

WETTING PROPERTIES OF BEECH, FIR AND POPLAR INTERACTING WITH DIFFERENT MOLAR-MASS UREA-FORMALDEHYDE RESINS

Ivana GAVRILOVIĆ-GRMUŠA

Assist.prof.dr. - University of Belgrade - Faculty of Forestry
Address: Kneza Višeslava 1, 11030 Belgrade, Serbia
E-mail: ivana.grmusa@sfb.bg.ac.rs

Milanka DJIPOROVIĆ-MOMČILOVIĆ

Assoc.prof.dr. - University of Belgrade - Faculty of Forestry
Address: Kneza Višeslava 1, 11030 Belgrade, Serbia
E-mail: milanka.djiporovic@sfb.bg.ac.rs

Mladjan POPOVIĆ

Assist.prof.dr. - University of Belgrade - Faculty of Forestry
Address: Kneza Višeslava 1, 11030 Belgrade, Serbia
E-mail: mladjan.popovic@sfb.bg.ac.rs

Jasmina POPOVIĆ

Assistant - University of Belgrade - Faculty of Forestry
Address: Kneza Višeslava 1, 11030 Belgrade, Serbia
E-mail: jasmina.popovic@sfb.bg.ac.rs

Sergej MEDVED

Assist.prof.dr. - University of Ljubljana - Faculty of Biotechnology
Address: Rožna dolina, Cesta VIII/34, 1000 Ljubljana, Slovenia
E-mail: sergej.medved@bf.uni-lj.si

Abstract:

The objective of this work was to evaluate the influence of the molar mass of the urea-formaldehyde (UF) resins on the wetting properties of beech, fir and poplar. It was achieved by monitoring the contact angle formation between the small resin drops and the radial and tangential surfaces of the selected wood species. Three UF resins samples were used, having different degree of condensation, and thus having different molar mass distribution. The results have showed relatively high contact angle at the zero position for all three UF resins, which could be observed by the formation of the so-called "sitting drops". Subsequent measurements at the selected time intervals (5, 10 and 15 seconds after the initial contact) have showed that the increase in the UF resins molar mass also increases the contact angle, resulting in the lower wetting of substrates from all three wood species. Comparing the effects of the wood species, the results showed that the fir wood has the highest wettability in regard to both beech and poplar. This was noticed with all of the UF resin samples and for all of the measuring intervals. Contrary, the beech substrate has resulted in the highest contact angle, under the same test conditions. The effects of wood substrate on the wettability can be addressed to the anatomical structure of the selected wood species and the percentage ratio of the early wood versus late wood.

Key words: urea formaldehyde (UF) resin; wettability; degree of condensation; contact angle; roughness.

INTRODUCTION

Surface properties of wood are important for its wetting characteristic, which is related to the glueability of wood. In fact, good wettability is often a predictor of high-quality adhesive bonding. Wood is a complex matrix that is hygroscopic, porous, rough and physically and chemically heterogeneous. The behavior of a water droplet placed on a wood surface is often different from behavior on a metal or polymer surface. In addition to spreading across the surface of wood, the liquid also penetrates into wood, therefore, wetting occurs at and under the wood surface. The shape and contact angle of a sessile droplet changes with time and becomes more or less stable after a certain period. A standardized procedure for contact angle measurement would permit reproducible measurements and comparable data for surface characterization. (Piao *et al.* 2010).

Surface properties of wood can be divided into two major groups: physical and chemical properties. Physical properties include morphology, roughness, smoothness, specific surface area and permeability. Chemical properties include elemental and molecular, or functional, group composition. Together, these two major groups of properties determine the thermodynamic characteristics of the wood surface, such as surface free energy and surface acid-base acceptor and donor numbers. Characterization of the surface properties of wood is a very complex and difficult undertaking. No single technique is adequate to completely characterize the surface chemistry of wood and related lignocellulosic materials. Rather, a combination of spectrometric techniques, used together with microscopic and thermodynamic techniques, can provide good insight into the chemical composition of lignocellulosic surfaces, the surface distribution and topography of acid-base sites, and the effect of chemical composition on the reactivity of lignocellulosic surfaces (Rowell 2005).

X-ray photoelectron spectroscopy (XPS) provides valuable information about chemical components on the surface (Sinn *et al.* 2001, Nzokou and Kamdem 2005, Inari *et al.* 2009). Nzokou and Kamdem (2005) determined contact angle and sorption isotherms of extracted and unextracted specimens in order to evaluate the role of wood extractives on the wettability and sorption properties of three wood species. They reported that extracted samples had contact angle values consistently lower than control samples for all three species, and the difference analyzed by one-way ANOVA (95% confidence level) was statistically significant between all extraction types and control samples. Lower contact angles for extracted samples reinforced the hypothesis of removal of hydrophobic compounds during the extraction process. This result is in agreement with results from Maldas and Kamdem (1999), who also obtained increased wettability in southern yellow pine. However, Nussbaum and Sterley (2002) obtained a more complex relationship between the contact angle, total extractives and storage time. They explained the variability in their results by the migration of extractives spreading on the wood surface and causing chemical changes to the surface.

