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#### FOREWORD

This special issue of Journal of Anatolian Environmental and Animal Sciences contains extended versions of selected papers of the ORENKO 2018 – International Forest Products Congress held on September 26-29, 2018 in Trabzon, Turkey. The congress, organized by the Department of Forest Industry Engineering at Karadeniz Technical University, has received about 200 abstracts from all over the world including United States, Canada, Australia, Slovenia, Slovakia, Serbia, Romania, Poland, South Korea, Italy, Iraq, Iran, Czech Republic, China, Chile, Bulgaria, Sweden, Bangladesh and Turkey. After an initial review of the submitted abstracts, about 180 abstracts were accepted for both oral and poster presentation.

The purpose of this congress was to provide an up-to-date discussion in the field of forest products in general. ORENKO 2018 was focused on the theme "Outlining the Forefront Research in The Field of Wood Science and Engineering". The topics that covered in the congress include wood science, technology and engineering, wood and wood-based products, wood anatomy, wood raw materials, wood composites, wood-plastic composites, engineered wood products, wood drying, biomaterials, wood constructions, physico-mechanical properties of wood and wood-based materials, nanotechnology applications in wood science, nondestructive evaluation of wood, sustainable utilization of forest products, wood preservation, wood modification, wood biomass, wood-inhabiting insects and fungi, marine borers, recycle/reuse/disposal of wood and wood based materials, non-wood forest products, wood chemistry, adhesives and bioresins, formaldehyde and VOC emission from wood based panels, pulp and paper, advanced cellulosic products, fiber resources from non-woody plants, furniture design and manufacturing, wood coatings, wood finishing, archaeological wooden structures, industry 4.0 in forest products industry, forest products ergonomics, environmental and ecological issues in forest products and occupational health and safety in forest products industry.

We would like to thank to all person of the organizing committee who have dedicated their constant support and countless time to organize this congress. The ORENKO 2018 is a credit to a large group of people, and everyone should be proud of outcome. We would also like to take this opportunity to express our sincere gratitude and thanks to our congress sponsors once again. Without their supports, it would not have been possible for us to organize this congress successfully.

Finally, we would like to specially thank to Prof. Dr. Bülent VEREP, Editor in Chief of the journal and Co-Editors in Chiefs Prof. Dr. Fikri BALTA and Prof. Dr. Turan YÜKSEK for providing an opportunity to bring out this special issue which composed of some selected papers of the conference.

> Assoc. Prof. Dr. Engin Derya GEZER, Section Editor of the JAES Chair of the ORENKO 2018 Congress



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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Optimization of Veneer Drying Temperature for the Best Mechanical Properties of Plywood via Artificial Neural Network

Şükrü ÖZŞAHİN<sup>\*1</sup> Aydın DEMİR<sup>2</sup> İsmail AYDIN<sup>2</sup>

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**Abstract:** The drying of veneer is an essential part of the veneer-producing process to aid the gluing during the manufacture of the plywood and laminated veneer lumber. Determining the optimum veneer drying temperature without decreasing of mechanical properties is also very important from industrial viewpoint. Due to the high drying costs, increased temperatures are being used commonly in plywood industry to reduce the overall drying time and increase capacity. However, high drying temperatures can alter some physical, mechanical and chemical characteristics of wood and cause some drying-related defects. In this study, it was aimed to predict the optimum drying temperature for alder and scots pine veneers via artificial neural network modelling for optimum mechanical properties. Therefore, mechanical strength values of plywood panels manufactured from alder and scots pine veneers were dried at temperatures of 110, 130, 150, 170, 190 and 210°C. Shear strength, bending strength and modulus of elasticity of the plywood panels were experimentally determined according to EN 314-1 and EN 310 standards. Then, the mechanical strength values based on veneer drying temperatures are subjected to prediction by artificial neural network modelling. As a results of this study, the optimum drying temperature values were obtained as 165, 162 and 161°C in Scots pine plywood and 190, 195 and 196°C in alder plywood, for best shear strength, bending strength and modulus of elasticity of uses shear strength and modulus of elasticity values.

Keywords: Alder, artificial neural network, mechanical properties, scots pine, veneer dying temperature

# Kontrplakların En İyi Mekanik Özellikleri için, Yapay Sinir Ağları ile Kaplama Kurutma Sıcaklığının Optimizasyonu

Öz: Kaplama kurutma işlemi, kontrplak ve LVL üretimi esnasında, tutkallama işlemini kolaylaştıran, kaplama üretiminin önemli parçalarından biridir. Yüksek kurutma maliyetleri nedeniyle, genel kurutma süresini azaltmak ve kapasiteyi artırmak için kontrplak endüstrisinde yaygın olarak yüksek sıcaklıklar kullanılmaktadır. Ancak, yüksek kurutma sıcaklıkları, ahşabın bazı fiziksel, mekanik ve kimyasal özelliklerini etkilemektedir ve kurumaya bağlı olarak bazı kusurlara neden olmaktadır. Bu çalışmada, yapay sinir ağları yardımıyla kurulan modellerle, en yüksek mekanik özellikleri sağlayan optimum kurutma sıcaklıklarının, kızılağaç (*Alnus glutinosa* subsp. *barbata*) ve sarıçam (*Pinus slyvestris*) kaplamaları için tahmin edilmesi amaçlanmıştır. Bu nedenle, kontrplak üretiminden önce kaplamalar, 110, 130, 150, 170, 190 ve 210 °C sıcaklıklarda kurutulmuştur. Üretilen kontrplak levhaların çekme-makaslama direnci; TS EN 314-1, eğilme direnci ve elastikiyet modülü; TS EN 310 standartlarına göre belirlenmiştir. Daha sonra, mekanik direnç değerleri kaplama kurutma sıcaklıklarına bağlı olarak, yapay sinir ağları modellemesi ile tahmin edilmiştir. Çalışmanın sonucunda, en yüksek çekme-makaslama direnci, eğilme direnci ve elastikiyet modülü değerlerini veren en iyi kurutma sıcaklıklarında sırasıyla, 165, 162 ve 161 °C, kızılağaç kontrplaklarında ise, sırasıyla 190, 195 ve 196 °C sıcaklıklardan elde edilmiştir.

Anahtar sözcükler: Kaplama kurutma sıcaklığı, kızılağaç, mekanik özellikler, sarıçam, yapay sinir ağları.

#### INTRODUCTION

Plywood is a wood composite with good physical and mechanical properties and can be used for different applications such as the manufacturing of furniture, musical instruments, means of transportation, packings, sporting goods, as well as in constructions (Bekhta & Salca, 2018). Its production in the World and Europe in 2016 exceeded 159 and 8 million cubic meters, respectively (FAO, 2018). European Union (EU) imports of panels (mainly plywood) increased 9% to Euro 2.79 billion in 2017. This follows a 3% rise in 2016 and an 11% increase in 2015. Most of this gain was due to a rise in plywood imports from Russia and other Eastern European countries. The value of EU plywood imports from China and tropical countries was generally stable or declining in 2017 (Industry News & Markets, 2018).

Drying veneer sheets is an essential part of the veneer manufacturing process. Veneer drying often becomes a production bottleneck because of inefficient equipment and methods. The purpose of veneer drying is to reduce its moisture content to a range suitable for gluing (Aydin & Colakoglu, 2008). Drying process accounts for some 70 percent of the thermal energy consumed in plywood production and approximately 60 percent of the mill's total energy requirement. Although drying temperatures of between 90-160°C may be considered normal, increased temperatures are being used to reduce the overall drying time and increase capacity (FAO, 1990). Reductions in drying time and energy consumption offer the wood industries a great potential for economic benefit (Aydin, 2014). It was concluded that the high-temperature drying practice can save energy and drying time by 44 and 25 %, respectively, in comparison with the conventional temperature drying (Theppaya & Prasertsan, 2004). Also, the economic benefits of quick drying of veneers at high temperature, this process can be effective on some mechanical, physical and chemical properties of wood (Ozsahin & Aydin, 2014).

Determination of the optimum veneer drying temperature without loss of mechanical strength is also very important from industrial view point. For this aim, a lot of temperature values need to be tested to determine the optimum values that cause the loss of much time and energy and high costs. Therefore, it is important to find more economic methods providing desirable results concerning technological properties (Demirkir et al., 2013). Artificial neural networks (ANNs) have been widely used in the field of wood (Esteban et al., 2011). The neural network most commonly used is the multilayer perception, whose nature as a universal function approximation makes it a powerful tool for modelling complex relations between variables (Fernandez et al., 2012). ANNs are capable of processing information in a parallel distributed manner, learning complex cause-and-effect relationships between input and output data, dealing with nonlinear problems, generalizing from known tasks or examples to unknown tasks. ANNs are good for tasks involving incomplete data sets, fuzzy or incomplete information, and for highly complex and illdefined problems, where people usually decide on an intuitional basis. Moreover, they can be faster, cheaper and more adaptable than traditional methods (Ceylan, 2008; Ozsahin & Aydin, 2014).

This study is aimed to predict the optimum drying temperature for alder and Scots pine veneers via artificial neural network for best mechanical properties by using experimental data. The main purpose was to obtain the intermediate values not measured from the experimental study by artificial neural network modelling and to estimate the optimum temperature values for each wood species giving the highest mechanical strength values.

#### **MATERIAL and METHODS**

Scots pine (Pinus slyvestris) and alder logs (Alnus glutinosa subsp. barbata) were used in this study. The logs were obtained from Trabzon region. While the alder logs were peeled freshly, scots pine logs were steamed for 12-16 hours before veneer production. A rotary type peeler (Valette & Garreau - Vichy, France) with a maximum horizontal holding capacity of 800 mm was used for veneer manufacturing and rotary cut veneer sheets with dimensions of 500x500 mm by 2 mm were clipped. Vertical opening was 0.5 mm and horizontal opening was 85% of the veneer thickness in veneer manufacturing process. After rotary peeling, spruce and beech veneer sheets were divided into six groups. The veneers were oven-dried at 110°C, 130°C, 150°C, 170°C, 190°C and 210°C for 5-7% moisture content in a laboratory scale jet veneer dryer (manufactured by Hildebrand Holztechnik GmbH).

Three-ply-plywood panels with 6 mm thick were manufactured by using urea formaldehyde (UF) glue resin with 55% solid content. The formulations of adhesive mixture are given in Table 1. Two replicate panels were manufactured for all test groups. Approximately 160 g/m2 adhesive mixture was spread on single surfaces of veneers by using a four roller gluing machine. In the manufacturing of plywood panels, hot pressing time was 6 minutes, press pressure were 1.2 MPa for alder and 0.8 MPa for scots pine, and press temperature was 110°C.

 Table 1. Formulations of adhesive mixtures used for the manufacturing of plywood panels.

Ingredients of Adhesive	Parts by weight
UF resin (with 55% solid)	100
Wheat flour	30
Hardener (NH <sub>4</sub> Cl with 15 % concentration)	10

Shear strength test was carried out for plywood panels manufactured according to EN 314-1 (1998) standard. Thirty samples were used for the evaluation of plywood shear strength. Before the shear strength tests, samples obtained from the panels manufactured with UF glue were immersed for 24 hours in water at  $20\pm3$ °C. Bending strength and modulus of elasticity tests was carried out for plywood panels manufactured according to EN 310 (1993) standard. Twelve samples were used for the evaluation of plywood bending strength and modulus of elasticity.

ANN was used to predict the shear strength, bending strength and modulus of elasticity values in response to the other veneer drying temperatures not obtained from this experimental study. In this way, it was aimed to reveal the optimum veneer drying temperature for each wood species to obtain the highest shear strength, bending strength and modulus of elasticity. In the ANN modelling for the present work; wood species and drying temperatures were considered as the prime processing variables. The proposed ANN models was designed by software developed using the MATLAB Neural Network Toolbox. The data were obtained from the experimental study. To examine the effects of wood species and drying temperatures on shear strength, bending strength and modulus of elasticity; the experimental data were grouped into training data and test data. Among these data, 8 samples were selected for ANN training process, while the remaining 4 samples were used to verify the generalization capability of ANN. The data sets used in the training and prediction models are shown in Table 2. The shear strength, bending strength and modulus of elasticity results obtained experimentally also presented in Table 2.

The obtained predicted values as a result of the testing process were compared with the real (measured) values. The models providing the best prediction values with respect to the root mean-square error (RMSE) ratio, calculated with Eq. 1, the mean absolute percentage error (MAPE) ratio, calculated with Eq. 2 and coefficient of determination ( $R^2$ ) with Eq. 3 was chosen as the prediction models.

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left(t_i - td_i\right)^2}$$
(1)

$$MAPE = \frac{1}{N} \left( \sum_{i=1}^{N} \left[ \left| \frac{t_i - td_i}{t_i} \right| \right] \right) \times 100$$
(2)

$$R^{2} = 1 - \frac{\sum_{i=1}^{N} (t_{i} - td_{i})^{2}}{\sum_{i=1}^{N} (t_{i} - \bar{t})^{2}}$$
(3)

In Equations 1, 2 and 3,  $t_i$  is the actual output values,  $td_i$  is the neural network predicted values, and N is the number of objects.

Table 2. Training and testing data set and shear strength, bending strength and modulus of elasticity prediction models results.

				T	raining Data					
Wood Spacias	Draing Temperature (°C)	S	hear Strength (N/m	n <sup>2</sup> )	Ber	ding Strength (N/n	nm²)	Modu	lus of Elasticity (N	/mm <sup>2</sup> )
wood species	Drying reinperature (C)	Actual	Predicted	Error (%)	Actual	Predicted	Error (%)	Actual	Predicted	Error (%)
Scots pine	110	1.342	1.342	0.002	65.733	65.753	-0.031	4868.228	4870.060	-0.038
Scots pine	150	1.447	1.448	-0.015	87.611	87.586	0.029	5509.160	5508.783	0.007
Scots pine	170	1.719	1.719	0.002	88.231	88.228	0.004	5518.530	5518.553	0.000
Scots pine	210	1.006	1.006	-0.002	56.018	56.013	0.008	4618.743	4618.774	-0.001
Alder	130	1.212	1.212	0.042	63.057	63.328	-0.430	4360.011	4377.383	-0.398
Alder	150	1.404	1.403	0.041	65.413	65.063	0.536	4424.940	4406.832	0.409
Alder	190	1.888	1.888	-0.016	71.250	71.331	-0.114	4878.719	4879.752	-0.021
Alder	210	1.181	1.181	-0.012	58.839	58.835	0.006	3701.773	3702.047	-0.007
	MAPE		0.017			0.145			0.110	
	RMSE		0.000			0.160			8.905	
				Т	<b>Testing Data</b>					
	Dervie a Terrer anatura (%C)	S	hear Strength (N/m	n <sup>2</sup> )	Bending Strength (N/mm <sup>2</sup> )			Modulus of Elasticity (N/mm <sup>2</sup> )		
	Drying reinperature (C)	Actual	Predicted	Error (%)	Actual	Predicted	Error (%)	Actual	Predicted	Error (%)
Scots pine	130	1.391	1.343	3.460	77.073	77.058	0.019	5201.858	5164.226	0.723
Scots pine	190	1.252	1.137	9.177	62.548	63.003	-0.728	5081.412	5128.929	-0.935
Alder	110	1.257	1.290	-2.626	64.471	62.341	3.303	4336.573	4371.079	-0.796
Alder	170	1.426	1.485	-4.089	65.550	67.787	-3.413	4499.033	4520.922	-0.487
	MAPE		4.838			1.866			0.735	
	RMSE		0.071			1.561			36.551	

Figure 1 shows the ANN models containing one input layer, one hidden layers and one output layer. The selected ANN models represents the prediction models that produced the closest values to the measured values for the shear strength, bending strength and modulus of elasticity. The wood species and drying temperatures were used as the input variables, while the shear strength, bending strength and modulus of elasticity values was used as the output variable in the ANN models. The processing element numbers (neurons) of the hidden layers were 4, 3 and 3 for the models in Figure 1.

A feed forward and back propagation multilayer ANN was used for solving problems, and the network training and testing was carried out using the MATLAB software package. In this study, the hyperbolic tangent sigmoid function (tansig) and the linear transfer function (purelin) were used as the activation transfer functions, the levenberg marquardt algorithm (trainlm) was used as the training algorithm, the gradient descent with a momentum back propagation algorithm (traingdm) was used as the learning rule, and the mean square error (MSE) with Eq. 4 was used as the performance function.



Figure 1. The ANN architecture selected as the prediction models.

MSE = 
$$\frac{1}{N} \sum_{i=1}^{N} (t_i - td_i)^2$$
 (4)

Where,  $t_i$  is the actual output (targeted values),  $td_i$  is the neural network output (predicted values), and N is the total number of training patterns.

To ensure an equal contribution of each parameter in the models, the training and test were normalized (-1, 1 range) due to the use of the hyperbolic tangent sigmoid function in the models and network, which allowed the data to be translated into the original value, with a reverse normalizing process for the interpretation of the results. The normalization (scaling) operations were carried out by using Eq. 5.

$$X_{\text{norm}} = 2 \times \frac{X - X_{\text{min}}}{X_{\text{max}} - X_{\text{min}}} - 1$$
(5)

Where,  $X_{norm}$  is the normalized value of a variable X (real value of the variable), and  $X_{max}$  and  $X_{min}$  are the maximum and minimum values of X, respectively.

#### **RESULTS and DISCUSSION**

*Experimental Results:* The shear strength, bending strength and modulus of elasticity values of plywood panels manufactured with UF glue were presented in Table 2, based on veneer wood species and veneer drying temperatures.

As can be seen from Table 2, the highest shear strength, bending strength and modulus of elasticity values were obtained experimentally from 170°C for Scots pine plywood, while 190°C drying temperature yielded the best shear strength, bending strength and modulus of elasticity values both for alder plywood.

Artificial Neural Network Results: Changes of shear strength, bending strength and modulus of elasticity depending on the veneer drying temperature were modelled with obtained network parameters. The amount of error variation depending on iteration of the selected ANN was shown in Figure 2.

Figure 3 shows the relationship between the real values and calculated values obtained by the prediction models. The comparative plots of these values are given in Figure 4.



Figure 2. A plot of error variation depending on iteration of the ANNs.



Figure 3. The relationship between experimental results and ANN predicted results.



Figure 4. The comparison of the real and calculated values.

In order to assess the validity of the networks and their accuracy, it is often useful to perform regression analysis between the network response and the corresponding target. The regression curves of the output variables for the experiment and ANN data set are shown in Figure 3 (shear strength, R = 0.98539; bending strength, R = 0.99585; modulus of elasticity, R = 0.99912). As the correlation coefficients approach 1, prediction accuracy increases and indicates good agreement between the experimental results and the models predication. This value supports the applicability of using ANNs in the present study.

Comparisons of the results between the outcomes of ANN modelling and experimental values for the shear strength, bending strength and modulus of elasticity values are plotted in Figure 4. The results of graphic comparisons showed similarities between the experimental study and the ANN models and supported the reliability of the models.

The results indicate a consistent agreement between the outcomes of the ANN modelling and the experimental results. MAPE was used to evaluate the performance of the proposed ANN in the prediction technique. The maximum absolute percentage errors (MAPE) for shear strength, bending strength and modulus of elasticity were 0.017, 0.145 and 0.110 % for training and 4.838%, 1.866% and 0.735 %for testing, respectively. These levels of error are satisfactory for the shear strength, bending strength and modulus of elasticity. As seen from the results, the ANN approach has a sufficient accuracy rate for the prediction of shear strength, bending strength and modulus of elasticity values of plywood.

The intermediate values not obtained from the experimental study for shear strength, bending strength and modulus of elasticity were predicted from the designed ANN modelling. The shear strength, bending strength and modulus of elasticity values predicted by the ANN models for different drying temperature are shown in Figure 5, 6 and 7.



Figure 5. The change of shear strength with increasing drying temperature.



Figure 6. The change of bending strength with increasing drying temperature.



Figure 7. The change of modulus of elasticity with increasing drying temperature.

According to Figure 5, 6 and 7, the optimum drying temperature values yielded the highest shear strength, bending strength and modulus of elasticity were found as 165, 162 and 161°C for Scots pine and 190, 195 and 196°C for alder, respectively. Plywood manufacturers prefer veneer to be dried at the highest possible temperature for a short time, without having the veneer degraded by excessive heat (Shen, 1958).Drying temperatures between 90-160°C are considered normal in veneer drying process, but the use of higher drying temperatures are giving rise to a reduction in drying time and increased capacity (FAO, 1990). On the other hand, high drying temperatures cause changes in the physical, mechanical and chemical characteristics of wood because of surface inactivation (Lehtinen et al., 1997; Sernek, 2002). In addition, veneer drying temperature affects the veneer-water relationships. Concerning this issue, Aydin (2014) studied the effects of veneer drying at high temperature on equilibrium moisture content, and reported that high temperature veneer drying caused a significant decrease in equilibrium moisture content values of plywood produced from beech, spruce and alder veneers. The waterabsorbing capacity of veneer is reduced by increasing the drying temperature with a constant time, or by increasing the drying time at a constant temperature. The reduced waterabsorbing capacity of the veneer could be a contributing factor in producing inferior glue bonds because of reduction

in wettability of the veneer by the glue (Currier, 1958). It was also indicated in literature that, during a drying process, significant reduction in bonding ability occurs at the end of drying, when the evaporative cooling effect decreases and the wood surface temperature approaches that of the air in the dryer (Suchsland & Stevens, 1968). However, severe surface inactivation occurs at the drying temperature of 160°C and higher (Christiansen, 1990). According to the ANN models used in the present study, the decreases in mechanical strength values with increasing drying temperature has also been started from about 165-170°C for Scots pine and 195-200°C for alder (See Figure 5-7).

Christiansen (1990) stated that surface inactivation is more prevalent in some wood species than in others and the highest safe drying temperatures for avoiding inactivation in wood varies with species. For example, southern pine was indicated to be the most susceptible species for inactivation, followed by ponderosa pine, then douglas-fir, western white pine and larch. Therefore, it can be considered normal that the optimum veneer drying temperature values (presenting the highest mechanical strength values) were different for Scots pine and alder plywood panels in the present study. Ozsahin & Aydin (2014) determined that the optimum drying temperature values yielded the highest bonding strength were obtained as 169°C for urea formaldehyde and 125°C for phenol formaldehyde adhesive in beech plywood while 162°C for urea formaldehyde and 151°C for phenol formaldehyde in spruce plywood panels. Demirkir et al. (2013) found that optimum veneer drying temperature ranges giving the highest bonding strength values were determined as 154 -160°C for panels with phenol formaldehyde. In this study, it was determined as 160-165°C for Scots pine plywood produced with urea formaldehyde. In a study conducted by Lehtinen (1998) on spruce plywood, it was also concluded that bending strength increased by 6.3-12% when the veneer drying temperature was increased from 110°C to 180°C.

At ANN design, some experimental results were used for training and some others were used for testing (Table 2). On the other hand, some data values for the Scots pine samples dried at 190°C was not available in training set. However, the strength values for this temperature was available for alder samples (Table 2). It was stated in literature that, ANNs are capable of processing information in a parallel distributed manner, learning complex cause and effect relationships between input and output data, dealing with nonlinear problems, generalizing from known tasks or examples to unknown tasks. ANNs are good for tasks involving incomplete data sets, fuzzy or incomplete information, and for highly complex and ill-defined problems for which humans would usually decide on an intuitional basis. Moreover, they can be more adaptable than traditional methods and ANNs technology brings completely different concepts to computing (Ceylan, 2008). As a consequence, the knowledge of the neural network is spread overall the links in

network with their weight values. So, the lack of some data in a trained ANN does not significantly affect the network to produce accurate information.

#### CONCLUSIONS

Increased temperatures are being used in plywood industry to reduce the overall drying time and increase capacity because of the high cost of drying process. However, high drying temperatures can cause some drying related defects. In the manufacturing of plywood, veneers should be dried as quickly as possible with high temperatures without losing mechanical properties. The optimum veneer drying temperature gives the highest mechanical strength values of plywood can be different depending on the wood species from which the veneers manufactured. These types of experimental studies need much time and high testing cost. Therefore, ANN modelling method can be used as a predictive method to determine the appropriate drying conditions for best mechanical properties. In the present study, the optimum drying temperatures for Scots pine and alder veneers were predicted by ANN modelling for best mechanical properties by using experimental data. As a results, the optimum drying temperature values were obtained as 165, 162 and 161°C in Scots pine plywood and 190, 195 and 196°C in alder plywood, for best shear strength, bending strength and modulus of elasticity values, respectively. It can be concluded from this study that the ANN method is reasonable for the modelling (the optimization) of shear strength, bending strength and modulus of elasticity at various drying temperature without needing the experimental study again and again.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Determination of the Optimum Feed Rate and Spindle Speed Depending on the Surface Roughness of Some Wood Species Processed with CNC Machine

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**Abstract:** In modern furniture industry, CNC machines are widely used, especially when high quality of product and flexibility of manufacturing process are expected. Even though there are many advanced computer-aided manufacturing systems for furniture producers, it is difficult to set process parameters according to obtain desired material surface properties because wood is a natural polymeric material with a heterogeneous structure. Wood surface properties are affected both material and machining factors, such as wood species, anatomical characteristics, moisture content, grain direction, feed rate, spindle speed, cutting depth, and tool geometry. In this study, it was aimed to determine of the optimum feed rate and spindle speed depending on the surface roughness of some wood species processed with CNC machine. Spruce, chestnut, larch and iroko were used as wood species. Three spindle speed (10000, 14000 and 18000 rpm) and feed rate (5000, 7000 and 9000 mm/min) were determined for CNC processing. The surface roughness (Rz) of wood samples were determined according to DIN 4768 standard. As a result of the study, the lowest surface roughness values were found in 10000 rpm spindle speed and 5000 mm/min feed rate for spruce and chestnut wood and 18000 rpm spindle speed and 7000 mm/min feed rate for larch and iroko wood. The highest values in the all of wood species were obtain from 10000 rpm spindle speed and 9000 mm/min feed rate.

Keywords: CNC, feed rate, spindle speed, surface roughness.

# CNC Tezgâhında İşlenmiş Bazı Ağaç Türlerinin Yüzey Pürüzlülüğüne Bağlı Olarak Optimum Besleme Hızı ve Dönüş Hızının Belirlenmesi

Öz: Modern mobilya endüstrisinde, özellikle yüksek kaliteli ürünün ve esnek üretim sürecinin beklendiği durumlarda CNC makineleri yaygın olarak kullanılmaktadır. Mobilya üreticileri için birçok gelişmiş bilgisayar destekli üretim sistemi olmasına rağmen, ahşap malzemenin heterojen bir yapıya sahip doğal bir polimerik malzeme olması nedeniyle, istenen malzeme yüzey özelliklerini elde etmek için işlem parametrelerinin ayarlanması zordur. Ahşap yüzey özellikleri, ağaç türleri, anatomik özellikler, rutubet, lif yönü, besleme hızı, dönüş hızı, kesme derinliği ve takım geometrisi gibi malzeme ve işleme faktörlerini etkilenmektedir. Bu çalışmada, CNC tezgahında işlenen bazı ağaç türlerinin yüzey pürüzlülüğüne bağlı olarak optimum besleme hızı ve dönüş hızının belirlenmesi amaçlanmıştır. Ağaç türü olarak ladin, kestane, melez ve iroko kullanılmıştır. CNC işlemi için, üç farklı dönüş hızı (10000, 14000 ve 18000 dev/dk) ve üç farklı besleme hızı (5000, 7000 and 9000 mm/dk) belirlenmiştir. Örneklerin yüzey pürüzlülüğü (Rz), DIN 4768 standardına göre belirlenmiştir. En düşük yüzey pürüzlülüğü değerleri, ladin ve kestane için; 10000 dev/dk dönüş hızı ve 5000 mm/dk besleme hızında bulunnuştur. Tüm ağaç türü gruplarında en yüksek değerler ise, 10000 dev/dk dönüş hızı ve 9000 mm/dk besleme hızında nelde edilmiştir.

Anahtar sözcükler: Besleme hızı, CNC, dönüş hızı, yüzey pürüzlülüğü.

#### INTRODUCTION

Wood and wood based materials have been used as a construction material for years, mostly because of low cost, renewability, strength and low processing energy requirements. In recent years, the machining of wood products has acquired great importance, due to the short supply of wood and increasing environmental awareness among users and manufacturers (Sofuoglu, 2017).

Working with automatic mechanical equipment demands precise, accuracy, speed, consistency and flexibility. In this case it takes the help of embedded computer applications to do the job. One of the mechanical equipment combined with microcomputer that has been widely used is a CNC machine (Computer Numerical Controlled). CNC machines are used for mechanical work such as cutting, engraving, drilling and others. The computer technology used to control, parse and execute certain objects based on user command. In the manufacturing industry, the use of CNC machines greatly affects the increased production (Jayachandraiah et al., 2014; Ginting et al., 2017). CNC machines are used in various manufacturing applications such as steel machining, plastic cutting, etc. and are widely used in the woodworking industry. In the furniture industry, CNC machines perform drilling, milling, sanding and cutting operations (Koc et al., 2017). These machines provide high productivity increasing the efficiency up to 2.5 times and flexibility in production and integration to automation systems (Raja & Baskar, 2011).

