Effect of Alkyl Ketene Dimer on the Physical, Mechanical, and Biological Durability of Plywood

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The effect of alkyl ketene dimer (AKD) on plywood properties was studied. AKD is widely used in the paper industry as a sizing agent and can esterify wood cell wall components. Two types of veneers obtained from alder (*Alnus glutinosa* subsp. *barbata* (C. A. Mey) Yalt.) and beech (*Fagus orientalis* L.) wood logs were used. Two different treatment processes and two different concentrations (1% and 3%) of AKD were tested. The first method was AKD-dispersion, which was mixed with glue and sprayed onto veneers. The second method was dipping veneers into the AKD solutions (1% and 3%) for 25 min. Water uptake after 2 h, 24 h, and 48 h was reduced by the AKD treatment. Plywood produced from AKD impregnated veneers showed the lowest thickness swelling *versus* untreated plywood and plywood produced from AKD blended in glue. The AKD treatment generally reduced the mechanical properties of the plywood. However, AKD treatment considerably improved the biological resistance against brown rot fungi (*Coniophera puteana* BAM Ebw. 15) and white rot fungi (*Trametes versicolor* CTB 863A). Increased methyl/methylene and carbonyl groups of the alkyl chain were determined in the Fourier transform infrared (FTIR) spectra of specimens subjected to the AKD-dispersion method.

Keywords: Alkyl ketene dimer; Mechanical properties; FTIR; Decay resistance

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INTRODUCTION

Plywood is made of wood veneers bonded in an odd number of layers and is one of the most used wooden products in the construction and furniture industries (Ferreira *et al.* 2017). Due to its structural properties, plywood has higher bending strength and screw holding capacity, and better physical properties than those of solid wood (Demir *et al.* 2017). The demand and production for plywood are increasing. However, some properties of plywood, including thermal properties, fire resistance, and dimensional stability, require improvement through modification processes (Cheng and Wang 2011). Poor dimensional stability and irreversible thickness swelling of wood-based panels restrict their use in crucial applications (Paul *et al.* 2006). These issues cause a weakened structure and decrease resistance against wood-destroying fungi. It is reported that different factors, such as board density, wood species, resin type and amount, pressing conditions, particle geometry, and hydrophobic agents affect the dimensional stability of the boards (Hundhausen *et al.* 2009). Paraffin wax emulsions of 0.3% to 1% based on oven-dry weight, are added into wood-based panels to improve moisture resistance. However, higher amounts of paraffin added into the boards decreases their bonding strength (Hundhausen *et al*. 2009).

Many efforts have been taken to improve the dimensional stability of wood-based panels (Filcock and Vinden 2000; Papadopoulos and Gkaraveli 2003; Hundhausen *et al.* 2009; Ferreira *et al*. 2017). For example, some researchers have reported that wood-based panels treated with isocyanate resin as a hydrophobic agent decreased the thickness swelling of wood-based composites (Filcock and Vinden 2000). Thermal treatment is an ecologically friendly alternative treatment method used to improve dimensional stability. During the thermal treatment process, dimensional stability increases due to the reduction of hydroxyl groups in the hemicellulose, the most hydrophilic polymers of wood (Hill 2006; Ferreira *et al*. 2017).

Another approach to improve dimensional stability of wood-based composites is chemical modification. Several researchers reported that chemical modification of wood-based panels caused an alteration of cell wall structure and led to improvements in dimensional stability (Hill 2006; Hundhausen *et al.* 2009; Mai 2016). Numerous chemicals were tested on the chemical modifications of wood-based panels such as phenol-formaldehyde resin (Kajita and Imamura 1991), acetylated chips (Rowell *et al.* 1986; Youngquist and Rowell 1986; Subiyanto *et al.* 1989), and particleboards made from chips modified with propionic anhydride (Papadopoulos and Gkaraveli 2003). These reports showed some improvements on the dimensional stability of wood-based panels through chemical modification. However, chemical modifications in such compounds are generally expensive and difficult to apply in the board production process.