Several investigators have found that wettability and glueability of a sanded surface is better than that of a planed surface. For a sanded surface, both cell walls and lumen surfaces are refreshed by sanding. A sanded surface often has cell-wall fibrillation, scratches and debris, which produce even more free hydroxyls at new surfaces than those produced by planing. Because most protruding, cross-sectional cell walls are crushed, a sanded surface is flatter (not necessarily smoother) than planed or microtome-prepared surfaces (De Moura and Hernandez 2005). Therefore, a sanded surface would have greater wettability and glueability than a planed or a microtome-prepared surface. Sanding alters distribution of nonwetable extractives on the lumen surface. A sanded surface may be contaminated by moisture in the air, polar volatile organic compounds (VOCs) and some solid extractives in the lumen as well (Gindl *et al.* 2004).

Urea-formaldehyde (UF) resins are common in the wood working and wood-based panels industries (Dunky 2003), and degree of condensation and molar mass distribution belong to their most essential characteristics. The observation of a resin droplet on the wood surface as a function of time (dynamic contact angle) is useful for a preliminary evaluation. Scheikl and Dunky (1996) showed that the higher the molar masses (more or less equivalent to the viscosity of the resin at the same solid content), the worse is the wettability and the lower is the penetration into the wood tissue (Gavrilovic-Grmusa *et al.* 2012). The lower molar masses are responsible for the good wettability, however a too high portion of low molar masses can cause over-penetration and hence starved bond lines. The higher molar masses remain at the wood surface and form the bond line. In order to achieve good bond strengths a certain ratio between low and high molar masses is necessary.

The objective of this study was to evaluate the influence of the condensation degree of urea formaldehyde (UF) resins on the wettability of three wood species in two cutting directions: radial and tangential. For this purpose an UF lab batch was prepared and samples were taken after three different time spans during the condensation reaction, hence showing three different degrees of condensation. In order not to falsify the influence of the length of the condensation phase, no low molar mass moieties were added at the end of this condensation phase; the molar ratio was kept at the original high value during condensation. The intention was to minimize all other chemical influences in the resins beside the degree of condensation itself. A higher degree of condensation means formation of greater molecules.

MATERIALS AND METHODS

Wood raw material

Beech (*Fagus Moesiaca*), fir (*Abies alba Mill.*) and poplar (*Populus x euramericana 'I-214'*) logs of length 1100mm were cut from the trees at a height of 1.3m from the ground and divided into boards of 42mm thickness in different cutting directions using a band-saw. After initial air drying the boards were further dried in a laboratory kiln drier and planned to the final dimensions of 1000mmx150mmx30 mm. These boards then were cut into radial and tangential blocks of 100x30x5mm in order to investigate the wetting properties in relation to urea-formaldehyde resins of different molar mass in tangential and radial direction. Before the various experiments all blocks were conditioned at 20±2°C at 65±5% relative humidity. After conditioning beech blocks has moisture content of 9.3% and density of 0.69g/cm³, fir blocks has moisture content of 9.2% and density of 0.43g/cm³ and poplar blocks has moisture content of 9.1% and density of 0.43g/cm³.

Urea formaldehyde (UF) resins

Three laboratory UF resins with different degrees of condensation according to recipes described in the literature (Kim *et al.* 2003, Pizzi 1999) were provided by DUKOL Ostrava, s r.o. (Ostrava, Czech Republic). The degree of condensation (DOC) increases as a consequence of the duration of the acidic condensation step from resin UF I (lowest DOC) to resin UF III (highest DOC). This determines the viscosity of a resin if the dry matter is the same. The viscosity of the three resins increased due to the larger molecules present in the resin from 218mPa·s for UF I to 281mPa·s for UF II and eventually to 555mPa·s for UF III, measured within two days after the lab cooks (Table 1). The molar ratio of formaldehyde to urea (F/U) of all resins was 2.0; no urea was added after the condensation step.

The adhesive mixes used in the investigations reported here and applied onto the various wood surfaces were prepared by addition of 10 mass % of wheat flour as the extender and 0.05 mass % of Safranin (Superlab, Belgrade) as the marker (both based on the adhesive dry matter).

A similar increase in viscosity as for the resins themselves was observed for the adhesive mixes (UF I: 545mPa·s; UF II: 745mPa·s; UF III: 1644mPa·s) just after mixing.

The average molar mass of each UF resin was determined by Matrix-Assisted Laser Desorption/Ionisation Time-of-Flight (MALDI-TOF) mass spectrometry (MS). MALDI TOF MS proved to be an appropriate technique for analyzing these types of polymers, bearing in mind that the results of the analysis correspond with previous physical and chemical measurements. This technique enables a relatively swift determination of the degree of polymerisation, through the monitoring of key changes in the structure of a polymer. Table 1 summarizes the characteristics of the UF resins and the adhesive mixes.