The surface quality of solid wood and wood based panels is one of the most important properties characterizing the wood machining process and its conditions, manufacturing processes, such as finishing or adhesive strength properties. The surface roughness of wood can be affected by different factors, such as annual ring variation, wood density, cell structure, earlywood ratio and latewood ratio, and humidity (Zhong et al., 2013; Sofuoglu, 2017). CNC machining process is a significant stage as it affects surface coating performance such as adhesion strength of coating, minimization of waste and appearance of wood products (Cool & Hernandez, 2011; Tan et al., 2012; Ozdemir et al., 2015). Surface roughness is a crucial quality indicator of cutting and coating process (Koc et al., 2017). Nas et al. (2012) reported that the surface roughness had changed significantly depending on the parameters as a result of milling the material surfaces in CNC machinery. Wilkowski et al. (2011) utilized Taguchi method to investigate effective factors for CNC processing parameters of wood surface roughness of oak and ash. The cutting parameters such as feed rate and spindle speed were resulted as significant parameters on the surface roughness.

The main purposes of process monitoring and control are to optimize manufacturing speeds and to reduce production times. An effective process monitoring system should alert the operator and shut the machine down when critical conditions are about to be reached. In wood machining, the control process should manage key control variables such as feed rate or spindle speed, and adjust them to approach optimum conditions for the chosen machining objectives (Iskra & Tanaka, 2005). In this study, it was aimed to determine of the optimum feed rate and spindle speed depending on the surface roughness of some wood species processed with CNC machine.

#### **MATERIAL and METHODS**

In this study, spruce, chestnut, larch and iroko were used as wood species. Three spindle speed (10000, 14000 and 18000 rpm) and feed rate (5000, 7000 and 9000 mm/min) were determined for CNC processing. It is given description of test groups according to wood species in Table 1.

Tahle	1	Descrit	ntion	of	test	orom	ns
rable	1.	Desch	puon	or	lest	grou	ps

Wood	Spindle	Feed Rate	Wood	Spindle	Feed Rate
Species	Speed (rpm)	(mm/min)	Species	Speed (rpm)	(mm/min)
		5000			5000
	10000	7000		10000	7000
		9000			9000
		5000			5000
Spruce	14000	7000	Chestnut	14000	7000
		9000			9000
		5000			5000
	18000	7000		18000	7000
		9000			9000
		5000			5000
	10000	7000		10000	7000
		9000			9000
		5000			5000
Larch	14000	7000	Iroko	14000	7000
		9000			9000
		5000			5000
	18000	7000		18000	7000
		9000			9000

The wood samples were processed on Megatron 2128, 4 Axis CNC Milling Machine with 9 kW spindle power, a maximum spindle speed of 24.000 rpm. Working area of the machine is 2100x2800x150 (mm). 2 mm diameter conical knife were used for all of the groups. The CNC machine and processing of the wood samples are shown in Figure 1.



Figure 1. CNC milling machine and Processing of the wood samples.

A fine stylus-type profilometer, Mitutoyo Surftest SJ-301 Surface Roughness Tester was used for the surface roughness test (Figure 2). This device consists of a main unit and a pickup. The pickup has a skid-type diamond stylus with a radius of 5  $\mu$ m and a tip angle of 90°. Cut off length (kc) was 2,5 mm, and tracing length was 12,5 mm. Ten replicates of wood samples for each group were used for surface roughness measurement. The samples cut at the dimensions of 50×50×10 mm were used for each test group to evaluate their surface roughness. The Rz roughness parameter (mean of the 10-point height of irregularities) was used to evaluate surface roughness of the samples according to DIN 4768 (1990).



Figure 2. Mitutoyo Surfest SJ-301 surface roughness meter.

#### **RESULTS and DISCUSSION**

The surface roughness mean values (Rz) are given in Figure 3 according to wood species and CNC parameters. The groups that given the lowest roughness values are shown with red arrow.



Figure 3. The change of surface roughness mean values according to wood species and CNC parameters.

As can be seen that Figure 2, the lowest surface roughness values were found in 10000 rpm spindle speed and 5000 mm/min feed rate for spruce and chestnut wood. Many researchers reported that the effect of feed rate and spindle speed on surface roughness in wood and wood based materials machining, and the results demonstrated that the surface roughness decreases with increasing spindle speed and increases with the feed rate (Iskra & Tanaka, 2005; Sutcu, 2013; Sutcu & Karagoz, 2012). In this study, smoothest surfaces for spruce and chestnut were obtained from the lowest spindle speed and feed rate.

However, it was found in 18000 rpm spindle speed and 7000 mm/min feed rate for larch and iroko wood. Davim et al. (2009) studied CNC processing parameters of MDF such as spindle speed, cutting speed and feed rate. Higher spindle speed and higher feed rate performed smother surfaces where in the present study lower feed rate and higher spindle speed performed minimum surface roughness. Koc et al. (2017) determined that minimum surface roughness values of MDF, ayous and beech wood were found as spindle speed of 18,000 rpm and feed rate of 2 m/min in their study.

The highest values in the all of wood species were obtain from 10000 rpm spindle speed and 9000 mm/min feed rate. Sofuoglu (2017) found that the surface roughness, both Ra and Rz, increased with increasing feed rate, but decreased with increasing spindle speed.

#### CONCLUSIONS

CNC wood working machinery has been widely introduced in wood industries, especially in the furniture industry. CNC has been used for the grooving, milling, patterning of furniture material etc. This technology presents many advantages related to output, surface quality and provide greater improvements in productivity, and increase the quality of the machined part. In this study, the effect of various CNC machine parameters such as spindle speed and feed rate in the wood working processing were investigated on the quality of wood surface. The ranges of CNC parameters studied in this research are spindle speeds of 10000, 14000 and 18000 rpm and feed rates of 5000, 7000 and 9000 mm/min. As a result of the study, the lowest surface roughness values were found in 10000 rpm spindle speed and 5000 mm/min feed rate for spruce and chestnut wood and 18000 rpm spindle speed and 7000 mm/min feed rate for larch and iroko wood. The highest values in the all of wood species were obtain from 10000 rpm spindle speed and 9000 mm/min feed rate.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

A Study on the Planning and Targeting Applications in Forest Products Industry Enterprises (Case of Istanbul Province)<sup>[\*]</sup>

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Abstract: Planning and targeting is the first step of business management function. The success of management activities in each business is achieved through effective planning and targeting. Managers can allocate more time to planning and targeting than other functions. Priorities should be defined when determining processes in a business. The first step in planning and targeting is to determine where you want to be reached. Also, procurement and selection of all kinds of sources which will achieve these targets are included in planning and targeting. In this study, it was tried to determine how much the managers in the different forest industry enterprises operating in İstanbul have an important role in achieving the success of the enterprise in terms of planning and targeting function. In the study, a scale consisting of 31 questions was set up for the planning and targeting to the employees in the enterprises. The number of surveys evaluated was 271. The results obtained from managers and other employees in different forest products sectors were tested in SPSS statistical program with some other factors and the results were presented in detail.

Keywords: Forest products industry, planning, targeting.

# Orman Ürünleri Sanayi İşletmelerinde Planlama ve Hedefleme Uygulamaları Üzerine Bir Araştırma (İstanbul İli Örneği)

Öz: Planlama ve hedefleme, işletmedeki yönetim fonksiyonunda ilk adımdır. Her işletmedeki yönetim faaliyetlerinin başarısı, etkili planlama ve hedeflemeden gecer. Bir isletmedeki üst düzev yöneticiler icin planlama ve hedefleme diğer fonksivonlardan daha fazla zaman alabilir. Alt kademe yöneticiler içinde, daha fazla zaman alabilir. Bir işletmedeki süreçleri belirlerken öncelikler tanımlanmalıdır. Planlama ve hedeflemede ilk adım, nereye ulaşmak istediğinizi belirlemektir. Ayrıca, bu hedeflere ulaşacak her türlü kaynağın alımı ve seçimi planlama ve hedeflemeye dahil edilir. Bu çalışmada, İstanbul'da faaliyet gösteren orman endüstrisi işletmelerinin yönetim aşamalarındaki yöneticilerin, işletmelerin planlama ve hedefleme işlevi açısından başarısını sağlamada ne kadar önemli bir rol oynadıklarının belirlenmesi amaçlanmıştır. Çalışmada, işletmelerdeki çalışanların planlanması ve hedeflenmesi için 31 sorudan oluşan bir ölçek oluşturulmuştur. 271 anket değerlendirmeye alınmıştır. Farklı orman ürünleri sektörlerindeki yöneticilerden ve diğer çalışanlardan elde edilen sonuçlar SPSS istatistik programında bazı faktörler ile test edilmiş ve sonuçlar ayrıntılı bir şekilde sunulmuştur.

Anahtar sözcükler: Hedefleme, orman ürünleri sanayi, planlama.

#### INTRODUCTION

As in any industry, enterprises operating in forest products industry should attach importance to "management" concept to increase their profits, maximize their sales and survive in a competitive environment. A good management is the most important element in the success of a business.

It is relatively new that management emerged as a scientific discipline even though management event is based on as old as human history. But concepts related to management science have not been fully clarified and a common management definition has not yet been reached among the authors (Şimşek, 2010). Therefore, management can be understood as a process, elements of this process are that people and groups, a certain information society, and decision-making and leadership activities (Saltürk, 2008).

According to thinkers who tend to perceive management as a process, management can be expressed as a process involving the whole of the efforts of directing a group of people towards designated targets and coordinating them with cooperation. It should not be forgotten that the management is not achieved by using only human resources. Because, in the concept of management, it is mentioned that all production factors, including the firstly human element, are used effectively and efficiently in accordance with the targets determined at the planning stage (Simşek, 2010).

As a process, the functions need to be handled one by one for management to review as required. In traditional sense, these management functions are divided into five. These are planning, organizing, directing, coordinating, and controlling (Şimşek & Çelik, 2014). In this study, it is informed only about planning in those functions.

In a simple sense, the planning is the first and most important of the management functions which is a bridge between the present situation and the future. If in a general sense, it is a conscious process that has the qualities of choosing and developing the best course of action to achieve an target. At this stage, answers to the following questions seek; "What?", "When?", "How?", "Where?", "Why? "," By whom? "," What cost? " (Bakan et al., 2013; Can & Güney, 2018; Tengilimoğlu et al., 2012).

Planning is done for a variety of reasons. The most important of these reasons is that scarce resources are used efficiently and profitably. Beside this, there are any other reasons for planning (Özevren, 2009). These (Özevren, 2009);

- To determination of the criteria for effective control,
- To guide the enterprise,
- To predict the future of the business more accurately,
- To direct other functions of the management,
- To prevent repetition of activities in enterprise,
- To respond more quickly to sudden changes for enterprise,
- To increase efficiency and effectiveness.

It should not be forgotten that some important points should be taken into consideration when making the planning which is important for enterprise. These: "who made the planning", "time dimension", "resource need", "alternative cost of the plan", "making alternative plans", "basing planning on real events and scientific predictions", and "the effect of environmental factors" (Özevren, 2009).

It is collected a number of pieces of information by planning function in order to determine what the targets and strategies of the enterprises will be. Accordingly, the planning activities involves a number of stages until the plan is clear, starting with the collection of information. One of these stages is to determine the targets (Tutar, 2005).

In the targeting stage of the planning process, it is determined where the enterprise wants to be in the future. The uncertain targets make practitioners unsuccessful. The targets may also be comprehensive enough to cover all enterprise. Main (general) targets are determined at the upper levels of the organization. In order to the targets to be carried out effectively and efficiently, everyone should be aware of the basic and special targets and adopt these targets. (Tengilimoğlu et al., 2012; Şimşek & Çelik, 2013; Şimşek & Çelik, 2015).

In this study, the perceptions of participants related to questions regarding targeting and planning were investigated. It was determined by using one-way variance analysis (ANOVA) that whether there was a significant difference between the positions of employees and questions regarding targeting and planning or not.

#### **MATERIAL and METHODS**

The scope of our study constitutes forest products enterprises operating in the Anatolian side of Istanbul. The survey form was planned to be applied to the employees of large and/or small scale enterprises in the Forest Products Industry. The survey form contains questions regarding the demographic characteristics and targeting and planning. 31 questions were prepared in accordance with a 5-point Likerttype scale (never:1, very little:2, undecided:3, normal level:4, high level:5) for questions regarding targeting and planning. The questions prepared regarding targeting and planning were given in Table 1. The surveys were applied by performing face to face interviews with people.

Table	1.	Questions	regarding	objective	setting	and p	olanning.
					<u> </u>		<u> </u>

	Questions
1	The objectives in our enterprise are clear.
2	Awareness of the objectives and plans increases the motivation of employees in the enterprise.
3	The objectives and plans in our business form a guide for the activities.
4	The objectives and plans in our enterprise form the basis for the decisions of the managers.
5	The objectives and plans in our enterprise are the success standard.
6	The market share to will be owned in our objectives and plans in our enterprise is investigated.
7	In our enterprise, new methods and product innovations are included in the objectives and plans.
8	Profitability is the forefront in objectives and plans in our enterprise.

9	In our enterprise, the cash flow in determining objective and plan is important.
10	In our enterprise, physical and financial resources are important in objectives and plans.
11	In our enterprise, importance is given to productivity performance in objectives and
11	plans.
12	In our enterprise, managerial and organizational growth is prioritized in objectives and
12	plans.
13	In our enterprise, social responsibilities towards customers and society are in the
15	forefront in objectives and plans.
14	The objectives in our enterprise were often digitized.
15	The objectives and plans in our enterprise are carried out on the main activities.
16	The objectives in our enterprise are very real.
17	The objectives and plans in our enterprise are realized within a certain period of time.
18	Planning is a financial burden for enterprise.
19	Planning is necessary for the sector we are in.
20	Planning increases the number of staff.
21	Planning limits the freedom of movement of managers.
22	Planning contributes to regular communication.
23	With the planning, the hierarchical system works properly from top to bottom.
24	Planning in our enterprise facilitates the transfer of authority.
25	Our enterprise has policies.
26	The methods available in our enterprise are used.
27	The rules and bases are obeyed in our enterprise.
28	There are always the desires for planning in our enterprise.
29	Alternative plans in our enterprise are constantly being developed.
30	The flexibility principle in planning in our enterprise is implemented.
31	Teamwork is fully implemented in our enterprise.

The following sample determination formula was used to determine the total number of the participants to whom the surveys would be applied (Naing et al., 2006):

$$n = \frac{N.Z^2.p.q}{(N-1).d^2 + Z^2.p.q}$$
(1)

In formula; n: sample size;

N: universe size;

p: p-value (0.5);

q: q- value (0.5);

Z: Z-score at 95 % confidence interval (1.96);

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d: margin of error (0.05).
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According to the Istanbul Chamber of Commerce, there are 1750 enterprises in the Forest Products Industry operating in the Istanbul. The "p" and "q" values were taken as 0.5 in a way that would enable the sample size to be more in our study. The error margin was accepted as 5%. As a result, the sample size was determined to be 315. However, 271 surveys were achieved. The survey forms were numbered and they were entered into the SPSS for Windows package program. One-way variance (ANOVA) analysis was used to determine whether there was a significant difference between the positions of employees and expressions about targeting and planning or not. Abbreviations of the positions of the employees used in the analyzes were given in Table 2.

Table 2. Th	ne positions	of employees	and abbreviations.
	1	1 2	

The positions of employees in enterprises	Abbreviations
Senior managers	SM
Middle-level managers	MLM
Lower-level managers	LLM
Labor-officer employees	LOE

RESULTS

**Results regarding demographic characteristics:** According to Table 3, 77.9% of personnel who participated in the survey were male, 22.1% were female. Majority of the participants (42.1%) were between 26-33 years of age. Majority of the participants (80.8%) worked at the same enterprise less than 10 years. More than half of them (57.2%) worked in the manufacture department. While 61.6% of enterprises operated in the furniture sector, other enterprises operated in the lumber (19.9%) and corrugated board sector (18.5%). Majority of the enterprises were limited liability companies. A great majority of the enterprises were small and medium-sized. 4.1% of participants who participated in the survey were senior manager, 16.2% were middle-level manager, 8.5% were lower-level manager and 71.2% were labor-officer employee

	Table 3.	The demo	ographic	charact	eristics	of	partici	oants
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Demographic characteris	tics	Frequency	Percentage (%)
Cardan	Male	211	77.9
Gender	Female	60	22.1
	18-25	42	15.5
1.00	26-33	114	42.1
Age	34-41	80	29.5
	42 and over	35	12.9
	Senior manager	11	4.1
De eldere	Middle-level manager	44	16.2
Position	Lower-level manager	23	8.5
	Worker, officer	193	71.2
	Limited company	195	72
Legal structure	Joint stock company	35	12.9
	Others	41	15.1
	0-5 year	128	47.2
Total working time in	6-10 year	91	33.6
enterprise	11-15 year	38	14
	16 year and over	14	5.2
	0-9 people	111	41
Total number of	10-49 people	94	34.7
employees in enterprise	50-99 people	30	11.1
	100 people and over	36	13.3
	Manufacture	155	57.2
Department where the	Sales & Marketing	76	28
participants work	Financial and administrative affairs	35	12.9
	Enterprise owner	5	1.8
The sector is subject the	Furniture	167	61.6
i ne sector in which the	Timber	54	19.9
enter prise is located	Corrugated cardboard	50	18.5

*Statistical analysis results:* It was determined that whether there was a significant different between questions regarding targeting and planning and manager types or not. For this, one-way variance (ANOVA) analysis was used and the results were given in Table 4.

Table 4.	Results	regarding	planning	and	targeting
		U . O			U. 0

Question	Manager type	Mean	F	р
	SM	4.18		
	MLM	3.98		
Question 1	LLM	4.00	0.971	0.407
	LOE	4.19		
	Total	4.09		
	SM	4.36		
	MLM	4.05		
Question 2	LLM	4.13	0.509	0.676
	LOE	4.17		
	Total	4.18		

Question	Manager type	Mean	F	р
Question 3	SM	4.36	1.205	0.308

	MLM	3.95		
_	LLM	4.09		
_	LOE	4.20		
	Iotal	4.15		
—	MIM	4.33		
Ouestion 4	LLM	4.22	0.923	0.430
Question 4	LOE	4.17	0.725	0.150
	Total	4.26		
	SM	4.45		
	MLM	4.09		
Question 5	LLM	4.26	0.727	0.537
	LOE	4.22		
	Total	4.26		
_	SM	4.27		
Ouestion 6	MLM	3.82	2 252	0.083
	LEM	4.50	2.232	0.005
	Total	4.13		
	SM	4.45		
	MLM	4.07		
Question 7	LLM	4.22	0.767	0.513
	LOE	4.19		
	Total	4.23		
	SM	4.45	3.737	$0.012^{\circ}$
o o	MLM	3.86		
Question 8	LLM	4.26		
	LUE	4.25		
	SM	4.21	3 206	0.024°
	MLM	3.86	5.200	0.024
Question 9	LLM	4.17		
-	LOE	4.24		
	Total	4.18		
	SM	4.64		
_	MLM	3.95		
Question 10	LLM	4.13	1.851	0.138
	LOE	4.17		
	Total	4.22		
	MIM	3.86		
Ouestion 11	LLM	3.91	1 468	0 224
·	LOE	4.12		
	Total	4.04		
	SM	4.36		
	MLM	3.73		
Question 12	LLM	4.35	4.065	0.008**
	LOE	4.16		
	Total	4.15		
_	MIM	4.18		
Ouestion 13	LLM	4 26	1 761	0.155
Question 10	LOE	4.12	11/01	0.100
	Total	4.10		
	SM	4.09		
	MLM	3.66		
Question 14	LLM	4.13	2.004	0.114
	LOE	3.98		
	Total	3.97		
	SM ML M	4.36		
Question 15		4.05	0.529	0.662
Question 15	LLM	4.09	0.528	0.003
	Total	4.17		
	SM	4.45		
	MLM	3.98		
Question 16	LLM	4.04	0.855	0.465
	LOE	4.09		
	Total	4.14		
_	SM	4.55		
0	MLM	3.86	1.02.	o · · · -
Question 17	LLM	3.91	1.824	0.143
	Total	3.99		
	SM	3.82		
	MLM	3.52		
Question 18	LLM	3.96	2.308	0.077
	LOE	3.96		

Question	Manager type	Mean	F	р
Question 10	SM	4.00	1.016	0.127
Question 19	MLM	3.93	1.910	0.127

	IIM	4 39		
-	LOF	4.39		
-	Total	4.13		
	SM	4.18		
-	MLM	3.75		
Question 20	LLM	4.13	1.349	0.259
	LOE	3.98		
-	Total	4.01		
	SM	4.18		
_	MLM	3.64		
Question 21	LLM	3.83	1.426	0.236
	LOE	3.98		
	Total	3.91		
_	SM	3.91		
_	MLM	3.80		
Question 22	LLM	4.09	1.274	0.284
_	LOE	4.06		
	Total	3.97		
_	SM	4.45		
	MLM	3.61		**
Question 23	LLM	4.35	8.473	0.000
_	LOE	4.11		
	Total	4.13		
-	SM	4.00		
Question 24	LLM	5.80	1 226	0.207
Question 24	LLM	4.13	1.230	0.297
_	Total	4.03		
	SM	4.03		
-	MLM	3.93		
Ouestion 25	LLM	3.96	3.048	0.029
·	LOE	4.13		
-	Total	4.19		
-	SM	4.64		
-	MLM	3.89		
Question 26	LLM	4.00	3.200	0.024
-	LOE	4.16		
-	Total	4.17		
	SM	4.45		
_	MLM	3.93		
Question 27	LLM	4.13	2.165	0.093
_	LOE	4.23		
	Total	4.19		
	SM	4.36		
	MLM	3.77		
Question 28	LLM	3.87	3.005	0.031
_	LOE	4.15		
	Total	4.04		
_	SM	4.09		
_	MLM	3.70		
Question 29	LLM	4.22	2.561	0.055
_	LOE	4.11		
	Total	4.03		
_	SM	4.18		
	MLM	3.70	0.1.7	0.007
	LLM	3.87	2.167	0.092
Question 30	LOD	~		
Question 30	LOE	4.09		
Question 30	LOE Total	3.96		
Question 30	LOE Total SM	4.09 3.96 4.36		
Question 30	LOE Total SM MLM	4.09 3.96 4.36 3.68	1 000	0.002*
Question 30 Question 31	LOE Total SM MLM LLM	4.09 3.96 4.36 3.68 4.13 4.25	4.808	0.003°

\*, p<0.05; \*\*p<0.01; \*\*\*, p<0.001

According to one-way variance analysis, it was found that there was significant different between the eight questions regarding targeting and planning and the opinions of managers. The questions which were found significant differences were 8, 9, 12, 23, 25, 26, 28 and 31. Senior managers said that more attend to the questions, which were found significant differences,. At the same time, when the Table 4 was examined, majority of the senior managers who participated in the survey said that the enterprise has policies (question 25). Majority of the middle-level managers who participated in the survey said that the enterprise does not attach much importance to targeting and planning. Because a great of majority of the questions were under 4. The lowerlevel managers who participated in the survey said that planning is necessary for the sector (question 19). The officer-workers said that profitability is the forefront in targets and plans in enterprise (question 8) and teamwork is fully implemented in enterprise (question 31). When the results examined in general, majority of the respondents said that the targets and plans form the basis for the decisions of the managers and the targets and plans are the success standard.

#### CONCLUSION

In this study, the perceptions of the employees related to questions regarding targeting and planning were investigated. Also, it was determined that whether there was a significant difference between the positions of the employees and questions regarding targeting and planning or not. In this study, the following results were obtained:

(1) A great of majority of the participants were male.

(2) Majority of the participants were under the age of 42.

(3) Majority of the participants were the worker or officer.

(4) Majority of the participants worked in smallscale enterprises and majority of the enterprises were in the status of limited liability company.

(5) Majority of the employees participating in the study were employed by enterprises operating in the furniture sector. Because there were the most furniture sectors in the forest products industry in the Anatolian side of Istanbul.

(6) Majority of the employees worked in manufacturing departments of enterprises.

(7) According to opinion of managers, it was found that some questions related to the planning and targeting have significant differences. These questionss were 8, 9, 12, 23, 25, 26, 28 and 31. In other words, the answers given varied according to the manager type.

(8) Majority of the participants said that the enterprises pay attention to planning and targeting.

In order to ensure the continuity of the enterprises and to keep the enterprises standing, they are necessary to give importance to targeting and planning. Therefore, more extensive researches can be done for future studies.

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**RESEARCH PAPER** 

#### ARAŞTIRMA MAKALESİ

The Effect of Vermiculite Usage on Surface Properties of Medium Density Fibreboard Abdullah İSTEK İsmail ÖZLÜSOYLU\* Ahmet CAN Saadettin Murat ONAT Bartin University, Department of Forest Industry Engineering, 74100, Bartin/Turkey. D: https://orcid.org/0000-0002-3357-9245, \*D: https://orcid.org/0000-0002-0391-4794 D: https://orcid.org/0000-0001-5926-6039, (D: https://orcid.org/0000-0003-1749-0619) Received date: 14.05.2018 Accepted date: 09.07.2019 How to cite: İstek, A., Özlüsoylu, İ., Can, A. & Onat, S.M. (2019). The Effect of Vermiculite Usage on Surface Properties of Medium Density Fibreboard. Anatolian Env. and Anim. Sciences, 4(4), 607-612. Attf yapmak için: İstek, A., Özlüsoylu, İ., Can, A. & Onat, S.M. (2019). Orta Yoğunluklu Lif Levhaların Yüzey Özellikleri Üzerine Vermikülit Kullanımının Etkisi. Anadolu Çev. ve Hay. Dergisi, 4(4), 607-612.

Abstract: In this study, the effects of vermiculite, which is one of volcanic minerals, usage in medium density fibreboard (MDF) production on the surface properties of these boards were investigated. The test boards were produced using the dry method with 12% urea formaldehyde resin. Additions of 10%, 15%, 20% and 30% vermiculite were used based on the oven-dry fibre weight. Surface roughness, colour and gloss values of both surfaces of the obtained boards were determined. Based on the results, the ratio of vermiculite increased, roughness values on the surfaces increased. The roughness values in the bottom surface of the produced boards were determined to be higher than the top surface. With the use of 30% vermiculite, the average minimum surface roughness (Ra) values were found to be 8.65  $\mu$ m on the upper surfaces and 15.44  $\mu$ m on the lower surface. It has been found that total color change and gloss are improved by the increase of vermiculite usage and the colour change on the bottom surface is found higher. In short, the use of vermiculite in the production of MDF negatively affects the surface roughness and discoloration of the boards, but it has been found to positively affect the gloss.

Keywords: Colour, gloss, MDF, surface roughness, vermiculite.

# Orta Yoğunluklu Lif LevhalarınYüzey Özellikleri Üzerine Vermikülit Kullanımının Etkisi

Öz: Bu çalışmada, orta yoğunlukta lif levha (MDF) üretiminde volkanik minerallerden vermikülit kullanımının yüzey özelliklerine etkisi araştırılmıştır. Deney levhaları kuru yöntemle %12 üre formaldehit tutkalı kullanılarak üretilmiştir. Tam kuru lif ağırlığına oranla %10, %15, %20 ve %30 oranlarında vermikülit kullanılmıştır. Levhaların her iki yüzeyinin yüzey pürüzlülük, renk ve parlaklık değerleri belirlenmiştir. Sonuçlara göre vermikülit oranının artmasıyla, pürüzlülük değerleri artmıştır. Levhaların alt yüzeylerindeki pürüzlülük değerleri ist yüzeylerinden daha yüksek olduğu belirlenmiştir. %30 vermikülit kullanımı ile ortalama minimum yüzey pürüzlülüğü (Ra) değerleri üst yüzeylerde 8,64 µm, alt yüzeylerde ise 15,44 µm olarak bulunmuştur. Toplam renk değişimi ve parlaklığın vermikülit kullanım oranının artmasıyla iyileştiği, alt yüzeylerindeki renk değişiminin daha fazla olduğu tespit edilmiştir. Kısaca, MDF üretiminde vermikülit kullanımının levhaların yüzey pürüzlülüğünü olumsuz, parlaklık ve renk değişimini ise olumlu olarak etkilediği sonucuna varılmıştır.

Anahtar sözcükler: MDF, parlaklık, renk, vermikülit, yüzey pürüzlülüğü.

# INTRODUCTION

Nowadays, board products are widely used as building materials in many areas such as construction, decoration, interior and exterior architecture, furniture production (Kim eal t., 2002; Seo et al., 2016). Medium density fibreboard (MDF) is one of wood-based panels produced by bonding wood fibres with resin under temperature and pressure and produced in high quantities (Krzysik, 2001). In recent years, MDF production has increased significantly and has a large market share in the wood composite sector (Koc & Aksu, 1999; Cabuk et al., 2013; İstek et al., 2017a). As with other wood-based boards, the disadvantages of MDF boards are poor resistance to moisture and low resistance to burning (Ustaömer et al., 2008; İstek et al., 2017b). Some measures must be taken against these disadvantages in order to make better use of wood based board products and to use these products efficiently. This is because the objective of board production is to improve the economic, aesthetic and technological properties of the material, as well as to increase the resistance against biotic and abiotic factors (Hall et al., 1982; Dix, 1997). It is reported that vermiculite can be used to increase the fire resistance of cellulose based composites in different studies (Kozlowski et al., 1999; Rider, 2015; Rider, 2016; Wang et al., 2016b; Aksogan et al., 2018).

Many methods and materials are used to improve the burning properties of wood materials. Important chemicals used as preservatives for this purpose are combustion retardants such as ammonium sulphate, ammonium chloride, dicyandiamide, borax and boric acid, and various phosphorus compounds (phosphoric acid, monoammonium phosphate and diammonium phosphates) (Ustaömer, 2008; Peker & Atılgan, 2015; İstek et al., 2012; İstek et al. 2013; İstek & Özlüsoylu, 2016; Özdemir & Tutuş, 2013).