Alkyl ketene dimer (AKD) is widely used in the paper industry as a sizing agent to build resistance against fluids, which increases the paper’s wet strength and printability (Lindström and Söderberg 1986; Quillin *et al.* 1992; Neimo *et al.* 1999). The hydrophobic effect of AKD is predominantly attributed to an esterification reaction with wood hydroxyl groups and subsequent orientation of hydrocarbon chains (Hundhausen *et al.* 2009; Mai 2016). Therefore, modification of wood and wood-based panels with AKD is expected to improve dimensional stability. While AKD has been extensively used in the paper industry, there are a few studies regarding AKD-treated wood and wood-based panels (Suttie *et al.* 1998; Hundhausen *et al.* 2009; Mai 2016).

In this study, researchers aimed to improve plywood properties with AKD, which can undergo an esterification reaction with wood hydroxyl groups and improve the hydrophobicity of plywood. Two different processes and two different concentrations (1% and 3%) of AKD were tested. In the first process, AKD-dispersion was mixed with glue and sprayed onto veneers prior to pressing. In the second process, veneers were dipped into AKD-dispersion. The hydrophobicity, mechanical properties, thermal properties, decay resistance, and FTIR spectra of plywood modified with AKD were determined.

EXPERIMENTAL

Materials

Veneer treatment and plywood production

Rotary cut veneers with dimensions of 400 mm × 400 mm × 2 mm and a moisture content of 7% were prepared from Alder (*Alnus glutinosa* subsp. *barbata* (C. A. Mey) Yalt.) and beech (*Fagus orientalis* L.) wood logs which are commonly used wood species for plywood manufacturing in Turkey.

The alkyl ketene dimer-dispersion with a solid content of 18% was obtained from SETAS Chemical Company/RD Center (Tekirdag City, Turkey).

Two different treatment processes and two different concentrations (1% and 3%) of AKD were tested. In the first process, the two different concentrations (1% and 3%) of AKD-dispersion were mixed with glue and sprayed onto veneers prior to pressing (Bürkle, City of Freudenstadt, Germany). In process two, two veneers for each group were dipped into two different concentrations (1% and 3%) of AKD-dispersion for 25 min. The average chemical retention was determined as shown in Eq. 1,

$$Retention = ((G \times C)/V) \times 10\ kg/m^3 \quad (1)$$

where G is the weight (g) of treating solution absorbed by veneer, C is the concentration of treating solution (%), and V is the volume of veneer (cm^3).

Three-ply laboratory plywood panels were manufactured and urea formaldehyde (UF)-Resin (solid content of 65%, Polisan, City of Kocaeli, Turkey) was used as the adhesive. The adhesive mixture used for gluing veneers was composed of 100 parts UF resin, 30 parts wheat flour and 10 parts hardener (NH_4Cl) by weight. Rotary cut veneers were glued with 160 g/m^2 of UF-resin. The plywood panels were assembled and hot-pressed under 1176.79 kPa at a temperature of 110 °C for 6 min. Two boards (400 mm × 400 mm × 6 mm) were manufactured for each group (totally 20 boards). Samples for thickness swelling (n = 30), decay test (n = 6), bonding shear strength (n = 20), bending strength, and modulus of elasticity values (n = 14) were cut from these panels.

Water uptake and thickness swelling of plywood

The samples with the dimensions of 10 mm × 20 mm × 6 mm were cyclically submerged in deionized water. The water was replaced with fresh water after 2 h, 24 h, and 48 h. After each cycle, the mass and thickness of the samples were determined. Experiments were conducted at room temperature. Water uptake and thickness swelling were calculated according to Eqs. 2 and 3, respectively, after each water replacement,

$$WA\ (\%) = ((W_2 - W_1) / W_1) \times 100 \quad (2)$$

$$TS\ (\%) = ((T_2 - T_1) / T_1) \times 100 \quad (3)$$

where W_1 and W_2 are the weight (g), of the wood specimens before and after the test, T_2 is the tangential dimension at any given time during water soaked condition (mm), and T_1 is the initial tangential dimensions of the specimen (mm).