Table 1

Characteristics of the resins and the adhesive mixes UF I, UF II, and UF III

Property	Unit	UF I		UF II		UF III	
		Resin	Adhesive mix	Resin	Adhesive mix	Resin	Adhesive mix
Dry matter	%	53.7	54.4	53.7	54.3	53.8	54.6
Brookfield Viscosity (20°C)	mPa·s	218	545	281	745	555	1644
pH	/	7.8	3.7	7.9	3.8	8.0	3.9
Gel time	s	58	59	59	60	58	59
Density	g/cm ³	1.24	1.20	1.24	1.20	1.25	1.21
Molar mass	g/mol	936		956		1052	

Wetting factors

Many factors potentially affect wettability, therefore the samples prepared for the evaluation of wetting properties, were also used for the measurements of the annual ring size and the percentage ratio of early and late wood. In addition, the surface characterization included the roughness measurements and analysis of the surface chemical composition.

Determination of annual ring size and the early and late wood ratio

The number of the annual rings per 1cm was chosen as the parameter describing the annual zone. It was measured in the radial direction of the cross section. The ratio of early and late wood was determined as

the percentage of total with of early or late zones in wood, in regard to total radial distance including the whole rings.

Having in mind that the ratio of early and late wood is variable throughout the height of the tree (the width of the ring increases and the ratio of the late wood decreases with height), the measurements were done on the both sides of the test pieces, and between each of the pieces the 2cm thick layer were cut out, perpendicularly to the fiber direction. Prior to measurements of the annual ring size and the ratio of early and late wood, the surface of the test pieces were sanded with the sanding papers of N°180 and N°240, in order to achieve more pronounced borderline between the early and late wood. Macroscopically observed, the annual rings are more pronounced in fir, as a conifer species, while in beech and poplar, as diffusive species, the differences between vessels inside the ring are less pronounced. The division of vessels across the ring is more uniform in those species, and the borderline of the annual ring is characterized by the thin zone at the end of the vegetation period.

Surface geometrical characteristics - determination of roughness

Measurements of the surface roughness were used to describe the surface geometrical characteristics of a given wood species. Roughness was measured on the conditioned samples ($t = 19^{\circ}\text{C}$), using digital instrument Surtronic Duo (Taylor/Hobson precision).

The roughness was expressed as the mean profile deviation (R_a), which is the arithmetical mean value of the recorded profile deviations on all measuring points in regard to the centerline, according to DIN 4768/1:1974 (equation 1).

$$R_a = \frac{1}{l_m} \int_{x=0}^{x=l_m} |y| dx \quad [\mu\text{m}] \quad (1)$$

R_a – arithmetical mean profile deviation (μm)

l_m – referent measuring length

x – centerline (crossing the geometrical profile)

Analysis of the wood surface chemical composition

Wood surface was analyzed for its chemical composition by the energy dispersive X-ray analysis (EDAX), using energy dispersive spectrometer (Oxford Instruments EDS), aided with the INCAX-sight software. The images were taken with the Jeol scanning microscope (JSM-6460LV, JEOL, Tokio, Japan).

The test pieces of 10x10x5mm were coated with 24-carad gold in the Sputter Coater Device SCD 005 (BAL-TEC) with the applied electric current of 30mA. The coating process has lasted for 90 seconds at the distance of 50mm.

In these measurements, the electron beam was set to pass the whole surface of the test pieces, during the 120s.

Determination of the contact angle between the liquid and the surface - wetting

The wetting is commonly described as the value of the contact angle between the small drops of liquid on the surface of wood. There are several methods for measuring the contact angle on wood (Wellons 1980, Hemingway 1969). The method used in this research was based on the direct dropping of liquid onto the wood surface. The wetting was determined on the conditioned samples and according the standard SRPS EN 828 (EN 828:2009). The first series of measurements were made with distilled water as the test liquid, while the second, third and fourth series were conducted using adhesives UF I, UF II and UF III, respectively. The UF I adhesive has the lowest molar mass, while the highest molar mass was determined in UF III adhesive. Each test piece has three measurements and the result was presented as the mean value.

The drops were formed using burette with the mounted syringe needle (Terumo 21G x 1 $\frac{1}{2}$ » 0.8x40 Nr.2). Dropping was made from the close proximity to the surface, at the height which allowed the drop formation. Immediately after the drop has reached the surface of wood specimen, the burette with the needle was moved aside. The contact angle between the liquid and the wood surface was monitored with the video camera in predetermined time sequences. Measurement has started at the moment when drop reaches the surface, marking the zero position of the contact angle. Subsequent measurements were taken at each 5s during 15s (total of 4 positions).

The purpose of these measurements was to determine the influence of the adhesive molar mass, wood species and cutting direction on the wetting and adhesion properties of the wood surface.

RESULTS AND DISCUSSION

Annual ring size and the early and late wood ratio

The size of the annual rings for all selected wood species, was expressed as the mean value of the annual rings number per 1cm. These values, together with the relevant ratios of early and late wood are given in the Table 2.

Table 2

The width of the annual ring and the percentage ratio of early and late wood

	Bukva	Jela	Topola
Number of annual rings / cm	4,61	2,42	1,10
Late wood ratio (%)	28,09	20,12	37,93
Early wood ratio (%)	71,91	79,88	62,07

The number of the annual rings per 1cm was the highest in beech, and the lowest in poplar. The width of the annual ring in poplar reaches almost 1cm wide, and is characterized by the highest late wood ratio.

Mean profile deviation – the roughness

Results of the mean profile deviation (Ra) of the test peces are presented in the Fig. 1, for each cutting direction and for all wood species.