Vermiculite is a magnesium aluminosilicate clay mineral formed by natural wear of mikan. It is a mineral obtained from volcanic magma rocks and it is expanded at high temperatures to increase volume and permeability. The bulk density value changes significantly by falling shape (Toksoy, 1997). Vermiculite has good sound and heat insulation properties and also has the ability to stick to different surfaces. When used as a fire retardant, the smoke and gases released are not toxic and do not pose a threat to the environment (Crawford et al., 1990). The expanded vermiculite has a density of between 80 kg /  $m^3$  and 120 kg /  $m^3$  with a heat transfer coefficient of 0.04 W / (m  $\cdot$  K) to 0.12 W / (m  $\cdot$  K) and a high melting point of 1240 ° C - 1430 ° C. It is also a chemically inert, stable and environmentally safe material (Suvorov & Skurikhin, 2003; Nguyen et al., 2013; Wang et al., 2016).

Surface properties are important in terms of woodbased boards, mainly medium density fibreboard (MDF) and chipboard, and board products are coated with liquid or solid coating materials to enhance their aesthetic, resistance properties and economic values (İstek et al., 2010; İstek et al., 2015; Atar, 2006; Nemli, 2003). Since the board products form the lower layer of the coating materials, surface properties such as surface roughness, bonding and quality of the final product are important. It is known that many factors are effective on the surface roughness depending on raw material and production conditions (Kılıc et al., 2009; İstek et al., 2012; Nemli et al., 2007; Dündar, et al., 2008; Özdemir 2016; Bozdogan Balcik & Özdemir, 2019).

Various studies indicated that fire-retardant materials have different effects on the physical, mechanical and surface properties of wood materials and wood-based composites (İstek & Özlüsoylu, 2016; Ustaömer, 2008; Winandy et al., 2002; Taghiyari et al., 2013; Winandy, 1998; Ayrilmis et al., 2007; Ayrilmis et al., 2005; Ayrilmis, 2007; Simsek et al., 2013; İstek et al., 2017c; İstek et al., 2017d).

The needle screening method is used in the metal and plastics industries for surface roughness. This method is more widely used in the measurement of wood composite boards and the roughness of solid wood product surfaces (Hızıroğlu, 1996; Burdurlu et al., 2005; Peters & Mergen, 1971). The use of combustion retardants affects the surface properties of the boards. In this study, the effects of vermiculite used as fire retardant on the surface properties of the boards were investigated. For this purpose, some surface properties such as roughness, colour and gloss of MDF boards produced by adding vermiculite at different ratios have been determined.

## MATERIAL and METHODS

In this study oriental beech (*Fagus orientalis*) and black pine (*Pinus nigra*) wood fibres were used as raw materials. Fibres were supplied from Kastamonu Integrated Company, MDF Plant and consists of 80% beech and 20% black pine wood fibre blends. Urea formaldehyde binder (57% solid content) was used with respect to 12%. total dry fibre weight. Vermiculite was commercially available and was used in MDF production at rates of 10%, 15%, 20% and 30% relative to the total dry fibre weight in this study. The vermiculite was milled in 0.5 mm mesh before addition.

*MDF Production:* In the study, the total of 15 boards were produced including vermiculite in 4 different ratios (10%, 15% 20% and 30%) and control boards without vermiculite as three boards of each group. The target density of the produced boards was 800 kg/m<sup>3</sup> and the form sizes were 400x400x12 mm. The fibres used in the production were supplied in a ready-made form and were brought to the laboratory in plastic bags in such a way that they do not get in contact with air. After the moisture control has been carried out, it was mixed with vermiculite at the specified ratios using a rotary drum gluing machine. The prepared

fibres were hand-laid out in a wooden shaping mold with dimensions of 400x400x300mm and a board mat was formed. The board mat was exposed to 180 bar pressure and the temperature of  $170^{\circ}$ C and 5 minutes at a hot press (SSP180 Cemil Usta, Turkey). The MDF boards were produced using a 12 mm thick metal thickness control sticks. The colour change, gloss values and the surface roughness values of the produced boards were determined and they were compared with the control group. The colour, gloss and surface roughness measurements of the vermiculite-added boards were carried out from two different surfaces, top and bottom, depending on the position of the boards in the formation.

**Colour measurements:** Colour measurement of the test samples were carried out in accordance with ISO 7724 standards by Konica Minolta CD- 600 colour meter. On the board samples, the colour measurements from 3 different points were measured and their mean value were calculated for three replicates in each variation (ISO-7724, 1984). The CIELab (Commission Internationale de l'Eclairage) system consists of three variants (ISO 7724). L\* refers to Light stability, a\* and b\* chromatographic coordinates (+a\* indicates red, -a\* green, +b\* yellow, -b\* blue). The values of  $L^*$ ,  $a^*$  and  $b^*$  were measured on the samples and the colour changes were determined according to the following Formula 1.

 $\Delta E^* = (\Delta L^* 2 + \Delta a^* 2 + \Delta b^* 2) \frac{1}{2}$ (1)

**Gloss measurements:** Gloss measurements were taken using a KONICA Minolta Multi gloss 268 plus. The angle of incidence of the radiation was  $60\pm0.1^{\circ}$ , as defined in ISO 2813.1994. Six measurements were made in each test panel.

*Surface roughness:* Mitutoyo Surftest SJ-301 was used for surface roughness measurements. The mean surface roughness (Ra), maximum height (Rz) and ten-point average roughness (Rq) values of the test samples were evaluated. Both of side measurements were made on the surfaces of the control and test samples according to the ISO 4287 standard. The measurements were used as a boundary wave length of 8 mm, a velocity of 0.5 mm/s and a scanning length of 12 mm. Ten measurements were made to evaluate the surface roughness of each group.

#### **RESULTS and DISCUSSION**

The results of the board groups with vermiculite added at different ratios and the results of the control group boards produced without vermiculite with respect to colour, gloss and surface roughness were discussed below.

**Colour change:** The colour change values in the test and control samples were calculated using the L (light intensity), a and b chromatographic coordinates (+ a \* red, -a green, + b yellow and -b blue) determined according to the CIELab system. Table 1 shows the bottom surface, top surface and average values and standard deviations of L \*, a \*, b \* values for the board groups.

According to the results, the average L \* value increased as the chemical substance added increased except for the addition of 20% vermiculite. An increase in L \* is an indication that the surfaces of the boards have colouring. Also, the L \* values obtained at the bottom surface are higher than the values measured from the top surface. This is due to the fact that some of the vermiculite used as a dust in the forming mold collapses on the bottom surface of the mat and is found to be higher than the surface of the mat. Figure 1 shows the mean change values of L\*, a\* and b\*.

Table 1. L\*, a\*, b\* values of test boards.

Type of h	orde	Control	10%	15%	20%	30%
Type of be	Jarus	Control	vermiculite	vermiculite	vermiculite	vermiculite
Dottom	L*	-	56.28±0.87	58.49±1.27	57.40±2.67	60.90±1.30
Bottom	a*	-	8.60±0.29	7.56±0.71	8.10±1.32	6.11±0.42
surface	b*	-	22.25±0.38	20.99±0.93	21.97±1.81	19.40±0.46
Tom	L*	-	56.09±0.98	53.99±1.56	53.47±2.25	54.92±1.58
Top	a*	-	9.29±0.24	9.47±0.44	9.50±0.37	8.85±0.27
surface	b*	-	24.08±0.53	23.46±0.64	23.25±0.73	22.18±0.56
Avorago	L*	52,01±2,14	56.18±0.88	56.24±0.92	55.44±0.82	57.91±1.02
Average	a*	9,69±0,47	8.95±0.19	8.51±0.39	$8.80 \pm 0.56$	7.48±0.25
values	b*	23,45±0,87	23.16±0.22	22.23±0.50	22.61±1.11	20.79±0.37

The a \* value decreased as the ratio of additional chemicals increased, except for the addition of 20% vermiculite for the average a \* values. Reduced a \* value is a sign of red colour on the board surfaces. Similarly, as the rate of added material increased, the b \* value decreased. The decrease in b \* indicates the increase in blue colour on the board surfaces.



Figure 1. Average L\*, a\*, b\* values change of board types.

Figure 2 shows the total colour change values ( $\Delta E$  \*).



Figure 2. Total colour change of board types.

The total colour change value indicates the colour change on the surface of the board compared to the control sample. As the utilization rate of vermiculite increased in board production, total colour change values increased except for the use of 20% vermiculite. The maximum total colour change was 6.84 in the addition of 30% vermiculite while the minimum total colour change value was 3.64 in 20% vermiculite use. Ustaömer et al. (2006) found that treatment with boric acid, borax and sodium perborate tetrahydrate at 1% and 3% concentrations increased the discoloration ( $\Delta E^*$ ) value of the fibreboard due to the increase in chemical concentration and that the highest 3% was sodium perborate tetrahydrate stated. In another study, it was reported that  $\Delta E^*$  values increased in direct proportion with increasing chemical concentration (Ustaömer, 2008).

*Glossiness:* Glossiness values of control group and groups of vermiculite added boards at different ratios are given in Table 2. It has been understood that the use of vermiculite as compared to the control sample increases the gloss value of the boards but this increase is not linear. The highest gloss value was achieved as 2.33 with 15% vermiculite use, while the lowest gloss was achieved with control as 2.09.

Table 2. Gloss value of test boards

Type of boards	Bottom surface	Top surface	Average values	
Control	-	-	2.09±0.03	
10% vermiculite	2.18±0.13	2.38±0.05	2.28±0.06	
15% vermiculite	2.38±0.17	$2.28 \pm 0.05$	2.33±0.06	
20% vermiculite	2.25±0.17	2.20±0.12	2.23±0.06	
30% vermiculite	2.15±0.10	$2.08 \pm 0.05$	2.11±0.05	

*Surface roughness:* The surface roughness test results of the boards produced in the study are given in Table 3.

 Table 3. Surface roughness of test boards

Type of bo	oards	Control	10% vermiculite	15% vermiculite	20% vermiculite	30% vermiculite
D - 44	Ra	-	8.69±1.42	9.39±1.08	11.76±1.63	15.44±3.92
Bottom	Rq	-	9.97±1.72	12.03±1.30	15.12±2.19	20.51±4.97
surface	Rz	-	72.42±6.31	77.63±10.71	95.83±15.35	124.92±14
<b>T</b>	Ra	-	8.63±1.06	8.71±1.37	8.84±1.04	8.65±1.40
Top	Rq	-	11.25±1.26	$11.14 \pm 1.78$	11.35±1.42	11.14±1.87
surface	Rz	-	$65.15 \pm 8.28$	69.25±12.47	$70.80{\pm}10.91$	73.29±12.20
A	Ra	8.06±1.58	8.66±1.24	8.94±0.80	10.09±0.86	10.97±1.34
Average	Rq	$10.27 \pm 2.05$	10.61±1.49	$11.40{\pm}0.88$	12.97±1.39	$14.48 \pm 1.81$
values	Rz	60.43±6.01	$68.78 \pm 7.30$	72.83±6.98	75.33±7.08	89.30±6.13





It was determined that vermiculite addition increased the surface roughness (Ra, Rz and Rq) properties and reduced the smoothness of the surfaces when compared to the control group of surface roughness properties. Moreover, the higher roughness values on the bottom surfaces were caused by the accumulation of more vermiculite on the bottom surface of the board compared to the pavement pattern during the formation of the board. Figure 3 shows the change of Ra, Rq and Rz values of the board groups.

As shown in Fig. 3, the lowest Ra value was 8.06 µm in control and the highest Ra was 10.97 µm in 30% vermiculite added boards. It has been concluded that vermiculite used in powder form did not disperse homogeneously in the board and adversely affected bonding between fibres and glue, preventing smooth surface formation during hot pressing. This has led to an increase in surface roughness values with increasing vermiculite use. This appears to be more pronounced on the bottom surfaces where vermiculite is distributed unevenly. It is thought that the grain size of the vermiculite used additionally may be effective on the surface roughness. Ustaömer et al. (2008) reported that surface roughness values of MDF boards produced were increased due to chemical types and concentration increase of 3%, 5% boric acid, borax, sodium perborate tetrahydrate, zinc borate and boric acid+borax mixtures of fire retardant treatment. In addition, different studies have reported that the morphological properties of the used fibre raw materials and the production parameters effect surface roughness (Nemli, et al., 2007; Dündar, et al., 2008; Özdemir, 2016).

#### CONCLUSION

In this study, colour, glossiness and surface roughness values of MDF produced by vermiculite addition at different ratios were determined. As a result of the study, L \* value increased with increasing vermiculite use rate, but a \* and b \* values decreased. This is an indication of the increase in white colour (+L\*) and blue colour (-b\*) and decrease in red colour (+a\*) on the boards. Total colour change was increased with increasing vermiculite usage rate except 20% vermiculite usage. When the glossiness values were examined, it was determined that vermiculite usage increased the glossiness compared to the control sample. The surface roughness values (Ra, Rq and Rz) increased as vermiculite usage increased and the surfaces became rougher.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

Antioxidant, Antimicrobial and Anti-Quorum Sensing Activities of Usnea filipendula and Viscum album<sup>[\*]</sup>

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**Abstract:** Many plants contain a variety of bioactive components. Therefore, it is important to know the bioactive properties of plant materials in order to be a reference for later researchers. In this study, it was investigated the antioxidant, antimicrobial and anti-quorum sensing activities of *Usnea filipendula* and *Viscum album*'s methanol extracts. To determine the antioxidant properties of the extracts; total phenolic, flavonoid and condensed tannin contents and ferric reducing antioxidant power analyses were performed. The antibacterial potential of plant extracts was tested by agar well diffusion method against *Staphylococcus aureus*, *Escherichia coli*, *Enterococcus faecalis*, *Pseudomonas aeruginosa*, *Salmonella typhimurium*, *Klebsiella pneumoniae*, *Proteus mirabilis*, *Listeria monocytogenes*, *Candida parapsilosis* and *Candida albicans* microorganisms. Anti-quorum sensing activity was investigated on *Chromobacterium violaceum* bacteria. The highest total phenolic and ferric reducing antioxidant power was determined in *U. filipendula* extract. This extract inhibited the growth of *S. aureus*, *K. pneumonia* and *L. monocytogenes*. None of the extracts showed anti-quorum sensing activity.

Keywords: Antioxidant, antimicrobial anti-quorum sensing, Usnea filipendula, Viscum album.

# *Usnea filipendula* ve *Viscum album*'un Antioksidant, Antimikrobiyal ve Çoğunluğu Algılama İnhibisyonu Aktiviteleri

Öz: Birçok bitki çeşitli biyoaktif bileşenler içerir. Bu nedenle, daha sonraki araştırmacılar için referans olması amacıyla bitki materyallerinin biyoaktif özelliklerini bilmek önemlidir. Bu çalışmada, *Usnea filipendula* ve *Viscum album*'ün metanol ekstrelerinin antioksidan, antimikrobiyal ve çoğunluğu algılama inhibisyonu aktiviteleri incelenmiştir. Ekstraktların antioksidan özelliklerini belirlemek için; toplam fenolik, flavonoid ve kondanse tanen içerikleri ve demir indirgeyici antioksidan gücü analizleri yapılmıştır. Bitki ekstraktlarının antibakteriyel potansiyeli, *Staphylococcus aureus, Escherichia coli, Enterococcus faecalis, Pseudomonas aeruginosa, Salmonella typhimurium, Klebsiella pneumoniae, Proteus mirabilis, Listeria monocytogenes, Candida parapsilosis ve Candida albicans* mikroorganizmalarına karşı agar kuyucuk yöntemi ile ölçülmüştür. *Chromobacterium violaceum* bakterileri üzerinde ise çoğunluğu algılama inhibisyonu aktivitesi araştırılmıştır. En yüksek toplam fenolik ve demir indirgeyici antioksidan gücü, *U. filipendula* özütünde belirlenmiştir. Bu özüt, *S. aureus, K. pneumonia* ve *L. monocytogenes*'in büyümesini önleyebilmiştir. Ekstraktların hiçbiri çoğunluğu algılama mekanizmasını inhibe edememiştir.

Anahtar sözcükler: Antioksidan, antimikrobiyal, çoğunluğu algılama inhibisyonu, Usnea filipendula, Viscum albüm.

#### INTRODUCTION

The plants can be described as 'a gift of nature' because they are therapeutic. Many kinds of them have played an active role in the treatment of different diseases for centuries (Farombi, 2003). Although many drugs or medical methods have been applied to treat diseases by the development of technology and science, some governments have made it compulsory to consume natural products for many aims (Ertürk et al., 2004). Plants produce secondary metabolites in their bodies and it has been proven by many scientific studies that secondary metabolites have antioxidant, antimicrobial, anticancer, antidiabetic, etc. properties (Rao & Kingston, 1982; Mensor et al., 2001; Srinivasan, 2005; González-Lamothe et al., 2009; Kılıçkaya Selvi et al., 2019).

Some chemical reactions in body tissues produce free radical molecules under certain conditions. Free radicals occur naturally in every step of the duration. These molecules cause metabolic problems and play a role in bringing damage to tissues. However, these unstable electron-laden chemicals are largely destroyed or removed by natural antioxidant defence systems normally found in the body. The use of antioxidant plants/foods supports the body's antioxidant defence mechanism (Gate et al., 1999; Srinivasan, 2005). Antioxidants can be considered as two major groups; synthetic and natural, generally. Despite the synthetic ones have been used in many places, there is still suspicion about their reliability (Ho & Shahidi, 2005; Taghvaei & Jafari, 2015) because of their possible toxic/side effects especially during long-term intake (Taghvaei & Jafari, 2015). On the other hand, it has been indicated that many natural additives have more antioxidants property and thermal stability than the synthetic ones.

Increased technology, unlimited consumption demand and pollution have also increased/diversified the disease. As it is known some plants species have been used to overcome the microorganisms that cause diseases. The therapeutic effects of plants are related to the synergistic effect of a large number of compounds. It has been reported that the herbal combinations provide more effective treatment against the resistance of microorganisms that are difficult to kill with a single antibiotic (Sree et al., 2010; Nazri et al., 2011).

While quorum sensing (QS) is the communication system between the bacterial cells by the signaling molecules, anti-quorum sensing is the name of the stopping this communication mechanism (Alvarez et al., 2012). Over the past few years, QS has become a very extensive field of research because of its promising results for the utilizations in industry, medicine and biotechnology (Taganna et al., 2011). According to research of some scientists that "plants are rich natural resource of quorum sensing agents" (Choo et al., 2006; Kohand Tham, 2011; Mohamed et al., 2014; Al-Haidari et al., 2016). The most likely benefit of the QS researchers is to disrupt the signal communication between microorganisms' communities and to keep their growth under control. It was seen that there is not much study in the literature about the quorum sensing-disrupting activity of plants. Therefore, this study will reveal whether the lichen and mistletoe plants investigated in this article have antiquorum sensing activity or not.

It has been known that the lichens and mistletoe have some special bioactive properties. Bioactive natural products obtained from lichens have been utilized for medicinal and cosmetic purposes. Six lichen species involving usnic acid in various amounts were found to be effective against various (Cansaran et al., 2006; Yıldız, 2017). On the other hand, biologically active components of mistletoe have been reviewed (Ochocka and Piotrowski, 2002). Ertürk et al. (2004) investigated anti-microbial properties of mistletoe (*Viscum album* L.) against a fungus and six bacteria species. They explained that different concentration of n-hexane extract of mistletoe was effectual against micro-organisms analyzed.

The phenolic components of plant origins have attracted attention because of their useful and nutritional properties including antioxidant and antimicrobial capacity, in recent years (Bubonja-Sonje et al., 2011). The plants that are found in abundant quantities and inexpensive such as lichen or mistletoe need to be investigated, firstly. In this study it was examined the antioxidant, antimicrobial and antiquorum sensing activities of *Usnea filipendula* and *Viscum album*'s methanol extracts.

#### **MATERIAL and METHODS**

Lichen (*Usnea filipendula*) was collected from wild areas in Trabzon province, Tonya district (Fig. 1). Mistletoe (*Viscum album* L.) was collected from *Pinus sylvestris* host tree in Trabzon province, Sürmene district (Fig. 2), located in the north-eastern of Turkey (Table 1). The plants were brought to laboratory for extraction process.

Table 1. Investigated plant samples.

Sample	Scientific description	Collected place		
	Scientific classification	Province	District	
Mistletoe	Viscum album L.	Trabzon	Tonya	
Lichen	Usnea filipendula	Trabzon	Sürmene	



Figure 1. Lichen (Usnea filipendula)



Figure 2. Mistletoe (Viscum album)

Sample Preparation: Whole of lichen and twigs of mistletoe were used for the analyses. Samples were dried in an oven at 60°C at 24 hours before grinding. A laboratory scale Wiley mill was utilized to grind. Approximately 5 g powdered samples were dissolved in 50 mL methanol (99%). The mixture was continuously stirred using a shaker (Heidolph Promax 2020, Schwabach, Germany) at room temperature for 24 h. Particles were removed using Whatman No. 4 filter paper (pore size 20-25  $\mu$ m). Then the solutions were filter sterilized using 0.45  $\mu$ m hydrophilic polyvinylidene fluoride (PVDF) filters.

#### Antioxidant Properties

*Ferric Reducing Antioxidant Power (FRAP):* The antioxidant capacity was determined using ferric reducing antioxidant power (FRAP). This method is based on the reduction of tripyridyltriazine complex (Fe  $(TPTZ)^{3+}$ ) to blue colored Fe $(TPTZ)^{2+}$  by antioxidants in acidic medium (Benzie and Strain, 1996). FRAP values were expressed in wet weight of the samples as µmol of ferrous equivalent Fe (II) per g of sample.

**Determination of Phenolic Contents:** The polyphenolic contents of the methanol extracts were evaluated by three different ways; total phenolic contents (TPC), total flavonoids (TF) and condensed tannin (CT) contents. For the determination of the total phenolic contents, the Folin-Ciocalteau procedure was employed and gallic acid was used as standard (Slinkard & Singleton, 1977). The results were expressed as mg Gallic Acid Equivalent (GAE) per g of methanolic extracts.

*Determination of Flavonoid Contents:* The concentration of the total flavonoid content in the methanol extracts was measured using a spectrometric assay. The total flavonoid concentration was expressed as mg equivalents of quercetin (QE) per g of sample (Fukumoto & Mazza, 2000).

Determination of Condensed Tannins Contents: The concentration of condensed tannins was determined according to the method previously used by Julkunen-Titto (Julkunen-Tiitto, 1985). The results were expressed as mg catechin equivalent (CE) per g of sample.

Antimicrobial Activity: The extracts were tested for antimicrobial activity by agar-well diffusion method

according to the Clinical & Laboratory Standards Institute (CLSI) guidelines (Wayne, 2002) against Staphylococcus aureus ATCC 25923, Escherichia coli ATCC 25922, Enterococcus faecalis ATCC 29212, Pseudomonas aeruginosa ATCC 27853, Salmonella typhimurium ATCC 14028, Klebsiella pneumoniae ATCC 13883, Proteus mirabilis ATCC 7002, Listeria monocytogenes ATCC 43251, Candida parapsilosis ATCC 22019 and Candida albicans ATCC 10231. The microorganisms were obtained from Department of Medical Microbiology, Faculty of Medicine, Karadeniz Technical University, Trabzon, Turkey. Bacteria and yeast were cultured in Luria Bertani (LB) and Sabouroud Dextrose agar (LABM, UK), respectively. Fresh cultures (18 h) of bacteria and yeast were used to make suspension in 5 mL of sterile isotonic sodium chloride and turbidity was adjusted to 0.5 McFarland. Agar plates were filled with suspension and 0.6 cm agar wells were cut out using a sterile pipette tip. 50 microliters of extracts were transferred into each agar well and cultures were incubated at 37°C for 24 hours. Ampicillin, gentamicin, cefotaxime, tetracycline and amphotericin B solutions and DMSO were used as positive and negative controls, respectively. The antimicrobial activity was determined by visual inspection and measurement of the diameter of inhibition zones around the agar-wells. The minimal inhibitory concentration (MIC) of the extracts showing a positive antimicrobial activity was determined using the liquid microdilution test method. The well with the lowest concentration that did not show any microbial growth was considered to be the MIC of the tested extract.

Anti-Ouorum Sensing Activity: Anti-quorum sensing activity was determined using microdilution method as described for the antimicrobial activity test above (Damte et al., 2013). The anti-QS activity of the extracts has been tested against Chromobacterium violaceum ATCC 12472, a violacein-producing strain. Briefly, MIC of each extract was determined as described above and sub-MIC concentrations were used for the inhibition of pigment production of C. violaceum. For anti-QS assay, to the fresh culture of the strains in LB broth was added for each extract and incubated for 24 h. At the end of the incubation, 1 mL of culture was centrifuged and pellet was resuspended in 1 ml of DMSO and vortexed at the high speed for pigment extraction. Supernatant was removed and absorbance values of the pigments were determined at OD 585 nm using a microplate reader (Damte et al., 2013; Norizan et al., 2013). Vanilla extract was used as positive control (Choo et al., 2006).

*Statistical Analysis:* The data were presented as means and standard deviations of three replicates for total phenolic content and antioxidant properties and ten replicates for metal composition analyzed by using Statistical Package for Social Sciences (SPSS version 23.0). The data were analyzed by ANOVA and tests of statistical significance were performed using Duncan's multiple range tests.

## **RESULTS and DISCUSSION**

Antioxidant Activity: In this study, antioxidant capacity was determined using ferric reducing antioxidant power (FRAP) method. The results are given Figure 3.

As shown in Figure 3, antioxidant capacity of *U*. *filipendula* (54.4  $\mu$ molFeSO<sub>4</sub>.7H<sub>2</sub>O/g) was found higher than antioxidant capacity of *V*. *album* (51.45  $\mu$ molFeSO<sub>4</sub>.7H<sub>2</sub>O/g). Vicas et al. (2009) investigated the hydrophilic and lipophilic

antioxidant activities of *V. album.* For this purpose, they collected the *V. album* leaves and stems from five host trees (*Acer campestre, Malus domestica, Fraxinus excelsior, Populus nigra* and *Robinia pseudoacacia*) and determined the antioxidant activity of methanol and acetone extracts of all collected samples. They reported that methanol extract of *V. album* leaves collected from *M. domestica* exhibited the highest activity.



Figure 3. Antioxidant capacity of samples

Önay-Uçar et al. (2006) found that *V. album* living in different trees had different antioxidant activity. So, they reported that *V. album* extract's antioxidant capacity can vary depending on the plant's harvest time and the configuration of the main tree at the same time. In this study, *V. album* was collected from *P. sylvestris* host tree. Therefore, it can be concluded that antioxidant capacity of *V. album* collected from different host tree can be differ from our results. In a study, the reducing power activity of *V. album* crude alcoholic extract was reported at 0.10 equivalent 1mM FeSO<sub>4</sub> by (Papuc et al., 2010). It was noticed that extracts of *V. album* obtained from cashew tree demonstrated a stronger Fe chelating ability (Oluwaseun & Ganiyu, 2008).

Oran et al. (2016) studied the antioxidant capacity of different lichen species' (Usnea intermedia, Usnea filipendula and Usnea fulvoreagens) methanol and ethanol

extracts. They reported that methanol extracts of lichen species showed higher antioxidant capacity from the ethanol extracts.

All the previous studies and the present study are evaluated together, it can be concluded that the antioxidant capacity is affected many factors such as extract type and method, extract concentration, plant harvesting time, host tree (for mistletoe) etc.

*Total Phenolic Contents:* In this study, the polyphenolic contents of the methanol extracts were evaluated by three different ways; total phenolic content, total flavonoids and condensed tannin contents. The polyphenolic contents of the methanol extracts of samples are given in Figure 4.



Figure 4. The polyphenolic contents of the methanol extracts of samples.

In this study, total phenolic contents of *V. album* and *U. filipendula* was determined as 6.114 and 6.377 mg (GAE)/g, respectively (Fig. 4) and the results were found to be statistically significant each other (p<0.05).

In a study, total phenolic contents of *V. album* methanol extract were determined as 19.43 mg GAE/g dried weight (Sengul et al., 2009). Vicas et al. (2009) reported that the total phenolic content of *V. album* methanolic and acetonic extracts' which collected from 5 host tree were between 0.40-0.65 mg GAE/g fresh weight and 0.002-0.015 mg GAE/g fresh weigh, respectively. It can be concluded that the total phenolic content of the mistletoe collected from different host trees is also different. (Papuc et al., 2010) informed that the polyphenols of *V. album* ethanolic extracts as 6.33 mg/g dry plant. Our results are comparable with just mentioned study. Total phenolic content of *U. filipendula* acetone, ethanol and methanol extracts was reported that 329.7, 197.4 and 291.5 mg GAE/100 g<sup>-1</sup> of dried lichen, respectively (Oran et al., 2016).

*Total Flavonoid Contents:* In this study, total flavonoid content of *V. album* and *U. filipendula* was calculated as 2.73 and

1.17 mg (QE/g), respectively (Fig. 4). In a previous study, it was (Papuc et al., 2010) reported that the flavonoid content of *V. album* ethanolic extracts was 9.72 mg/g dry plant. It can be said that the solvent type affects the flavonoid content. There are some studies in the literature reported that some lichen species have important flavonoid contents (Kosanić et al., 2011).

**Condensed Tannins Contents:** In this study, condensed tannin content of *V. album* extract (2.304 mg (CE/g)) was found 4 times higher than the *U. filipendula* extract (0.575 mg (CE/g).

