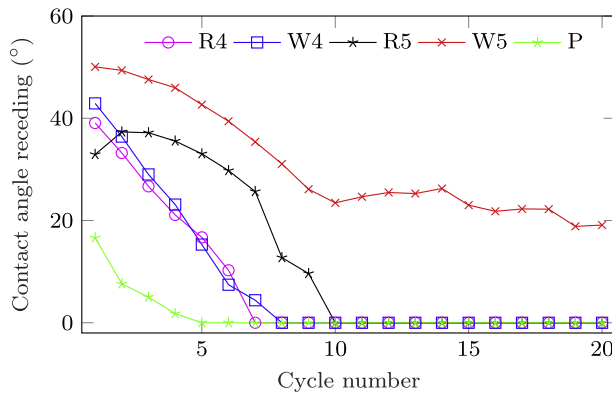
Figure 2 shows the water uptake as a function of the number of cycles. Dynamic sorption by the wood specimens was determined by extrapolation of the final forces  $F_f$  in Wilhelmy curves (Figure 1) to zero depth in water (ibid.). The average sorption of water by the specimens welded with wollastonite was less than that of the specimens welded without wollastonite (controls). The sorption of water by non-welded pine was initially very high but it decreased rapidly after some cycles.



**Figure 3:** Average perimeter change vs. cycle number. W4 denotes welded pine with wollastonite for 4 s, W5 welded pine with wollastonite for 5 s, R4 welded pine without wollastonite for 4 s, R5 welded pine without wollastonite for 5 s, and P non-welded pine.



(a) Advancing contact angle

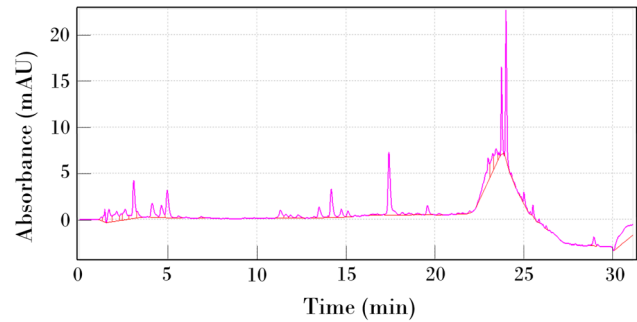


(b) Receding contact angle

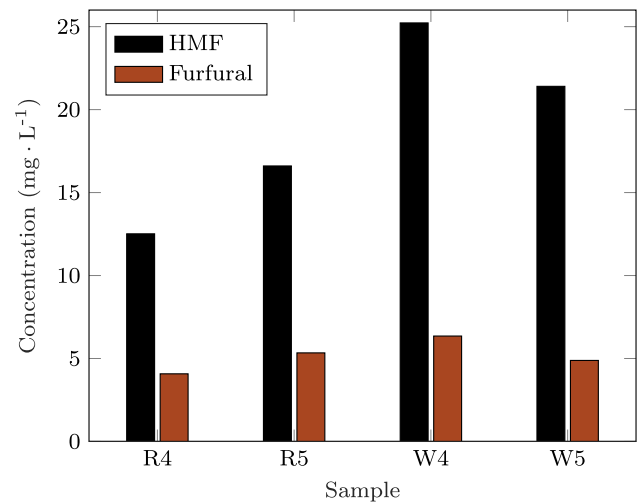
**Figure 4:** Dynamic contact angles (CAs) of the welded specimens for 20 cycles. (a) Advancing CA, (b) Receding CA. R4 denotes welded pine without wollastonite for 4 s, W4 welded pine with wollastonite for 4 s, R5 welded pine without wollastonite for 5 s, W5 welded pine with wollastonite for 5 s, and P non-welded pine.

To study the swelling associated with the absorption of water, the perimeter of the specimens welded with and without wollastonite were measured before and after the specimens had been immersed in water for different cycles. The relative change in the perimeter of the specimens after each of the 20 cycles of water immersion is presented in Figure 3. A significantly higher degree of swelling for W4 and R4 were noticed.

