ГЛАСНИК ШУМАРСКОГ ФАКУЛТЕТА, БЕОГРАД, 2005, бр. 92, стр. 111-124

BIBLID: 0353-4537, (2005), 92, p 111-124

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THE INFLUENCE OF FIRE RETARDANTS ON THE PROPER-TIES OF BEECH AND POPLAR VENEERS AND PLYWOOD

Abstract: Rising demands for fire resistance properties of wood construction and elements matching new standards have been an important part of building codes during the last decade. On the other side, lack of more detailed research on interaction between wood species and selected fire retardant chemicals even with basical one is evident. This is particularly truth with domestic wood species. In this research, beech and poplar veneers were immersed in 25% solutions of monoammonium phosphate (MP) and sodium acetate (SA) and impregnated for different periods of time. To determine the preliminary level of fire retardancy achieved in veneers before manufacturing of finished plywood, thermogravimetric (TG) and derivative thermogravimetric (DTG) methods were used. TG and DTG analyses of treated and untreated wood, as well as of fire retardants alone, were performed. The next properties of impregnated and nonimpregnated veneers and plywood were determined: absorption of impregnant solution (A), weight percent gain (WPG) of impregnant, equilibrium moisture content (EMC), pH values, and in the case of plywood, strength and fire resistance. Fire resistance of plywood was tested in accordance with standard test for resistance to the effects of fire and the most efficient fire retardant, monoammonium phosphate, had the same result as TG/DTG analyses, which pointed out the validity of TG methods in predicting fire resistance of future products.

Key words: veneers, plywood, fire retardants, beech, poplar, termogravimetry

УТИЦАЈ УСПОРИВАЧА ГОРЕЊА НА СВОЈСТВА ФУРНИРА И СЛОЈЕВИТИХ ПЛОЧА ОД БУКОВОГ И ТОПОЛОВОГ ДРВЕТА

Извод: Последњих десетак година, у складу са новим стандардима и прописима, знатно повећана је потреба за ватроотпорним дрвеним конструкцијама и елементима. С друге стране, евидентан је недостатак детаљних истраживања

Dr Jovan Miljković, Full Prof., Faculty of Forestry, The University of Belgrade, Belgrade Mr Ivana Grmuša, assistant, Faculty of Forestry, The University of Belgrade, Belgrade Dr Milanka Điporović, docent, Faculty of Forestry, The University of Belgrade, Belgrade Dr Zorica Kačarević-Popović, researcher, Institute of Nuclear Science "Vinča", Belgrade интеракције између дрвних врста, посебно домаћих и изабраних, чак и основних, ватроотпорних хемикалија. У овом истраживању, букови и тополови фурнири су различито време импрегнисани потапањем у 25% раствор моноамонијум-фосфата (MP) и натријум-ацетата (SA). У циљу прелиминарног одређивања постигнуте ватроотпорности у фурнирима, пре израде готове фурнирске плоче, спроведене су динамичке методе термалне анализе и то: термогравиметријска (TG) анализа и деривативна термогравиметријска (DTG) анализа импрегнисаног и неимпрегнисаног дрвета као и самих ватроотпорних средстава. За импрегнисане и неимпрегнисане фурнире су такође одређене и следеће карактеристике: абсорпција импрегнационог раствора (А), масени процентуални принос ватроотпорних средстава (WPG), равнотежна влага (EMC), pH вредност, а за фурнирске плоче још и смицајна чврстоћа и ватроотпорност. Ватроотпорност фурнирских плоча је тестирана према ЈУС стандарду и као ефикаснији се показао моноамонијум-фосфат што су показале и TG/DTG анализе из чега се може закључити да TG методе имају значајну улогу у предвиђању ватроотпорности будућег производа.

Кључне речи: фурнир, фурнирска плоча, ватроотпорна средства, буква, топола, термогравиметрија

1. INTRODUCTION

Wood construction for many years has been classified in building codes under three standard types - heavy timber, ordinary and light frame. Heavy timber and ordinary types have been used widely in industrial, commercial and assembly buildings. However, light frame accounts for about 80% of the world wide dwellings and buildings. The self-insulating properties of wood, particularly in the large wood sections used in heavy timber construction, are an important factor in providing a good degree of fire resistance.