When all the polyphenolic contents and antioxidant capacity of methanol extracts of studied samples are evaluated together, the higher total phenolic content and antioxidant capacity was determined in *U. filipendula* extract while the higher total flavonoid content and condensed tannin content was determined in *V. album* extract.

**Antimicrobial Activity:** The antimicrobial activity of studied samples and used antibiotics are given Table 2.

Bacteria isolates	Agar Well Diffusion (mm zone diameter)						
Bucteria isolates	V. album	U. filipendula	Ampicillin	Gentamicin	Amphotericin B	Tetracycline	Cefotaxime
S. aureus	0	4	> 30	-	-	-	-
E. coli	0	0	16-17	-	-	-	-
P. aeruginosa	0	0	-	21-22	-	-	-
E. faecalis	0	0	>30	-	-	-	-
C. albicans	0	0	-	-	30		
C. parapsilosis	0	0	-	-	-	-	-
S. typhimurium	0	0	27	-	-	-	-
P. mirabilis	0	0	-	-	-	-	37
K. pneumoniae	2	1	-	-	-	-	-
L. monocytogenes	2	1	-	-	-	25	-

**Table 2.** Antimicrobial activity and used antibiotics

As can be seen in Table 2, *V. album* methanol extract inhibited *K. pneumoniae*, L. monocytogenes and *U. filipendula* methanol extract inhibited *S. aureus, K. pneumoniae* and L. monocytogenes microorganisms. Also, *V. album* extract was shown better antimicrobial zone than *U. filipendula* extract against *K. pneumoniae* and *L. monocytogenes* microorganisms.

Minimum inhibition concentration (MIC) values of extracts which show antimicrobial property are given in Table 3.

Table 3. Minimum inhibition concentration (MIC) values of extracts ( $\mu g/mL$ ).

Bacteria used in the test	V. album	U. filipendula	
S. aureus	-	312.5	
K. pneumoniae	1250	1250	
L. monocytogenes	625	625	

The lower MIC value means the stronger antimicrobial effect of extract. In this study, *U. filipendula* methanol extract has the best antimicrobial activity against *S*.

*aureus* microorganism with 312.5  $\mu$ g/mL concentration. It can be said that *V. album* extract was more effective than *U. filipendula* extract, because when same MIC values of extract was tested (Table 3), *V. album* extract showed higher (twice times) antimicrobial activity (mm zone diameter) from *U. filipendula* extract (Table 2).

Sengul et al. (2009) reported that both methanol and aqueous extracts of V. album inhibited many organisms. Methanol extracts showed better antimicrobial activity than aqueous extracts. In a study, it was investigated the antimicrobial activity of different extracts (acetone, petroleum ether, ethyl acetate, chloroform, ethanol, methanol, water) of V. album collected from Rialy, Muzaffarabad Azad Jammu and Kashmir. According to the reported results all extracts inhibited many of studied microorganisms except from acetone and petroleum ether extracts (Hussain et al., 2011). In another study; it was investigated that antimicrobial activity of V. album against 6 bacteria and 1 fungus (Bacillus Staphylococcus subtilis, aureus, Escherichia coli, Pseudomonas aeruginosa, Enterobacter cloacae, Proteus vulgaris and Candida albicans). The results showed that the

different concentrations of n-hexane extract of mistletoe were effective against micro-organisms analyzed (Ertürk et al., 2003).

Oran et al. (2016) reported that the MIC values of all analyzed extracts ranged from 64  $\mu$ g/mL to 512  $\mu$ g/mL for all the bacterial strains and all the Fluoro quinolone-resistant Escherichia coli isolates (except for E101) were sensitive to the methanol extracts of the three *Usnea filipendula*. In another study, it was reported that *U. filipendula* have antimutagenic and antigenotoxic effects.

Antiquorum Sensing Activity: The communication mechanism between microorganisms has called "quorum sensing" (QS). Anti-QS activity charts of positive control (vanilla) and *U. filipendula* and *V. album* are given Figure 5-7, respectively.



Figure 5. Anti-QS activity chart of vanilla.



Figure 6. Anti-QS activity chart of U. filipendula





Bacteria used in this study (*C. violaceum*) for anti-QS assay produces the purple pigment. If the extract we use is not killing bacteria or killing very little, the intensity of pigment production is not reduce, or if it is very low, it can be considered that used extract have anti-QS activity. A good functioning of this mentioned state is seen in the positive control extract (Fig. 5). Vanilla showed anti-QS activity from 312.5 to 625 µg/mL while inhibited the bacteria when 1250 µg/mL. In this study, both of *U. filipendula* and *V. album* inhibited the *C. violaceum* at 625 Mg/mL extract concentration so they did not show anti-QS activity.

Kenar et al. (2016) investigated that the methanolic and dicloromethanolic extracts of fruits, leaves, and stem of *V. album*. They used agar well and disc diffusion assay for anti-QS activity using *Chromobacterium violaceum* (CV12472 and CVO26) strains. They reported that the effect of *V. album* extracts on anti-biofilm and anti-QS was very effective over biofilms produced by pathogens and these extracts were good sources for new antimicrobial components.

#### CONCLUSION

In this study antioxidant, antimicrobial and antiquorum sensing activities of *U. filipendula* and *V album*'s methanol extracts were investigated. As a result; the higher total phenolic content and antioxidant capacity was determined in *U. filipendula* extract. The higher total flavonoid content and condensed tannin content was determined in *V. album* extract. *U. filipendula* methanol extract has the best antimicrobial activity against *S. aureus* microorganism with 312.5 µg/mL concentration. *V. album* extract was more effective than *U. filipendula* extract against *K. pneumoniae* and *L. monocytogenes*. Both of extracts inhibited *C. violaceum*, they did not show anti-QS activity.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# The Effect of Modification with Epoxy and Polyester Resins on Some Mechanical Properties of Pine and Chestnut Woods <sup>[\*]</sup>

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**Abstract:** In this study, the effects of epoxy and polyester resin applications on some mechanical strength properties of scots pine (*Pinus sylvestris* L.) and chestnut (*Castanea sativa*) wood samples were investigated. Firstly, in the circular sawing machine, a different numbers of channels (2+1 and 3+2) were opened on the surfaces of wood samples. Then, these channels were filled with casting type epoxy and polyester resins. The density, modulus of rupture (MOR), modulus of elasticity (MOE), and compression strength parallel to the grain (CS) tests were performed on the samples. According to the results of the study, the density values of pine and chestnut samples increased by 25% and 42%, respectively, depending on the number of channels after resin application. In addition, compared with epoxy resin applied samples, the density values of chestnut samples. However, CS values tend to decrease in these samples. In pine samples with resin applied, the strength properties (MOR, MOE and CS) decreased depending on the increase in the number of channels. Furthermore, the effect of the resin type on all strength properties of the samples was statistically insignificant.

Keywords: Epoxy resin, mechanical properties, polyester resin, wood material.

# Çam ve Kestane Odunlarının Bazı Mekanik Özellikleri Üzerine Epoksi ve Polyester Reçineler İle Modifikasyonun Etkisi

Öz: Bu çalışmada, sarıçam (*Pinus sylvestris* L.) ve kestane (*Castanea sativa*) odunu örneklerinin bazı mekanik direnç özellikleri üzerine epoksi ve polyester reçine uygulamalarının etkisi incelenmiştir. Öncelikle daire testere makinesinde örneklere farklı sayılarda (2+1 ve 3+2) kanallar açılmıştır. Daha sonra bu kanallar döküm tipi epoksi ve polyester reçineler ile doldurulmuştur. Örneklerde meydana gelen değişiklikleri belirlemek için hava kurusu yoğunluk, eğilme direnci (ED), elastikiyet modülü (EM) ve liflere paralel basınç direnci (BD) testleri gerçekleştirilmiştir. Çalışmanın sonuçlarına göre, kanal sayısı artışına bağlı olarak reçine uygulamalarından sonra çam ve kestane örneklerin yoğunluk değerleri sırası ile %25 ve %42'ye kadar artmıştır. Ayrıca, epoksi reçineye göre polyester reçine uygulanmış örneklerde yoğunluk artışı daha yüksektir. Kestane örneklerin ED ve EM direncinde reçine uygulama işlemlerinden sonra biraz artış gözlenmiştir. Ancak bu örneklerde BD direnci azalmıştır. Reçine uygulanmış olan çam örneklerde ise tüm mekanik direnç özellikleri (ED, EM ve BD) kanal sayısındaki artışa bağlı olarak azalmıştır. Ayrıca, mekanik direnç özellikler üzerinde reçine tipinin etkisi genel olarak önemsizdir.

Anahtar sözcükler: Ağaç malzeme, Epoksi reçine, Mekanik özellikler, Polyester reçine.
## INTRODUCTION

Wood is a material used by mankind since the dawn of time due to its high-quality properties. Wood which used to be used as a basic material in the construction of houses is now being used in designing the living spaces. Wood is indispensable both for living spaces and for its healthy and durable structure.

The warm image of natural wood let it be widely used in interior spaces, which led to new ideas. Since the beginning of the 21st century, interest in products such as wooden desks and tables has increased. When the products produced by natural wood are touched, they give the feeling of being just inside nature where the wood has been taken from. With concrete becoming more and more widespread, people's desire to get back to nature has motivated these designs to be even more involved in our life (Web-1). In recent years, products made from natural wood materials are very popular. Wood materials and various resins are generally used in combination in the manufacture of these products. Thus, very different types of designs are emerging and especially the aesthetic look is emphasized. In addition, with the use of resins, different wood defects can be removed. Many types of furniture and decoration elements are produced using natural wood. Some of these products are; table, coffee table, TV units, consoles, bookshelves, pendant lamps and wooden wall designs. In the production of these products, the most commonly used wood species are pine, maple, juniper, plane, walnut, oak, chestnut, mulberry, and ash. Epoxy and polyester resins are generally used to remove various wood defects (cracks, slits, knot fall, etc.) in furniture and decoration elements produced from natural wood. In addition, these resins are now often preferred for their aesthetic and decorative reasons, together with wood.

Epoxy resins can strongly adhere to many materials such as metal, plastic, glass, and wood (Bulmuş & Pişkin, 2000; Şahmetlioğlu, 2000). In addition, it has many important properties such as high mechanical strength, chemical resistance, corrosion resistance, dimensional stability, wetting and filling ability of fiber reinforcements (Kaw, 2005; Chung, 2010). Epoxy resins are used in marine, automotive, aerospace and construction industries due to their superior properties. It is also widely used in many applications such as coatings, adhesives, insulation materials and composite materials (Güzel, 2016). Polyester resins have high resistance to chemical and environmental influences. They also have high dimensional stability and low viscosity. They are less costly and harden faster than epoxy resins (Sezgin, 2018) but have higher shrinkage ratios after curing (Seyhan et al., 2007). Polyester resins are used in automotive industry, building constructions, marine applications and composite materials as a matrix. When compared to epoxy resin, their thermal, air and mechanical strength properties are low, so their use in high-performance composites is limited (Campbell, 2010; Sezgin, 2018).

The aim of this study is to determine the density and the mechanical strength properties of scots pine (*Pinus sylvestris* L.) and chestnut (*Castanea sativa*) woods modified with epoxy and polyester resins.

### **MATERIAL and METHODS**

**Wood material:** In this study, scotch pine (*Pinus sylvestris* L.) and chestnut (*Castanea sativa*) woods were used. Wood materials were supplied by random selection method from a timber company in the Düzce city in Turkey. Attention was paid to ensure that no rot, knot, crack, color, or density differences were present in the samples (TS 2470, 1976). A sufficient number of experimental samples were prepared with  $20 \times 20 \times 340$  mm (tangential direction × radial direction × longitudinal direction) dimensions from the sapwood parts of the wood materials which have air-dry moisture. Thereafter, a different number of channels (2+1 and 3+2) were opened with 2.4 mm wide and 7 mm depth to the samples subjected to be treated by resin, at corresponding pairs of surfaces (tangential section) using a circular saw machine (Figure 1).



Figure 1. Appearance of cross sections of the samples.

After opening channels, samples remained at a temperature of  $20\pm2$  °C and relative humidity of  $65\pm3\%$  until they reached a stable weight (TS 2471, 1976).

**Resin Application:** In the treatment of samples casting type epoxy and polyester resins were used. Some properties of these resins are given in Table 1.

Table 1. Properties of resins

Resin type	Viscosity (cpl)	Density (g/cm3) (20 °C)	Gelling time (min.)	Elongation at break (%)	Hardness (Barkol)
Epoxy	156	1.2	110-130	1.0	66
Polyester	450	1.6	8	2.3	45

Before the resin processing, cross sections of the experimental samples were closed using paper tape to prevent resin leakage. When the resins were prepared, the hardener and/or accelerator mixture ratios were carried out in accordance with the manufacturers' recommendations. The channels opening into the samples were filled with the resin using an injector with the appropriate tip clearance under room conditions ( $20 \pm 2$  °C) (Figure 2).



Figure 2. Application of resins to wood samples.

After filling the channels on the first surface with resin, the samples were left on the counter with a balanced scale for 48 hours. Then resin filling was performed on the channels on the second surface. The samples, in this case, were kept for about three weeks. After the resins had been thoroughly dried, the samples were passed through a calibrating sanding machine to remove excess resin on the surfaces. After, samples remained in a conditioning cabin (RH 65±3% and 20±2°C) until they reached a stable weight (TS 2471, 1976). Then, samples were cut into smaller samples according to the specified test standard. The test samples were prepared in the number as to eight repetitions (n = 8) for each variable.

**Determination of Density:** Air-dry density of the samples were determined according to TS 2472, (1976). The mass of each sample ( $M_{12}$ ) was measured on an analytical balance, with a sensitivity of ±0.01 g. Dimensions (length, width, thickness) were measured with a vernier caliper having ±0.01 mm sensitivity, and volumes ( $V_{12}$ ) were determined. The air-dry density ( $\delta_{12}$ ) was calculated using Eqs. 1.

$$\delta_{12} = M_{12} / V_{12} \quad (g/cm^3) \tag{1}$$

*Determination of MOR, MOE and CS Strength:* Modulus of rupture (MOR) (or bending strength) and modulus of elasticity (MOE) of the samples were determined according to TS 2474, (1976). The MOR and MOE values were calculated by using following Eqs. 2 and 3.

$$MOR = 3P_{max}L/2bd^2 \quad (N/mm^2)$$
<sup>(2)</sup>

$$MOE = PL^3 / 4bd^3 \Delta \qquad (N/mm^2) \tag{3}$$

Where  $P_{\text{max}}$  is the maximum load when the sample is broken (N), *L* is the supporting span (mm), *b* is the width of the samples (mm), *d* is the thickness (depth) of the samples (mm),  $\Delta$  deflection at mid-length below the proportion deflection limit (mm), and *P* is the load in N within the proportional deflection (N).

Compression strength parallel to the grain (CS) of the samples were determined according to ISO/DIS 13061-

17, (2015). The CS values were calculated by using following Eqs. 4.

$$CS = P_{max} / bd \qquad (N/mm^2) \tag{4}$$

where  $P_{\text{max}}$  is the maximum load applied to the samples (N), *b* is the width of the samples (mm), *d* is the thickness of the samples (mm).

*Statistical Analysis:* The MSTAT-C software program was used for the evaluation of data. ANOVA (analysis of variance) tests were performed to determine the effect of resin type and number of channel on the density and mechanical properties (MOR, MOE and CS) of scotch pine and chestnut woods at the 0.05 significance level. Duncan's tests were conducted for comparisons of the means of the density and mechanical strength values of the wood samples.

#### **RESULTS and DISCUSSION**

Arithmetic means of density, modulus of rupture (MOR), modulus of elasticity (MOE), and compression strength parallel to the grain (CS) values of the wood samples are given in Table 2. Additionally, analysis of variance results of density, MOR, MOE, and CS measurements from wood samples resin applied are shown in Table 3.

According to Table 3, the effects of resin type and number of channel factors on density in both wood species were found to be statistically significant. In addition, the effect of number of channel on MOR, MOE, and CS of the pine wood samples was significant, while the effect of the resin type was insignificant. On the other hand, both resin type and effect of number of channel on MOR, MOE, and CS of the chestnut wood samples were found to be insignificant ( $P \le 0.05$ ). In the pine and chestnut woods, comparison results of the Duncan's tests conducted for the factors of resin type and number of channel are given in Table 4.

Regarding resin type, the highest density for both wood species was found to be in the samples applied polyester resin and the lowest was found in the samples applied epoxy resin (Table 4). It is shown in Table 1 that the polyester resin used in the work has a higher density than the epoxy resin. With respect to number of channel, the highest density was found to be in the 3+2 channels opened samples, while the lowest was obtained in the control samples (Table 4). Depending on the increase in the number of channels, the density value of the samples also increased. In other words, the increase in the amount of resin applied to wood materials caused an increase in density. This can be explained by the fact that the densities of the resin filled in the opened channels are higher than the density of the wood materials. After resin application, density value of pine and chestnut samples increased up to 25% and 42% compared to control samples (Table 2).

#### Table 2. Arithmetic means of the density, MOR, MOE and CS values.

Wood	Desin trms	Number of		Te	sts	
Wood species	Kesin type	channel	Density (g/cm <sup>3</sup> )	MOR (N/mm <sup>2</sup> )	MOE (N/mm <sup>2</sup> )	CS (N/mm <sup>2</sup> )
		Control	0.545 (0.022)	95.69 (7.34)	10537 (758)	46.59 (3.57)
	Epoxy	2 + 1	0.611 (0.024)	87.73 (8.46)	10019 (684)	40.29 (3.72)
Sootah mina		3 + 2	0.660 (0.025)	89.51 (8.51)	9598 (724)	38.25 (3.16)
Scotch pine		Control	0.545 (0.022)	95.69 (7.34)	10537 (758)	46.59 (3.57)
	Polyester	2 + 1	0.636 (0.022)	94.75 (7.31)	9827 (636)	40.57 (3.29)
		3 + 2	0.682 (0.031)	86.47 (6.98)	8709 (631)	37.10 (2.51)
		Control	0.438 (0.017)	55.61 (6.14)	4630 (354)	32.24 (2.13)
	Epoxy	2 + 1	0.537 (0.010)	59.35 (5.71)	4659 (265)	31.34 (1.57)
Charter		3 + 2	0.614 (0.007)	61.34 (3.59)	4760 (169)	31.09 (1.22)
Chestnut		Control	0.438 (0.017)	55.61 (6.14)	4630 (354)	32.24 (2.13)
	Polyester	2 + 1	0.556 (0.023)	60.35 (7.10)	4887 (439)	31.25 (1.70)
	•	3 + 2	0.622 (0.018)	55.22 (7.31)	4541 (447)	30.50 (1.58)

Values in parenthesis are standard deviations.

Table 3. Analysis of variance results for density, MOE, MOR, and CS of resin applied wood samples

Weederseine	Course	Density		MOR		MOE		CS	
wood species	Source	F-ratio	p-value	F-ratio	p-value	F-ratio	p-value	F-ratio	p-value
	Factor A	4.871	0.032*	0.372	-	3.365	0.073	0.091	-
Scotch pine	Factor B	106.472	0.000*	4.239	0.021*	16.593	0.000*	30.541	0.000*
-	A*B	1.241	0.299	1.884	0.164	1.889	0.163	0.207	-
	Factor A	4.018	0.050*	0.933	-	0.001	-	0.200	-
Chestnut	Factor B	496.561	0.000*	1.967	0.152	0.773	-	2.819	0.071
	A*B	1.482	0.238	1.587	0.216	1.616	0.210	0.132	-

A: Resin type, B: Number of channel, \*:Significant at 95% confidence level.

#### Table 4. Duncan's test results for mean values

Wood species	Factor	Density (g/cm <sup>3</sup> )		MOR (N/m	m <sup>2</sup> )	MOE (N/mm <sup>2</sup> ) CS (N/mm <sup>2</sup> )			
	Factor	Mean	HG	Mean	HG	Mean	HG	Mean	HG
	Resin type								
	Epoxy	0.606	b	90.98	a	10051	a	41.71	a
	Polyester	0.621	a	92.30	a	9691	a	41.42	a
Scotch pine	Number of channel								
	Control	0.545	с	95.69	a	10537	a	46.59	а
	2 + 1	0.623	b	91.24	ab	9923	b	40.43	b
	3 + 2	0.671	a	87.99	b	9154	c	37.67	с
	Resin type								
	Epoxy	0.529	b	58.77	a	4683	a	31.56	а
	Polyester	0.539	a	57.06	a	4686	a	31.33	а
Chestnut	Number of channel								
	Control	0.438	с	55.61	a	4630	a	32.24	а
	2 + 1	0.547	b	59.85	a	4773	а	31.29	ab
	3 + 2	0.618	a	58.28	a	4650	а	30.80	b

HG: Homogeneous group (different letters denote a significant difference).

According to Table 4, the difference between the MOR values of pine and chestnut wood samples at the resin type level was found to be statistically insignificant. The same applies to MOE and CS values. For pine wood samples, the highest MOR, MOE, and CS values at the number of channel level were found to be in the control samples, while the lowest values were obtained in the 3+2 channels opened samples. The increase in the number of channels caused a decrease in the mechanical strength properties (MOR, MOE, and CS) of the pine wood samples. MOR, MOE, and CS values decreased by 8%, 13%, and 19%, respectively, in the 3+2 channels opened samples. The increase in the number of channels in the samples. The increase in the number of channels in the samples.

Because of this, it may be effective in decreasing the strength values of pine samples in resins which are harder and brittle than wood materials. In the literature, it is reported that the resulting epoxy or polyester resins in liquid form transform into solid with a layer that is a hard, brittle and thermosetting cross-linked structure (Velde, 1992; Miyoshi, 2001; Sönmez & Budakçı, 2004; Campbell, 2010; Ting et al., 2011). Also, their structures which are prone to brittle (Ersoy, 2001), combustible and fragile, restrict the use of such resins (Güzel, 2016). Hardened resin layer's physical and mechanical properties depend on the functional group types of hardener, cross-link density between resin and hardener, molecular structure and hardening conditions of functional group bonds

between resin and hardener (Şahmetlioğlu, 2000; Nohales et al., 2006; Montero et al., 2013; Güzel, 2016).

For the chestnut wood samples, no statistically significant difference was observed in terms of both MOR and MOE values between resin applied (channel opened) samples and control (no channel opened) samples (Table 4). It can be said that the chestnut wood samples and the resins exhibited similar behavior in terms of strength properties. In addition, with respect to number of channel, the highest CS value was obtained in the control samples, while the lowest value was found to be in the 3+2 channels opened samples. The CS value of chestnut wood samples tends to decrease due to the increase in the number of channel. CS value decreased by 4.5% in the 3+2 channels opened samples compared to control chestnut wood samples (Table 4).

## CONCLUSION

In this study, the density and the some mechanical strength properties of pine and chestnut wood samples modified with epoxy and polyester resins were investigated. After resin applications, the density values of pine and chestnut samples increased up to 25% and 42%, respectively, depending on the number of channels. Compared to epoxy resin applied samples, the density increase was higher in polyester resin applied samples.

The effect of the type of resin on all selected mechanical properties (MOR, MOE, and CS) of pine and chestnut wood samples was found to be statistically insignificant. In other words, similar strength values were obtained in both epoxy and polyester resin applied samples.

In pine wood samples with resin applied, the strength values decrease depending on the increase in the number of channels. The MOR, MOE and CS values of these samples decrease by 8%, 13%, 19% respectively when compared to the control (untreated) pine samples. In chestnut samples, the effect of the number of channels on the mechanical strength was found to be statistically insignificant.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

Effect of Cement and Accelerator Types on the Physico-Mechanical Properties of Cement-Bonded Particleboards<sup>[\*]</sup>

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**Abstract:** In this study, it is aimed to determine the effect of the use of different types cement and accelerator on the physicomechanical properties of cement-bonded particleboards. Within this scope, two types of cements (calcium aluminate cement and Super white CEM I 52.5 R) and accelerators (aluminum sulfate and calcium chloride) were used in the production of boards. Therefore, CBPBs with 1200 kg/m<sup>3</sup> target density and 1/2.75 wood-cement ratio were produced. Based on cement weight 1.5% accelerators were used. The test results obtained were evaluated according to EN 634-2 (2009). According to result, density values of the boards were changed with using depending on cement and accelerator types. The use of super white cement and calcium chloride positively affected the both mechanical properties and dimensional stability of the boards. The use of calcium aluminate cement and aluminum sulfate resulted in lower strength properties.

Keywords: Cement-bonded particleboard, cement types, accelerator, physico-mechanical properties.

# Çimento ve Priz Hızlandırıcı Tipinin Çimentolu Yongalevhaların Fiziksel ve Mekanik Özellikleri Üzerine Etkisi

Öz: Bu çalışmada farklı tip çimento ve priz hızlandırıcı kullanımının çimentolu yongalevhaların fiziksel ve mekanik özellikleri üzerine etkisi araştırılmıştır. Bu kapsamda yobgalevhaların üretiminde iki tip çimento (kalsiyum alüminat çimento ve Süper beyaz CEM I 52.5 R) ve priz hızlandırıcı (alüminyum sülfat ve kalsiyum klorür) kullanılmıştır. Üretilen levhaların hedef yoğunluğu 1200 kg/m<sup>3</sup> ve odun-çimento oranı 1/2.75'dir. Çimento ağırlığına göre %1.5 priz hızlandırıcı kullanılmıştır. Elde edilen test sonuçları EN 634-2 (2009)'a göre değerlenirmiştir. Elde edilen sonuçlara göre levhaların yoğunluğu çimento ve priz hızlandırıcı türüne göre farklılık göstermiştir. Süper beyaz çimento ve kalsiyum klorür kullanımı, levhaların hem mekanik özelliklerini hem de boyutsal stabilitesini olumlu yönde etkilemiştir Kalsiyum alüminat çimento ve alüminyum sülfat kullanımı, daha düşük direnç özellikleri ile sonuçlanmıştır.

Anahtar sözcükler: Çimentolu yongalevhalar, Çimento türü, Priz hızlandırıcı, fiziksel ve mekanik özellikler.

## INTRODUCTION

Wood composites are products obtained by combining wood (fiber, particle or particle) with other materials (plastics, synthetic fibers, glue, fillers, cement etc.) under temperature and pressure (Moloney, 1993). Nowadays, the decline of forest areas and the increase in wood prices, as well as the development of the chemical and adhesive industry, have evolved in wood composite materials (Ayrılmıs et al., 2012).

Cement-Bonded Particleboards (CBPBs) are much more resistant to fire, fungi and insects, as well as strong resistance to outdoor weather conditions, sound and heat insulation and wood composites are advantageous both in terms of production line and binder cost. They are advantageous both in terms of production line and binder cost. Moreover, the ability of the cement to self-harden makes the production process more economical. Because simple machines can be used during the production phase and high temperature pressing is not required. Cement is also a much cheaper binder than adhesives. Therefore, the use of wood-cement composites is increasing (Tittelein et al., 2012).

Depending on the developments in concrete technology, high-performance concretes (HPC), which are superior in terms of strength and durability, are currently being developed from concrete produced from traditional portland cement (NSC) (Khaliq & Khan, 2015). The calcium aluminate cement (CAC) is composed of calcium oxide and alumina oxides. It has applications not only in infrastructure works such as sewerage networks but also in hydraulic dams where wear resistance is required at the same time. It is also a used as refractory material in kilns and steel industry (Karadeniz et al., 2007). The studies have shown that concretes produced with CAC are resistant to aggressive environmental conditions and corrosion (Scrivener at al., 1999). While the super white cement (SWC) gives a very high early strength compared to the gray cement with the superior strength characteristic. Stabilization value allows the production of stabilized products. In addition, the Product provides advantages in cement dosage as less cement is used to achieve the same target strength values. Super white cement is highly resistant to alkaline-silica reactions and has a long service life (Web-1, 2018)

Compatibility in wood-cement composites can be expressed as the hardening level of the cement after mixing water and a certain amount of wood with cement. It is stated that when the cement is mixed with the wood, it is compatible if there is no restriction in cement hardening, otherwise it is incompatible. The extractive substances and sugars in the wood delay the cement hydration and cause the crystal structures to change (Jorge et al., 2004). So the accelerators are used in wood-cement composites to reduce the adverse effect of wood on the cement hydration reaction. The studies have shown that different types of accelerators are significantly effective on hydration heat changes and technological properties of cementitious wood composites (Semple et al., 2000; Soriano et al., 2000; Yel, 2014).

Therefore, the main goal of this work was to find out the possibility of using calcium aluminate and super white cement for making cement-bonded particleboards. In addition, the effects of accelerator types on some mechanical and physical properties of boards produced have been investigated.

#### **MATERIAL and METHODS**

*Materials:* The particles obtained from the poplar woods (*Populus Tremula L.*) were used in production of the boards. It was supplied as sawmill wastes by Trabzon Organized Industrial Zone, Turkey. ISIDAÇ 40 (calcium aluminate cement) and Super White CEM I 52.5 R cements used in the production were supplied by Çimsa Cement Industry and Trade Co., Turkey. The chemical properties of the CAC and SWC are given in Table 1.

Table 1. Chemical properties of the CAC and SWC.

