

**IMPACT OF PF AND MUF ADHESIVES MODIFIED WITH
TiO₂ AND SiO₂ ON THE ADHESION STRENGTH**

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ABSTRACT

The purpose of this study was to evaluate adhesion strength of phenol formaldehyde (PF) and melamine urea formaldehyde (MUF) adhesives modified with nano-technological products on the adhesion strength of different wood species. For this purpose, the effect of nano-TiO₂ and nano-SiO₂ on bonding performance and structural properties of PF and MUF were researched. And also, TiO₂ and SiO₂ chemicals were chosen as a rate of 2%, 4%, 6%, 8% within the adhesives. The bonding strength tests of the acquired Uludag fir and aspen boards were measured with a Universal Zwick Roell brand testing device in accordance with TS EN 205 standards. The obtained results showed that the highest bonding strength for Uludag fir wood was 8.27 N·mm⁻² with PF adhesive mixed as 8% of SiO₂ and the lowest was 5.91 N·mm⁻² with MUF adhesive mixed as 2% TiO₂, respectively. For aspen wood, the highest value was determined as 7.32 N·mm⁻² with PF adhesive into which 8% of TiO₂ had been added and the lowest was as 5.55 N·mm⁻² with MUF adhesive into which % 6 TiO₂ had been added. In conclusion it was determined that compared to the control samples the bonding strength of wood materials manufactured with the addition of nanoparticle into the PF adhesive enhanced the bonding strength by approximately 30% and 40% within MUF adhesive.

KEYWORDS: Adhesives, strength, fir, aspen, nanotechnology.

INTRODUCTION

In the polymer composites, different types of filler are used for improving the thermal, mechanical, as well as other properties. Among them, clay is widely used as filler. The surface characteristics of nanopowders play a key role in their fundamental properties from phase transformation to reactivity. A dramatic increase in the interfacial area between fillers and polymer can significantly improve the properties of the polymer (Song 1996). Different types of metal oxide nanoparticles such as SiO₂, TiO₂, ZnO, etc., are widely used for these purposes. These are non-toxic, stable and highly thermostable inorganic filler (Deka and Maji 2013).

Nanoscience and nanotechnology are rapidly entering our lives in various areas. For composites, SiO₂ nanopowder is one of the widely used filler. SiO₂ can enhance the mechanical as well as thermal properties of the composite. Polymer-SiO₂ composites have been explored as technological importance due to their potential applications in electrochromic windows, fuel cells, chemical separation, electrochemical sensing, and water treatment (Wu et al. 2005). TiO₂ and SiO₂ nanoparticles extensively increase the tensile and impact strength of epoxy nanocomposite (Zheng, Ning 2003). In order to increase the hydrophobicity of the inorganic MUF and PF adhesives, the mixtures has been modified by different nanotechnological chemicals.

Nanotechnological chemicals, wood materials and adhesives can be used by combining with each other or other materials, laminating or blending with other polymers to gain the benefit of their various attributes.

The impact of nanoparticles on the physical, mechanical, combustion and formaldehyde emission characteristics of the acquired new materials have been determined with the study carried out for the strengthening of wooden sandwich panels and laminate flooring with nanoparticles. Urea formaldehyde and melamine urea formaldehyde adhesive were reinforced with nano-silicon dioxide SiO₂, nano-aluminum oxide Al₂O₃ and nano-zinc oxide ZnO within the scope of the study. With the use of nanoparticles the performance of wood sandwich panels and laminated parquets in various places will be enhanced, more appropriate products in terms of human health will be manufactured and thus these products will contribute usage in place of massive wood material (Candan 2012, Huang et al. 2012, Bello et al. 2014, Liu and Zhu 2014, Hemmilä et al. 2017).

A study of nanochemicals, it was stated that the addition of nano-SiO₂ may improve the age resistant properties of starch and polyvinyl alcohol films (Yao et al 2011). When nano-SiO₂ is added in percentages from 0.75% to 1%, the interfacial shear strength ultrahigh - molecular weight polyethylene fibers increases from 1.29 to 1.83 MPa (Zhang et al. 2010). In other studies, it has been found that added nano-SiO₂ particles can improve the mechanical properties and water resistance of starch and polyvinyl alcohol blend films (Xiong et al. 2008, Tang et al. 2009).

As discussed above, there may be a stronged relationship between the quality of these kinds of composites and the mechanical properties of the cell wall in the bond interface for PF and MUF adhesives gluing wood. The aim of this study was determine the adhesion strength of PF and MUF adhesives modified with nanotechnological products, TiO₂ and SiO₂ 2% - 4% - 6% - 8% for Uludağ fir and Quaking aspen. Thus the possibilities for the use of nanotechnological products in the wood materials were tested.