The multicycle Wilhelmy method was used to investigate the dynamic contact angles of the welded specimens after each of the 20 cycles (Figure 4). By linear extrapolation of the advancing and receding curves to zero depth ( $h=0$ ), the advancing CA,  $\theta_A$ , and receding CA,  $\theta_R$ , of water on the specimens were calculated. The average CAs of the specimens welded for the longer time (W5) were higher than that of the other specimens, although the differences were not statistically significant. Nevertheless, the CAs of the welded specimens became zero after a couple of cycles,



**Figure 5:** HPLC Chromatogram (retention time) of a specimen welded with wollastonite for 5 s (W5); 5-hydroxymethyl-furfural (3.1 min); furfural (4.1 min); vanillyl alcohol (5.6 min); vanillic acid (11.3 min); vanillin (13.5 min); acetovanil-lone (15.1 min).



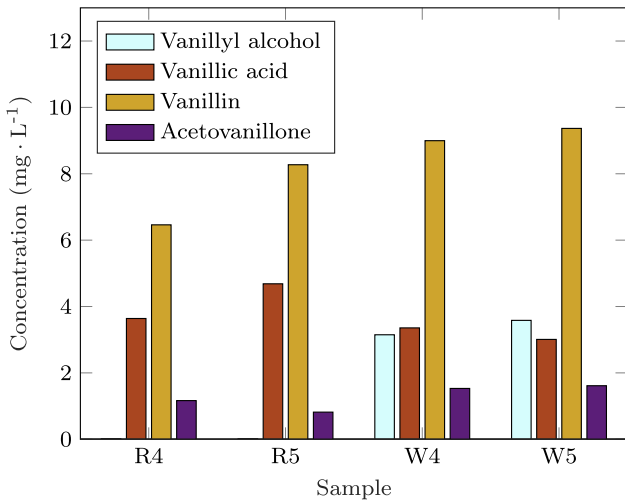
**Figure 6:** Concentration of HMF and furfural in welded joint extracts for specimens welded with wollastonite (W4, W5) and without wollastonite, controls (R4, R5).

except for W5 that still showed a considerable CA after 20 cycles, attributable to a higher hydrophobicity of the wood surface. The  $\theta_R$  of all the specimens was generally lower than  $\theta_A$ .

### 3.2 Chromatography (HPLC and GC-MS)

Furfurals and phenols in the methanol extracts from the welded pine-joints were analysed using HPLC. The compounds were identified by comparison with control substances and their UV-spectra as shown in the example (Figure 5). The amount of 5-hydroxymethylfurfural (HMF) found in the extracts was higher than the amount of furfural (Figure 6), in line with previously published statement by Karlsson et al. (2012) that the concentration of HMF was considerably higher than furfural in the methanol

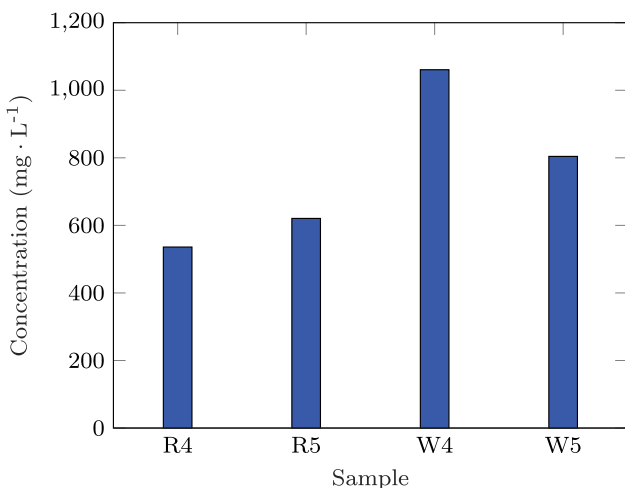




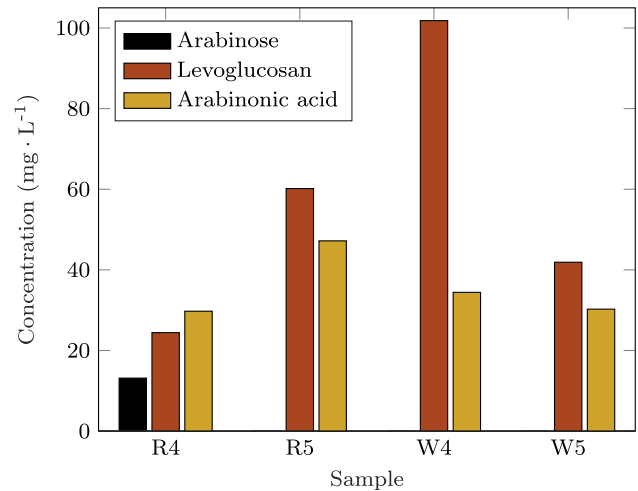
**Figure 7:** Concentration of phenolic compounds in welded joint extract for specimens welded with wollastonite (W4, W5) and controls (R4, R5).

extract from thermally modified wood under saturated steam conditions at 170 °C. HMF can be formed from hexosans by thermal treatment and the level of hexosans is higher than that of pentosans in softwood. HMF is also less volatile than furfural. The average amounts of furfural and HMF in the specimens welded with wollastonite were higher than that in the specimens welded without wollastonite and the standard deviation of the data for HMF was larger.