Light wood-frame construction, also doors, windows, flooring and paneling, can be protected to provide a high degree of fire performance. Fire-resistance rating of 30-60 minutes are readily attained for such products. Treatment of wood with fire-retardant chemicals or fire-retardant coatings is an effective means of preventing flame spread.

Forms of wood found in construction, have expanded from timber and decking to a wide variety of wood-based materials. Products such as plywood, laminated veneer lumber and more recently composites of plywood and particleboard (such as wafer board and strand board) are now commonly used. Fire-retardants are applied to wood and woodbased materials in two mentioned ways. One method involves surface coating to protect the underlying wood members. Such treatments increase time to ignition and reduce flame spread following ignition. However, such materials are of little benefit in a post-flashover fire. The second and more important application is by soak treatment. This application assumed absorption of fire-retardant compounds, such as different inorganic salts.

Such impregnating treatments both reduce flame spread and produce wood-based products which are accepted in the building codes, since they have improved fire endurance. The function of fire-retardant salts and other absorbed chemical compounds mostly in the form of water solution is complex, and lack of literature data on absorption rate, fire-retardant weight gain, equilibrium moisture content and pH value of treated wood, as well as thermogravimetric properties of treated wood in relation to wood species and the type of fire-retardant compound do exist (Rowell M.R. *et al.*, 1984, Woo J.K., Schniewind A.P., 1987).

On the contrary, in the past decade a number of patented fire-retardant formulations have been developed and numerous trade names appeared at the market (1989). Synergical action of impregnant mixtures diffused in wood tissue by pressurized or vacuum equipment were in research focus for a long time (Wang S-Y., Rao Y.C., 1999). However, the lack of initial information on particular impregnant and its absorptivity, gain, influence on adhesive joint and fire retardant effects concerning specific wood species are evident. Thus, in this study some factors affecting absorption step and thermal degradation of impregnated beech and poplar veneers as common domestic wood species, with monoammonium phosphate (*MP*) and with sodium acetate (*SA*) have been investigated.

2. MATERIALS AND METHODS

2.1. Materials

For the purpose of this research, rotary-cut 1,5 *mm* thick beech and poplar veneers (*Fagus moesiaca* - phytocen. *Fagetum Montanum* from East Serbia and *Populus Euramericana* Dode cv. *Robusta* from Danube riverside in Vojvodina) have been used as representative of common wood species in Serbia.

Salt Co	Molecular mass Молекулска	Melting point Тачка топљења	Solub. in H ₂ O Растворљивост у H ₂ O <i>g /</i> 100 <i>g</i>		pH	
	маса	°C	20°C	100°C	50 g·L -, 20 C	
MP	115.00	190	36.8	174.0	3.8-4.4	
SA	82.03	324	124.0	170.15	7.5 - 9.0	

 Table 1. The main characteristics of impregnants

 Табела 1. Главне карактеристике импрегнаната

Known fire retardant salts:

- monoammonium phosphate (MP), $NH_4H_2PO_4$ (proanalysi), "Zorka-pharma" Šabac, and
- sodium acetate (SA), CH₃COONa, (proanalysi), Instit. for technology of nuclear and other mineral raw materials, Beograd, have been used as impregnants.

2.2. Impregnation treatment

Treatment of veneers with water solution of fire retardant salts i.e. impregnants was as follow: veneers were dried to constant weight at $55\pm5^{\circ}C$ in convection dryer. Dry veneers were then submerged in 25% water solution of impregnants for 1, 2, 3, and 4 hours at $20^{\circ}C$.

After submersion for said periods, veneer specimens were suspended to drain for 5 minutes, at the end of wich time the excess surface water was removed and specimens were weighted with accuracy of $\pm 0.2\%$. Furthermore, impregnated veneers were dried to constant weight at 55 \pm 5°*C* and weighted again.

For each set of parameters nine veneers were impregnated and average values of absorption and weight percent gain were calculated.

2.3. Plywood production

Impregnated and nonimpregnated beech and poplar veneers were used to produce 3 layer plywood (Nikolić S.M., 1988). Veneers submerged for 1 hour according to procedure mentioned before in the previous paragraph, were used for this purpose. Urea-formaldehyde adhesive (dry matter $67\pm1\%$, viscosity $100\pm10 \ s$ (F420) and gel time $50\pm5 \ s$ with 1% of NH₄Cl catalyst added, PKS-Latex, Cacak) was applied in quantity of $180 \ g \cdot m^{-2}$.