MATERIALS AND METHODS

Uludağ fir (*Abies bornmülleriana* Mattf.) and Quaking aspen (*Populu tremula*) woods used in this study were obtained from the Çatalzeytinve Devrekani regions of Kastamonu province.

Adhesives

The phenol formaldehyde (PF: Polifen 47) and melamine-urea formaldehyde (MUF: P03) adhesives to be used in the manufacturing of the test panels were acquired from Polisan Co in 25 kg drums. The PF adhesive together with the appropriate hardener was defined by the manufacturing company as adhesive used in external facades and the manufacturing of laminated plywood. The MUF adhesive was defined by the manufacturing company as adhesive used in the manufacturing of water resistant E1 plywood used for the furniture and wood industry. PF adhesive is dark brown in color with a solid material rate of $47\% \text{ g}\cdot\text{cm}^{-3}$ a formaldehyde/urea mole fraction of 0.15%. MUF adhesive has the appearance of a clean white liquid with a solid material rate of $56\% \text{ g}\cdot\text{cm}^{-3}$, formaldehyde/urea mole fraction of 1.15%. The density of both adhesives is between $1200\text{-}1220 \text{ g}\cdot\text{cm}^{-3}$ at 20°C .

Nanotechnological products

Nanotechnology products titanium dioxide TiO_2 and silicon dioxide SiO_2 are sold on the market in powder form as 25-30 kg packages. Out of these products Titanium dioxide is insoluble in water and organic solvents, hydrofluoric acid and slowly dissolves in hot concentrated sulfuric acid solvent. They are used as filler or pigment in thermosetting resin compositions. It is usually known as white pigment and it enhances the strength of hardened resins in terms of weather conditions. Their density is $3.5\text{-}4 \text{ g}\cdot\text{cm}^{-3}$ at 20°C and melting temperature is 1830°C . Silicon is one of the most abundantly found elements on earth and has semiconductor properties. The crystallized silicon, gray-brown in color is quite hard and has a metallic luster. After oxygen it is the most abundant element in the world. In nature it is generally formed with oxygen compounds and displayed in the form of silica or quartz known as silicon dioxide (SiO_2). Density at 20°C is $2.34 \text{ g}\cdot\text{cm}^{-3}$ at with a melting temperature of 420°C .

Preparation of the test samples

All of wood test samples, cut from sapwood were acclimatized at the temperature of $20\pm 2^\circ\text{C}$ and $65\pm 3\%$ relative moisture content until equilibrium moisture content was gotten by storing them in a climate room for 3 months. Totally 360 samples were produced. The test samples were cut in dimensions of 150 mm long \times 20 mm wide \times 5 mm thick samples with tangential, and approximately $200 \text{ g}\cdot\text{m}^{-2}$ of the adhesives were applied to the bonding surfaces of the samples. The pressure and the duration were 0.8 MPa and 25°C and 30 min. Preparing test samples are two-layered in accordance with the principles of Turkish Standards (TS EN 386:2006). MUF and PF adhesives were applied to the test samples at the temperature of $20\pm 2^\circ\text{C}$.

The surfaces of the massive panels obtained from the fir and aspen woods were smoothed and readied for the gluing procedure at the wood work workshop of Karabük University Faculty of Technology. The adhesiving procedure was carried out by the application of PF and MUF adhesive respectively into which nano-technological products SiO_2 and TiO_2 were added at the rate of 2%, 4%, 6%, 8% and mixed with a hand blender for 5 minutes in $200 \text{ g}\cdot\text{cm}^{-2}$. After mixing the adhesive and nano-technology product the mixture was removed from the mixture container with a syringe and applied to the test panels on a precision scale of 0.05%; the mixture was applied on only one panel with a brush.

After the adhesiving procedure was completed the pressing was launched. The pressing procedure was carried out with the electrically heated hydraulic pressure press machine.

Duration of pressure for PF adhesive was 10 min, for MUF adhesive was 15 min and pressure was 100 bar, press temperature was 150°C for both adhesives.

Bonding strength test

The bonding strengths of the test specimens manufactured in this study were measured with related Turkish Standards (TS EN 205:2017) using a Zwick Roell universal testing device.

A total of 10 measurements from each sample was taken to calculate average bonding strength of each adhesive applied to the surface of samples.

