Mlađan Popović[1], Jasmina Popović[1], Milanka Điporovic-Momčilović[1], Nevena Vukić[2], Jaroslava Budinski-Simendić[2], Ivana Gavrilović-Grmuša[1], Fadhil Hamid[1]*

[1]University of Belgrade, Faculty of Forestry, Belgrade, Serbia,
[2]University of Novi Sad, Faculty of Technology,Novi Sad, Serbia

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The curing behavior of urea-formaldehyde adhesive in the presence of chemicaly treated narrow-leaved ash

ABSTRACT

The influence of Narrow Leaved Ash (Fraxinus angustifolia Vahl. ssp. Pannonica Soo & Simon) pre-treatments on the curing behavior of urea-formaldehyde (UF) adhesive was studied. Differential scanning calorimetry (DSC) was used to monitor the curing reaction of UF adhesive mixed with non-treated wood flour (control series) and wood flour of Narrow leaved Ash obtained after different pre-treatments of wood particles. Three different pre-treatments were applied, one with sodium carbonate solution (0.03 g/g of dry wood), one with acetic acid solution (0.06 g/g of dry wood) and one with distilled water. The curing reaction of different adhesive systems showed no apparent differences in peak temperature values, and there were no significant differences in the enthalpy of the reaction. However, it was assessed that the activation energy has lower values for the UF systems with alkaline and acid treated wood flour (76.64 and 76.92 kJ/mol) in regard to the UF systems with control (untreated) wood flour and water treated wood flour (79.28 and 79.14 kJ/mol). In addition, the application of iso-conversional models have shown different curing behavior of UF adhesive systems.

Keywords: *chemical treatment, narrow-leaved ash, curing kinetics, UF adhesive.*

1. INTRODUCTION

During this decade, the global particleboard production holds a steady upward course, resulting in more than 93 million m^3 of produced panels in 2017, according to FAOSTAT [1] data. Most of these panels are interior based particleboards glued with urea-formaldehyde (UF) adhesive. It is estimated that the UF adhesive has about 90-92 % share of all the adhesives used in European particleboard production [2]. Hence, the popularity of UF adhesive does not seem to give away, despite the more stringent requirements in terms of formaldehyde emission from the panels produced with this type of resin.

Adhesion of natural fibers and thermosetting resins may involve chemical interactions, which can be achieved through hydrogen or covalent bonding between the reactive hydroxyl (-OH) groups available on the lignocellulosic fibers and compatible functional groups on the thermosetting resins.

Hence, the chemical composition of wood may affect the quality of bond forming between wood and adhesive. In that aspect, the OH groups of wood have a significant role in adhesion [3,4]. The number of available OH groups can be increased by modifying the fiber surface, for which purpose, the alkaline treatments present the widely used technique [5,6]. Due to its acidic nature, the acetyl groups in hemicelluloses are especially susceptible to hydrolytic degradation, and the -OH groups are formed in their place [7]. In this way, the application of pre-treatments in alkaline condition can increase the number of OH groups available for creating bonds with the adhesive.

The treatments with NaOH solutions of various concentrations are very common practice for improving the certain properties of lignocellulose materials. The appropriate ionic radius of Na ions and their embedding into the crystalline cellulose lattice allow the NaOH solutions to expand the crystallographic planes and change the conformation of cellulose, thus changing the arrangement of the cellulose chains inside the crystal lattice [8]. Simultaneously with swelling of the cellulose fibers, their specific surface increase. The effect of this alkaline treatment is the breakup of existing hydrogen bonds between cellulose macromolecular chains, with simultaneous formation of new hydrogen bonds between cellulose chains. Decomposition and removal of

*Corresponding author: Mlađan Popović
E-mail: mladjan.popovic@sfb.bg.ac.rs
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hemicellulose, lignin and extraneous materials also occure [9]. However, higher concentrations of alkalis may cause the delignification, which may weaken or damage the wood fibers [10]. It was found that the treatment with sodium hydroxide, with 10 % concentration or higher, inevitably starts to degrade the cellulose fibers, regardless of the treatment duration. However, the treatment of cellulose fibers with 5 % NaOH during 2 hours, beside the increase in swelling of microfibrils and fibers also increases the ratio of amorphous regions, i.e. decreases crystallinity, which improves wetting and thermal stability. The swelled fibers, treated this way, show increased reactivity in comparison to initial fibers, and also have larger effective surface available for chemical interactions [11].