Pressing conditions: press temperature was 120°C, specific pressure of 9,8 bars and pressing time of 9,5 *min* vs. specific pressure of 5,9 bars and pressing time of 6,5 *min* were applied for pressing beech and poplar plywood respectively.

2.4. Determination of veneer and plywood characteristics

The absorption (A) of veneer samples was calculated according to equation (1) and weight percent gain (*WPG*) according to equation (2):

$$A = \frac{m_1 - m_0}{m_0} \cdot 100 \, [\%], \quad \dots \quad \dots \quad \dots \quad (1)$$

where are: m_0 - mass of nonimpregnated dry veneer [g], m_1 - mass of impregnated wet veneer [g] and m_2 - mass of impregnated dry veneer [g].

Equilibrium moisture content (*EMC*) of impregnated and nonimpregnated veneer samples, was determined after their conditioning in an atmosphere of $65\pm2\%$ relative air humidity at the temperature of $20^{\circ}C\pm1\%$.

The pH value of solution of fire retardant salts (impregnants) and veneers was measured by Iskra MA-5725 pH-meter. The pH value of impregnated and nonimpregnated







veneers was completed by the next procedure: the mass of 30 g of dried, milled and screened particles prepared from each veneer and passing through 12 mesh sieve (0,5 mm, DIN 4188), was mixed with 400 cm^3 of distilled H₂O free of CO₂, and pH value was measured after 30 minutes.

The shear strength of adhesive joint in plywood was determined on a conditioned standard specimens (Fig. 1) according to Yugoslav standard (JUS D. A8.067). Each force at failure was recorded and shear strength was expressed in Newtons per *mm*² of shear area [*N*·*mm*⁻²]. For each group of specimens, average shear strength and average percentage wood failure was calculated.

Thermogravimetric (TG) and Derivative thermogravimetric (DTG) analyses of veneer were completed by Perkin-Elmer TGS-2 equipment in nitrogen atmosphere. Heating rate was $10^{\circ}C \cdot min^{-1}$ and heating range from $30-450^{\circ}C$ was used. The same particle size of milled veneers as for pH measurements was used.



Fig. 2. Aparature for testing of fire retardant properties of plywood

For the sake of better comparison, veneers of the same species having approximate WPG of SA and MP impregnants were used for these analyses.

Слика 2. Апаратура за испитивање ватроотпорних својстава шперплоче

Comparative evaluation of fire resistance of plywood was accomplished by simple introductionary procedure according to Yugoslav standard (JUS D.T4.039). Square specimens of plywood panel made of treated and nontreated veneers were placed on horizontal metal frame and exposed to butan/propan flame from a bottom plywood surface for 30 minutes. Equipment consists of stand (1) with frame holders for panel specimen (2) and butan/propan burner (3) at the distance of 70 *mm* down the lower panel surface (Fig. 2).

3. RESULTS AND DISSCUSION

3.1. Absorption and weight percent gain of impregnant in veneers

Absorption (A) and weight percent gain (WPG) of impregnant in veneers are shown in Fig. 3 for poplar veneers (a) and for beech veneers (b). Both absorption (A) and weight percent gain (WPG) increased with extended time of immersion of veneers in 25% solution of impregnants.

The absorption (A) of the initial 25% solution of impregnants i.e. monoammonium phosphate (MP) and sodiumacetate (SA) was permanently higher than residual quantity of dry impregnants in veneers (WPG). Poplar veneers showed better absorptivity (A) and they retained more dry impregnants (WPG) than beech veneers, presumably because of more porosive structure of poplar tissue.

While absorption (A) of both salt solutions (SA and MP) in poplar veneers was a approximate, absorption of SA solution was better than absorption of MP solution in beech veneers, which might be consequence of higher density (and lower porosity) of beech-wood, as well as of differences in chemical character of these two salts.

In addition, larger quantity of dry monoammonium phosphate (MP) retained in poplar veneers (i.e. larger WPG), opposite to beech veneers where larger quantity of dry sodium acetate retained.



Fig. 3. Absorption (A) and weight percent gain (WPG) of impregnant in veneers for poplar veneers (a) and for beech veneers (b)

Слика 3. Апсорпција (A) и масени процентуални принос (*WPG*) импрегнаната у тополовим (a) и буковим (b) фурнирима

3.2. Equilibrium moisture content (EMC) of veneers and plywood

The results of determination of equilibrium moisture content (EMC) of impregnated and nonimpregnated beech and poplar veneers, as well as plywood made from such veneers, are presented in Table 2.