Statistical analysis

The data obtained with the bonding strength tests was analyzed with SPSS analysis program. The impact on the bonding strength of the PF and MUF adhesives modified at certain rates by mixing them with nanotechnological products obtained from fir (*Abies nordmanniana*) and aspen (*Populus tremula*) woods were manifested with a multi-purpose ANOVA analysis. A DUNCAN test was carried out to determine the differences between the factors and it was determined with a 0.95 reliability whether or not the differences were statistically significant.

RESULTS AND DISCUSSION

The average values for bonding strength are given in Tab. 1.

Tab. 1: Bonding strength values of the test samples ($N\cdot mm^{-2}$).

Adhesive	Woods		Pure	SiO ₂	SiO ₂	SiO ₂	SiO ₂	TiO ₂	TiO ₂	TiO ₂	TiO ₂
			%	2	4	6	8	2	4	6	8
			($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)	($N\cdot mm^{-2}$)
FF	Fir	X _{max.}	6.89	7.08	7.62	7.65	9.06	6.87	7.64	8.36	8.88
		X _{min.}	5.40	5.71	6.49	6.07	7.46	4.95	5.60	5.54	6.52
		X _{main.}	5.97	6.40	7.02	6.93	8.27	6.19	6.31	6.72	7.41
		std	.44	0.52	0.39	0.41	0.52	0.59	0.60	0.77	0.70
	Aspen	X _{max.}	6.54	6.55	6.61	6.77	7.41	7.94	7.25	7.17	8.08
		X _{min.}	5.33	5.73	6.28	6.00	5.64	5.51	5.85	5.75	6.25
		X _{main.}	6.02	6.08	6.51	6.34	6.46	6.94	6.42	6.38	7.32
		std	0.49	0.26	0.27	0.28	0.67	0.70	0.40	0.40	0.60
MUF	Fir	X _{max.}	7.05	6.72	6.86	7.52	7.59	6.77	6.84	6.46	6.55
		X _{min.}	4.51	4.87	4.78	5.38	6.71	4.98	5.49	5.44	5.60
		X _{main.}	5.75	6.03	6.01	6.36	7.23	5.91	6.12	5.97	6.07
		std	0.88	0.69	0.64	0.89	0.25	0.62	0.46	0.32	0.36
	Aspen	X _{max.}	6.21	7.74	7.26	6.52	6.85	6.53	6.65	6.27	6.86
		X _{min.}	4.27	5.62	5.19	5.48	4.77	5.16	5.40	5.11	5.67
		X _{main.}	5.34	6.81	6.51	5.90	5.66	5.64	5.82	5.55	6.20
		std	0.57	0.78	0.65	0.35	0.78	0.38	0.39	0.40	0.41

According to Tab. 1, with increasing of nanotechnological chemical, in the first sample of SiO₂ the shear strength increased, however TiO₂ had the negative effect on the bonding strength of the composites. When the chemical percent have arrived at 8%, the dry shear strengths of fir is the highest. In addition, shear strength of fir with 8% percent nanotechnological chemical was 8.27 $N\cdot m^{-2}$.

As observed in Tab. 1 when the average data regarding the bonding strength values are evaluated it has been determined that the wood material acquired from different wood species and the use of adhesives modified to a specific rate with nanotechnological products has a positive impact on bonding strength. The results of the variance analysis test carried out to determine the impact of dependent variables on the bonding strength of the generated wood materials are indicated in Tab. 2.

Tab. 2: Results of multiple variance analysis.

Sources	Sum of squares	Deg. of freedom.	Mean square	F Value	Significance $\alpha \leq 0.05$
Corrected Model	125.98	35	3.6	11.818	0.00
Intercept	12506.8	1	12506.77	41060.39	0.00
Adhesive type	26.616	1	26.616	87.38	0.00
Wood species	5.15	1	5.15	16.90	0.00
Nano product	3.943	1	3.943	12.944	0.00
Nano percentage	17.84	3	5.947	19.52	0.00
Types of adhesive * wood	0.003	1	0.003	0.01	0.92
Adhesive type * Nano product	2.65	1	2.65	8.7	0.003
Adhesive type * Nano percentage	6.7	3	2.233	7.332	0.00
Wood type * Nano product	3.925	1	3.925	12.886	0.00
Wood type * Nano percentage	13.129	3	4.376	14.367	0.00
Nano product * Nano percentage	0.475	3	0.158	0.52	0.669
Adhesive type * Wood type * Nano product	4.446	1	4.446	14.59	0.00
Adhesive type * Wood type * Nano percentage	0.275	3	0.092	0.301	0.825
Adhesive type * Nano product * Nano percentage	3.02	3	1.007	3.305	0.021
Wood type * Nano product * Nano percentage	10.776	3	3.592	11.79	0.00
Adhesive type * Wood type * Nano product * Nano percentage	3.796	3	1.265	4.154	0.007
Error	98.689	324	0.305		
Total	14737.221	360			
Corrected Total	224.67	359			