Chemical properties (%)	CAC	SWC
SiO <sub>2</sub>	3,60	21,6
Al <sub>2</sub> O <sub>3</sub>	39,80	4,05
Fe <sub>2</sub> O <sub>3</sub>	17,05	0,26
CaO	36,20	65,7
MgO	0,65	1,30
SO <sub>3</sub>	0,04	3,30
Loss of ignition	0,30	3,20
Na <sub>2</sub> O	0,16	0,30
Chloride (CI-)	0,009	0,01

*Methods:* Firstly, sawmill wastes chipped using a drum chipper before grinded into smaller particles in a knife ring flaker. Then the wood particles were classified using a laboratory type-vibrating screen. The particle size used in production is 0.5-3 mm. The wood-cement ratios were 1/2.75 based on the oven dry weight for the single layer CBPB manufacture. Solid Aluminum sulphate  $(Al_2(SO_4)_3)$ , calcium chloride(CaCI<sub>2</sub>) was prepared 25% solution and the mixture was added. 1.5% used on both accelerator types based on the cement weight. Hand formed mats were compressed in a laboratory type hot press using a pressure of 18-20 kg/cm<sup>3</sup> for 4hrs. The press temperature is 60 °C. The amount of water used in production is determined according to the following formula (Simatumpang, 1979),

Water (liter) = 
$$0.35C + (0.30-MC)W$$
 (1)

where C is the cement weight (kg), MC is moisture content (oven dry basis) of wood particles, and W is oven dry wood particle weight (kg). The dimensions of the CBPBs were 42,5 x 42,5 x 1 cm. The target density was 1200 g/cm<sup>3</sup>. Two panels were made for each group. After pressing, the particleboards were conditioned for 30 days at a temperature of  $20 \pm 2^{\circ}$ C and  $65 \pm 5\%$  relative humidity and then cut to obtain test samples according to the European Standards. Experimental design is given in Table 2.

Board types	Cement types	Accelerator types
А	CAC	$Al_2(SO_4)_3$
В	CAC	CaCI <sub>2</sub>
С	SWC	$Al_2(SO_4)_3$
D	SWC	CaCI <sub>2</sub>

Density (D), water absorption (WA) and thickness swelling (TS), modulus of elasticity (MOE), modulus of rupture (MOR), internal bonding strength (IB) and screw withdrawal strength (WS) properties of the produced boards were determined according to EN 323, ASTM D1037, EN 317, EN 310, EN 319, EN 320, respectively. The test results obtained were evaluated according to EN 634-2 (2009). The data were analysed using SPSS 22 procedure for the analysis of variance (ANOVA) at 95% confident level (P  $\leq$  0.05). Duncan test was performed to determine the difference between the groups. The general view of the CBPBs produced is given in figure 1.



Figure 1. General view of produced CBPBs.

#### **RESULTS and USSION**

Physical properties of the cement bonded particleboards are presented in figure 2. The physical properties of the plates using SWC and ABC were higher when the results were examined. It was determined that the MC of the boards produced with  $Al_2(SO_4)_3$  was higher than those produced with  $CaCI_2$ . This may be due to the hygroscopic nature of the  $Al_2(SO_4)_3$  and its inability to form sufficient bonds with the wood phenol groups. According to EN 634-1 (1999); the MC of the CBPB should be between 6% and 12%. MC values of all board groups conform to the standard. The D values were found particularly higher in boards produced using SWC. It has been determined that the thicknesses of the boards are very effective on the D values. It has been found that the CAC produced boards have much more spring back than the SWC At the end of the 4 hour pressing period. Zhou & Kandem (2002) have determined that thickness changes due to spring back after pressing process. It has been found that the use of CaCI<sub>2</sub> in wood cement composites produced using waste railway sleepers reduced the WA values (Ashori, 2012). The maximum TS of CBPB should be 1,5% according to EN 634-2 (2009) standard. TS values of boards produced with SWC conformed to the EN standard. The dimensional stability of the boards was greatly increased with the use of SWC. The use of SWC in boards increased dimensional stability compared to CAC.



Figure 2. Properties of physical properties of the CBPBs.

Mechanical properties of the CBPBs are presented in figure 3. It was determined that the curing accelerator and cement types affected the mechanical properties of the boards. The MOR, MOE, IB and WS values of CBPB increased parallel with using CaCl<sub>2</sub> and SWC. The MOR and MOE values should be 9 N/mm<sup>2</sup> and 4000 N/mm<sup>2</sup> according to EN 634-2 standard. The MOR and MOE values of boards produced with SWC conformed to the EN standard. The use of SWC and CaCl<sub>2</sub> increased the MOR and MOE by 40% and 45%, respectively. The Al<sup>+3</sup> ion in the Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> complexes with polyphenol groups in the wood to prevent the delay of cement hydration. However, CaCl<sub>2</sub> has a more inhibitory effect on polyphenols (Yousuf, 1995). Therefore, the use of CaCl<sub>2</sub> in the boards resulted in higher mechanical properties. The SWC provides superiority to NSC with advantages such as aesthetics, high early strength, low alkalinity, durability and faster processing. It has been determined that the same performance is achieved even when using less than 50% of the NSC. When the dosage rate is increased, very high compressive strengths are obtained (Delibaş & Kırca, 2017).



Figure 3. Properties of mechanical properties of the CBPBs.

IB values should be 0,5 N/mm<sup>2</sup> according to EN 634-2 standard. Except for A type of the panels, other groups met the required level of IB values. The WS values are highest when CACl<sub>2</sub> and SWC are used. The SWC is a very hydraulically reactive linker due to the high content of  $C_3A$  and  $C_3S$  compounds in the structure. It has much higher performance than NSC due to its high reaction rate and gains earlier strength (Web-2, 2018). Cement and accelerator types have a significant effect on physical and mechanical properties based on the results of statistical analysis. The results of homogenous subsets of the board were given in Table 3.

 Table 3. Homogenous subsets for physico-mechanical properties of boards.

Board types	MC	D	WA	TS	MOR	MOE	IB	WS
А	а	b	d	d	d	d	d	d
В	b	b	с	с	с	с	с	c
С	b	a	b	b	b	b	b	b
D	b	а	a	a	а	а	а	а

### CONCLUSION

The effects of cement and accelerator types on the physical and mechanical properties of CBPBs were investigated. Nowadays, the use of recycled materials is of great importance due to the decreasing natural resources. It has been determined that poplar sawmill waste used for this purpose is suitable for wood cement composites. Cement and accelerator types and their interaction significantly influenced the strength of CBPBs. It has been determined that the use of CAC is not suitable for CBPB for the under current production conditions. Therefore, the pressing time can be extended to produce with CAC. In addition, the laying height of boards produced using CAC is less than on SWC. This also leads to a sufficient bond between particle and cement due to the reduction in the amount of connecting. On the contrary, SWC has been found to be very suitable for CBPB production. CaCl<sub>2</sub> has been more compatible with CBPBs and improved properties as an accelerator type. The use of CaCl<sub>2</sub> increased the compatibility of cement with wood particles (figure 1).

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#### ARAŞTIRMA MAKALESİ

**RESEARCH PAPER** 

# Some Properties of Fiberboard Manufactured with Wastes of Kraft Pulp Mill [\*]

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**Abstract:** In this study, it was aimed to investigate the usability of some wastes such as wood chip screening rejects and kraft pulp screening rejects as fibrous materials in the manufacture of medium density fiberboard (MDF). Chip screening rejects were refined with a laboratory type refiner; however, pulp screening rejects were not refined. These materials were added to commercial fibers with the rates of 10%, 30%. Fiberboards were manufactured using urea formaldehyde (UF) adhesive. Some properties of these fiberboards such as water absorption (WA), thickness swelling (TS), surface roughness parameters, color change, modulus of rupture (MOR) and modulus of elasticity (MOE) values were determined. As a result of these, it was found that all values showed different trend depending on rates and types of Kraft mill wastes. Generally, the use of these materials had positive effects on the panel properties.

Keywords: Fiberboard, kraft process, pulp fiber, screening rejects, wood chips.

# Kraft Hamuru Fabrika Atıkları ile Üretilen Lif levhaların Bazı Özellikleri

Öz: Bu çalışmada, odun yonga eleme atıklarının ve kraft hamuru elek atıklarının lifsel materyaller olarak orta yoğunlukta lif levha (MDF) üretiminde kullanımının uygunluğu araştırılmıştır. Yonga eleme atıkları, laboratuvar tipi bir rafinörde liflendirilirken; hamur eleme atıkları ise rafinör işlemine uğratılmadan kullanılmıştır. Elde edilen lifsel materyaller ticari liflere %10 ve %30 oranlarında ilave edilmiş ve üre formaldehit (UF) tutkalı kullanılarak lif levha üretimleri gerçekleştirilmiştir. Üretilen bu levhaların su alma (SA), kalınlığına şişme (KŞ) değerleri, yüzey pürüzlülük parametreleri, renk değişim değerleri, eğilme direnci (MOR) ve eğilmede elastikiyet modülü (MOE) gibi bazı özellikleri belirlenmiştir. Bu çalışmanın sonucu olarak, tüm değerlerin, kraft fabrika atıklarının türü ve kullanım oranına bağlı olarak değişim gösterdiği belirlenmiştir. Genel olarak, bu atık materyallerin kullanımının levha özellikleri üzerinde olumlu yönde etkisinin olduğu tespit edilmiştir.

Anahtar sözcükler: Eleme atıkları, hamur lifi, kraft prosesi, liflevha, odun yongaları.

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## INTRODUCTION

The growing scarcity of raw materials and disruptions of industrial waste management are some of the main problems that cause inevitable negative effects for all industries and need to be solved with economically and reasonably.

Like other industries, the MDF industry faces the same problems and is also negatively affected by the decrease in the availability of raw materials (Akgül & Tozluoğlu, 2008). This important problem has led researchers to search alternative sources. Residues or by-products of forest, wood or paper products such as annual plants, harvesting residues, agricultural wastes, lumber plant wastes, furniture plant wastes, wood shavings or shreds of paper etc. can reuse as raw material (Akgül & Çamlibel, 2008).

Kraft is the most common chemical pulping method used in the worldwide (Vaaler & Moe, 2001; Enqvist, 2006). Plenty of wastes such as barks, pins, fines, oversize and overthick wood chips of screening, pulp and paper sludge, pulp screening reject, black liquor, etc. occur during all stages of this process (Gavrilescu, 2004; Bajpai, 2015). Although these wastes are considered as a problem, they could be useful for many industries as resources.

The size and shape of wood chips are very important in chemical pulping and especially in kraft pulping (Gullichsen, 1999). Unsuitable wood chips cause some problems such as pulping chemical penetration and lower pulp yield (Gullichsen, 1999; Eriksen et al., 1981; Tian, 2017). Acceptable sizes of wood chips in kraft process are approximately 15-25 mm length, a width of 20 mm and 3-5 mm thickness (Gerald, 2006). In kraft pulping process, the oversized wood chips are rejected for pulping and sent to rechipping while the fines are sent to burn for heat production (Bajpai, 2010). It is possible to produce refiner mechanical pulp (RMP) with small size of wood chips such as sawdust and shavings which are less suitable for kraft pulping due to their bulk (Lewis, 1971). Low cost, high values of yield (range of 85-95%), brightness, light scattering properties, smoothness, bulk and good formation are the major advantages of mechanical pulps (Bierman, 1996; Sundholm, 1999).

The pulp screening reject is another waste of the kraft pulping process. After separation of black liquor from the fiber, pulp screening performs to separate the undercooked, the coarser and non fibrilized fiber and fiber bundles from the pulp to produce accepted high-value pulp (Tikka et al., 1993; Kırcı, 2000; Hart, 2011). Rejects from the pulp screening process are usually refined, screened and the final rejects are thickened and burned (Tikka et al., 1993; Bierman, 1996; Hart, 2011).

The objective of this study was to evaluate the usability of small size of wood chips, which are not suitable in sizes for pulping standards and pulp screening rejects obtained from kraft process, for the manufacture of medium density fiberboard (MDF) and determine the some properties such as water absorption (WA), thickness swelling (TS), surface roughness parameters, color change, modulus of rupture (MOR), modulus of elasticity for these MDF panels.

#### **MATERIAL and METHODS**

In this study, commercial fibers (mixture of pine and beech) were used as raw material. Also, refiner mechanical pulp (RMP) fibers produced from wood chip screening rejects (the pin-chips) and kraft pulp screening rejects (PSR) obtained from Kraft pulp mill were taken as additive raw materials to commercial fibers.

Wood chip screening rejects (pin-chips) were refined by using disc refiner in a laboratory scale for refiner mechanical pulp (RMP) fibers. These fibers were air-dried and separated using laboratory mixer with 18.000 rpm speed for 2 minutes. Refiner mechanical pulp (RMP) fibers and pulp screening reject (PSR) fibers were mixed into commercial fibers with the additional rates of 10% and 30% for panel manufacturing. Fibers were dried to 2-3% moisture content. Urea formaldehyde (UF) at 12% rate was used as an adhesive. Paraffin emulsion as water repellent and ammonium chloride as hardener at 1% rates were added to UF adhesive. After the application of the adhesive, manually formed fiber mats were pressed at the hot press at 180 °C temperature for 7 min. Fiberboards were manufactured with 8 mm thickness and 750 kg/m3 target density. These MDF panels were conditioned at 65  $\pm$  5% RH and 20  $\pm$  1 °C, in accordance with TS-642-ISO 554 (1997) and dimensioned for the tests according to TS-EN 326-1 (1999). Panel types and contents were represented in Table 1.

Table 1. Panel	types	and	contents.
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Panel type	Content
A1	90% fiber +10 %RMP*
A2	70% fiber +30 % RMP*
B1	90% fiber +10 %PSR**
B2	70% fiber +30 %PSR**
Control	100% fiber

\*RMP: Refiner mechanical pulp, \*\*PSR: Pulp screening rejec.

*Water Absorption and Thickness Swelling*: The water absorption (WA) and thickness swelling (TS) values of MDF samples for 2h-24h were determined according to EN 317 (1993) standard.

*Modulus of Rupture and Modulus of Elasticity:* The modulus of rupture (MOR) and modulus of elasticity (MOE) values of MDF samples were determined according to EN 310 (1993) standard.

*Surface Roughness Parameters:* The surface roughness parameters such as *Ra*, *Rq* and *Rz* of MDF samples

were measured using Mitutoya Surftest SJ-210 instrument according to DIN 4768 (1990) standart.

**Color Measurements:** The color measurements of the MDF samples were carried out by using Konica Minolta CM-2600d spectrophotometer according to the CIE  $L^*a^*b^*$  system (HunterLab, 2008). The  $\Delta a^*$ ,  $\Delta b^*$ ,  $\Delta L^*$  and total color change ( $\Delta E^*$ ) of the MDF samples were determined.

## **RESULTS and DISCUSSION**

*Water Absorption and Thickness Swelling:* The water absorption (WA) and thickness swelling (TS) values of MDF samples after water immersion for 2 and 24 h are represented in Figure 1 and 2, respectively.



**Figure 1.** (a)-WA values of MDF samples for 2h, (b)- WA values of MDF samples for 24h.

As can be seen in Figure 1a and 1b, average WA values changed depend on the type and rate of fibers. WA values of MDF samples for 2-24 h were found lower than WA values of control samples except for A2 group (2h) manufactured with rate of 30% RMP fibers. This group had slightly higher value. B1 group was found having the lowest WA values for both 2-24h. The best results were obtained with B group manufactured with PSR fibers compared to A group manufactured with RMP fibers. This situation could be reasoned because of fiber properties. The pulping process has an important effect on fiber properties (Clark, 1985; Smook, 2002; Migneault et.al., 2010). According to Luukko and Maloney (1996), mechanical pulp fibers are prone to swelling because of beaten fines.



**Figure 2.** (a)-TS values of MDF samples for 2h (b) - TS values of MDF samples for 24h

As can be seen in Figure 2a and 2b, average TS values of MDF samples showed similar trend with WA values of MDF samples. These values also changed depend on the type and rate of fibers. TS values(2-24h) of A and B groups manufactured with RMP and PSR fibers were found more lower than those of control samples except for A2

group manufactured with rate of 30% RMP fiber (2h). A notable decrease was observed on the TS values (2-24h) of B1 groups manufactured with 10% addition of PSR fibers. Generally, higher TS values were obtained with RMP fibers. This could be attributed to the increased amount of fine in mechanical pulp.

*Modulus of Rupture and Modulus of Elasticity:* The modulus of rupture (MOR) and modulus of elasticity (MOE) values of MDF samples are represented in Figure 3 and 4, respectively.



Figure 3. MOR values of MDF samples.

As represented in Figure 3, the type and rate of raw material showed notable effect on the MOR values of MDF samples. All MOR values were recorded to be higher than control values. These values increased with increasing rate of RMP and PSR fibers. The better results were recorded with B group compared to A group. The highest MOR value of MDF samples was obtained from B2 group manufactured with rate of 30% PSR fibers. The addition of PSR fibers provided more improvement than the addition of RMP fibers on the MOR values of MDF samples.



Figure 4. MOE values of MDF samples.

As can be seen in Figure 4, MOE values showed similar tendency with MOR values of MDF samples. All MOE values of the samples clearly improved as rates of RMP and PSR fibers were increased from 10% to 30%. The highest MOE value of MDF samples was obtained from B2 group manufactured with rate of 30% PSR fibers.

Evaluating both MOR and MOE values together; it is clearly seen that the rate and type of fibers had an

important effect on these values. All MOR and MOE values of A and B groups were found higher than those of control groups. PSR fibers provided better results. These results could be attributed to the structural properties of the fibers. PSR fibers are longer than RMP fibers because of their production process. It is well known that fiber length is a crucial parameter. Long fiber can have more fiber joints and therefore this could affect the strength properties of the final material. Also, chemical pulp fibers are more flexible compared to mechanical pulp fibers (Johansonn, 2011). Similar trend was observed by Nourbakhsh & Ashori (2009). According to their study, the usage of long fibers with high aspect ratio is one of the considerable parameters controlling the mechanical properties of composites.

#### **Surface Roughness Parameters**

The changes in surface roughness parameters such as Ra, Rq and Rz of MDF samples are represented in Figure 5, 6 and 7, respectively.



Figure 5. Ra values of MDF samples.



Figure 6. Rq values of MDF samples.



Figure 7. Rz values of MDF samples.

As can be seen in Figure 5, 6 and 7, generally, all surface roughness parameters ( $R_a$ ,  $R_q$ ,  $R_z$ ) of A and B groups showed differences depend on the rate and type of fibers used

for MDF manufacturing. These values of A and B groups were generally found to be higher than those of control groups except for B1 group manufactured with rate of 10% PSR fibers. The higher surface roughness parameters ( $R_a$ ,  $R_q$ ,  $R_z$ ) were obtained with A2 and B2 groups. The B1 group resulted in the smoothest surfaces with the Ra value of 4.07  $\mu$ m, Rq value of 5.08  $\mu$ m and an Rz value of 25.14  $\mu$ m, while corresponding  $R_a$ ,  $R_q$  and  $R_z$  values for the control samples were 4.21 $\mu$ m, 5.63 and 28.98  $\mu$ m, respectively.

Especially,  $R_a$ ,  $R_q$ ,  $R_z$  were found higher for A group. As the additive rate of RMP was increased, surface roughness values might be increased due to the more fines and small fibers. The changes on the surface roughness parameters of samples could be reasoned from the structural properties of RMP and PSR fibers. It is clear to say that, structural properties of raw materials might cause some irregularities on the material surface, and these irregularities also affect surface parameters of final material. It is reported that the surface roughness degree is a function of production parameters and raw material properties (Hiziroglu & Kosonkorn, 2006).

**Color Measurements:** The color measurement parameters ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ,  $\Delta E^*$ ) of MDF samples are given in Table 2, and Figure 8. The pictures of color changes occurred on the surfaces of MDF samples are presented in Figure 9.

Table 2. Color measurement parameters	$(\Delta L^*, \Delta a^*, \Delta b^*)$
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	1	( /	· · ·
Panel type	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$
A1	6.30	-1.60	-2.65
A2	8.48	-2.19	-0.53
B1	-1.68	-0.65	-2.11
B2	5.32	-0.98	-0.20
Control values we	re taken as referenc	es.	

Referring to the results in Table 2, positive and negative  $\Delta L^*$  values were recorded for MDF samples.  $\Delta L^*$ value represents difference in darkness and lightness; positive "+" value of  $\Delta L^*$  indicates lighter and negative "-" value of  $\Delta L^*$  indicates darker (Konica Minolta, 2018). According to results in Table 2, the  $\Delta L^*$  values increased with increasing rates of RMP and PSR fibers. While the highest positive  $\Delta L^*$ value was found to be 8.48 for A2 group manufactured with rate of 30% RMP fibers, the lowest value was found to be -1.68 for B1 group manufactured with rate of 10% PSR fibers.  $\Delta L^*$  values indicated that all MDF samples turned to lighter color except for B1 group. These differences between lightness or darkness of MDF panel groups can be clearly observed from Figure 9.

All  $\Delta a^*$  values of MDF samples were found as negative and ranged from -0.65 to -2.19. This means that MDF samples had a tendency to green direction. Similarly,  $\Delta b^*$  values were also found as negative and ranged from -0.20 to -2.65. These values were found in blue direction. In the color scale, + a<sup>\*</sup> and - a<sup>\*</sup> represent red and green directions; +b\* and -b\* represent yellow and blue directions, respectively (HunterLab, 2008).



**Figure 8.**  $\Delta E^*$  values of MDF samples.

As represented in Figure 8, it was found that the type and rate of raw material for panel manufacturing had effect on the total color change values of MDF samples. Especially,  $\Delta E^*$  values increased with the increasing rates of RMP and PSR fibers. The highest  $\Delta E^*$  value was determined to be 8.72 from A2 group manufactured with rate of 30% RMP fibers. The lowest  $\Delta E^*$  value was recorded to be 2.78 from B1 group manufactured with rate of 10% PSR fibers. The highest color changes were obtained from A group manufactured with RMP fibers compared to B group manufactured with PSR fibers.



Figure 9. Pictures of control and MDF samples manufactured with RMP and PSR fibers

The color changes of panel groups were cleary seen in Figure 9. A1 and A2 groups had higher color change than B1, B2 groups and control group. This could be probably due to the differences in the structural properties of fibers. Mechanically produced pulp have some optical advantages such as brightness, light scattering properties (Sundholm, 1999). However, in kraft pulp production, chemical reactions of residual lignin with pulping chemicals cause dark brown color of pulp (Twede et al., 2014).

## CONCLUSIONS

The fiber of refined chip screening rejects and pulp screening rejects obtained from kraft pulping process had positive effects on the MDF panel properties. In general, WA and TS values of each group for 2-24 h were found lower than those of control groups except for MDF panel manufactured with rate of 30% RMP. A considerable decrease was recorded on the WA and TS values (2-24h) of MDF panels manufactured with the rate of 10 % PSR fibers. All MOR and MOE values of each group were found higher than those of control groups and these values improved increasing rate of RMP and PSR fiber. The highest MOR and MOE values were obtained with rate of 30% PSR fibers. Surface roughness parameters of each group were generally found to be higher than those of control groups except for MDF panel manufactured with rate of 10% PSR fibers. The rougher surfaces were obtained with increasing rate of RMP and PSR fibers. The highest color changes were observed with the additive rates of RMP fibers. Generally, the best results for all tests were recorded from MDF panels manufactured with PSR fibers. Results indicated that, some wastes of Kraft pulping process such as wood chip screening rejects and kraft pulp screening rejects as fibrous materials have potential for reuse as raw materials for medium density fiberboard (MDF) manufacturing.

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#### ARAŞTIRMA MAKALESİ

**RESEARCH PAPER** 

Brand Management Strategies For Furniture Enterprises: Case Study Of Ordu And Giresun [\*]

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Abstract: In today's dynamic business world, enterprises which are innovative, open-minded, knowledge-creating can keep their assets as powerful players on the market. In this challenging competitive environment, enterprises need a realistic and sustainable vision. Sustainability, recognisability and easy accessibility are important for enterprises. One of the necessities for achievement is an effectively managed brand. Creating effective brand value in the domestic and foreign markets requires strategic moves. Branding is one of the most critical concepts for businesses to reflect themselves and reach their customers.

In this study, it is aimed to determine brand management strategies of furniture enterprises in Turkey. Accordingly, current branding works, brand assets, brand related future plans, how enterprises see their brands in comparison with other brands in the sector, and their views on how their brands are transforming have been determined. In this context, data were collected with survey method from 45 furniture enterprises in Ordu and Giresun provinces in Black Sea Region, Turkey.

As the result of the analysis, 78% of the enterprises have the moderate technology. The results show that Ordu and Giresun furniture firms are in good communication with their customers, they perceive their expectations / demands, and are easily accessible. 89% agree that it is important to pay attention to the feelings and needs of the target group and to recognize them well. 73.3% of the companies make content on social media in the name of branding. It is clear that most popular social media tools are Facebook and Instagram

Keywords: Brand management, Furniture enterprises, SMEs, Turkish furniture industry.

# Mobilya İşletmelerinin Marka Yönetim Stratejileri: Ordu-Giresun Örneği

Öz: Günümüzün dinamik iş dünyasında yenilikçi, değişime açık, bilgi ve değer yaratan işletmeler, bulundukları pazarda güçlü birer oyuncu olarak varlıklarını sürdürebilirler. Bu zorlu rekabet ortamında, işletmelerin gerçekçi ve sürdürülebilir bir vizyon sahibi olmaları gereklidir. Sürdürülebilirlik, tanınırlık ve kolay ulasılabilirlik kavramları isletmeler icin önemli odak noktalarındandır. Bunu sağlamanın gerekliliklerinden biri, pazarda etkin yönetilen bir markaya sahip olmaktır. Yurtiçi ve yurtdışı pazarlarda etkin marka değeri oluşturmak, stratejik çalışmaların varlığını gerektirir. marka yönetimi, işletmelerin kendilerini anlatmaları ve müşterilerine ulaşabilmeleri için en kritik kavramların başında gelmektedir.

Bu çalışma ile Türkiye mobilya işletmelerinin marka yönetim stratejilerinin belirlenmesi amaçlanmıştır. Buna göre mevcut marka çalışmaları, marka varlıkları, markaları ile ilgili gelecek planları, kendi markalarını sektörde diğer markalar ile karşılaştırıldığında nerede gördükleri, markalarının nasıl bir dönüşüm içinde olduğuna dair görüşlerinin belirlenmesine çalışılmıştır. Bu kapsamda Türkiye'nin Karadeniz Bölgesi'nde yer alan Ordu ve Giresun illerinden seçilen mobilya işletmelerinden, ulaşılan toplam 45 işletmeden anket yöntemi ile veri elde edilmistir.

Yapılan analiz sonucunda, %78'inin orta düzey teknolojiye sahip olan Ordu ve Giresun mobilya işletmelerinin müşterileri ile iyi iletişimde oldukları, beklentilerini / taleplerini doğru algılayabildikleri ve markalarının kolay erişebilir olduğu görülmektedir. Marka imajı, müşterilerin karar alma süreçlerini olumlu etkilemekte ve şirketler (%89) hedef gruplarına önem vermektedir. İşletmelerin %56' sı marka "dönüşüm/ yeniden yapılanma" sürecinde bulunmaktadır. Şirketlerin %73,3' ü sosyal medyada markalaşma / marka geliştirme/ sürdürülebilirlik tanıma/ geliştirme adına internet kanallarında içerik ürettiklerini belirtmişlerdir. En popüler sosyal medya araçları Facebook ve Instagram olmuştur.

Anahtar sözcükler: KOBİ, Marka Yönetimi, Mobilya İşletmeleri, Türkiye Mobilya Endüstrisi.

## INTRODUCTION

Nowadays, the use of the brand as a marketing variable is an important competitive tool for businesses. For this reason, it is important for businesses to decide which brand strategy to apply. Whatever brand strategy is used, the foundation for the implementation of brand strategies (brand architecture) for businesses is a strategic tool. The selected brand strategy will be a significant influence on the effectiveness of the marketing efforts of the enterprises (Sönmez, 2010).

The traditional approach is to communicate with the customer through only a few channels such as radio, television, printed media, etc., as it is not too difficult to convince the customer, and because the competition is not too much; most of the company's products could easily be sold, and the customer itself is not in big expectation. The customer basically concerns about the functional benefits of the product buys the product or service accordingly. Consumers can compare all brands in a very short time. It basically focuses on its own benefit. Even in the face of a small mistake, the brand can leave. In such a challenging environment, the best means of competition is to 'become a brand'. The main goal of every company should be building the brand in minds. The most competitive advantage in the current business world is to become a brand and to have branded products (Ünalan, 2015).

The main aim of the branding is to gain competitive advantage through distinctiveness, awareness and the emotional plus benefit-based relationship established by the consumer and the customer. The branding effort may also aim to provide a distinction based on a product, idea or image. In order to create and manage a successful brand, it is necessary to recognize the target consumer or potential customers according to their motivation, enjoyment, preferences, needs, and expectations. In order to ensure successful branding, the processes such as identity, positioning and awareness of the brand are carried out without supporting the objectives of the campaign and supporting each other (Alikişioğlu, 2012).

Whatever the size of their enterprise is, there are certain features that make sense for current and potential employees. These features are the reasons why they prefer the enterprise. These features cause a sense of branding in the minds of people about the business. The studies have shown that employees who have a strong employer brand increase their level of commitment, their motivation and performances, the number and quality of applications and accumulate more qualified employees in their organizations, where also decrease recruitment costs, make long term impact, increase managerial satisfaction, culture, and their competitiveness (Baş, 2011).

The strategy is a kind of plan. It is more dynamic than routine plans and requires t the competitors' possible actions that can affect the outcome of the business (Baş, 2011). Markets should have a vision with awareness of change, and can make the brand live in the long run with integrated strategies by taking the appropriate steps (Ünalan, 2015).