 Table 2. Equilibrium moisture content (*EMC*) of impregnated and nonimpregnated beech and poplar veneers and plywood

 Table 2. Equilibrium moisture content (*EMC*)

Табела 2. Равнотежни садржај	влаге (ЕМС) импрегнисаних	и неимпрегнисаних	букових и
тополових фурнира			

	Equilibrium moisture content <i>EMC</i> [%] Равнотежни садржај влаге <i>EMC</i> [%]				
Impregnant Импрегнант	Beech Буква		Poplar Топола		
	veneer фурнир	plywood шперплоча	veneer фурнир	plywood шперплоча	
/	7.98	6.83	7.73	6.86	
SA	9.70	8.18	11.29	12.05	
MP	7.24	7.46	6.22	7.94	

Impregnation with *SA* raised EMC of both beech and poplar veneers, contrary to impregnation of these veneers with MP, which has to be objective of further research. On the other side, impregnation with mentioned salts raised *EMC* of both beech and poplar plywood unanimously and with *SA* creating higher *EMC* than *MP*. Simultaneously, this means higher hydroscopicity of impregnated plywood.

3.3. pH values

pH values of solution of fire retardant salts for impregnation, of impregnated and nonimpregnated veneers and of plywood are shown in Table 3.

Considering the acidic nature of *MP* and alkalic nature of *SA*, it was logical to expect the enlargement of acidity of veneers impregnated with *MP* solution and enlargement of alkalinity of veneers impregnated with *SA* solution.

Since cured urea-formaldehyde adhesive was acid catalyzed polymer system, *MP* impregnated superponed adhesive's acidity and still further decreased pH value of *MP* impregnated plywood. Alkalic *SA* impregnate on the other side, partly neutralized acidity of adhesive, increasing at the same time pH value of *SA* impregnated plywood. Thus, concentration of catalyst and/or pressing time and temperature must be adjusted, having in mind influence of different impregnants on pH of pressed plywood i.e. on curing time. Otherwise, precure or on the other side retard and postpone of curing might happened. Jovan Miljković, Ivana Grmuša, Milanka Điporović, Z.M. Kačarević-Popović

 Table 3. pH value of impregnant solutions, of impregnated and nonimpregnated veneers and of plywood

Табела 3. рН вредност раствора импрегнанта, импрегнисаних и неимпрегнисаних фурнира и шперплоча

		рН					
Impregnant Импрегнант	pH of 25% solution pH	Be Fy	ech ква	Poplar Топола			
	25% раствора	veneer фурнир	plywood шперплоча	veneer фурнир	plywood шперплоча		
/	/	6.47	4.7	5.79	4.85		
SA	9.45	8.37	5.81	8.51	6.09		
MP	4.09	5.61	4.23	5.52	3.67		

3.4. Shear strength of plywood

Both sodium acetate (SA) and monoammonium phosphate (MP) impregnants weakened adhesive bonding in the beech and in the poplar plywood (Table 4).

Decrease of shear strength of plywood made of impregnated veneers compared to reference plywood made of nonimpregnated veneers, was more noticeable with *SA* (about 7% for beech and 16% for poplar plywood) than with *MP* (about 3% for beech and 12% for poplar plywood). In addition, shear strength of *SA* impregnated poplar plywood was more than twice, and *MP* impregnated poplar plywood more than three times less comparing to corresponding *SA* or *MP* impregnated beech plywood.

Table 4. The influence of impregnant on glue joint shear strength of plywood

Табела 4. Утицај импрегнанта на затезну чврстоћу лепљене везе у шперплочи

Impregnant	Shear strength [N·mm ⁻²] Затезна чврстоћа [N·mm ⁻²]			
Импрегнант	Beech Буква	Poplar Топола		
/	3.87	2.55		
SA	3.58	2.14		
MP	3.74	2.24		

Nevertheless, shear strength of weakened impregnated plywood still exceeded minimal standard limit of $1.2 N \cdot mm^{-2}$ (JUS D.C5.041). Percentage of wood failure was over 60% with all tested specimens.