Methods

Mechanical properties of plywood

Bonding shear strength, bending strength, and modulus of elasticity values of plywood panels were determined according to EN 314-1 (1993) and EN 310 (1993), respectively. Samples were conditioned to a constant weight at a relative humidity of 65% ± 5% and at a temperature of 20 °C ± 1 °C prior to mechanical testing. Bonding shear strength samples were tested after being soaked in water at 20 °C for 24 h.

Thermal conductivity

The Fox-314 Heat Flow Meter (Laser Comp., City of New Castle, USA) was used to determine thermal conductivity according to ASTM C518 (2017) standard.

Thermal conductivity (λ) can be defined as a measurement of heat conduction ability of a material. It is stated as heat flow and expressed as Watts per meter (W/mK) thicknesses of material for a temperature gradient of 1 °K (Kelvin). Thermal conductivity is calculated by Eq. 4,

$$\lambda = Q.e / A.z.\Delta t \ (W/mK) \tag{4}$$

where λ is thermal conductivity (W/mK), Q is heat quantity (kcal), e is the thickness of material (m), Δt is the temperature difference between T_1 and T_2 (°C), A is the surface area (m^2), and z is time of duration (h).

FTIR Spectroscopy

The FTIR spectra were determined using a Bruker Tensor 37 Spectrophotometer (Bruker, City of Billerica, USA) in the range of 3600 cm^{-1} to 400 cm^{-1}, with a 4 cm^{-1} resolution over 16 scans, using Diamond ATR (Attenuated Total Reflectance).

Decay test

A mini-block test was conducted to decay the resistance of plywood in plastic sterile petri dishes with a 90-mm diameter. Samples (10 mm × 25 mm × 6 mm) were tested against brown rot fungi (*Coniophera puteana* BAM Ebw. 15) and white rot fungi (*Trametes versicolor* CTB 863A).

The samples were leached according to the AWPA E11-06 (2006) standard before the decay test. The water was replaced with fresh water after 6 h, 24 h, and 48 h and thereafter at 48-h intervals for a total of 14 days of leach testing. Fungi cultures were obtained from the Forest Products Laboratory, Madison, WI, USA. Malt extract agar of 4% (w/v) concentration and samples were sterilized in an autoclave at a pressure of approximately 0.1 MPa at 120 °C for 25 min. Each plate contained one treated test sample and one untreated control sample. Six replicates for each treatment were used. The incubation time was 8 weeks at 25 °C ± 1 °C and 65% ± 5% relative humidity (RH). After incubation, fungal mycelium were removed from the samples and dried at 103 °C ± 2 °C. The mass loss caused by the fungus was calculated according to Eq. 5,

$$\textit{Mass loss}\ (\%) = [(w_o - w_d) / w_o] \times 100 \tag{5}$$

where w_o is the oven-dry mass prior to testing (g) and w_d is the oven-dry mass after testing (g).

RESULTS AND DISCUSSION

The average retentions of 1% and 3% of AKD in beech veneers were 2.07 kg/m^3 ± 0.32 kg/m^3 and 6.15 kg/m^3 ± 0.59 kg/m^3, respectively, while 1% and 3% of AKD in alder veneers were 3.77 kg/m^3 ± 0.28 kg/m^3 and 9.80 kg/m^3 ± 0.91 kg/m^3, respectively.

Water Uptake and Thickness Swelling

The water absorption and thickness swelling results of the treated and untreated wood samples are listed in Table 1.