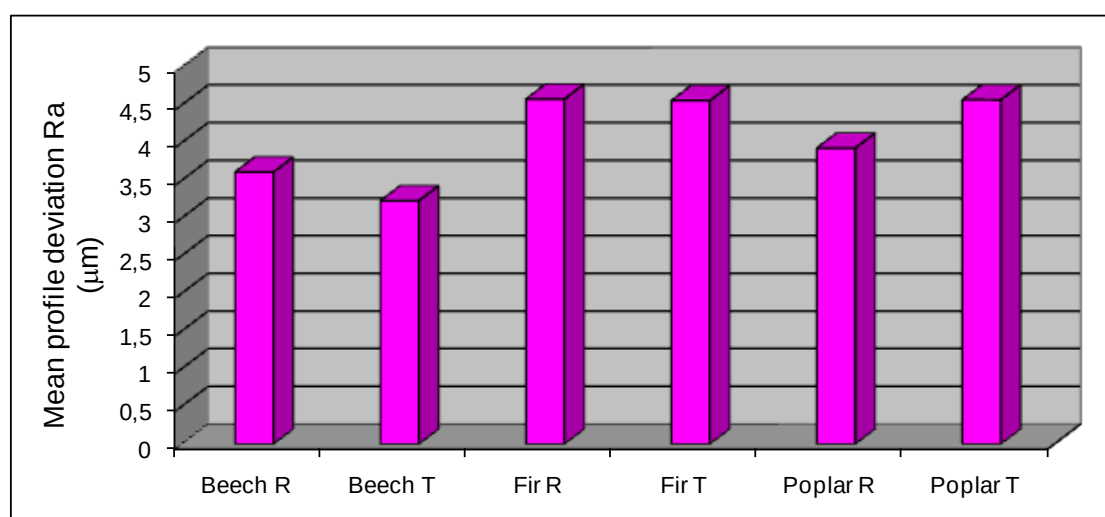


Fig. 1

Mean profile deviation (Ra) for beech, fir and poplar test pieces in radial and tangential direction.

The Fig. 1 shows that the cutting direction (tangential and radial) has no significant influence in the roughness of the test pieces. The roughness was similar between the fir and poplar, while it was lower in beech wood, at the same preparation conditions and cutting directions. This was expected according to the structural properties of the relevant wood species (higher density results in the lower porosity).

The standard DIN 4768/1:1974 divides the roughness into four categories, in regard to the Re values. The measured Re values of the samples from all three wood species and for both radial and tangential direction fall in the range of 3.23 - 4.58μm, which selects them into the third category (Ra = 2 – 10μm). This category of roughness is common for the bonding surfaces in the wood products industry, and does not require further sanding before the application of relevant wooden elements.

Surface chemical composition

For the purpose of analysis of the surface chemical composition it was very important to determine the quantity of carbon and oxygen elements, i.e. the ratio of oxygen to carbon atoms (O/C).

The Fig. 2 presents the characteristic spectres of the radial and tangential surfaces of beech wood. The peaks in the spectrums correspond to the percentile ratio of each element on the wood surface. The highest peak on all spectrums corresponds to the gold used for coating, and therefore it is excluded from the calculation of elements ratio.

The Table 3 presents the results of the mass and the atomic percentile ratio of carbon and oxygen, as well as the O/C ratio on both the radial and tangential surfaces of each of the three selected wood species.

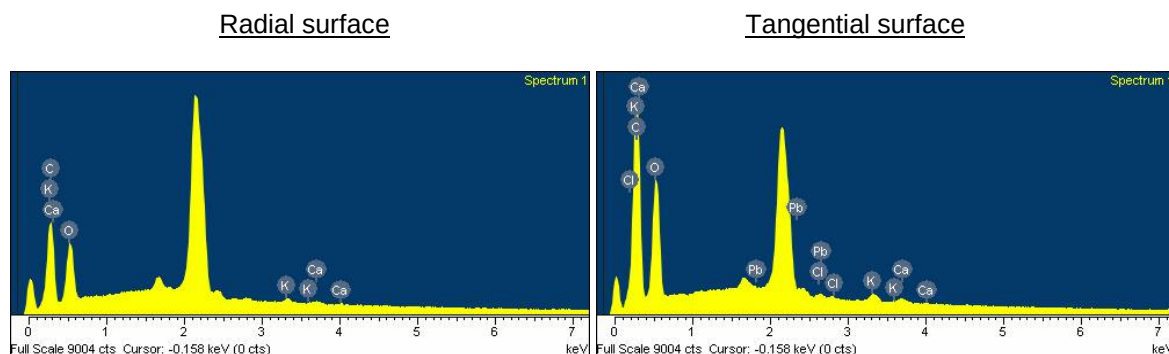


Fig. 2
Energy dispersive X-ray spectres of radial and tangential surface of beech samples.