The acidic environment also leads to hydrolytic degradation of lignocellulosic material. The rate and degree of degradation of polysaccharides are proportional to the concentration of reagents, treatment temperature and pressure and to process duration. One of the main effects of wood treatments at low pH values, such as the water treatment [12-15] and treatment with dissolved acids [16-18] is the removal of significant amounts of hemicelluloses from wood tissue. However, degradation of treated samples can be reduced while improving their properties, by using the appropriate selection of reagents and processing parameters. The most studied treatments have utilized the sulfuric acid solutions of 0.5 to 1.5 %, at the temperatures of 120-160 °C, followed by the solutions of hydrochloric, nitric and phosphoric acids. Wood having modified structure and properties can be utilized for further processing [19]. For instance, medium density fiberboard (MDF) panels produced by treated wood chips had their water resistance improved [20]. Another study showed that MDF panels produced of oxalic acid treated wood exhibited decreased water absorption due to the partial removal of hemicelluloses in regard to water treated and untreated samples [21]. The same samples also showed decreased thickness swelling, but at the expense of worsening their mechanical properties. Decrease in mechanical properties of wood plastic composites made from oxalic acid treated fibers, was explained by chemical changes in cell wall as a consequence of extraction of hemicelluloses, which affected the compatibility between treated wood fibers and UF adhesive [22]. In addition, another factor that causes decrease in mechanical properties is the lower density of treated samples, due to the loss of wood mass during treatment [23,24].

Water treatment of lignocellulosic material, at elevated temperatures, results in reactions of autocatalytic hydrolysis of polysaccharides, due to the catalytic effect of acids already present in wood molecular structure, such as the acetic acid in hemicelluloses. Besides the degradation of hemicelluloses, the water treatment also results in losses of extractive materials, as well as lignin in lower extent [25]. Extraction of hemicelluloses decreases the number of OH groups, thus decreasing the water absorption and increasing dimensional stability of treated wood samples [26]. Oriented strand board (OSB) panels produced from water treated samples of red maple showed decreased swelling in comparison to untreated wood samples [27]. The hydro-thermal treatment of beech samples also resulted in reduced water absorption, however, with decreased shear strength and increased wood failure percentage at the bond line [28]. Namely, the OH groups of wood present the potential sites for bond formation with adhesive [3]. By reducing the number of available OH groups, the number of possible connections between the wood and adhesive is also reduced. In addition, the changes of pH of wood also affect the bond formation with adhesive, due to removal of "acidic" acetyl groups.

The chemical treatments lead to the changes in structure and composition of wood, thus affecting its chemical, mechanical and physical properties, but also the adhesion processes and strength of the adhesive bond. In this study, the narrow-leaved ash wood samples were treated with sodium carbonate and acetic acid solutions, as well as with distilled water. The goal was to evaluate the influence of these treatments on the curing of UF adhesive. Differential scanning calorimetry (DSC) was applied to examine the curing behavior of UF adhesive mixed with non-treated (control) wood flour and the UF adhesive systems with wood flour obtained from treated wood particles.

2. MATERIALS AND METHODS

2.1. Pre-treatment of wood particles

Three samples of Narrow leaved Ash mature trees were cut into the logs and the wood particles were obtained on the industrial flaker. The air dried wood particles were used for the chemical pre-treatments in autoclaves. The pre-treatments were performed in 2.5 L autoclaves, at 100 °C, during 1 hour. The alkaline pre-treatment was obtained with the addition of 0.03 g/g sodium carbonate per dry wood, while the acid pre-treatment was obtained with 0.06 g/g of acetic acid per dry wood. The third pre-treatment was performed with pure distilled water (hot water treatment). Treated and non-treated wood particles were further cominuted using a laboratory mill. The fraction of wood flour passing through the No 100 sive (0.14 mm) was chosen for the DSC measurements.

The reagents in chemical pre-treatments were: sodium carbonate (Na_2CO_3), anhydrous (99.9%),

AnalaR NORMAPUR (Belgium) and acetic acid (CH_3COOH), glacial (99.5%) Zorka Pharma - Hemija d.o.o. (Serbia). Commerial UF adhesive was used with the following characteristics: dry content was 66.92 % (EN 827); density was 1.27 (measured with areometer); viscosity by Brookfield was 415 mPa·s (EN 12092) and the pH value was 8.05 (EN 1245).

2.2. DSC measurements

Test samples for evaluation of the influence of wood species were obtained by adding wood flour into the adhesive 24 h prior testing and stored into refrigerator at 5 °C. The content of wood flour was 10 % by weight (od wood flour vs. od adhesive). The addition of NH_4Cl was 0.5 % by weight (od catalyst vs. od adhesive) for all samples mixed with wood flour. For all DSC tests, the UF adhesive was diluted to a concentration of 50 %.