As seen in Tab. 2 there are significant differences between the bonding strength data of the separate dependent variables as well as the interaction among themselves. According to the results of the executed DUNCAN test (see Tab. 4) there are significant differences between wood species, adhesive, nanoproduct types as well as the percentage utilization rate combinations with an error possibility of $\alpha \leq 0.05$.

According to the Tab. 3 which was carried out it has been determined that the use of nanotechnological products and the percentages that they are included in adhesives has a significant statistical impact on bonding strength value.

Tab. 3: Duncan test regarding the use of nanotechnological products and the merging rate with adhesive on bonding strength.

Factor	Groups	n	Mean	Std.Dev.	F	t	p
Glue type	PF	180	6.65	0.76		7.743	<0.001
	MUF	180	6.05	0.71			
Tree species	Fir	180	6.48	0.85		3.226	0.001
	Aspen	180	6.22	0.70			
Nano product	Pure	40	5.77	0.65	16.55		<0.001
	SiO ₂	160	6.53	0.81			
	TiO ₂	160	6.30	0.73			
Nano percentage	Pure	40	5.77	0.65	15.26		<0.001
	2%	80	6.25	0.70			
	4%	80	6.34	0.59			
	6%	80	6.27	0.65			
	8%	80	6.82	0.98			
Nano product& percentage	Pure	40	5.77	0.65	8.757		<0.001
	SiO ₂ (2%)	40	6.33	0.65			
	SiO ₂ (4%)	40	6.51	0.61			
	SiO ₂ (6%)	40	6.38	0.64			
	SiO ₂ (8%)	40	6.90	1.13			
	TiO ₂ (2%)	40	6.17	0.75			
	TiO ₂ (4%)	40	6.17	0.51			
	TiO ₂ (6%)	40	6.16	0.66			
	TiO ₂ (8%)	40	6.75	0.81			

Accordingly, when the bonding strength values are compared with control samples the highest bonding strength values were observed in test plates manufactured with SiO₂. Furthermore, it was determined that the bonding strength increased with the increase of nanotechnological products used that there were no important differences in 2-4-6 percentage rates and that there was a distinct increase compared with control samples with an 8% purity. There is a statistically significant difference between the bonding strength of PF adhesive compared to MUF adhesive.

The bonding strength test results indicate that wood type, adhesive type and the percentage of nano products mixed with adhesive are important in terms of the bonding strength of composite wood materials. It has been noted that all three factors have an impact on bonding strength. Accordingly, the highest bonding strength with 8.27 N·mm⁻² was observed with Uludağ fir (*Abies bornmülleriana* Mattf.) wood which was modified with PF adhesive modified with 8% nano product SiO₂. These results are consistent with the literature. In literature, Candan (2012) stated that nano-silicon dioxide SiO₂ was reinforced with MUF adhesive. In addition to this, Zheng and Ning (2003) determined that TiO₂ and SiO₂ nanoparticles extensively increase the share strength of epoxy nanocomposite.

The results obtained in this present study are supported by a previous work by Candan and Akbulut (2015). It was stated that nano SiO₂, nano Al₂O₃, and nanoZnO were used to reinforce particleboard at loading level of 0%, 1%, and 3%. It was reported that modulus of rupture values of all the nanoreinforced composites were higher than those of the unreinforced composites, except than the composites reinforced with 3% nanoZnO.

The modulus of rupture values of the composites reinforced with nano SiO_2 or nano Al_2O_3 increased with increasing the nanomaterial loading level from 1% to 3% (Candan and Akbulut 2014, Liu and Zhu 2014).

The lowest value with $5.55 \text{ N}\cdot\text{mm}^{-2}$ was observed in Quaking aspen (*Populus tremula*) wood material bonded with MUF adhesive modified with 6% TiO_2 nano product. The results indicate that the bondline of bonding strength was affected via the percentage of nanotechnological products. Thus, when the increase in nano chemicals resulted in the highest bonding strength. In the same study, Celiker (2005) stated that with the addition of nano-molecules into products better mechanical properties, better resistance to gases can be achieved. Furthermore, Ghosal and Freeman (1994) determined that nano fillers make available thin bond lines on surface of material and therefore they can rise the bonding strength.