Table 3
The mass and the atomic percentile ratio of carbon and oxygen, and the O/C ratio on the radial and tangential surfaces of beech, fir and poplar

Wood species	Surface	Element	Mass ratio (%)	Atomic ratio (%)	O/C atoms ratio
Beech	radial	C	52.37	59.55	0.68
		O	47.20	40.30	
	tangential	C	54.86	62.11	0.61
		O	44.36	37.71	
Fir	radial	C	55.41	62.62	0.59
		O	43.90	37.25	
	tangential	C	51.65	58.88	0.69
		O	47.85	40.95	
Poplar	radial	C	53.13	60.79	0.64
		O	45.14	38.77	
	tangential	C	52.60	60.06	0.66
		O	46.33	39.72	

The results of the chemical composition analysis given in the Fig. 2 and Table 3, clearly shows that the carbon and oxygen preset the dominant elements on the wood surface of all three species, which was expected according to the basic wood composition (approx. 50% C, 44% O, 6% H). In addition, the results suggest that there is no significant difference in the O/C ratio between the radial and tangential surfaces for all three selected wood species. Relatively high O/C ratio (0.59-0.69) observed in all measurements, also suggests that there are no significant build up of of extractive material on the surface, which could affect the wetting. According to the literature data (Dorris and Gray 1978) concerning individual chemical components, the cellulose O/C ratio is 0.83 and for the lignin it is 0.37, while the extracts have very low O/C ratio (0.1 - 0.2).

Contact angles

The results for wetting properties are graphically presented (Fig. 3, 4, 5 and 6) as the $\cos \theta$ change. Fig. 7 represents the change of the contact angle in time.

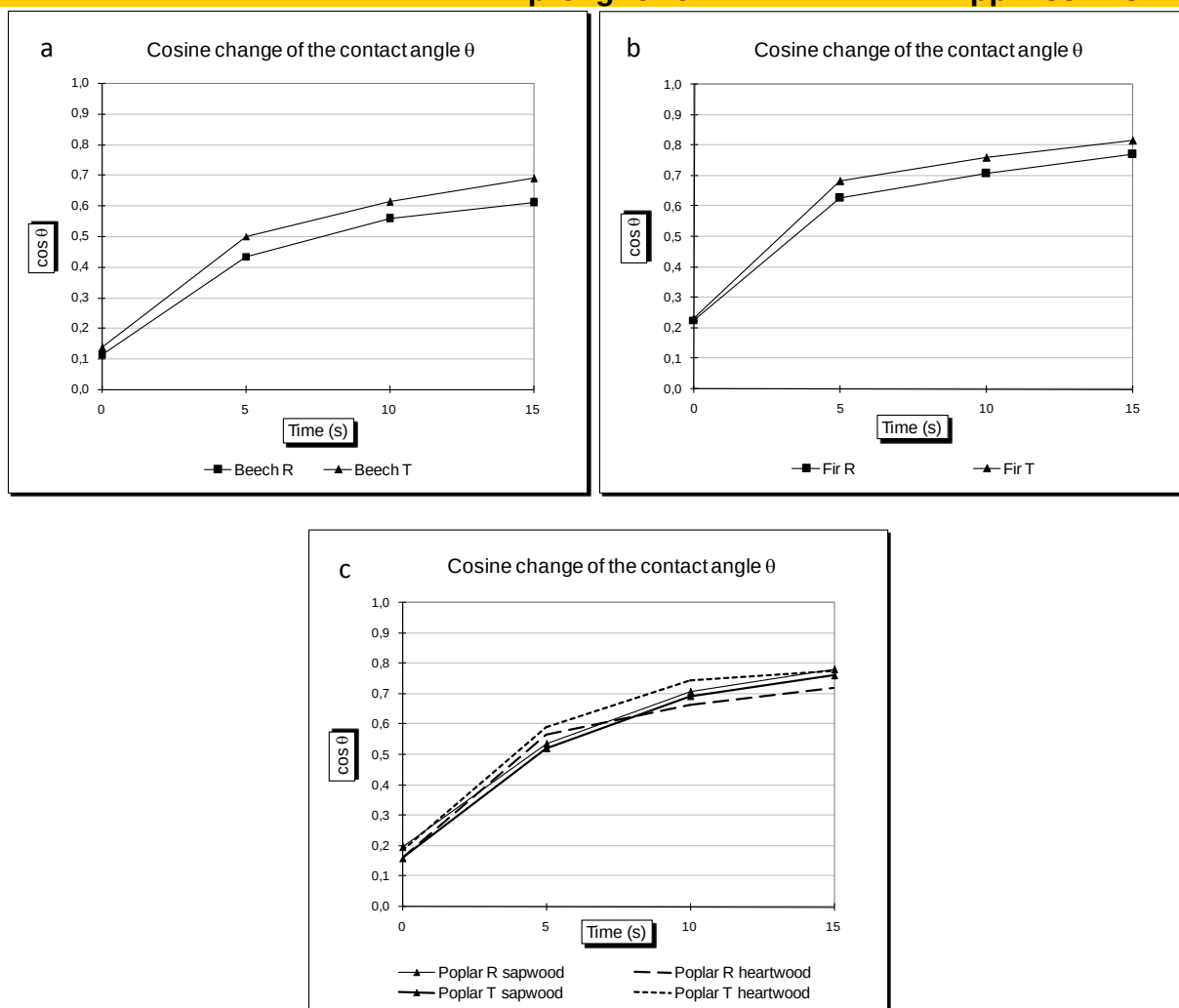
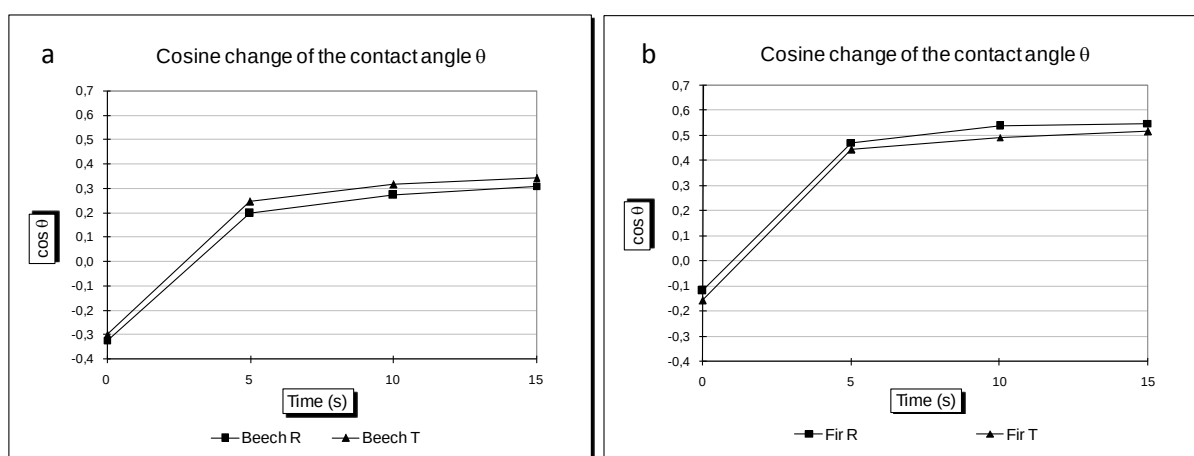