The curing reactions of UF adhesive / wood flour systems were monitored by DSC Q20 differential scanning calorimeter (TA Instruments, USA). Approximately 4 to 5 mg of test samples were hermetically sealed in aluminum pans. Dynamical scanning regime was used in the temperature range from 30 °C to 200 °C. In order to obtain kinetic parameters, different heating rates (β) were used: 5, 10 and 20 °C/min. Before each scan, the heating blocks inside the DSC instrument were adjusted to 30 °C, and the nitrogen was used as a purge gas. The instrument software, TA Universal Analysis, was used to calculate peak temperatures and the enthalpy of reaction, with the exothermic peak plotting in upward direction.

In order to present the curing of UF adhesive systems with wood flour in isothermal environment, the obtained DSC data were subjected to iso-conversional modeling. The Kisisinger-Akahira-Sunose (KAS) and the Friedman iso-conversional methods were used, as presented elsewhere [29-31].

3. RESULTS AND DISCUSSION

The basic parameters for the curing reaction of UF adhesive mixed with different wood flour are given in the table 1. The values for peak temperatures and enthalpy of reaction for each adhesive system are obtained by DSC instrument software, while the energy of activation (E_a) was calculated using the general Kissinger model. It can be noticed that no apparent differences exist in peak temperature values among the tested series, as the temperature difference for the maximum of reaction at all three different heating rates do not differ more than 1°C. Similarly the results of the reaction enthalpy also do not differ significantly among the test series. Hence, those two parameters do not show any influence of pre-treatments of wood material on the curing reaction of UF adhesive. On the other hand, there is a slight decrease of the activation energy (E_a) for the UF adhesive systems with wood flour treated with alkali (0.03 g/g sodium carbonate) and acid (0.06 g/g acetic acid).

Table 1. Peak temperatures, enthalpy (ΔH) and activation energy (E_a) of the curing reaction of different UF adhesive / wood flour systems: control (non-treated wood flour); alkaline (wood flour treated with sodium carbonate); acid (wood flour treated with acetic acid) and water (wood flour treated with distilled water)

Tabela 1. Temperature maksimuma reakcije, entalpija (ΔH) i energija aktivacije (Ea) reakcije očvršćavanja različitih sistema UF veziva sa drvnim brašnom: kontrolni (netretirano drvno brašno); alkalni (tretman natrijum karbonatom); kiseli (tretman sirćetnom kiselinom) i vodeni (tretman vodom)

UF adhesive / wood flour sample series	Peak temperature (°C)			ΔH (J/g)	Ea (kJ/mol)	R^2
	β = 5 °C/min	β = 10 °C/min	β = 20 °C/min			
UF + control wf.	80.37	89.98	98.03	77.03 ±2.61	79.29	0.9952
UF + alkali wf.	80.15	89.06	98.46	77.88 ±8.21	76.64	1
UF + acid wf.	79.36	88.96	97.48	78.15 ±7.54	76.92	0.9973
UF + water wf.	79.52	87.92	97.20	74.97 ±11.74	79.14	0.9997

The data from DSC experiments were further analysed using the KAS and Friedman iso-conversional methods, which enabled the calculation of activation energy as a function of conversion, presented in Figures 1a and 1b.

The application of KAS method showed fairly constant values of E_a in the conversion range from 10 % to 70 %, while from the 80 % until the end of conversion they exhibited the steep increase (Figure 1a). The increase of E_a at the later stage of curing may be the consequence of the diffusion controlled reaction of UF adhesive [32].

Such behavior was similar for all UF adhesive systems. However, the E_a values were at different levels depending on the wood flour type mixed with UF adhesive. The UF adhesive with non-treated (control) wood flour achieved the highest activation energy throughout the conversion, while the acetic acid treatment influenced in the lowest E_a values for the relevant UF adhesive system. The application of Friedman method showed decrease

of E_a values at the beginning of the conversion, and significant increase after 60 % of conversion until the end of reaction, for all UF adhesive systems (Figure 1b). In the range of conversion from 50 % to 95 %, the UF adhesive systems with acetic acid and water treated wood flour exhibited lower E_a values. Then the UF adhesive systems with control wood flour and alkaline treaded wood flour.