It has been determined that the use of nano product in PF adhesive improved the bonding strength of wood materials by approximately 40% compared to control samples and improved usage by 30% when used with MUF adhesive. In literature, Song (1996) revealed that an important increase in the interfacial area between fillers and polymer can significantly improve the properties of the polymer.

In literature, it was stated that nano clay were used to reinforce oriented strand board (OSB) at loading level of 0%, 1%, 3% and 5%. It was reported that the MOE values of the OSB boards were higher than those of the unreinforced ones. Also it was reported that the MOE properties of the OSB boards was improved when the nano clay content increased from 1 to 3% and 5%. In another study by Salari et al. (2013) investigated effect of nano- SiO_2 on some applied properties of oriented strand board (OSB) made from underutilized low quality Paulownia wood. It was stated that nano- SiO_2 were used to reinforce oriented strand board (OSB) at loading level of 0%, 1%, 3% and 5%. It was reported that with incorporation of nano- SiO_2 up to 3% into UF resin mechanical and physical properties of the resulting panels improved and formaldehyde emission decreased.

Fig. 1 indicates scanning electron microscopy (SEM) micrographs of glulam beams manufactured with PF adhesive and nanomaterials.

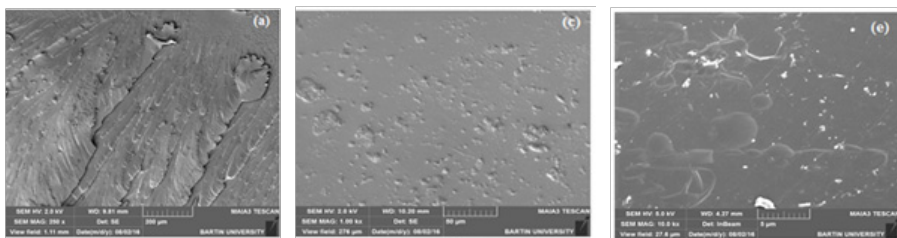


Fig. 1: SEM micrographs of glulam beams manufactured with PF adhesive and nano materials a) Control (b) %4 TiO_2 , (c) %4 SiO_2 .

The SEM micrographs show that the nanomaterials were found to be embedded in the adhesive. It was also determined that the nanomaterials used to reinforce the composites were well dispersed in the matrix. On the other hand, it was observed that the nanomaterials tended to accumulation when its loading level increased to 4%. Moreover nano- SiO_2 may influence the structure of woods' crystalline regions and fibrils, strengthening the interfacial interaction of the composites (Zhang et al. 2010).

While the wood species was not significant in terms of bonding strength the type of adhesive and wood species as well as the interaction of the adhesive type turned out to have statistical significance. The difference in terms of wood species turned out to be small regarding bonding strength yet it was deemed insignificant. This could be due to the fact that in terms of adherence fir and aspen woods display characteristic similarities and display close values.

This study concluded that the results for Uludağ fir (*Abies bornmülleriana* Mattf.) were superior to those of the Quaking aspen (*Populus tremula*) wood. In terms of adhesive type it was noted that PF adhesive gave better results compared to MUF adhesive and in terms of nanotechnological products the performance of SiO₂ was better compared to the performance of TiO₂.

CONCLUSIONS

The impact of modifying PF and MUF adhesives with a specific percentage of nanotechnological products to prepare wood materials from the wood of Uludağ fir (*Abies bornmülleriana* Mattf.) and Quaking aspen (*Populus tremula*) on bonding strength has been determined in this study. So, there was close relationship between the mechanical properties of the cell walls at the wood-adhesive interface and the percentage of nano chemicals in the PF or MUF. The bonding strength of wood or adhesives gradually increased when the content of these two type of nano chemicals was increased from 0% to 8%. Because of this, PF adhesive with SiO₂ is able to permeate into lumen to influence the mechanical properties of cell wall in the bond line. Mixing the adhesive and nanotechnological chemical succeeded the bonding strength.

These factors can affect the mechanical bond and adhesion strength between the wood surface and the adhesive. In respect to type of adhesive, percentage of nanotechnological chemicals, there is no significant difference between the woods and adhesives. However, when evaluating the adhesive and percentage of chemical factors, the %8 rate gave more bonding strength than the other percentages. As for the woods, the results indicated that fir wood with PF adhesive gave better results in the MUF and aspen wood. Moreover, Fir wood has a smoother structural surface and density than the aspen. Consequently, fir wood and PF adhesive strengthened with 8 SiO₂% could be advised for manufacturing of wood elements.

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