Fig. 3

*Change of the contact angle of water drop on the radial and tangential surface in the function of time
a - beech; b - fir; c - poplar.*



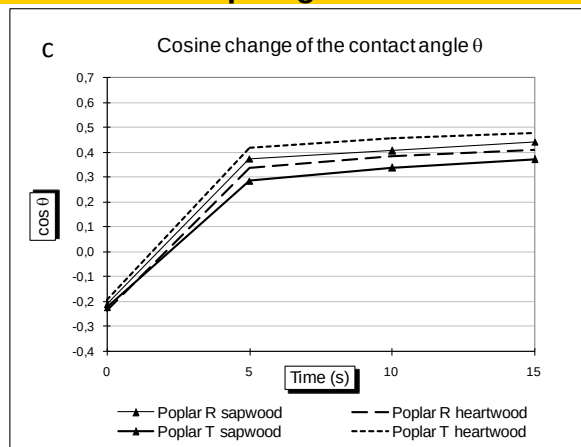


Fig. 4

Change of the contact angle of the UF I adhesive drop on the radial and tangential surface in the function of time
a - beech; b - fir; c – poplar.

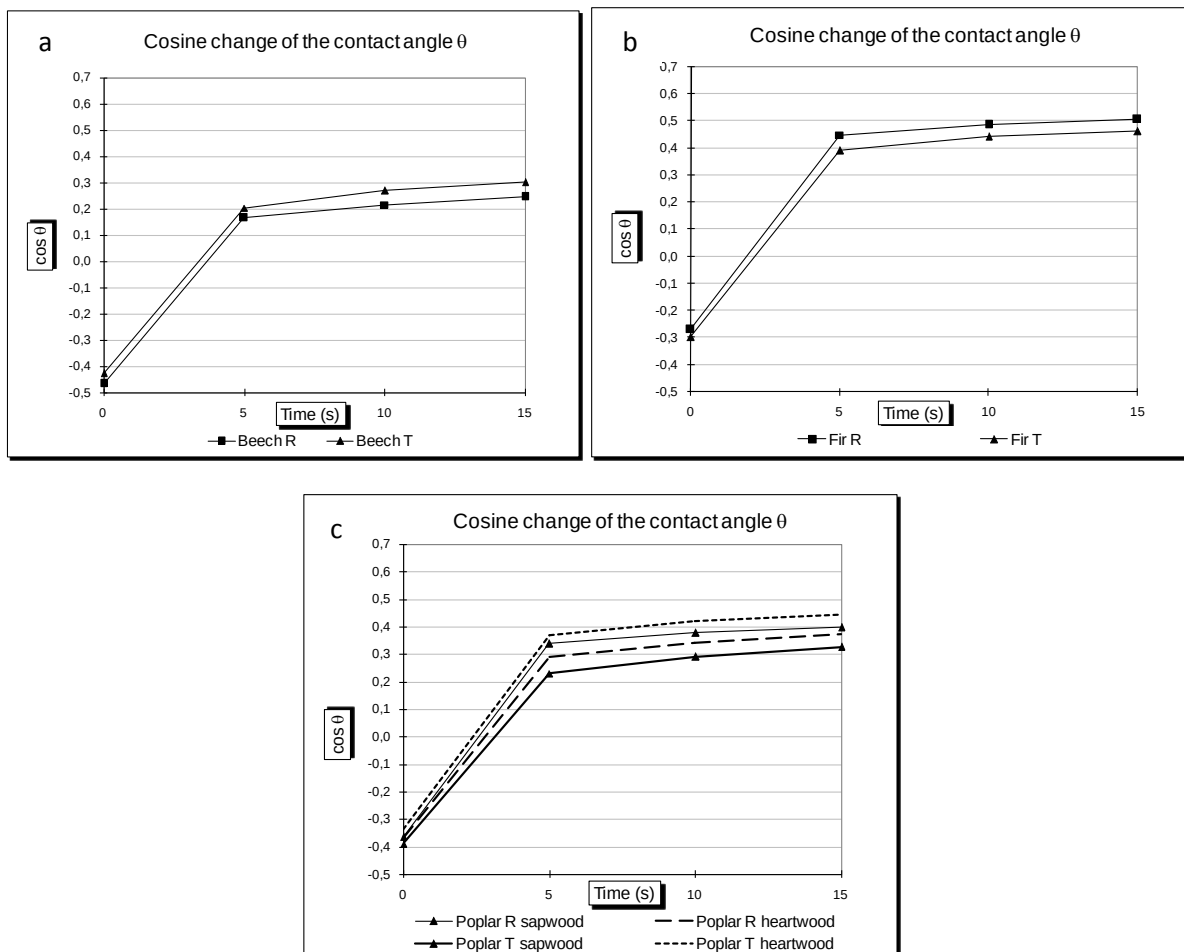