The brand strategy is expressed as a long-term plan to achieve the brand's goals. However, if there is a confusion that the brand is a product, name, website or logos in the company, it is necessary to understand that the concept of the brand is not only a complementary element, but also includes the values, feelings, habits and place of the consumer. Every day, a new brand enters to the market and consumers come across a physical or digital brand bombardment. Brands willing to exist without being victims of the consumer's tiredness need to reflect their company's goals on their brands and determine their commitment to the consumer's contribution to life with clear lines. As new channels appear every day, the communication, the word, the message and the visual in each channel need to show a consistent line, attitude and expression. Consistent activities that increase the brand's recognition acceleration also serve as a reinforcer in terms of customer loyalty. To carry out the communication demanded by the new generation consumers, to smooth the transition between the electronic devices, to adopt the language of the mark with the platform's own language and to make the messages invisible but powerful, are the notes leading to the success of the consistency move (Moralioğlu, 2016).

Global companies, that is, brands operating in international markets, need to apply strategies that are in line with the local values of the different countries in which they operate. When the local customer expectations, culture structure, habits, traditions, lifestyle etc, are understood and are not adapted accordingly, the products can not be kept in new and different markets, can't be successful and in a short time, they may have to leave the market or change products. While products are adapted accordingly to socio-cultural differences, the factors such as climatic conditions of the local market, development levels of countries, religious structure, income level, packaging and labeling, taste should be considered. In addition, it is also possible for companies to maintain their brand identities using global messages, while making appropriate adjustments to their market. However, if the given current message is contradictory to the cultural or product that is adapted to the country, it may be necessary to create a completely new message (Moralioğlu, 2015). It is stated that Turkish firms do not follow a certain analysis method when they enter new export markets and therefore major parts of the big picture are not noticed (Gider, 2016).

One of the most important branches of the Turkish forest products industry is the furniture industry. Turkish furniture industry has a heterogeneous structure which is consisted numerous enterprises with micro and macro structure. Because of these heterogeneous structures and the multiplicity of structural and economic problems they have, enterprises need the change and improvement. Customers are at the forefront of effective determining factor when most of the business are taking a decision. This study is aimed to determine brand management strategies of furniture enterprises. Accordingly, current brand studies, brand assets, future plans related to brands, how their brands are seen in comparison with other brands in the sector, and their views on how their brands are transforming have been determined.

## **MATERIAL and METHODS**

The main purpose of the research is to determine brand management strategies of furniture companies. The brand assets of the furniture enterprises, the importance of perceiving the brand value and the importance given to the brand have been questioned.

The sample of the survey is limited to the provinces of Ordu and Giresun in the Black Sea region of Turkey. It has been targeted to reach all the furniture enterprises operating in Ordu and Giresun.

The survey form was used as data and information gathering tool in the research. The prepared questionnaire was applied on a sample and the clarity and clarity of the questions on the questionnaire were tested. With the information gathered, necessary regulations were made in the questionnaire form and the questionnaire form was put into practice. Surveys were conducted with authorized interviewers at face-to-face meetings. Survey work was created via Google Drive and responded online by sending them to their email address. 45 enterprises responded the questionnaire forms. The survey was conducted in July and August 2018.

The data considered in the study consist of demographic information for determining the characteristics of the enterprises and statements aimed at evaluating the brand management strategies of the enterprises 5 Likert scale (Strongly Agree, Agree, Undecided, Disagree, Strongly Disagree), yes / no questions and multiple choice questions were used to the evaluations of the enterprises being questioned with 23 items.

## RESULTS

**Demographic** Information: The furniture enterprises participating in the survey, 66,7% were in Ordu (30 enterprises) and 33,3% were in Giresun (15 enterprises). 62,2% of Participating Enterprises are Private Company, 33,3% are Limited Companies and 4,4% are Joint Stock Companies. When the distribution of enterprises according to the years of establishment is examined, it is observed that the enterprises established between 2001 and 2010 (33,3%) and the enterprises established between 1981 and 1990 (31,1%) are concentrated. Then, 15,6% are from 1991-2000, 15,6% are from 2011 and after, and 4,4% are from 1980 and before.

55,6% of the respondents are business owners, 22,2% are professional managers, 11,1% are engineers / architects / technicians, 11,1% are employees. 84,4% were

male and 15,6% were female. 33,3% of the respondents were aged 33-39, 28,9% were aged 40-46, 13,3% were aged 47-53, 11,1% were aged 26-32, 6,7% are 19-25, and 6,7% are in the 53 and over age group. According to educational status, 42,2% of respondents were university graduates, 26,7% were vocational high school graduates, 15,6% were high school graduates, 8,9% were primary education graduates, 4,4% were secondary education and 2,2% is postgraduate people.

31,1% of the enterprises were in the organized industrial zone, 31,1% were in the urban neighbourhood, 31,1% were the small industrial sites and 6,7% were in the free zones. Number of employees was between 1-9 in 64,4%, between 10-49 in 33,3%, and between 50-249 in 2,2%. There were no businesses with 250 employees. There were 9 engineers, 12 architects, 28 technicians and 11 industrial designers. It was stated by managers that 77,8% of the enterprises had medium level technology, 20% had high level technology and 2,2% had low level technology. The average capacity rate of the enterprises in the last one year was 63% on average.

Assessment of Brands and Process for Orientation to National/International Target Markets Conceptual Awareness: The ratios of strongly agree and agree responses from 23 questions prepared at the 5-Likert scale (Strongly Agree, Agree, Undecided, Disagree, Strongly Disagree) are shown in Table 1.

In the brand communication, the percentage of the enterprises using social media is 86,7%. The most used social media channels in brand communication are Facebook and Twitter. This is followed by WhatsApp, Twitter, Google+ and Linkedin respectively. YouTube, Pinterest, Skype, Snapchat, social media channels have never been used. Firms that did not use any social media channels accounted for 4,4%. The distribution of social media usage according to the establishment years of the enterprises is shown in Table 2 and 3.

The factors that play an important role in the continuity of the brand from the enterprises were required to be marked. According to this, the answers given by the enterprises are shown in Table 4 as percentage and frequency distribution. Enterprises are most likely to attach importance to an innovative, sustainable vision and trust-based criterion.

73,3% of the enterprises gave a positive answer where 26,7% answered no, for the question "Do you produce content in the name of branding / brand development / sustainability recognition / development in social media?".

The first 3 tool markings which are most important for the brand promotion are requested to mark from the enterprises. According to the this, in decreasing order, 73,3% brochures / catalogs, 60% social media groups, 53,3% magazines / newspapers / other printed publications, 20% fairs / invitations and similar events, 20% internet advertising, 17,8% TV commercials, 8,9% cinema take place.

#### Table 1. Brand evaluations of enterprises.

	Percentage (%)
We are recognized by our customers (our target kit).	91%
It is important to give importance to the feelings and needs of the target group and to recognize them well.	89%
Customers can easily contact us when they have problems with our brand.	87%
Our mark affects our target customers' purchasing decision positively.	84%
We can perceive our customers' expectations / demands correctly	84%
We can quickly catch up with our clients' new demands or even more difficult expectations.	84%
It is important to whom our mark will address (target not set).	82%
Meeting our customer requirements is our high performance	82%
Positive relations with major customers / units in the sector	82%
Producing the right content for branding / branding is very important.	80%
We are striving to develop products and services that will strengthen our reputation and market perception	80%
We are a brand that can provide effective solutions to our customers' problems in short time.	80%
We follow the judgments and values created by our products and services in the market.	76%
We are recognized in the market we are in.	76%
We are a powerful player in the market we find.	62%
We have efforts to increase brand value.	60%
Our mark is in a "transformation / restructuring" process.	56%
We have an actively managed brand.	51%
We are actively using advertising channels on the domestic market.	40%
We have a brand value at Turkey market.	24%
We receive corporate communication consulting.	20%
We have a brand value in the overseas market.	13%
We are actively using advertising channels in international markets.	9%

#### **Table 2:** Distribution of social media usage by establishment's years.

	Facebook	Instagram	WhatsApp	Twitter	Google+	LinkedIn	Not Using
1980 and before	2	2	1	-	-	-	-
1981-1990	10	12	4	1	1	3	1
1991-2000	4	4	3	-	1	-	-
2001-2010	13	11	8	3	1	1	-
2011 and after	6	5	3	1	-	-	1
Total	35	34	19	5	3	4	2

Table 3: Usage rates of social media channels in communication.

Facebook	Instagram	WhatsApp	Twitter	Google+	LinkedIn	Not Using
78%	76%	42%	11%	7%	9%	4%

Table 4: Distribution of factors that play a role in the continuity of the brand.

	Number of answers	Percentage %
Innovative, sustainable vision based on strong foundations	32	71,1%
To be dependable	28	62,2%
To be able to update the business according to the trends of the day	26	57,8%
Gaining reputation	25	55,6%
To be open to innovation	19	42,2%
To create a better impression	17	37,8%

## **DISCUSSION and CONCLUSION**

In today's dynamic business world, enterprises which are innovative, open-minded, knowledge-creating can be keep their assets as powerful players on the market they are in. In this challenging competitive environment, enterprises need to have a realistic and sustainable vision. Sustainability is an important focus for enterprises. Being recognizable and easy accessibility are the goals of each enterprise. One of the necessities to achieve this is to have a brand that is effectively managed on the market. Creating effective brand value in the domestic and foreign markets requires the existence of strategic studies. In the competitive environment where there is a rapid change and transformation, brand concept is one of the most critical concepts for enterprises to express themselves and reach their customers. In this context, data were collected with survey method from 45 furniture enterprises reached from Ordu and Giresun provinces which are located in Black Sea Region of Turkey. Given the demographic structure of the respondents, two-thirds of the enterprises are in Ordu and one-third of those are in Giresun. Private companies (62,2%) and enterprises established between 1981 and 2010 are concentrated. The vast majority of respondents were business owners and university graduates. It has been determined that 77,8% of the enterprises have the moderate technology, 20% have high technology and average capacity utilization rate is 63% in the last one year.

Most of the furniture companies of Ordu and Giresun (91%) stated that they are recognized by the customers, namely the target groups. A similar majority (89%) stated that they agree that "it is important to pay attention to the feelings and needs of the target group and to

recognize them well." The results show that Ordu and Giresun furniture firms are in good communication with their customers, they can correctly perceive their expectations / demands, and they are easily accessible to brands.

It is seen that brand images have a positive effect on the decision-making process of the customers and that they attach importance to the target groups of the companies. Enterprises stated that their customers are able to keep pace with their new demands or more difficult expectations and those they have a high level of success in meeting customer needs and are able to provide effective solutions to their customers' problems in a short period of time. It is seen that the relations with important customers/units in the sector are also positive.

Enterprises are aware that producing the right content for branding / branding is very important. They are also endeavouring to develop products and services that will strengthen their reputation and market perceptions. Enterprises are known in the market and they are following the judgments and values of the products and services in the market. 62% of the enterprises see themselves as a strong player on the market they are in. There are studies to increase brand value, and again at similar rates (56%), their brands are in a "transformation / restructuring" process. Considering all these positive views, only 51% of companies have indicated that the brands were effectively managed. The effective use of advertising channels in the domestic market (40%) was found to be quite low in spite of the high recognition of their company in the market. The proportion of businesses indicating that they have a brand name in Turkey's market value was 24%. Participants in the survey indicated that only one of the furniture companies had corporate communication consultancy. Only 13% of firms have a brand value in the international market and 9% have used advertising channels effectively in international markets.

73,3% of the companies stated that they were producing content on the internet in the name of branding / brand development / sustainability recognition / development on social media.

Brochures / catalogues (73,3%), social media groups (60%), magazines / newspapers / other printed publications (53,3%) are the most important means of brand promotion of businesses. Fair / invitation etc. events, internet advertising, TV commercials, cinema have become the least preferred brand promotion channels.

According to the general structures, firms that are seen as micro, small and medium scale are known as brands on the market they are in, but they are not yet located in the domestic market all over the country. The fact that they are working on brand management shows that businesses are conscious of this issue and their awareness levels are in good condition. There are fewer companies that target overseas markets and work in this field. This may be attributed to the fact that the majority of businesses are micro and small-scale local businesses. It seems that YouTube channel has never been used by enterprises. However, the prospective customer group in the near future is now heavily following YouTube. Enterprises should also use the social media tools to reach young people in their 18-24 of age group who are the new generation, that is called the future customers.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Mold Resistance of Nano and Micronized Particles-Treated Wood After Artificial Weathering Process [\*]

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**Abstract:** Wood and wood based materials under outdoor conditions may lose their appearance and performance properties by time. Color differences, cracks, rough surface may occur within a few years in unprotected wood. These undesired formations could be avoided by technical applications such as coatings on wooden surfaces by paints and varnish since such coating materials include special additives in their formulations such as UV absorbents, pigments, etc. Novel systems with nano or micronized particles may have superior properties when compared to conventional wood preservatives.

In this study, it was investigated the effectiveness of Scots pine wood samples impregnated with nano-scale preservatives, micro-scale preservatives and common wood preservatives, against mold fungi before and after artificial weathering process. Accelerated weathering tests of the specimens were performed in a Xenon arc radiation cabinet with water spraying property. Artificially weathered and un-weathered wood specimens were evaluated for resistance to mold fungi according to the ASTM D4445. General mold score results collected before and after artificial weathering process showed that CCA and MCQ-treated specimens had the best performance properties; however, nano zinc oxide-treated wood specimens had the weakest performance against mold fungi.

Keywords: .Artificial weathering, micronized copper, mold growth, nano copper, nano zinc.

# Mikronize ve Nano Sistemlerle Emprenye Edilmiş Odunun Yapay Yaşlandırma İşlemi Sonrası Küflenmeye Karşı Dayanıklılığı

Öz: Dış hava koşullarında odun ve odun esaslı malzemeler zaman içerisinde görünüm ve performans özelliklerini kaybedebilmekte, korumasız ahşapta birkaç yıl içerisinde renk farklılıkları, çatlaklar, pürüzlü yüzeyler oluşabilmektedir. Bu istenmeyen oluşumlar, ahşap yüzeylerine UV absorbe eden maddeler, pigment gibi özel katkı maddeleri içeren boya ve vernik uygulanmasıyla önlenebilmektedir. Nano veya mikronize parçacıklara sahip yeni sistemler, geleneksel ahşap koruyucularla karşılaştırıldığında üstün özelliklere sahip olabilmektedir.

Bu çalışmada nano ölçekli, mikro ölçekli ve geleneksel ahşap koruyucular ile emprenye edilmiş ahşabın yapay yaşlandırma işlemi öncesi ve sonrası küf mantarlarına karşı performansı incelenmiştir. Test numunelerinin hızlandırılmış yaşlandırma testleri, yağmurlama özelliği olan Xenon-ark yaşlandırma kabininde gerçekleştirilmiştir. Yaşlandırma işlemine maruz bırakılmamış ve yapay olarak yaşlandırılmış odun örnekleri ASTM D4445 standart test metoduna göre küflenme testlerine tabi tutulmuştur. Elde edilen sonuçlar, CCA ve MCQ ile emprenye edilen örneklerin en iyi küflenme karşıtı performans özelliklerine sahip olduğunu göstermiştir. Bununla birlikte, nano çinko oksitle emprenye edilmiş ahşap numuneleri küf mantarlarına karşı en hassas grup olarak belirlenmiştir.

Anahtar sözcükler: Küf gelişimi, micronize bakır, nano bakır, nano çinko, yapay yaşlandırma.

## INTRODUCTION

Wood is susceptible to biological degradation by fungi, insects and marine borers, depending on its natural durability. Because of this reason, wood protection industry and researchers focus on nano and micro scale chemicals to produce more effective new generation wood protection chemicals, nowadays. (Clausen, 2007; Matsunaga et al., 2007; Clausen et al., 2010, 2011; Akhtari & Nicholas, 2013; Lykidis et al., 2013; Mantanis et al., 2014). The main usage purpose of nano- or macro-scale wood preservation systems is to allow chemical substances to penetrate deeper into the wood uniformly (Mantanis et al., 2014; Mantanis & Jones, 2012; Freeman & McIntyre, 2008; Kartal et al., 2009). Investigations are continuing to better explain the performance of nano- and micro-based wood preservative chemicals against leaching and weathering (Terzi et al., 2016; Mantanis et al., 2014). Terzi et al. (2016) reported that nano-particle forms of some metal oxides for wood protection were successful in terms of biological resistance and leaching. In this study, only nano-B2O3 treated and weathered specimens were not effective in preventing decay by G. trabeum. Mantanis et al. (2014) focused on the resistance of pine wood treated with zinc and copper based nano chemicals and mold rating results showed that only nanozinc borate could inhibit the mold growth. CCA treated wood were more effective then MCQ and ACQ and nano-CuO treated wood specimens against decay fungi in soil block tests (Kartal et al. 2015). According to recent studies, the effectiveness of nano and micronized particles-treated wood has not been fully elucidated and further research is needed.

The objective of this study to evaluate mold resistance of nano and micronized particles-treated wood specimens after artificial weathering process and compare with common preservatives.

## **MATERIAL and METHODS**

*Wood specimens and treatments:* The following commercial wood preservatives and nano particles were tested in the study (% m/m):

i) ACQ - water soluble form of Cu: Osmose Celcure AC-500 (Osmose Naturewood) (Osmose UK Protim Solignum Ltd): Quat (benzalkonium chloride) (4.8%), copper carbonate hydroxide (16.53%), boric acid (5%).

ii) Micronized ACQ - micronized form of Cu: Osmose Micro Pro, (Celcure MC) (Osmose UK Protim Solignum Ltd): Quat (benzalkonium chloride (10%), micronized copper carbonate hydroxide (17.39%), boric acid (5.23%).

iii) Nano-CuO - nano form of Cu: (NanoArc, 97.5%, 23-37 nm APS Powder, Alfa Aesar, Germany): CuO (97.5%).

iv) CCA: Osmose K-33 e water soluble form of Cu: (Osmose UK Protim Solignum Ltd) CuO (10.5%), chromic acid (29.9%), arsenic penta oxide  $As_2O_5$  (20%), water (39.6%).

v) Nano-ZnO – nano form of Zn: (NanoArc, 99%, 40-100 nm APS Powder, Alfa Aesar, Germany): ZnO (99%).

Water-based preservative solutions were adjusted in order to reach a target elemental Cu and Zn retention level of 0.64 kg m-3 in treated wood specimens.

Wood specimens (3 mm x 45 mm x 135 mm) were cut from the sapwood portions of Scots pine (*Pinus sylvestris L*.) lumber. The wood specimens (2 - 4 growth rings/cm) were free of knots and visible deposits of resins, and showed no visible evidence of infection from mold, stain or wooddegrading fungi. The specimens were conditioned to a moisture content of 10-12% in a conditioning room at 22 °C and 65% relative humidity (RH) for 2 weeks before preservative treatment. Pre-weighed wood specimens were vacuum treated for 40 min at 100 mm Hg with each treatment according to the AWPA standard E10 method (AWPA, 2012). All the specimens were treated to a target retention of 0.64 kg/m<sup>3</sup> based on Cu and Zn elements, individually. After treatment, the specimens were blotted-dry and re-weighed to check solution uptake.

Accelerated weathering tests: Accelerated weathering tests were performed in a Xenon arc radiation cabinet (Atlas Xenotest Alpha+, ATLAS), which included water spraying according to ISO 4892-2-A1 (ISO, 2006). The radiation source was a Xenon arc lamp (300 to 400 nm), using borosilicate filters. The test consisted of cycles of 102 min of radiation, a black body temperature of  $65\pm5\%$  followed by 18 min of water spray at the same radiation conditions, and a relative approximately 100%. The total duration of the test was 288 h (144 h x 2 periods) for 3 specimens (3 mm x 45 mm x 135 mm) per each test group.

Mold resistance tests: Artificially weathered and treated wood specimens were evaluated for resistance to mold fungi according to the ASTM D4445 (ASTM, 2012). Wood specimens (7 mm tangential x 20 mm radial x 7 cm long) for mold resistance tests cut from the artificially aged wood specimens (3 mm x 45 mm x 135 mm). Three mold fungi, Aspergillus niger, Penicillium fellutanum, and Trichoderma harzianum were grown and maintained on 2% malt agar. Wood specimens (5 specimens per group) were sprayed with 1 ml of mixed mold spore suspension of the three fungi and incubated at 27 °C and 80% RH for 4 weeks. All fungi were obtained from the USDA Forest Service Forest Products Laboratory, Madison, WI, USA. A mixed spore suspension of the three test fungi was prepared by washing the surface of individual 2-wk-old petri plate cultures with 10-15 ml of sterile DI water. Washings were combined in a spray bottle and diluted to approximately 100 ml with DI water to yield approximately  $3 \times 10^7$  spores ml-1.

The spray bottle was adjusted to deliver 1 ml of inoculum per spray. Wood specimens were sprayed with 1 ml of mixed mold spore suspension and incubated at 27 °C and 80% RH for 12 wk. Following incubation, specimens were visually rated on a scale of 0-5, with 0 indicating the specimen is completely free of mold growth and 5 indicating the specimen was completely covered with mold growth (0: no growth, 1: 20% coverage with mold fungi, 2: 40%, 3: 60%, 4: 80%, 5:100%).

### **RESULTS and DISCUSSION**

Average ratings of weathered and un-weathered wood specimens for resistance to the mold fungi are shown in Fig. 1. Generally, artificial weathering and treatment processes improved the mold resistance of specimens except that ACQ treated - weathered and nano ZnO treated - unweathered specimens. Mold resistance of control specimens were improved after the artificial weathering process and average mold ratings down under 1.0 from 5.0. Similar results were obtained in previous studies made by researchers. These results could be supported by the products of lignin photodegradation. Because the lignin sub-units that are released during photodegradation are aromatic and therefore fungitoxic (Schoeman & Dickinson 1997: Cerniglia & Crow 1981). The nano or micronized particle based preservatives used in this study have not prevented the growth of mold fungi on un-weathered wood specimens as well as CCA. Mold development was not observed in the unweathered specimens impregnated with CCA during the test period. MCQ and CCA treated and weathered specimen groups have similar mold inhibition effects on wood specimens. Nano CuO treated specimens have also a good performance against mold growth. The worst mold inhibition performance was observed in nano ZnO treated wood specimens, whether weathered or not. Mold inhibitor effectiveness of CCA and MCQ treated wood specimens were as good as weathered control specimens.



**Figure 1.** Average mold ratings for un-weathered (dotted bars) and weathered (filled bars) wood specimens (after 4 weeks): 0 indicates no mold growth, 1 = 20%, 2 = 40%, 3 = 60%, 4 = 80%, and 5 = 100% mold coverage.

#### CONCLUSION

In this study, mold resistance in laboratory conditions of Scots pine wood samples impregnated with nano-scale preservatives, micro-scale preservatives and common wood preservatives were evaluated. Experiments were performed on artificial weathered and un-weathered samples. Results showed that the mold fungi were noticeably inhibited by MCQ, CCA and also nano CuO, while the nano ZnO did not inhibit the mold growth on wood specimens. It was also shown that the artificial weathering process inhibit the mold growth. This can be attributed to the removal or degradation of nutrients on the surface of the wood during the weathering process.

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### ARAŞTIRMA MAKALESİ



#### **RESEARCH PAPER**

# Investigation of Thermal Conductivity of Wood Sandwich Panels with Aluminium and Polypropylene Core [\*]

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**Abstract:** Sandwich panels are obtained by placing thick but rather light core material between two thin and rigid lower and upper surface layers. Sandwich panels, especially due to their light weight, high "strength / weight" ratio and durability compared to conventional materials have many use areas such as aviation and space industry, maritime, automotive and building industry. It is one of the biggest advantages that sandwich materials can be obtained from different materials and geometric structures by choosing the lower and upper surface layers and the core for various applications. The aim of this study is to investigate the thermal conductivity values of the sandwich panels, which are manufactured with different types of surface and core materials in sandwich panels. An aluminium and polypropylene as a core materials and alder, birch and scots pine wood veneers were used as wood species for surface layers. Thermal conductivity of sandwich panels was determined according to ASTM C 518 & ISO 8301. As a result of the study, the highest thermal conductivity values were obtained from aluminium core sandwich panels. The highest values were obtained from alder for the aluminium core panels and scots pine for the polypropylene core panels as wood species.

Keywords: Aluminium, core, polypropylene, sandwich panel, thermal conductivity.

# Alüminyum ve Polipropilen Çekirdekli Sandviç Panellerin Isıl İletkenliklerinin Araştırılması

Öz: Sandviç paneller, iki ince ve sert alt ve üst yüzey tabakasının arasına kalın fakat hafif çekirdek malzeme yerleştirilerek elde edilir. Sandviç paneller, özellikle hafiflikleri, yüksek "kuvvet / ağırlık" oranları ve geleneksel malzemelere göre dayanıklılıkları nedeniyle havacılık ve uzay endüstrisi, denizcilik, otomotiv ve inşaat endüstrisi gibi birçok kullanım alanına sahiptir. Sandviç malzemelerin, farklı uygulamalar için alt ve üst yüzey katmanları ve çekirdeği seçilerek farklı malzemelerden ve geometrik yapılardan elde edilebileceği, en büyük avantajlardan biridir. Bu çalışmanın amacı, farklı yüzey ve çekirdek malzemeleri ile üretilen sandviç panellerin ısıl iletkenlik değerlerini araştırmaktır. Çekirdek malzeme olarak, alüminyum ve polipropilen; kızılağaç, huş ve sarıçam kaplamaları, sandviç panellerin üretiminde yüzey panelleri olarak kullanılmıştır. Çekirdek tabakanın her iki yüzey tabakasına yapışması için poliüretan ile modifiye edilmiş bir epoksi yapıştırıcı kullanılmıştır. Sandviç panellerin ısıl iletkenliği ASTM C 518 ve ISO 8301'e göre belirlenmiştir. Çalışma sonucunda, en yüksek ısıl iletkenlik değerleri, alüminyum çekirdekli sandviç panellerinden elde edilmiştir. Alüminyum çekirdekli paneller için kızılağaçtan en yüksek değerler elde edilmiş ve polipropilen çekirdekli paneller için çam ağacından elde edilmiştir.

Anahtar sözcükler: Alüminyum, çekirdek, ısıl iletkenlik, polipropilen, sandviç panel.

## INTRODUCTION

Sandwich panels are used for variety of structural applications, including building construction, transportation, decking, marine and aerospace because of their high energy absorption ability, high bending stiffness and light weight (Li et al., 2014; Aslan et al., 2017). They are favoured for their high specific strength and stiffness, corrosion resistance, tailor ability, and stability. Sandwich panels are very suitable for lightweight structures requiring high in-plane and flexural stiffness (Gustin et al., 2005). Additionally, they are usually based on two thin face sheets with high stiffness and strength, and a compliant and light-weight core that maintains the distance between the faces and sustains deformation, often with insulation properties. By varying the material and thickness of core and face sheets, it is possible to obtain sandwich panels with different properties and performance (Steeves and Fleck, 2004). The properties of interest for core materials include, among others, low density and good thermal and acoustic insulation characteristics. Commonly used core materials are honeycombs, foams and balsa wood, but other alternatives of cellular core structures are being proposed (Lakreb et al., 2015).

Most of the sandwich panels are made of honeycomb and foam cores with surface materials (Pan et al., 2008; Vaziri et al., 2006; Qin & Wang, 2013). Especially, the sandwich panel composites that have open cells provide the multifunctional benefits to composite such as the high stiffness strength and high specific strength (Xiong et al., 2010; Joo et al., 2011; Xiong et al., 2016). Honeycomb sandwich structure consist of a thick layer (core) intercalated between two thin-stiff surface layers (skin). Honeycomb sandwich core is cellular solid that used void to decrease mass, while maintain qualities of stiffness and energy absorption. Honeycomb sandwich composite are widely used to replace traditional material in highly loaded application such as automotive and aviation industry (Newstead et al., 2008; Forsberg et al., 2006; Aslan et al., 2017).

With the increasing adversity of climate changes from global warming, discussions within the international

community for establishing an appropriate response policy have become more urgent (Seo et al., 2011). In facing the global warming trend, there is a dire need for more effective measures to sustain comfortable temperatures in living environments. To sustain an indoor temperature that is independent of outdoor temperature fluctuations, materials need to be developed that have superior thermal insulation abilities (Kawasaki & Kawai, 2006). Thermal conductivity is a very important parameter in determining heat transfer rate and is required for development of thermal insulation of materials (Sahin Kol & Altun, 2009). Several studies about thermal conductivity of composite materials showed that thermal conductivity was influenced thickness of composite materials, density, moisture content, temperature, material space ratio and flow direction of heat (Suleiman et al., 1999; Bader et al., 2007; Sonderegger & Niemz, 2009; Aydin et al., 2015).

The aim of this study is to investigate the thermal conductivity values of the sandwich panels, which are manufactured with different types of surface and core materials in sandwich panels. An aluminium and polypropylene as a core materials and alder, birch and scots pine wood veneers were used as wood species for surface panels for manufacturing of sandwich panels.

#### **MATERIAL and METHODS**

In this study, aluminium and polypropylene (PP) as honeycomb core materials and alder, birch and scots pine veneers as face sheet materials were prepared in  $30 \times 30$ dimensions for the manufacture of sandwich panels. The polyurethane modified epoxy- adhesive was used for binding of core layer to both surface layers. The hardener was added in the adhesive at 20%. Figure 1 shows a sandwich panel production.



Figure 1. Sandwich panel production.

Depending on the adhesive and surface materials used in this method, the sandwich composite panels were pressed at 80°C, under pressure of 10 kg/cm<sup>2</sup> and for 15 minutes and then are waited 24 hours in the press without temperature and pressure.