3.5. Thermogravimetric (TG) analyses

For the sake of better comparison, veneers with different immersion time but having approximate weight percent gain (*WPG*) were used for thermogravimetric (TG) and Differential thermogravimetric (DTG) analyses (Table 5).

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Since TG analyse follows change of mass of the sample with temperature increase, it gives valuable information on thermal stability and decomposition process of sample material. Momentary rate of change of the mass of sample, with increase of temperature (DTG) is commonly calculated (Blažek A., 1972).

Table 5. Immersion time and *WPG* of veneers for TG analyses **Табела 5.** Време потапања и *WPG* фурнира за TG анализе

	Веесһ Буква		Poplar Топола		
Impregnant Импрегнант	Immersion time Време потапања <i>WPG</i>		Immersion time Време потапања	WPG	
	h	%	h	%	
SA	2	4.5	4	14.7	
MP	1	4.3	1	14.8	

Fig. 4 and Fig. 5 show the TG and DTG curves for untreated beech and poplar veneers in the temperature range of $20-450^{\circ}C$, while Fig. 6, 7, 8 and 9 show the same curves for treated veneers. The results of TG and DTG analyses of pure impregnants, are shown in Fig. 10 and Fig. 11. It was observed that *TG* curves for untreated veneers first slightly fall due to evaporation of volatile materials, and than rapidly fall which indicates the beginning of pirolyses at the temperature of about $250^{\circ}C$. It was obvious that poplar is less stable at elevated temperature than beech, since the mass loss of untreated poplar was about 20% greater at $450^{\circ}C$ than that of the untreated beech. Also, the maximum rate of mass loss (DTG) for poplar was higher than that of the beech and performed at the lower temperature.

However, poplar and beech veneers treated with *SA* and *MP* impregnants showed better thermal stability than that of untreated veneers, as shown in Fig. 6, 7, 8 and 9.



Fig. 4. TG/DTG curves for the untreated beech Fig. 5. TG/DTG curves for the untreated poplar veneer veneer

Слика 4. ТG/DTG криве за нетретиране буко- Слика 5. ТG/DTG криве за нетретиране топове фурнире лове фурнире

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in MP solution in SA solution





Fig. 8. TG/DTG curves for poplar veneer treated Fig. 9. TG/DTG curves for poplar veneer treated in SA solution in MP solution

Слика 8. ТС/DTС криве за тополове фурнире Слика 9. ТС/DTС криве за тополове фурнире третиране у SA раствору третиране у *MF* раствору



Слика 10. TG/DTG криве за чист SA



Generally TG curves indicate that the initial temperature of thermal degradation of treated veneers in the both cases, were lower than that of the untreated veneers the weight loss was slower, but the residual mass at $450^{\circ}C$ was greater. The same effects with other impregnants were noticed before (Wang S-Y., Rao Y.C., 1999).

The rate of mass loss (DTG curves) of treated samples was lower than that of the untreated, and the maximum rate of mass loss occurred at the lower temperatures for treated samples. As a matter of fact, diminution of integral surface down the dW/dt curve and the increase of residual mass of the sample at $450^{\circ}C$, indicates protective and fire retardant efficiency of impregnant.

Factor of efficiency (Table 6) indicates better protection against thermal degradation of veneers impregnated with *MP* (compared to *SA*), and better effects of poplar compared to beech veneers. Thus, combination of poplar veneers impregnated with *MP* gave better results.

 Table 6. The characteristic values of TG i DTG analysis of impregnated and non impregnated veneers

Wood species Врста дрвета	Fire retardant Ватро-отпорно средство	Max. de- grad. rate Max. брзина деград. wt%·min ⁻¹	<i>t</i> ° at max. de- grad. rate <i>t</i> ° max. брзине деградације ° <i>C</i>	Loss/resi- due at t=450°С Губитак/ остатак на t=450°С %	Reduce of loss Смањење губитака %	Factor of effi- ciency Фактор ефикас.
Poplar Топола	nonimpregnated неимпрегнисано	9.6	329.6	89.5 / 10.5	0	1
	SA	7.4	282.9	74.8 / 25.2	16.4	2.4
	MP	7.1	267.4	66.6 / 33.4	25.6	3.2
Beech Буква	nonimpregnated неимпрегнисано	7.3	356.9	70.7 / 29.3	0	1
	SA	7.9	296.4	68.7 / 31.3	2.8	1.1
	MP	7.1	273.3	59.3 / 40.7	16.1	1.4

Табела 6. Карактеристичне вредности TG и DTG анализе импрегнисаних и неимпрегнисаних фурнира

3.6. Fire retardant properties of plywood

Fire retardant properties of plywood produced of treated and nontreated veneers and submitted to fire during 30 minutes according to standard (JUS D.T4.039) were used for final confirmation of cited analysis.