Fig. 5

Change of the contact angle of the UF II adhesive drop on the radial and tangential surface in the function of time
a - beech; b - fir; c – poplar.

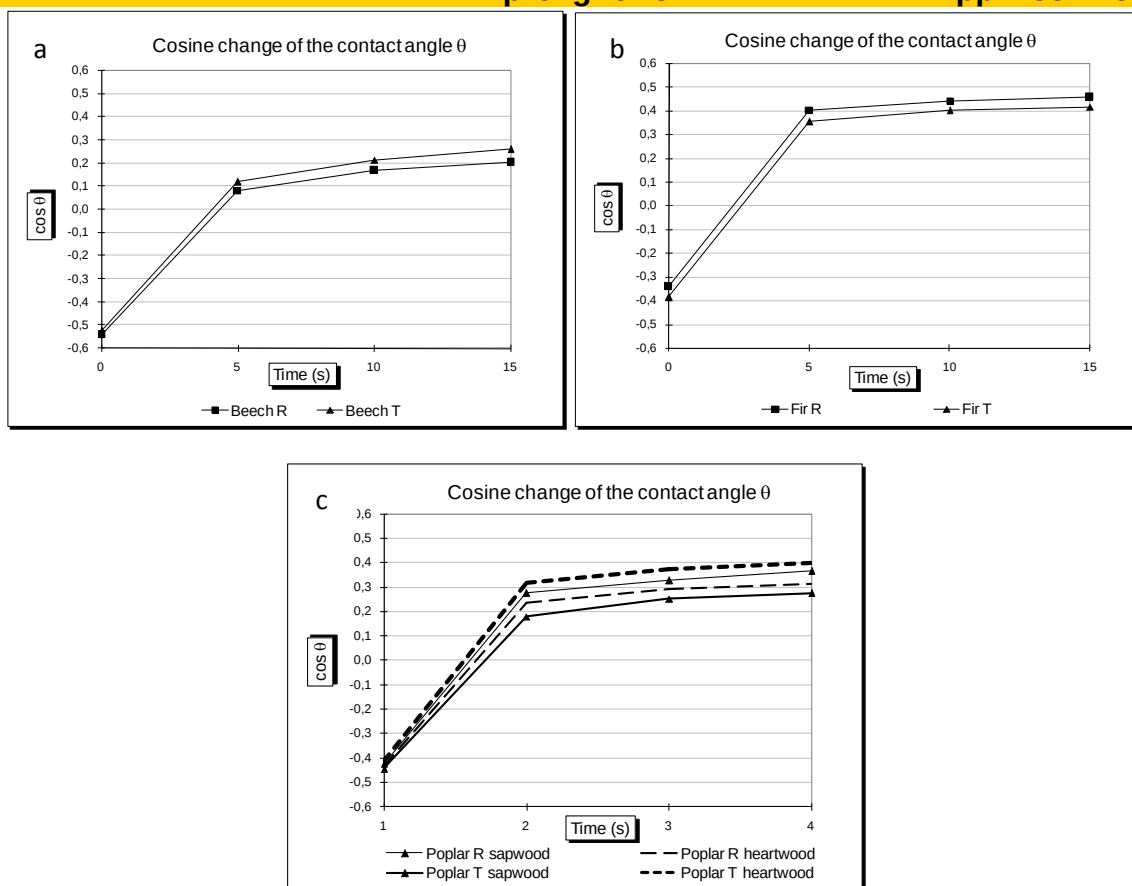


Fig. 6
Change of the contact angle of the UF III adhesive drop on the radial and tangential surface in the function of time
a - beech; b - fir; c - poplar.