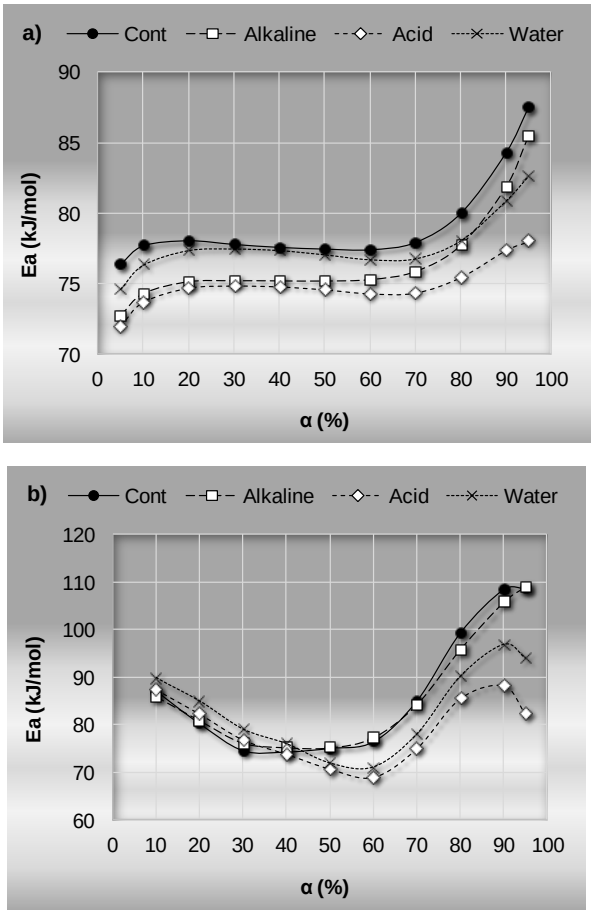

Figure 1. Activation energy (E_a) dependance on conversion obtained by KAS (a) and Friedman (b) methods for the curing of different UF adhesive systems with wood flour: control (non-treated wood flour); alkaline (wood flour treated with sodium carbonate); acid (wood flour treated with acetic acid) and water (wood flour treated with distilled water)

Slika 1. Zavisnost energije aktivacije (Ea) od stepena konverzije, dobijena KAS (a) i Friedman (b) metodama, pri očvršćavanju različitih sistema UF veziva sa drvnim brašnom: kontrolni (netretirano drvno brašno); alkalni (tretman natrijum karbonatom); kiseli (tretman sirćetnom kiselinom) i vodeni (tretman vodom)

The obtained non-isothermal kinetic data were also used for isothermal kinetic calculations, i.e. to predict the conversion degree variation in function of time, at the given constant temperature [33]. Hence, the KAS and Freidman methods were applied to calculate these predictions, and the conversion dependence on time for each method is given for the temperature of 80 °C (Figures 2a and 2b).

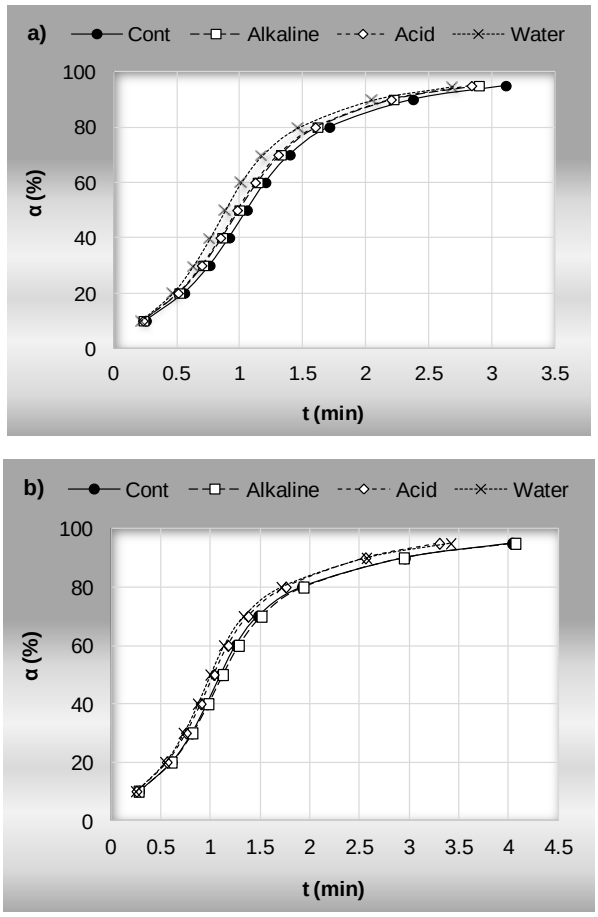

Figure 2. Conversion degree in function of time at the constant temperature of 80°C obtained by KAS (a) and Friedman (b) methods, for the UF adhesive systems with wood flour: control (non-treated wood flour); alkaline (wood flour treated with sodium carbonate); acid (wood flour treated with acetic acid) and water (wood flour treated with distilled water)