Table 1. Mean Values of Thickness Swelling and Water Absorption of AKD-treated Plywood

Alder Plywood						
	Immersion Time (h)	Control	AKD Glue (1%)	AKD Glue (3%)	AKD Imp (1%)	AKD Imp (3%)
Thickness Swelling (%)	2 h	5.55 (1.07)*	4.64 (1.73)	3.34 (0.76)	2.39 (1.47)	2.91 (1.77)
	24 h	6.42 (1.22)	5.97 (1.67)	7.50 (1.05)	6.21 (1.76)	4.92 (1.40)
	48 h	6.58 (1.24)	5.99 (1.54)	7.68 (1.05)	6.64 (1.72)	5.41 (1.32)
Water Absorption (%)	2 h	53.57 (3.35)	51.76 (4.33)	53.22 (4.02)	26.05 (2.78)	18.01 (1.33)
	24 h	77.57 (1.60)	72.01 (3.72)	73.64 (3.61)	62.78 (2.55)	50.84 (1.97)
	48 h	89.69 (2.81)	77.67 (3.11)	83.13 (3.22)	75.31 (1.87)	68.56 (2.80)
Beech Plywood						
Thickness Swelling (%)	2 h	5.43 (2.90)	5.14 (1.77)	7.78 (1.74)	2.23 (1.15)	1.04 (0.87)
	24 h	8.45 (3.62)	6.52 (1.90)	9.82 (1.46)	6.23 (1.56)	2.71 (1.33)
	48 h	8.79 (3.55)	6.58 (1.66)	10.16 (1.49)	6.86 (1.72)	3.08 (1.24)
Water Absorption (%)	2 h	34.52 (2.56)	35.49 (1.77)	30.55 (1.41)	15.99 (1.23)	15.89 (0.56)
	24 h	52.03 (1.64)	49.54 (0.87)	47.37 (1.00)	34.98 (3.12)	37.58 (1.53)
	48 h	54.97 (1.90)	53.69 (0.95)	52.10 (0.99)	44.16 (2.87)	49.46 (2.09)

* Numbers in parenthesis are standard deviation; AKD glue = blended in glue; AKD imp = veneer impregnated

As shown in Table 1, control plywood panels (made of untreated veneers) showed the highest water uptake for all immersion times. Alder plywood panels had higher water uptake than beech plywood panels. This was mainly attributed to the higher density of beech plywood panels.

It should also be noted that the addition of AKD reduced water absorption. In comparison, plywood produced from veneer impregnated with AKD showed lower water uptake than that of plywood produced from AKD blended in glue.

Regarding the thickness swelling, plywood produced from 3% of AKD blended in glue generally caused higher thickness swelling than the control groups for both alder and beech plywood groups. At the same time, the plywood produced from AKD-impregnated veneer showed lower thickness swelling than both the untreated plywood and plywood produced from AKD blended in glue.

The reason for the different effects of AKD blended in glue and veneer impregnated might be a physical blocking by UF-resin, which impedes the spreading of AKD on the veneer surface during gluing.

Some researchers reported that the combination of an esterification reaction and a subsequent orientation of AKD plays a critical role for sizing effect (Neimo *et al.* 1999; Hubbe 2006; Hundhausen *et al.* 2009). β-keto esters binding is the primary mechanism between cellulose and AKD. The β-keto ester bonding occurs between the four-membered strained lactone ring in AKD and cellulose hydroxyl groups (Zhang 2014).

The hydrophobicity of AKD is strongly related to an orientation of alkyl chains regardless of whether an esterification reaction occurs or not (Hundhausen *et al*. 2009). This might emphasize that the pressing time and temperature (6 min and 110 °C) are not enough for an orientation.

Mechanical Properties

Table 2 shows the mechanical properties of plywood samples treated with AKD.

Table 2. Mean Values of Mechanical Properties of AKD-treated Plywood

	Control	AKD Glue (1%)	AKD Glue (3%)	AKD Imp (1%)	AKD Imp (3%)
Alder Plywood					
Bonding Shear Strength (N/mm²)	2.08 (0.19) *	1.89 (0.36)	2.07 (0.35)	1.30 (0.16)	1.69 (0.30)
Bending Strength (N/mm²)	63.46 (2.64)	58.19 (6.30)	62.83 (5.47)	58.46 (6.20)	57.75 (4.84)
Modulus of Elasticity (N/mm²)	5347.31 (230.99)	4785.19 (252.73)	5233.33 (389.87)	4463.20 (833.69)	5146.13 (263.21)
Beech Plywood					
Bonding Shear Strength (N/mm²)	2.06 (0.16)	2.30 (0.18)	2.47 (0.28)	1.96 (0.18)	1.87 (0.26)
Bending Strength (N/mm²)	104.43 (7.71)	107.44 (8.73)	103.01 (7.27)	100.59 (6.50)	91.70 (4.11)
Modulus of Elasticity (N/mm²)	8624.70 (389.98)	8259.13 (776.41)	8429.69 (508.06)	9113.76 (435.86)	6869.81 (527.37)