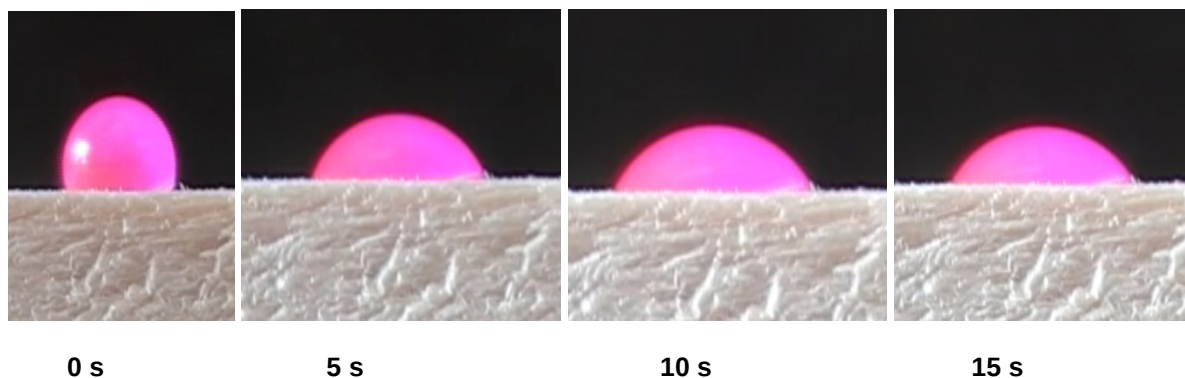


Fig. 7
Change of the contact angle of the UF I adhesive with the marking supstace, on the radial and tangential surface, and in the function of time (measurements obtained at the zero point and for each 5s untill 15s).

Distilled water had the lowest contact angle (θ) with wood surface in regard to all three prepared UF adhesives, in both radial and tangential surfaces and for all three selected wood species. This was expected having in mind the lower density and the viscosity of water in regard to UF adhesives.

Graphics also show relatively high contact angle at the zero position for all three UF adhesives (UF I, UFII i UF III) which resulted in the formation of so-called "sitting drops". Contact angle abruptly decreases for 30-35% in the first 5s on all three wood species, and both for water and the adhesives. Decrease in the contact angle is much slower during next 5s, especially for the adhesives (approx. 4-5% for all three wood species), while in the case of water, the contact angle decreases for 16% in the same period. During last 5s,

decrease of the contact angle of water is 11%, and for adhesives it is almost negligible, amounting only 2% on all wood species.

The increase in the adhesive molar mass resulted in the increased contact angle, thus lowering the wetting ability. At the zero position, the contact angle of UF II was 7-8% higher in regard to the UF I, for all three wood species (mean values were taken from both radial and tangential surfaces, since there was no significant difference between them). UF III adhesive had the 11-12% higher contact angle at the zero position than the UF I, and 5% higher contact angle in regard to UF II. This is the obvious consequence of the viscosity of the selected adhesives, since this property has the critical effects on the wetting and in turn on the formation of the contact angle. In this research, the increase in the contact angle has followed the increase in the viscosity of the selected adhesives.

In the case of the wood species effect, and for all three adhesives, the contact angle was the lowest on the fir substrates, having 9% lower values in regard to the beech wood. Poplar has resulted in 5% lower contact angle than beech. Therefore, the wetting was the best on the fir substrate and worst on beech. This is expected according to the anatomical structure of the selected wood species and the percentage ratio of early and late wood (Table 2). Namely, the fir wood is highly porous, its xylem consists of 93% of tracheides (conductive vessels) and has the highest ratio of early to late wood. The poplar, which is also highly porous wood species, consists of the 70% of mechanical elements but with the wide lumens and thin walls. Contrary, the beech wood has approximately 50% of mechanical elements, characterized by very small lumens and lignified walls.

CONCLUSIONS

The initial measurements of the contact angle (at the zero position) have showed high values for all of the three UF adhesive samples, resulting in the formation of characteristic "sitting drops".

Contact angle quickly decreases for 30-35% in the first 5s, for all tested combinations of wood surface and liquids (water and UF adhesives). Later, during next 5s and especially during last 5s, decrease in the contact angle is much slower, in particular for adhesives.

Adhesives with the lower molar mass resulted in the lower values of contact angle, thus clearly suggesting that the decrease in the adhesive molar mass increases the wettability.

For instance, the results of the contact angle measurements, at the zero position, have showed the highest values for the adhesive UF III, having the highest molar mass. The UF III adhesive had 5% higher contact angle in regard to UF II, and 11-12% higher contact angle in regard to UF I (with the lowest molar mass) at the zero position. In compliance with this, distilled water had the lowest contact angle in regard to all three prepared UF adhesives, on both radial and tangential surfaces of all selected wood species.

The relatively high O/C ratio on the radial and tangential surfaces of all three wood species suggests that there are no significant amounts of extractive material which could affect the wetting.

The roughness, expressed as the mean profile deviation (Ra), was similar between the fir and poplar, while it was lower in beech wood.

However, the contact angle was the lowest on highly porous fir wood surface, having the highest ratio of early to late wood. Contrary, the beech wood surface had the higher contact angle in regard to poplar and especially to fir. The beech wood consists of about 50% of fiber tracheids and wood fibers, which are much smaller elements than the softwood tracheids.

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