Slika 2. Stepen konverzije u funkciji vremena pri konstantnoj temperaturi od 80°C, dobijen KAS (a) i Friedman (b) metodama, pri očvršćavanju različitih sistema UF veziva sa drvnim brašnom: kontrolni (netretirano drvno brašno); alkalni (tretman natrijum karbonatom); kiseli (tretman sirćetnom kiselinom) i vodeni (tretman vodom)

The isothermal kinetic calculations according to KAS model showed that the UF adhesive mixed with water treated wood flour exhibited the shortest conversion time. In other words, this UF adhesive system had the highest rate of curing (Figure 2a). The same diagram also shows that the slowest

curing system is the UF adhesive with non-treated wood flour (control). However, the application of the Friedman model showed that both water and acid treatments of wood material had positive effects on the curing reaction of UF adhesive (Figure 2b). In the case of Friedman model, the alkaline treatment showed no effects on the curing reaction of UF adhesive, i.e. curing behavior was similar between the UF adhesive mixed with alkaline treated wood and the one mixed with control wood flour.

4. CONCLUSIONS

The influence of different chemical pre-treatments of wood particles on the curing reaction of UF adhesive was evaluated using general kinetic data obtained from non-isothermal DSC measurement. The obtained data were further analyzed by application of KAS and Friedman iso-conversioanl methods.

The results of peak temperatures and enthalpy of the reaction showed no apparent influence of chemical pre-treatments of wood material on the curing reaction of UF adhesive. Even though, the lowest enthalpy of the reaction was obtained for the UF adhesive system mixed with water treated wood flour, this difference was not statistically significant.

On the other hand, the energy of activation was lower for the UF adhesive systems with alkaline and acid treated wood flour (76.64 and 76.92 kJ/mol) in comparison to the activation energy of 79.29 kJ/mol, obtained for the UF adhesive system with non-treated wood flour (control series). The water treated wood flour has shown no effects on the activation energy.

The curing reaction of UF adhesive in the presence of treated and non-treated wood flour was further evaluated by KAS and Friedman iso-conversional methods. The isothermal kinetic predictions obtained by KAS method showed that the water treatment of wood particles resulted in the shortest curing time of UF adhesive. The application of Friedman method showed that both water and acetic acid treatment have decreased curing time in regard to the UF adhesive mixed with non-treated wood flour.

According to the iso-conversional methods, it could be concluded that the water treatment can be successfully applied in the processes involving the bonding of wood furnish with UF adhesive, such as the particleboard and fiberboard production.

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IZVOD

OČVRŠĆAVANJE UREA-FORMALDEHIDNOG ADHEZIVA U PRISUSTVU HEMIJSKI TRETIRANOG DRVETA POLJSKOG JASENA

U ovom radu istraživan je uticaj pred-tretmana poljskog jasena (Fraxinus angustifolia Vahl. ssp. Pannonica Soo & Simon) na tok očvršćavanja urea-formaldehidnog (UF) adheziva. Metoda diferecijalne skenirajuće kalorimetrije (DSC) korišćena je za praćenje reakcije očvršćavanja UF adheziva u smeši sa netretiranim drvnim brašnom (kontrolna serija) i drvnim brašnom dobijenim od različito tretiranog iverja poljskog jasena. Primenjena su tri različita pred-tretmana: tretman rastvorom natrijum karbonata (0,03 g/g apsolutno suvog drveta), tretman rastvorom sirćetne kiseline (0,06 g/g apsolutno suvog drveta) i tretman destilovanom vodom. Rezultati praćenja očvršćavanja različitih adhezivnih sistema nisu ukazali na značajne razlike u temperaturama maksimuma reakcije, kao niti u vrednostima entalpije reakcije. Međutim, relativno niže vrednosti energije aktivacije uočene su kod UF adhezivnih sistema sa drvnim brašnom dobijenim nakon alkalnog i kiselog tretmana (76,64 and 76,92 kJ/mol) u odnosu na UF adhezive sa kontrolnim (netretiranim) drvnim brašnom i drvnim brašnom tretiranim vodom (79,28 and 79,14 kJ/mol). Pored toga, primenjene izo-konverzione metode pokazale su različito ponašanje ispitivanih UF adhezivnih sistema tokom očvršćavanja.

Ključne reči: *hemijski tretman, poljski jasen, kinetika očvršćavanja, UF adheziv.*

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