The thermal conductivity of the sandwich panels was determined according to ASTM C 518 & ISO 8301 (2004). Sample size required is 300x300xpanel thickness mm. Two specimens were used for each group. The tests were made at laboratory of Forest Industry Engineering in KTU. The Lasercomp Fox-314 Heat Flow Meter shown in Figure 2 was used for the determination of thermal conductivity.



Figure 2. Lasercomp Fox-314 Heat.

## **RESULTS and DISCUSSION**

The thermal conductivity values of the sandwich panels compared to other building materials (sand, gravel, cement, glass, concrete) are given in Figure 3 according to wood species and core materials.



**Figure 3.** Thermal conductivity values of the sandwich panels and other materials (W/mK).

As can be seen that Figure 3, the highest values were obtained from alder for the aluminium core panels and scots pine for the polypropylene core panels as wood species. The highest thermal conductivity values were obtained from aluminum core sandwich panels. The thermal conductivity values of all sandwich panels were lower than the thermal conductivity values of other building materials. Thermal conductivity of wood based materials is vary according to wood species, direction of wood fiber, resin type, addictive members, impregnate materials and processes used in the manufacturing of wood based composite panels (Kamke et al.,1989; Sahin Kol et al.,2010; Kol et al., 2010; Demirkir et al., 2013). Also, several studies about thermal conductivity of composite materials showed that thermal conductivity was influenced thickness of composite materials, density, moisture content, temperature, material space ratio and flow direction of heat (Suleiman et al., 1999; Bader et al., 2007; Sonderegger & Niemz, 2009; Aydin et al., 2015).

#### CONCLUSIONS

The aim of this study is to investigate the thermal conductivity values of the sandwich panels, which are manufactured with different types of surface and core materials in sandwich panels. The thermal conductivity values of all sandwich panels were lower than the thermal conductivity values of other building materials. The thermal conductivity values of alder and scots pine sandwich panels were higher than those of birch sandwich panels as similar as density values. This study showed that sandwich panels produced from aluminium and polypropylene as core materials and alder, birch and scots pine wood veneers can be used as an alternative insulation material.

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#### ARAŞTIRMA MAKALESİ

**RESEARCH PAPER** 

The Reaction to Fire of Some Chemicals Treated Pine Wood Product Surface\*

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**Abstract:** Wooden materials have been extensively used for furniture, outdoor and indoor cladding, buildings, terrace, fence, garden furniture and interior decoration and to decorate the roofline of houses. However, wood which is used outdoors or in areas exposed to moisture needs to be protected against wood-destroying organisms and to be shielded from water, marine pests, fungi, fire and weather conditions. Untreated wood materials have no resistance to the spread of fire and many buildings which constructed from wood based materials need to fire resistance. It is possible that the wooden material ensures very durable and resistant against physical effects by surface applications such as wood preservative paint and acrylic resin-based varnish. The application of fire retardant chemicals can also provide to satisfy regulatory requirements for wood products.

In this study, titanium dioxide and antimony trioxide were applied on pine (*Pinus sylvestris L.*) solid wood material to determine durability of reaction to fire using by oxygen index test technique (ASTM D 2863-6) and real fire test. These chemicals were added to the wood preservative paint which is a commercial product as concentrations of 2%, 5% and 10% for titanium dioxide and 2% and 5% for antimony trioxide. The effects on color change of their surfaces, brightness and surface roughness measurements, water absorption and thickness swelling of this wood material were also carried out. The results obtained were analyzed statistically and compared with the related standards. Addition of these chemicals to used wood preservative paint showed positive effect on the fire properties of the pine wooden surface.

Keywords: Antimony trioxide, Fire retardant, Titanium dioxide, Wood preservative paint.

## Yüzeyi Bazı Kimyasallar ile Muamele Edilen Çam Odunun Yanma Performansı

Öz: Ahşap malzemeler, mobilya, dış ve iç cephe kaplamaları, binalar, teras, çit, bahçe mobilyaları, iç dekorasyonda ve evlerin çatı hattını dekore etmek için yaygın olarak kullanılmaktadır. Bununla birlikte, açık havada veya neme maruz kalan alanlarda kullanılan odun odunu tahrip eden organizmalara karşı, sudan, deniz zararlılarından, yangın ve hava koşullarından korunmalıdır. İşlem görmemiş ahşap malzemelerin yangının yayılmasına karşı direnci yoktur. Ahşap esaslı malzemelerden yapılmış birçok binanın yangına dayanıklı olması gerekir. Ahşap malzemenin, ahşap koruyucu boya ve akrilik reçine bazlı vernik gibi yüzey uygulamalarında fiziksel etkilere karşı çok dayanıklı ve dayanıklı olması mümkündür. Yangın geciktirici kimyasalların uygulanması, ahşap ürünler için yasal gereklilikleri karşılamayı da sağlayabilir.

Bu çalışmada, sınırlayıcı oksijen indeksi testi (ASTM D 2863-6) ve gerçek yanma testi kullanılarak yangına karşı dayanıklılığın belirlenmesi amacıyla çam (Pinus sylvestris L.) odunu malzemesine titanyum dioksit ve antimon trioksit uygulanmıştır. Bu kimyasallar, titanyum dioksit için %2, %5 ve %10 ve antimon trioksit için %2 ve %5 konsantrasyonlarında ticari bir ürün olarak ahşap koruyucu boyaya eklenmiştir. Ahşap yüzeylerdeki renk değişimleri, parlaklık ve yüzey pürüzlülüğü ölçümleri, su absorpsiyonu ve kalınlığına şişme üzerine etkileri araştırılmıştır. Elde edilen sonuçlar istatistiksel olarak analiz edilmiş olup ilgili standartlarla karşılaştırılmıştır. Bu kimyasalların ahşap koruyucu boyaya eklenmesi, çam odun yüzeyinin yanma özellikleri üzerinde olumlu etki göstermiştir.

Anahtar sözcükler: Antimon trioksit, yangın geciktirici, titanyum dioksit, ahşap koruyucu boya.

## INTRODUCTION

Wood is renewable material, easily workable, compatible with its outstanding physical and mechanical properties. Since it is used flats, window and door, furniture, ceilings and floors, stairs, benches, its preservation is important significantly. But the use of wood can be restricted by safety regulations concerned with its ignitability and flame spreading characteristics. The application of a suitable fire retardant system can prevent these problems, thus can be used in many areas of use (Russell et al., 2004).

The resistance of wood to burning and flame spread have been researched for many years. A wood consists of carbon and hydrogen which make it highly combustible. A fuel, an oxidizer and a source of heat are three main elements in a combustion reaction. The combustion of wood occurs by the pyrolysis of the cellulose and its reaction to oxygen. When the temperature is increased, the pyrolysis starts. The combustion stops if any of the elements is removed (Pabelina et al., 2012). Many fire retardant techniques include surface treatment with fire retardant chemicals such as fire resisting coatings and pressure impregnations of chemical solutions into wood (White, 1999).

Fire retardants are mainly based on organic phosphorus, halogens, and metallic oxides. A fire retardant such as phosphorus, aluminum, antimony, chlorides, bromides, and boron-containing compounds can act in physically or chemically. It can cause a charred layer of carbon to form on the surface (Demir et al., 2005). The fire retardant chemicals act as by conversion of volatile gases to on flammable gases, promotion of char formation, forming an intumescent foam barrier at the surface, free radical termination in the gaseous phase, and occurring a glaze barrier at the surface (Ötsman & Tsantaridis, 2016). The fire retardants can affect the reaction to fire properties, however this effect is lower for the fully developed fire. When intumescent paints are used the time for start of charring can be delay and the fire resistance can be increased. The fact is that fire retardants cannot make wood non-combustible. The fire performance of fire retardant treated the virgin wood products may degrade depending on time in outdoor applications. The fire retardant chemicals can be removed in the wood surface when the wood surface is exposed to high humidity (Nussbaum, 1988; Östman et al., 2001; Ötsman & Tsantaridis, 2016). Mineral fillers such as titanium dioxide for intumescent fire retardant coating have attracted much attention in recent years. Titanium dioxide (TiO<sub>2</sub>) can be also used in coating industry as pigments. Some researchers were found that the presence of rutile type  $TiO_2$  could improve the fire resistance of the coating by enhancing char formation (Li et al., 2015; Lam et al., 2011). Antimony trioxide is usually used as a synergistic energy to improve fire retardancy (Giúdice & Benítez, 2001).

Pine wood which is a type of softwood grown in many varieties in world has a uniform texture, finishes well,

and east to work. It is usually light yellow in color and also some resistance against shrinkage, sharping and swelling (Zhong et al., 2013). It is considered that these properties will be improved by the use of some chemicals treated wood preservative paint. The wood preservatives contain pentachlorophenol or creosote in oil, water-borne salt treatments, copper, zinc, chromium, arsenic, and other compounds can protect wood against attack by fungi, bacteria, and insects (Thamasson et al., 2006).

There is a need to search new fire retardant treated wood products with improved long term durability of the reaction to fire performance in many usage areas, recently. The main aim of the study is to obtain an improved fire performance of pine wood surface. In this study, the effects on the flammability (the limiting oxygen index (LOI) and real fire test) of pine wood surface with applied of titanium dioxide and antimony trioxide were investigated owing to the synergy effect of wood preservative paint and these chemicals. The surface roughness, color change, brightness and physical properties of the pine wood surfaces were also measured in according to the relative standards.

## **MATERIAL and METHODS**

*Materials: Pinus sylvestris L.* solid wood surface was used for improving its flammability properties in this study. The wood preservative paint was obtained from Polisan, Turkey. Titanium dioxide and antimony trioxide were purchased from Sigma-Aldrich for this study. The purity grades of these chemicals are above 99,99%. Table 1 and 2 show average size of chemicals and sample codes, respectively.

Table	1.	Average	size	of	chemica	ls.
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Chemical type	Average size (nm)
Titanium dioxide	30≥
Antimony trioxide	30≥

Table	2.	Composit	ion	of fire	retardant	coatings,	%	by	volume	on
wood p	ore	servative j	pain	t						

Sample Code	2%	5%	10%
Control	-	-	-
WPP	-	-	-
A1	х	-	-
A2	-	х	-
A10	-	-	х
T1	х	-	-
T2	-	х	-

WPP: Wood preservative paint; A: Antimony trioxide; T: Titanium dioxide.

**Preparation of wood surfaces:** Firstly, the wood preservative paint is applied onto the pine wood surface. Amounts of fire retardant chemicals were changed in proportion to amounts of titanium dioxide (2% and 5%) and which of antimony trioxide (2%, 5% and 10%). Secondly, they were mixed for 5 minutes with the wood preservative

paint, and then applied by a brush onto solid pine surface. Once again was painted by the wood preservative paint after the wood surfaces were dried for 12 hours.

*Fire properties:* The limiting oxygen index (LOI) test method was determined using a Dynisco Limiting Oxygen Index Chamber according to ASTM D 2863 to measure the minimum oxygen concentration. Four samples with the dimensions of 10 mmx15mmx5 mm were used for each group. All wood samples were placed in vertical glass column and then adjusted oxygen and nitrogen flow. The LOI test determines minimum concentration of oxygen and nitrogen flowing upward in a test column. The five samples for each group were tested.

The real fire test was also performed. The sample dimensions were the same as the dimensions of the LOI sample. All samples were burned to the mark in mm/min. The blowtorch was kept at a distance of 10 cm from the wood surface. The flame was applied for 5 seconds. The flame extinguishing time was recorded after the flame source cut off. Four samples for each group were tested.

**Color measurements:** All surfaces were removed from dust and dirt to minimize the risk of variation of color values by differences in surface structure. The color measurement was carried out according to CIE L\*a\*b\* method by parameters L\*a\*b\* and  $\Delta E$ \* with a PCE-TCD 100 (CM10P Color Meter). Measurements were realized on both sides of wood samples. The arithmetic mean of these measurements was calculated for each sample. The coordinates L\* (lightness or black-white relation), a\* (coordinate red-green), b\* (coordinate yellow-blue), measured on unpainted and painted solid pine wood were used to determine overall color change  $\Delta E$ \* by using the CIE L\*a\*b\* color measuring system.

*Surface roughness:* The residual particles remaining after painting were removed by cleaning the surface. Surface roughness of painted or unpainted samples was measured with a profilometer (Mitutoyo SJ-210). Equipment has stylus with  $5\mu$  radius and  $90^\circ$  contact angle running at speed of 0.5 mm/s. A total of 25 random measurements with a span of 15 mm were taken from the surface. Mean peak to valley height ( $R_z$ ) which is well accepted roughness parameter was used an indicator of the surface quality of wood samples. The measurements were performed in different areas, along two different perpendicular directions (longitudinal and tangential).

*Surface brightness*: The brightness measurements were measured by PCE-GM 100. Measurements were done with a gloss meter which measured at 20°, 60°, and 80° as parallel and perpendicular to the fibers for each wood surface before and after painting.

*Physical testing:* The thickness swelling (TS) and water absorption (WA) were determined after 2h, 24h, 48h, 72h, 96h and 120h soaking in water in accordance with EN 317.

Statistical Analysis: The statistical analysis was carried out with using SPSS 21.0 statistical package software. The results were statistically tested with the one-way analysis of variance. The significance (P<0.05) between the samples was compared with Duncan homogeneity groups. Each value is an average of 6 specimens and the values in the parentheses are standard deviations.

## **RESULTS and DISCUSSION**

Reaction to fire properties: The results of LOI and real fire test are shown in Table 3. LOI levels of the samples were in the range of 26,5-27 for pine wood and wood preservative paint. The LOI levels of antimony trioxide and titanium dioxide with samples were ranging from 28,5 to 30. A maximum LOI level was obtained with A10 sample including antimony trioxide of 10%. Antimony trioxide is a well-known pigment for the interference on flame spreading velocity. It shows an adequate fire-retardant behavior during the fire spreading reaction. But it is not effective it by itself. It should be used in combination with halogenated organic compounds. The antimony trioxide increases smoke production since the char analysis shows that about 80-95% of antimony volatilizes. It also decreases concern for the toxicity of antimony oxide (Giúdice & Benítez, 2001). According to ISO 4589, control and WPP samples were located in "Limited fire retardant material" class while A1, A10, T1 and T2 were classified in "Fire retardant material" class, as seen in Table 4. For real fire test, the effect of A10 and T2 was important to decrease the flame spreading. The fire retardant chemicals can act combining one or more mechanisms. Titanium dioxide shows a significant physical activity generated by reducing the concentration of the organic part which divides the material in isolated pieces by occupation of pores and amorphous polymer regions. As a result of which a higher amount of heat is required to attain the pyrolysis temperature (Giúdice & Benítez, 2001). Hashim et al., (2009) reported that lignocellulosic material reduced heat release rate and increased charring rate. When compared with char weight of all samples, a minimum value was obtained with T2 sample, followed by A10 sample.

Table 3. LOI and real fire test results of samples.

Sample ID	LOI	After flame time (sec.)	After glow time (sec.)	Weight loss (%)	Smoking
Control	26,5	60	60	1,689	+
WPP	~27	60	29	0,799	
A1	28,5	60	17	0,759	
A2	-	60	4	0,750	
A10	~30	60	0	0,729	
T1	28,5	60	20	1,124	+
T2	28,5	60	10	0,558	+

LOI level	Classification of fire	Chemical
≤23	Flammable material	
24-28	Limited fire retardant material	Control, WPP
29-35	Fire retardant material	A1, A10, T1, T2



Figure 1. The images of samples after LOI test.

**Brightness and Color Properties:** The brightness (80°) values were increased by usage of antimony trioxide and titanium dioxide according to that of pine wood. This is clearly seen Figure 2. The brightness values of the samples were increased when ratio of antimony trioxide increased from 2% to 10% for antimony trioxide. Similar results were also seen in 2% and 5% ratios of titanium dioxide. The highest values of the brightness were obtained from A10 samples. No significant difference was found between  $\Delta E$  values of samples with antimony trioxide and titanium dioxide with samples based on Duncan test as displayed in Table 5.

**Table 5.** Statistical analysis results and homogeneity groups of the brightness,  $\Delta E$  and roughness test results.

Sample designation	Brightness			AF	Dra (uma)
	20°	60°	80°	- <u>A</u> E	κz (μm)
Control	0,24a*	1,76a	69,66a	2,80a	8,90b
	(0,05)**	(0,59)	(4,43)	(1,49)	(1,51)
WPP	4,96ab	24,32b	80,08b	2,30a	6,02ab
	(2,15)	(5,01)	(2,61)	(1,04)	(3,26)
A1	9,25bc	38,36cd	93,03c	2,38a	5,90ab
	(5,75)	(13,24)	(1,16)	(1,48)	(4,02)
A2	9,75bc	41,73d	98,02d	2,44a	5,51ab
	(1,24)	(3,85)	(2,84)	(1,26)	(3,90)
A10	9,17bc	38,95cd	112,74f***	2,40a	2,78a
	(2,59)	(5,55)	(3,74)	(0,53)	(1,53)
T1	11,37c	39,62d	107,81e***	3,32a	4,10a
	(8,59)	(10,91)	(2,21)	(0,31)	(1,34)
T2	7,15bc	29,64bc	110,61ef***	2,48a	3,98a
	(2,97)	(7,53)	(3,33)	(1,16)	(1,93)
Groups with same letter	s in column indi	cate that there	s no statistical dif	ference (p <	0.05) between

the samples according to Duncan's multiply range tests which were performed separately for each group.

\*\* The values in the parentheses are standard deviations.

\*\*\* Because UV filter is not used, the results are more than a hundred.

Figure 3 shows  $\Delta E$  values of all samples. There were no apparent differences between the WPP and antimony trioxide for  $\Delta E$ . As you can see Table 6, all samples are taken part into so little differences group. It was observed that  $\Delta E$  is not significantly changed by the addition of 5 wt % of antimony trioxide ratios. However, the addition of antimony trioxide content above 5 wt%,  $\Delta E$  decreased gradually.  $\Delta E$  was found to be 2,38-2,44 for WPP with applied by 10% antimony trioxide, and 2,48-3,32 for WPP with applied by 5% titanium dioxide. While titanium dioxide ratios increased,  $\Delta E$  increased slightly. The reason of this is that titanium dioxide improves brightness and smoothness (Stoneburner, 2014).



Figure 2. Brightness values (80°) of the samples.



**Figure 3.**  $\Delta E$  values of the samples.

**Table 6.** Color  $\Delta E$  differences (Heidelberg, 2008)

	( 8)
0-1	Unseen differences
1-2	So little differences
2-3,5	Intermediate differences
3,5-5	Apparent differences
>5	Obvious differences

Surface roughness: Table 5 and Figure 4 indicates  $R_z$  values of the samples. The highest  $R_z$  value of 8,90 $\mu$ m was found for control sample. Rz values of WPP samples with added antimony trioxide were decreased ranging from 2% to 10%. Rz values of WPP samples with added titanium dioxide were also reduced ranging from 2% to 5%. The minimum R<sub>z</sub> value was found for A10 samples while the control had the highest R<sub>z</sub> value which was indicated by statistical test. Both WPP samples with antimony trioxide and titanium dioxide for the minimum Rz value resulted in 2,78 µm and 3,98 µm, respectively. The quality of coating applied to surface of wood which being a nonhomogeneous material is affected by several factors such as roughness, porosity, species, density and interaction between coating and the substrate (Cheng & Sun et al., 2006; Ozdemir et al., 2015). The component of used fire retardants chemicals would be considered one of the mentioned factors influencing roughness as well as bonding between wood preservative paint and the pine wood surface in this study.



Figure 4. R<sub>z</sub> values of the samples.

Physical properties: The water absorption and thickness swelling results of samples after immersion in distilled water for 120 hours are given in Figure 5 and Figure 6. It was seen that the increasing of water immersion time resulted in WA and TS values to increase for all samples. While the water absorption and thickness swelling of control sample were higher than those of painted samples. The usage of antimony trioxide and titanium dioxide decreased WA and TS. The minimum value for WA was achieved from T2 sample. It is known that fire retardant chemicals prevent the samples to intake water by penetrating to the lignocellulosic material (Donmez et al., 2016). Some researchers have reported that fire retardants such as zinc borate, aluminum trihydrate, aluminum trihydroxide, sodium aluminate) reduced water absorption and thickness swelling (Gnatowski & Burnaby, 2005; Hashim et al., 2009; Donmez et al., 2016).



Figure 5. Long-term water absorption of the samples



Figure 6. Long-term thickness swelling of the samples.

#### CONCLUSIONS

Consequently, the LOI results showed that pine wood surface painted with antimony trioxide and titanium dioxide had affirmative effects in comparison with control and WPP. A1, A10, T1 and T2 were in class of "fire retardant material" according to ASTM D 2863. Using A10 as a fire retardant exhibited the best result in terms of fire performance. As the increase in ratio of used chemicals for real fire test, it was concluded that the char weight of the samples was decreased. The long-term thickness swelling and water absorption values of painted with the chemicals have given better results than control. The  $\Delta E$  of samples painted by using antimony trioxide and titanium dioxide did not change significantly. Increasing titanium dioxide has little improved the  $\Delta E$ . The brightness value of samples increased while increasing ratio of chemicals. However, Rz values of the samples painted using antimony trioxide and titanium dioxide had a lower in comparison with control and WPP. With an increase in antimony trioxide and titanium dioxide loading, the R<sub>z</sub> values decreased slightly.

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#### ARAŞTIRMA MAKALESİ

**RESEARCH PAPER** 

The Oxidative Pretreatments of Cellulose for Cellulosic Superabsorbents

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Abstract: Cellulosic superabsorbents (SAP) are produced by using cellulose and cellulose derivative blends with different binding methods, generally. But cellulose in SAP leads decrease in water absorption and water bonding ability because of nonreactive character of cellulose based on its chemical structure. The oxidative pretreatments of cellulose were attempted for the resolve of disadvantage of cellulose in SAP, in this study. Hydrogen peroxide and TEMPO (2,2,6,6- tetramethylpiperidine-1-oxyl) were used for oxidative pretreatments of cellulose. Oxidized cellulose and carboxymethylcellulose (CMC) blended and cross-linked by epicholohydrin for cellulosic SAP production. Water absorption capacity of cellulosic SAP were determined in pH:2, 7 and 10. The results show that the oxidative pretreatments of cellulose had affirmative effect of water absorption capacity of cellulosic SAP according to control samples.

Keywords: Cellulose, carboxymethylcellulose, crosslinking, epicholohydrin, superabsorbents.

# Selülozik Süperabsorbent Üretiminde Oksidatif Ön İşlemler

Öz: Selülozik superabsorbent (SAP) üretiminde genelde selüloz ve selüloz türevi farklı oranlarda karıştırılarak kullanılmaktadır. Ancak kullanılan selüloz oranı arttıkça selülozun kimyasal yapısına bağlı olarak süperabsorbentlerin su alma ve absorplama kapasitesi azalmaktadır. Bu çalışmada selüloza uygulanan oksidatif ön işlemlerle bu olumsuz etkinin azaltılması amaçlanmıştır. Selülozun oksidasyon reksiyonları için hidrojen peroxide ve TEMPO (2,2,6,6- tetramethylpiperidine-1-oxyl) kullanılmıştır. Okside selüloz ile karboksimetil selüloz epiklorohidrin çapraz bağlama yöntemi ile işleme tabi tutularak süperabsorbentler üretilmiştir. Üretilen selülozik SAP'ların su alma ve absorplama kapasiteleri pH:2,7 ve 10 koşullarında test ediliştir. Kontrol örnekleriyle yapılan karşılaştırmalarda selüloza uygulanan oksidatif ön işlemlerin SAP'ların su alma kapasitelerinde olumlu etki gösterdiği tespit edilmiştir.

Anahtar sözcükler: Çapraz bağlama, epiklorohidrin, karboksimetil selüloz, selüloz, süperabsorbent.
# INTRODUCTION

Superabsorbent hydrogels (SAP) are threedimensional cross-linked hydrophilic polymers with the ability to absorb large quantities of water, saline or physiological saline solutions compared to ordinary absorbing materials (Chang et al., 2010). They are able to absorb water and other liquids tens to thousands times their own weight in a relatively short time. They can also retain a swollen state even under some pressure. They have superabsorbent properties conferred by their hydrophilic groups or domains. Given all these advantages, superabsorbent polymers are widely applied in various fields, such as in hygiene, medicine, nutrition, petrochemistry, agriculture, and horticulture (Sannino, 2009; Wu et al., 2012).

Most of the current SAPs available commercially are synthetic polymers based on acrylic acid or acrylamide, which are expensive, poorly degradable and harmful to the environment (Khoo et al., 2014). Some natural resources such as polysaccharides and inorganic clay minerals have also been used to produce superabsorbent polymers (Li et al., 2012). Cellulose and its derivatives is often used in the biomedical field, and cellulose-based superabsorbents (cel-SAP) have been prepared using radiation-induced and chemical crosslinking. (Rémond et al., 2010). In previous studies, it was determined that the main drawback of cel-SAPs is the decrease in swelling rate depend on cellulose content increases in cel-SAP (Bao et al., 2011; Chang et al., 2010; Hubbe, 2013).

In this study, it was aimed to be minimize the negative effect of high cellulose content in cel-SAP on swelling rate by using hydrogen peroxide and TEMPO (2,2,6,6- tetramethylpiperidine-1-oxyl) oxidative pretreatments of cellulose.

### **MATERIAL and METHODS**

*Materials:* Spruce dissolving pulp was used as cellulose resource. Alpha cellulose content, Kappa no and DP of spruce dissolving pulp were determined 95.2%,  $\leq 1$  and 1406, respectively. Carboxymethylcellulose (CMC) was used as Mw: 700000. Epichlorohydrin (ECH), NaOH, urea, hydrogen peroxide and TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) were obtained as analytical-grade.

#### Methods

**Oxidative Pretreatments of Cellulose:** Hydrogen peroxide and TEMPO were used for oxidative pretreatments of cellulose (spruce dissolving pulp). Hydrogen peroxide, NaOH (o.d. dry pulp) and pulp consistency were selected as 10%, 3% and 10%, respectively. Temperature and pretreatment time were applied with 80 °C and 180 min, respectively.

Cellulose sample (5 g) was dispersed in water (375 mL), containing TEMPO (0.0625 g) and sodium bromide (0.625 g). TEMPO mediated oxidation was applied by adding NaClO solution containing 5 mmol NaClO to the cellulose suspension at room temperature. The cellulose suspension was stirred at pH 10 by continuous addition of 0.5 M NaOH. The TEMPO-oxidized cellulose thus obtained was washed thoroughly with ethanol by filtration (Puangsin et al., 2013).

**Preparation of cel-SAP:** Untreated cellulose (UC) and oxidative pretreated cellulose (OPC) solutions were prepared as follows: 5 g spruce dissolving pulp cellulose samples (UC and OPC) were suspended into 195 g of 6 wt% NaOH/4 wt% urea/90 wt% water mixture with stirring and then was stored under refrigeration (-20 °C) for 12 h. The frozen solid was thawed and stirred with IKA Ultraturrax at 10000 rpm at room temperature to obtain a transparent cellulose solution. CMC was dissolved in the same solvent to obtain a polymer concentration. ECH and NH<sub>4</sub>OH (25 ml) was added to the mixture as cross-linker, stirred at 40 °C for 4 h to obtain a homogeneous solution, and to prepare hydrogels. Cellulosic hydrogels were washed with ethanol and water to obtain hydrogels (Chang, 2010). The conditions of cel-SAP preparation are presented in Table 1.

Table 1. Cel-SAP preparation conditions.

Cellulose (%)	CMC (%)	Suspension (ml)	ECH (ml)	NH4OH (ml)	Temperature (°C)	Time (hour)
10	90	200	25	25	40	4
30	70	200	25	25	40	4
50	50	200	25	25	40	4

**Determination of Water Absorption Capacity:** The synthesized cel-SAPs were immersed in water at constant room temperature ( $25 \circ C$ ) for 12 h. Water absorption capacity and swelling in acidic, alkaline and neutral conditions of cel-SAPs was determined by adjusting the water to pH 2, 7 and 10. Cel-SAPs were filtrated to remove excess distilled water. Water absorption capacity was calculated by the following equation:

WAC =
$$(M_2 - M_1) / M_1$$
 (1)

Where,  $M_1$  and  $M_2$  are the mass of the dried and swollen sample, respectively. The WAC value was calculated as grams of water per gram of sample.

*SEM Analysis:* Surface properties of superabsorbents were investigated by scanning electron microscopy images (Jeol JSM-6060- Zeiss Evo LS-10). Cel-SAP sample were coated with Au before SEM observation.

# **RESULTS and DISCUSSION**

*Water Absorption Capacity of Cel-SAPs:* Cel-SAPs were prepared cellulose and CMC at different ratios (10/90, 30/70 and 50/50) by using ECH crosslinking method, as can

be seen in Table 1. HCl (pH:2) and NaOH (pH:10) were used for acidic and basic adjustment, before water absorption capacity determination. The results of water absorption capacity in distilled water at 25 °C for cel-SAPs which were prepared with untreated cellulose (UC) and CMC mixture are presented in Figure 1.



Figure 1. Water Absorption Capacity of Cel-SAPs which were prepared UC and CMC mixture.