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Fig. 12. The appearance of plywood after burning test Слика 12. Изглед узорка шперплоче након теста горења

Plywood made of nonimpregnated veneers of booth wood species took fire easily and burned away rapidly within about 10 minutes. Plywood made of beech veneers impregnated with *SA* lasted longer but burned away too. The appearance of poplar and beech plywood made of treated veneers which showed fire resistant properties to the certain extent is shown in Fig. 12.

The best fire retardant properties showed combination of poplar veneers impregnated with monoammonium phosphate (MP), than beech veneer impregnated with the same impregnant, and after all combination of poplar veneers impregnated with sodium acetate (SA) impregnant. These results were in accordance with TG and DTG analyses.

4. CONCLUSION

- The impregnation of poplar and beech veneers by their immersion into 25% solution of monoammonium phosphate (*MP*) or sodium acetate (*SA*) under normal atmospheric condition showed that both, absorption of solutions (*A*) and weight percent gain of dry impregnants (*WPG*), were significantly higher in the poplar veneers. Simultaneously, MP showed significantly better *WPG* than *SA* in the poplar veneers, while *WPG* of *SA* in the beech veneers was just somewhat better than that of *MP*.
- Generally, impregnated veneers showed better fire resistance in comparison to nonimpregnated. It is interesting that fire resistance effect of used fire retardant salts (impregnants) was better with poplar, than with beech veneers, although the original beech wood was more resistant itself than the original poplar wood.
- *MP* as impregnant showed better fire retardant effect than *SA* in combinations with both wood species, which was approved by burning tests of corresponding

plywood. The best fire resistance was realised by combination of *MP* and poplar veneers.

- Previous thermogravimetric (TG) analysis of impregnated veneers can be useful tool in predicting fire retardant properties of plywood made of such veneers.
- The presence of *MP* in wood increased its acidity, while *SA* increased wood alkalinity. Adhesive curing might be affected during hot pressing of plywood, since impregnants changed original pH value of veneers. Thus, certain adjustments of press temperature, pressing time and/or addition of catalyst might be necessary.
- The both fire retardant impregnants used in this research, weakened mechanical strength of plywood. This was indicated by lower shear strength of adhesive joint in impregnated plywood in comparison with nonimpregnated one. Still, their shear strength was above standard demands.

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УТИЦАЈ УСПОРИВАЧА ГОРЕЊА НА СВОЈСТВА ФУРНИРА И СЛОЈЕВИТИХ ПЛОЧА ОД БУКОВОГ И ТОПОЛОВОГ ДРВЕТА

Резиме

Импрегнација буковог и тополовог фурнира потапањем у 25%-тни раствор моноамонијум-фосфата (*MP*) или натријум ацетата (*SA*), под нормалним атмосферским условима, показује да су апсорбција раствора (*A*) и масени принос импрегнаната значајно већи у фурнирима тополе. Принос *MP* је значајно већи у фурнирима тополе док је принос *SA* нешто већи у фурнирима букве. Уопште. импрегнисани фурнири су показали бољу ватроотпорност у поређењу са неимпрегнисаним. Боље ефекте су показале обе изабране соли примењене на тополи него на букви иако је дрво букве, незаштићено, знатно отпорније на ватру од тополе. *MP* је показао бољу ватроотпорност него *SA* у комбинацији са обе врсте дрвета, а посебно са тополом.

Термогравиметриске анализе (TG) импрегнисаних фурнира су се показале као веома корисне у предвиђању ватроотпорности фурнирских плоча.

Присуство *MP* у дрвету повећава његову киселост, док *SA* смањује базност дрвета. Пошто импрегнанти мењају pH вредност фурнира, то може утицати на очвршћавање везива током врелог пресовања фурнирске плоче, што условљава подешавање параметара пресовања.

Изабране соли умањују смицајну чврстоћу готових плоча, мада је она још увек у оквиру стандардних захтева.