* Numbers in parenthesis are standard deviation; AKD glue = blended in glue; AKD imp = veneer impregnated

In comparison to the control (untreated) samples, the veneers impregnated with AKD (AKD imp) exhibited lower mechanical properties than the treatment groups with the addition of AKD in glue (AKD glue). This result could be explained by the destruction of hydrogen bonds in the composite membrane by the β-keto esters. This result was consistent with results in a study of the preparation and characterization of AKD-modified cellulose composite membrane (Song *et al.* 2012).

In beech plywood panels with AKD glue, the mechanical properties were slightly improved over the control samples. It is thought that the reaction between AKD and cellulose fibers helps internal bonding. Therefore, positive results were observed in the bonding strength of the manufactured panels.

FTIR Spectroscopy

The FTIR spectra of beech and alder wood modified with AKD are presented in Figs. 1 and 2, respectively.

In AKD treatment, important modifications were seen on the IR spectra between 2800 cm^{-1} and 3000 cm^{-1}, which represents the methyl and methylene groups of the alkyl chain in the AKD-molecule and 1700 cm^{-1} and 1750 cm^{-1} that indicate carbonyl groups originating from both AKD and wood compounds as reported in previous literature (Hergert 1971; Hundhausen *et al.* 2009).

The FTIR results showed that beech and alder wood veneer impregnation with AKD did not display any differences on the IR spectra. However, the peaks at 2800 cm^{-1} to 3000 cm^{-1} and 1700 cm^{-1} to 1750 cm^{-1} increased in the AKD-dispersions, mixed with glue and sprayed onto veneers.