This can mean that the formation and further reactions of the furfurals vary in the presence of wollastonite. In contrast to the control specimens, the amounts of furfural and HMF from W4 and W5 decreased with increasing welding time. The reaction patterns may be difficult to



**Figure 8:** Concentration of total phenolic compounds in the welded joint extract for specimens welded with wollastonite (W4, W5) and controls (R4, R5).



**Figure 9:** Concentration of phenolic compounds in extracts from welded joints for specimens welded with wollastonite (W4, W5) and controls (R4, R5). The standard deviation values show that the results vary considerably.

interpret due to different formation and breakdown/polymerization rates of the furfurals, but a previous finding of Cristescu and Karlsson (2013) showed that when beech veneer was hot pressed at 200–250 °C (*Fagus sylvatica* L.) the amount of HMF decreased with increasing press temperature and the furfural content remained constant. Similar behaviour was found when the modification temperature was raised to 200 °C for thermally modified timber (TMT) of beech, but in the case of spruce, higher amounts of furfural were found at the higher treatment temperature indicating that hardwood and softwood xylan had different reactivities (Peters et al. 2009). The amount of volatile gases like furfural decreased after a more extensive thermal modification (200 °C) of beech.

Vaziri et al., 2015 showed that a number of phenols were formed as degradation products during welding. The concentrations of some characteristic phenolic compounds detected with HPLC, are presented in Figure 7. The amount of phenols such as vanillin, vanillyl alcohol, and acetovanillone were higher in the extracts from the specimens welded with wollastonite than in the controls.

Other peaks in the HPL chromatogram (Figure 5) may be due to other phenols than those identified and presented in Figure 7. Therefore, an analysis using the Folin–Ciocalteu (FC) method was carried out to estimate the total concentration of phenols. The level of these phenols was higher than could be expected from the analysis of individual phenolic compounds (Figures 7 and 8). The phenol concentration seemed to be higher when wollastonite was present, but it decreased when the welding time was increased.

Figure 9 shows that the amount of levoglucosan, which is a dehydration product from the thermal treatment of cellulose (Fengel and Wegener 1984) was higher in the specimens welded with wollastonite. Arabinose was the only monosugar that was detected in the control specimens (R4).

Wollastonite seemed to enhance the formation of hydrophobic polymers such as furfurals and levoglucosan as well as phenols that result in an increase in the contact angle and a decrease in the water uptake, but increasing the welding time seemed to decrease the content of these products. It is possible that the condensation or polymerisation reaction between aldehydes and phenols forms insoluble materials during welding (Belleville et al. 2013; Delmotte et al. 2008; Mariscal et al. 2016). The presence of more of these materials together with the strength-bearing wollastonite particles can provide matrix of composite materials that increase the strength of the welded joint. These phenomena may offset the hygroscopic character of wollastonite that usually increases the moisture content of wood that is impregnated with wollastonite.

## 4 Conclusions

The results of this study indicate that welding pine with wollastonite resulted in a decrease in water uptake and swelling, and an increase in the contact angle of the welded joint compared to welded pine without wollastonite. The results of HPLC and GC-MS showed that slightly larger amounts of dehydration products such as furfural, HMF and levoglucosan together with phenolic compounds were found in the welded joints of specimens welded with wollastonite than in the specimens welded without wollastonite. Longer welding of the specimens with wollastonite (5 s) seemed to decrease the content of such products, probably due to formation of a more condensed matrix of these materials around the wollastonite particles that resulted in a decrease in the permeability of the welded joint. Wollastonite with a higher specific surface/smaller particles (nano-wollastonite) may increase the presence and the effect of residues materials. This knowledge will provide the basis for enhancing the moisture resistance of welded wood through wood modification techniques. The results of this study are indicative of the ability of a welded joint to withstand water or outdoor climatic variations, and for future work it is recommended to study the moisture resistance of specimens welded with wollastonite of smaller particle sizes by both laboratory immersion tests and exposure of specimens to extreme outdoor climate conditions.

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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