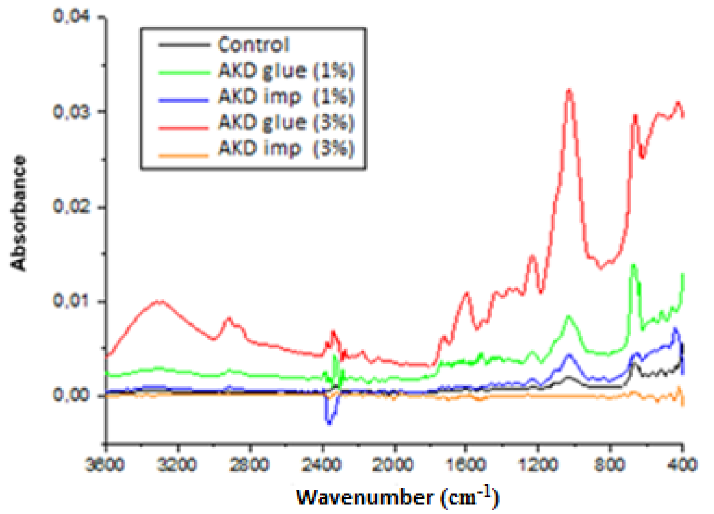

Fig. 1. FTIR spectra of beech wood

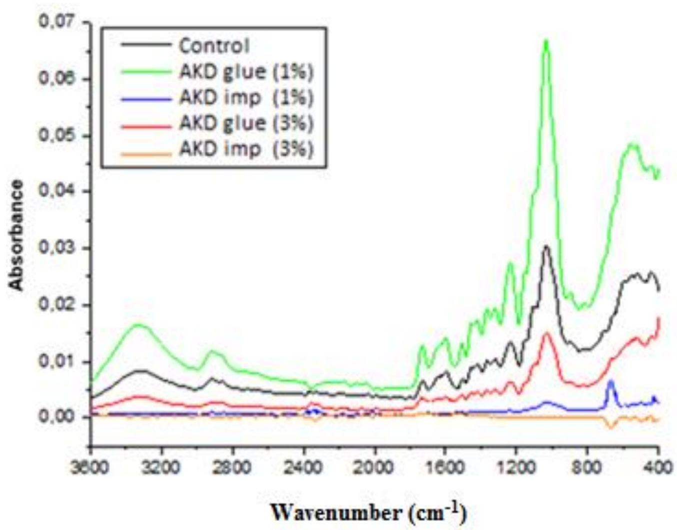

Fig. 2. FTIR spectra of alder wood

Decay Test

The samples were leached according to AWPA E11-06 (2006) for 2 weeks before the decay test. The weight loss of the leached wood samples against the test brown and white rot fungi are shown in Table 3.

Table 3. Weight Loss of Samples After Decay Test

C. puteana					
	Control	AKD Glue (1%)	AKD Glue (3%)	AKD Imp (1%)	AKD Imp (3%)
Alder plywood	31.70 (8.03)*	6.09 (3.92)	1.27 (0.93)	5.33 (2.43)	2.75 (1.37)
Beech plywood	23.80 (9.79)	4.09 (1.46)	5.68 (1.14)	5.40 (2.32)	4.10 (0.72)
T. versicolor					
Alder plywood	38.48 (2.66)	9.09 (0.77)	1.27 (0.93)	5.33 (2.43)	2.75 (1.37)
Beech plywood	27.25 (6.56)	0.71 (1.29)	2.58 (1.60)	1.38 (1.20)	1.41 (0.78)
* Numbers in parenthesis are standard deviation					

All treated samples had significantly less weight loss than the untreated samples. The mass loss of the control samples was higher than 20%, which confirmed the validity of the test.

The weight loss in the samples impregnated with AKD was slightly lower than that of the samples with AKD glue when these two methods were compared.

Thermal Conductivity

The average values of the thermal conductivity of plywood are given Table 4.

Table 4. Average Thermal Conductivity Value (W/mK)

	Thermal Conductivity (W/mK)	
	Beech Plywood	**Alder Plywood**
Control	0.04998	0.06635
AKD imp (1%)	0.05013	0.06493
AKD imp (3%)	0.06329	0.06717
AKD glue (1%)	0.06262	0.06500
AKD glue (3%)	0.04999	0.06560

As shown in Table 4, the highest thermal conductivity values were determined from AKD imp (3%) beech and alder plywood. The lowest thermal conductivity values were obtained from both the control and AKD glue (3%) in beech plywood

In alder plywood, the control samples had a higher thermal conductivity than the test samples, except for AKD imp 3% (0.06717 w/mK) due to a higher density of alder.

The thermal conductivity of wood and wood composite varied based on the wood species, direction of wood fiber, ratio of early and late wood, thickness of composite materials, density, moisture content, resin type and additives, temperature and flow direction of heat, and impregnate materials and processes (Kamke and Zylkowoski 1989; Suleiman *et al.* 1999; Kol *et al.* 2008; Sonderegger and Niemz 2009).

CONCLUSIONS

In this study, the effect of alkyl ketene dimer (AKD) on water uptake, thickness swelling, mechanical properties, biological durability against wood destroying fungi, and thermal conductivity of plywood was studied. The main findings were as follows:

1. Alkyl ketene dimer (AKD) treatment reduced water uptake after 2 h, 24 h, and 48 h. Beech plywood panels showed lower water uptake than those of alder plywood panels.
2. AKD blended in glue generally resulted in higher thickness swelling than control panels due to possible a blocking effect by UF resin. However, AKD impregnation decreased the thickness swelling.
3. The AKD treatment noticeably improved the biological properties against brown rot fungi (*C. puteana* BAM Ebw. 15) and white rot fungi (*T. versicolor* CTB 863A), while it reduced mechanical properties.

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