It was determined that water absorption capacity of cel-SAPs increased evidently with an increase CMC content in cel-SAPs. CMC has highly hydrophilic carboxyl group. These groups could absorb to enhance WAC of cel-SAPs (Wüstenberg, 2015). As can be seen in Fig 1, high UC content in cel-SAPs had negative effect on WAC of hydrogels. Chang et al., (2010) studied on water absorption capacity of cellulose/CMC hydrogel. They found that the highest WAC for cellulose /CMC (90/10) hydrogel was 1000% after immersing in distilled water for a week at 25 °C. CMC is a polyelectrolyte, which shows sensitivity to pH and ionic strength variations. Indeed the presence of CMC in a cellulose-based hydrogel provides the hydrogel itself with electrostatic charges anchored to the network, which have a double effect on the swelling capability. On one side, the electrostatic repulsion established between charges of the same sign forces the polymer chains to a more elongated state than that found in a neutral network, thus increasing the swelling. On the other, the counterions that are present in the gel to ensure macroscopic electrical neutrality induce more water to enter the network (Sannino, 2009).

WAC ratios of cel-SAPs which were prepared with hydrogen peroxide treated cellulose (HP-PC) and CMC mixture are presented in Fig 2. The results showed WAC in distilled water at 25 °C of cel-SAPs increased for pH:2 and pH:7, when it decreased for pH:10. Wu et al., (2012) investigated the absorption capacity of cellulosic superabsorbent composites in various solutions between pH:1 and pH:13. They found that absorption capacity of SAP composite was lower at pH:1 and pH:13 than neutral pH. They explained this finding that the screening effect of the Na+ counterions in the swelling medium led to decreased water absorption in highly alkaline solutions. WAC ratio found higher at 30% HP-PC content in cel-SAP than other HP-PC contents for pH:2 and pH:10, in this study.

TEMPO oxidative pretreatment applied to spruce dissolving pulp samples (T-PC), after blended with CMC at different ratios. Cel-SAPs formed T-PC and CMC mixture produced with epicholohydrin crosslinking method. WAC of cel-SAPs which formed T-PC and CMC mixture are presented in Fig 3.



**Figure 2.** Water Absorption Capacity of Cel-SAPs which were prepared HP-PC and CMC mixture.



**Figure 3.** Water Absorption Capacity of Cel-SAPs which were prepared T-PC and CMC mixture.

As can be seen in Fig 3, It was determined TEMPO oxidation pretreatment had more effective on WAC of cel-SAPs than hydrogen peroxide pretreatment. Saito & Isogai, (2004) found that crystallinities and crystal sizes of cellulose I were nearly unchanged during the oxidation, and thus, carboxylate and aldehyde groups were introduced selectively on crystal surfaces and in disordered regions of the water-insoluble fractions. Water retention values of cotton linter could be increased from 60% to about 280% through the introduction of hydrophilic carboxylate groups and morphological changes from fibrous forms to short fragments by the TEMPO-mediated oxidation.

*SEM Analysis:* SEM photos of cel-SAPs which were formed UC, HP-PC and T-PC, and CMC are presented in Fig 4. SEM photos showed that its surface were uneven and wide surface area. It was determined that surface SEM

photos of cel-SAPs were changed with oxidative pretreatments (hydrogen peroxide and TEMPO) of cellulose. It can be said that the ECH crosslinking mechanism between

cellulose and CMC changes according to oxidative pretreatments.





(B)

(B)



Figure 4. SEM photos of cel-SAPs (A)which were prepared UC and CMC mixture, (B) which prepared HP-PC and CMC mixture, (C) which were prepared T-PC and CMC mixture.

### CONCLUSION

It was determined that spruce dissolving pulp had affirmative effect on cel-SAPs compared to previous studies. TEMPO pretreatment had more effective on WAC at pH:7 of cel-SAPs than hydrogen peroxide pretreatment, but not at acidic medium (pH:2). It was found that WAC at basic medium (pH:10) of cel-SAP decreased with TEMPO and hydrogen peroxide pretreatments of cellulose.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Effects of Pressing Time on Some Technological Properties of Laminated Veneer Lumber (LVL) Produced Using Polythene Waste as Adhesive

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**Abstract:** It is desired the wood composite materials which have broaden using areas in human residence have no negative effects on human health and environment. But because of formaldehyde content of most of resins used in wood based composite production, wood composite materials have been seen a threat on human health and environment. Therefore, numerous studies have been made to develop an effective method to decrease formaldehyde emission from the wood based panels and various methods have been tried.

The aim of the study was to investigate those effects of pressing time on some technological properties of polythene composite LVL. Poplar (*Populus deltoides*) veneers were used to produce polythene composite LVL. Three different pressing time (8, 10, 12 minutes) and pressing temperature 140 °C were chosen as press parameters in the manufacturing of polythene composite LVL. Bonding strength, bending strength, modulus of elasticity, density and equilibrium moisture content of polythene composite LVL panels were determined according to TS EN 314-1, TS EN 310, TS EN 323 and TS EN 322, respectively. The experimental results showed that technological properties of panels generally increased with decreasing pressing time.

Keywords: Polythene composite LVL, pressing time, technological properties.

# Presleme Süresinin Yapıştırıcı Olarak Polietilen Atık Kullanılarak Üretilen Lamine Kaplama Kerestenin (LVL) Bazı Teknolojik Özellikleri Üzerine Etkisi

Öz: Yaşam alanlarındaki kullanımı genişleyen ahşap kompozit malzemelerin, insan sağlığı ve çevre üzerinde olumsuz bir etkisinin olmaması istenmektedir. Ancak odun esaslı kompozit üretiminde kullanılan tutkalların çoğunun formaldehit içeriği nedeniyle, odun kompozit malzemelerinin insan sağlığı ve çevre üzerinde bir tehdit oluşturduğu görülmüştür. Bu nedenle, ahşap esaslı panellerden formaldehit emisyonunu azaltmak için, etkili bir yöntem geliştirme amacıyla çok sayıda çalışma yapılmış ve çeşitli yöntemler denenmiştir.

Bu çalışmanın amacı, presleme süresinin polietilen kompozit LVL'nin bazı teknolojik özellikleri üzerindeki etkilerini araştırmaktır. Polietilen kompozit LVL üretmek için Kavak (*Populus deltoides*) kaplamaları kullanılmıştır. Polietilen kompozit LVL imalatında pres parametreleri olarak üç farklı presleme süresi (8, 10, 12 dakika) ve presleme sıcaklığı 140 °C seçilmiştir. Polietilen kompozit LVL panellerin yapışma dayanımı, eğilme dayanımı, elastikiyet modülü, yoğunluk ve denge rutubet miktarı sırasıyla TS EN 314-1, TS EN 310, TS EN 323 ve TS EN 322'ye göre belirlenmiştir. Deneysel sonuçlar panellerin teknolojik özelliklerinin genellikle presleme süresinin azalmasıyla arttığını göstermiştir.

Anahtar sözcükler: Polietilen kompozit LVL, presleme zamanı, teknolojik özellikler.

# INTRODUCTION

Wood has characteristics that make it suitable for many uses. Laminated veneer lumber (LVL) is manufactured from veneers with equal or different thicknesses or wood species glued together, ensuring that grains are glued parallel (Gaff and Gašparík, 2015;Shukla and Kamdem, 2009). LVL panels are of great structural use due to their high mechanical strength, great dimensional stability and ability to receive preservative treatment (Kamala et al., 1999). It has potential for use in structural and nonstructural applications such as in the construction and furniture industries, as a material for flooring, as structural beams, headers, columns and in both residential and commercial applications and numerous interior and exterior application areas (Burdurlu et al., 2007; Souza et al., 2011; Melo and Menezzi, 2014).

LVL may be one of the most important solutions concerning raw material economy. It is also one of the wellknown and commercially produced engineered wood products (EWPs) in the forest products market in North America and Europe. LVLs can be used for structural and non-structural purposes due to their high strength, dimensional stability, consistency, and treatability (Nelson, 1997).

Importance of wood based materials gradually increases due to growing demand to wood materials and existence and decreasing quality of raw materials. Therefore, use of adhesive extensively increases and use of raw material source develops in forest product industry. It is stated that adhesives used in about 70% of application in woodworking industry (Aydin et al., 2010). At present, urea formaldehyde and phenolic resins are the adhesives used mainly in plywood production and account for 87.1% and 9.6% of all adhesives used in plywood manufacture, respectively, in 2004 (Qian, 2006). Urea formaldehyde resin is non-flammable and resistant to changes in high temperature, light and corrosion. It has good adhesive strength is, a short curing time, simple production technology and low production costs. But it also has a number of disadvantages, such as a high curing shrinkage ratio, a brittle colloidal property, weak water resistance and formaldehyde emission. Phenolic resins are able to enhance bonding strength and water resistance, but they require a long curing time, high curing temperatures, and have high production costs and emit formaldehyde and phenol (Cui et al., 2010). Formaldehyde release depended on content of release causes adverse health effects such as eve and respiratory irritation, irritability, inability to concentrate and sleepiness (Colak and Colakoglu, 2004). Also, The International Agency for Research on Cancer (IARC) in 1995 attached to formaldehyde in terms of human health "Possible Substances" and Carcinogenic class the ratio of formaldehyde can release from wood based materials was limited in most of country (IARC, 2004; Colakoglu, 1993). After this area investigated comprehensively, IARC in June 2004 removed formaldehyde from "Possible Carcinogenic

Substances" class and identified as an agent caused directly carcinogen for human (Jianying et al., 2010). It is started to prefer alternative adhesives or using formaldehyde scavenger prevented formaldehyde release in industry due to this disadvantage of formaldehyde based resins. Although some of these new adhesives have already been used in industrial applications, their supply is limited which may be due to the high modification costs or some their poor properties, for example, low wood resistance (Fang et al., 2013). Therefore, the chemicals and adhesives used are both cheap and easily accessible and its technological properties qualify according to usage of wood based panels (Colak et al., 2016).

Solid waste composition varies with changing consumption habits in Turkey, population growth, rising living standards, the increase in packaged product sales. Overall, 20% by weight and 50% by volume of the waste consists of packaging waste is formed (Official Gazette, 2014). Recycle of polythene constitutes an important part of packaging waste is gained importance due to both dissolution of nature for a long time and harmful gases are released by burning into the atmosphere (Colak et al., 2016). In literature, it was shown that the wood based panels obtain from using plastic and textile fiber waste are petrochemical materials as an adhesive gave successful results (Cui et al., 2010; Kajaks et al., 2012; Kofi, 2014). From there, it is thought that the polythene constitutes a serious waste of potential for our country and is a petrochemical is evaluated in LVL industry.

## **MATERIAL and METHODS**

Poplar logs were selected in the study. The LVLs used in this study were manufactured using rotary peeled veneers. Logs were not steamed or boiled prior to peeling. Rotary peeled veneers clipped into 500 mm long by 500 mm wide by 2 mm thickness and shipped to a manufacturing site. The veneers were pre-selected for strength and appearance. They were conditioned in an environmentally controlled room in relative humidity of  $65 \pm 5\%$  and a temperature of 20  $\pm 2$  °C until they reached the equilibrium moisture content of 12%.

The veneers were then dried to 6-8% moisture content with a veneer dryer. After drying, it was formed polythene composite LVL panel drafts. The draft of polythene composite LVL is shown in Figure 1.



Figure 1.Draft of polythene composite LVL

Three-ply-LVL panels with 6 mm thick were manufactured by polythene wastes. The polythene wastes were lay outed at rates of 160 g/m<sup>2</sup> to the single surface of veneer. Hot press pressure was 8 kg/cm<sup>2</sup> for poplar while hot pressing time and temperature were 8, 10, 12 minute and 140°C, respectively. Two replicate panels were manufactured for each test groups.

#### Table 1. Press parameters.

GROUP	Bonding Types	Pressing Time (m)	Press Pressure (kg/cm <sup>2</sup> )	Press Temperature (°C)
Control	UF	6	8	110
А	Polythene	8	8	140
В	Polythene	10	8	140
С	Polythene	12	8	140

The specific gravity, shear strength, bending strength and modulus of elasticity of polythene composite LVL panels were determined according to TS EN 323-1 (1999), TS EN 314-1 (1998), and TS EN 310 (1999) standards, respectively.

# **RESULTS and DISCUSSION**

Technological properties test results of polythene composite LVL panels according to press parameters were presented in Table 2 and Figure 2-3.

Table 2	2.	Technological	properties	of	polythene	composite	LVL
panels.							

Test Methods	Control (UF)	8 minutes	10 minutes	12 minutes
Banding strength (N/mm2)	3,99	2.647	3.107	2.787
Bonding strength (N/min <sup>2</sup> )	(0,42)	(0,61)	(0,25)	(0,39)
Bending Strength	100,44	95,43	85,76	54,79
(N/mm <sup>2</sup> )	(17,20)	(7,05)	(8,54)	(17,76)
Madulus of Electicity (N/mm <sup>2</sup> )	7338,01	6719,31	6095,12	4687,38)
Modulus of Elasticity (N/IIIII-)	(695,88)	(302,49)	(233,95)	(772,39)
Demaitry (an/ann3)	0,497	0,495	0,493	0,499
Density (gr/cm <sup>-</sup> )	(0,03)	(0,05)	(0,02)	(0,06)
Equilibrium Maisture Contant (9()	6,034	4,825	4,469	4,966
Equilibrium Moisture Content (%)	(1,38)	(0,31)	(0,32)	(0,65)
*Values in perenthesis are standard de	wintions			







Figure 3. Effects of wood pressing time on the bonding strength of panels

The all of bonding strength values of polythene composite LVL panels were higher than 1 N/mm<sup>2</sup> determined according to DIN 68705-3 (2003). The bending strength and modulus of elasticity values of polythene composite LVL panels were higher than 40 and 4000 N/mm<sup>2</sup> determined according to DIN 68705-3 (2003), respectively. Generally, the lowest result is the C group. Colak et al. (2016) stated that molten state polythene, depend on both wood processing and wood anatomical structure, penetrates into porous structure of wood and filled cracks and cavities on surfaces, and this helps to have smoother veneer surfaces. In literature, it was stated that smooth surface veneer bonded better than rough surface veneer and so it shows better performance on mechanical properties (Frihart, 2005).

As a result; formaldehyde-free polythene composite LVL has been successfully produced using polythene waste as wood adhesive. This novel product shows considerable mechanical properties. The panels produced using 160 gr/m<sup>2</sup> polythene amount and 8 minute gave the best mechanical strength values. When this study applies in the LVL industry, can provide to both recycle polythene waste and prevent formaldehyde release. In addition, Polythene composite LVL waste can be used production of wood plastic composite panels.

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#### ARAŞTIRMA MAKALESİ

**RESEARCH PAPER** 

Industry 4.0 Awareness In Furniture Enterprises: Case Study Of Ordu And Giresun<sup>[\*]</sup>

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Abstract: Industry 4.0 forces enterprises to set up new teams in which know the software systems well, catch up with the new technologies, plan and program them, open for change and development. The Turkish furniture industry, having a suitable structure for development, may become an important sector where digital technologies stand out in the near future. Nevertheless, the structural and economic problems caused by the fact that the sector is composed of small and micro-sized enterprises, are emerging as a huge impediment in the use of digital technologies. This study aims the awareness of the furniture industry about the Industry 4.0 approach, the present possibilities, plans, and where they see themselves in terms of these developments. As a target group of the study, furniture enterprises of Ordu and Giresun selected. Data were collected through questionnaires from the 45 enterprises in these provinces and analyzed in a computer environment. The possible effects of Industry 4.0 on the furniture industry highlighted. What should be done in this transition process was emphasized. Suggestions developed and discussed.

Keywords: Furniture industry, industry 4.0, industry 4.0 awareness, SMEs.

# Mobilya İşletmelerinde Endüstri 4.0 Farkındalığı: Ordu ve Giresun Örneği

Öz: Endüstri 4.0, işletmeleri, yazılımı iyi tanıyan, yeni teknolojileri takip eden, planlayan ve programlayan, değişime ve gelişime açık yeni ekipler kurmaya zorlamaktadır. Mobilya endüstrisi yapısı gereği gelişmeye açık olması dolayısıyla, yakın gelecekte dijital teknolojilerin ön plana çıktığı bir sektör haline gelebilir. Bununla birlikte, sektörün küçük ve mikro ölçekli işletmelerden oluşması ve küçük olmasının neden olduğu yapısal ve ekonomik sorunlar dijital teknolojilerin kullanımında büyük bir engel olarak ortaya çıkmaktadır. Bu çalışma ile mobilya işletmelerinin Endüstri 4.0 yaklaşımına yönelik farkındalıkları, mevcut imkânları, gelecek planları ve kendilerini bu gelişmelerin neresinde gördükleri ortaya konmaya çalışılmıştır. Hedef kitle olarak Ordu ve Giresun illerindeki mobilya işletmeleri seçilmiştir. Bu illerde 45 işletmeye gidilerek anket yardımıyla veriler toplanmış ve bilgisayar ortamında analiz edilmiştir. Sonuç olarak, Endüstri 4.0'ın mobilya endüstrisine olası etkileri, işletmelerin bu geçiş ve dönüşüm sürecinde neler yapması gerektiği vurgulanmış ve öneriler geliştirilerek konu tartışmaya açılmıştır.

Anahtar sözcükler: Endüstri 4.0, endüstri 4.0 farkındalığı, KOBİ, mobilya endüstrisi.

# INTRODUCTION

With the onset of the industrial age towards the last quarter of the eighteenth century, a great change and development occurred in the industry. These changes and developments defined as industrial revolutions. There are four industrial revolutions to date. The first one took place in the nineteenth century, and in addition to human power in the industry, machinery, water, and steam power also used. After the first quarter of the 1900s, the concept of mass production started to the use of integrated machines together with electricity, and this change called the second industrial revolution. From the third quarter of the twentieth century, electrical and computerized machines started to use in the production line with the appearance of computers. This change also defined as the third revolution in the industry. Since then, countries and societies still have been experiencing the changes brought about by the Third Industrial Revolution. While this change has not been completed, while especially as the SMEs continued to resist this change, the industry has faced a new revolution. Since the middle of the first quarter of the 21st century, powerful sensors, artificial intelligence, learning, communicating, and managing processes, also known as the digital revolution or Industry 4.0, have named The Fourth Industrial Revolution.

Industry 4.0 brings a whole new way of life. It has occurred many technological concepts with this new concept that such as the internet of objects (IoT), the internet of systems, powerful sensors, three-dimensional printers, robotics, intelligent manufacturing systems, large data, cloud computing, artificial intelligence (Dirsehan, 2017). Keeping up with the rapid development of these newly emerging concepts is very important for the countries. Evaluating our country especially in terms of SMEs, it is seen that they are between the 2nd and 3rd Industrial Revolution, and far away from a deal with these new concepts. While machine investments, mass production technologies and production capacities in the 20th century seem to be a competitive advantage for companies, nowadays knowledge is providing a competitive advantage (Dirsehan, 2016).

In one of World Economic Forum reports published in 2015, 21-turning points that will shape our future's digital were mentioned. All of these turning points are expected to take place until 2025. These transformations that will occur in such a short period will significantly affect the competitiveness of our country's industrial sector. These mentioned turning points and expectation percentages are shown in the table below (Schwab, 2017).

It is almost impossible to predict the scope and breadth of this digital revolution, which will cause the economic, social, and cultural change in large dimensions. One of the biggest impacts in all of these areas is the strengthening of the governments, citizens, businesses' employees, shareholders, customers, and superpower relationships in smaller countries. Due to the disruptive impact of Industry 4.0 on the existing political, economic, and social models, these actors need to be aware that they have become part of a dispersed power system that demands more collaborative forms of interaction for them to succeed (Schwab, 2017).

Table 1. Turning points expected to occur by 2025.

Turning Points	Expected
	(%)
10% of people wearing clothes connected to the internet	91.2
90% of people having unlimited and free (advertising-supported) storage	91.0
1 trillion sensors connected to the internet	89.2
The first robotic pharmacist in the US	86.5
10% of reading glasses connected to the internet	85.5
80% of people with a digital presence on the internet	84.4
The first 3D-printed car in production	84.1
The first government to replace its census with big-data sources	82.9
The first implantable mobile phone available commercially	81.7
5% of consumer products printed in 3D	81.1
90% of the population using smartphones	80.7
90% of the population with regular access to the internet	78.8
Driverless cars equalling 10% of all cars on US roads	78.2
The first transplant of a 3D-printed liver	76.4
30% of corporate audits performed by AI	75.4
The tax collected for the first time by a government via a blockchain	73.1
Over 50% of internet traffic to homes for appliances and devices	69.9
Globally more trips/journeys via car-sharing than in private cars	67.2
The first city with more than 50,000 people and no traffic lights	63.7
10% of global gross domestic product stored on blockchain technology	57.9
The first AI machine on a corporate board of directors	45.2

Source: Deep Shift - Technology Tipping Points and Societal Impact, Global Agenda Council on the Future of Software & Society, World Economic Forum, Survey Report, September 2015, pp. 7."

It is almost impossible to predict the scope and breadth of this digital revolution, which will cause the economic, social, and cultural change in large dimensions. One of the biggest impacts in all of these areas is the strengthening of the governments, citizens, businesses' employees, shareholders, customers, and superpower relationships in smaller countries. Due to the disruptive impact of Industry 4.0 on the existing political, economic, and social models, these actors need to be aware that they have become part of a dispersed power system that demands more collaborative forms of interaction for them to succeed (Schwab, 2017).

It is not enough for enterprises to make only the production lines and technology compatible with the transition to Industry 4.0. Complete planning of production, marketing, delivery, after-sales services, customer relations, and feedback processes are required for a complete transition and success. Industry 4.0, which stands before us as a complex structure, is a long process starting from the ordering stage of the product, from designing, developing, and manufacturing to delivery to the end-user and feedback. This means that businesses need to completely renew themselves and integrate all systems. The positive effects of this revolution, which integrates people, objects and systems into one another and transform the world into a great information system in this process, can be summarized as follows (Kılıç, 2016):

• More automation in manufacturing with industry 4.0, mass production more connected to customer preferences, maximum manufacturing quality, localized manufacturing processes, rapid innovation process, and less resource use,

• Identification of the possibilities of production processes more flexible and free of system and applications,

• The minimum cost of producing highly personalized products for customers,

• Increase in data transmission speed and simplification of logistics operations by enabling products to be produced at closer centers utilizing 3D printers,

• The efficient use of energy resources.

A qualified workforce is one of the essential elements of Industry 4.0. Most of today's professions will disappear soon, and some will change. Surviving ability of today's operating systems, and aligning with Industry 4.0 depend on the ability to innovate from scratch, to perform digital transformations, and to become a new player on the market. To comply with Industry 4.0, changes in the qualifications of existing employees, work patterns, and job descriptions need to redefine. Soon, employees will be asked to have higher qualifications than today's employees. For these high qualities required, it will be necessary to restructure the education system and introduce radical change and transformation from beginning to end (Öztürk & Koç, 2017).

Industry 4.0 has entered into business life in the industry as internet technologies started to offer business solutions in production. "In this way, digitization and mutual relations have increased in business models. Companies in the manufacturing and engineering, automotive, process industries, electronics, information, and communication industries believe that digitalization will lead to transformation. This digitalization requires an investment of 40 Billion Euros in Germany and 140 Billion Euros in the EU each year until 2020. "(Koch et al., 2015; cited by Şenkayas et al., 2016). The biggest problem at this point is that there are ambiguities in the way that countries cannot afford these huge investments, how soon SMEs can benefit from this system, and the industrial internet can be fully understood. Particularly SMEs need to establish partnerships with technology and infrastructure companies, even with their competitors, to carry on their vital activities. In this context, the EU plans to support SMEs for high value-added and technologically advanced manufacturing (Davies, 2015; cited by Şenkayas et al., 2016).

When Turkey evaluates in terms of Industry 4.0, it seems to be quite far away from the desired point in this subject. Looking at the technological infrastructures of the enterprises in our country, we see that they could not complete the Industry 3.0 revolution yet, and they are in a position between Industry 2.0 and Industry 3.0. When TSI 2016 statistics examine, it seems that the total number of enterprises is 2.689.910, and the number of SMEs is 2.684.838. Proportionately, 99.81% of the total number of enterprises is SME. It is known that large-scale enterprises do not have difficulty in the transition to Industry 4.0 and are

already beginning to implement the transformations. However, the fact that the enterprise structure is SMEdominated, and especially the majority of SMEs is also micro-scale, it is a sign that we are quite far from the reality of Industry 4.0. As a country, we are still discussing the issue conceptually.

According to TSI 2016 data, when we look at the operating structure of the furniture sector, it is seen that the total number of enterprises is 34.477, and the number of SMEs is 34.417. When the enterprise numbers in the sector are evaluated proportionally, it is determined that 99.83% of the total number of enterprises is SME. The sector stands as a sector with producing in 61 provinces at the country scale, interacting with 19 different sectors, receiving input or giving input, having a high sector connection, and having a market share of 10 billion dollars (Öztürk & Koç, 2017).

"One of the factors that the furniture industry should consider in its forward-looking strategies is the growth of digital consumer mass. The result of the study is shown that 65% of consumers in Turkey are digitalized and digital consumers use every channel. It has been determined that digital consumers prefer products and services that make life easier for them, with competitive pricing and campaigns as a priority, and it has been determined that 60% use of them both digital and traditional channels." (Dumansızoğlu, 2017).

"Today's furniture is now seen as a high-tech design object. Large-scale enterprises in the sector know the concept of "Smart Factory" and Industry 4.0. They have started production processes with a new generation of project-based lines consisting of full automation and robotic processing. Branded furniture companies representing 75% of the sector are turning their production facilities to the AR-GE base. The use of full-automatic production lines with CNC-controlled and ERP systems has become a necessity, not the luxury, for brands anymore." (Anonymous, 2017). On the contrary, It is a question mark that whether SMEs understand this new concept or not, and do they know what is waiting for them in the future or not. "It is stated that in the first 6 months of 2018, the economic situation in the country, and the increase in the prices of raw materials are not reflected in the final product sales prices. It seems that enterprises are trying to reduce the number of workers to resist for surviving the adverse effects of this downsizing" (Anonymous, 2018).

This study aims that while putting great digital changes in the world until the year 2025, to look at the current situation of our country -especially in terms of SMEs. While the changes and transformations started at Industry 4.0 in terms of large-scale enterprises, it is aimed to create a conceptual, business-based, and country-based awareness for SMEs, especially small-scale enterprises. At this point, the furniture sector, which we consider to be one of the locomotive sectors of our country's economy, has been chosen as the subject of the study.

# **MATERIAL and METHODS**

The main aim of the study is to measure awareness and to compose awareness about Industry 4.0 in SME furniture enterprises. The universe of the study limits to Ordu and Giresun located in the Black Sea Region. The absence of large and medium-sized furniture enterprises in these provinces is an important influence in the election. Social Security Institution (SSI), Turkey Employment Agency (TEA), and Small and Medium Enterprises Development Organization's (SMEDO) records have been taken into consideration in determining business numbers. It determines that there are 43 enterprises in Ordu and 22 enterprises in Giresun. It was tried to reach all the furniture enterprises actively operating and producing in Ordu and Giresun.

The survey method was selected to collect data in the study. Firstly literature survey about awareness concepts, awareness scales and Industry 4.0 were done. As a result of the literature search, a previously prepared scale related to the topic didn't find. For this reason, a form with 80 questions organized with the help of the information obtained from a literature review. This form consists of four parts: demographic information, conceptual awareness, businessbased awareness, and country-based awareness. The prepared questionnaire applied to a sample enterprise, and the clarity of the questions on the questionnaire was tested. Factor analysis conducted to measure the consistency of these four sections on the questionnaire. With the information gathered, necessary regulations made in the questionnaire form and were put into practice. This prepared form was sent to the chosen enterprises via the internet environment. 45 enterprises responded to the questionnaire forms. 30 of these enterprises located in Ordu and 15 of them located in Giresun.

The survey was implemented in June-August 2018. 5 Likert Scale (Strongly Agree, Agree, Undecided, Disagree, Strongly Disagree) were used to evaluate the awareness of the businesses.

#### FINDINGS

**Demographic Information:** A total of 45 enterprises responded to the survey. 66.7% of these enterprises are in Ordu (30 enterprises), and 33.3% are in Giresun (15 enterprises). 62.2% of the enterprises are named as Private Company, 33.3% as Limited Company, and 4.4% as Joint Stock Company. 48.9% of the enterprises were established after 2000.

55.6% of the respondents are business owners, 22.2% are professional managers, 11.1% are engineers/architects/technicians, and 11.1% are the employee. When the respondents' gender distribution is examined, it is seen that 84.4% is male and 15.6% is female. When we look at age distribution, approximately 80% of respondents are 33 years of age or older. 45% of the

respondents are university graduates, and 42% are vocational or high school graduates. Only 13% are primary and secondary schools graduated.

31.1% of the enterprises are in the organized industrial zone, 31.1% are in the urban neighbourhood, and 37.8% are in the small industrial zone. 64.4% of enterprises responding to the survey are micro-scale enterprises, and 33.3% are small-scale enterprises. Only 1 of the enterprises, and 33.3% are small-scale enterprises. Only 1 of the enterprises, and industrial designers at 12 enterprises, technicians at 28 enterprises, and industrial designers at 11 enterprises are employed. It states that 77.8% of the enterprises have a medium level technology, 20% have a high-level technology, and 2.2% have the low-level technology. Approximately 75% of the participating enterprises say that they do not use CNC or NC looms in production. Up to now, 65% of the enterprises have not benefited from any support such as incentive, credit or KOSGEB support.

#### Industry 4.0 Awareness in Furniture Enterprises

*Conceptual Awareness:* The ratios of "strongly agree" and "agree" responses among the 20 questions prepared at 5 points Likert scale (Strongly Agree, Agree, Undecided, Disagree, Strongly Disagree) are as follows.

The average of the responses to the questions was calculated as 3.436, standard deviation score of 0.985, and standard error score of 0.033.

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I able	<i>L</i> .	Conce	ptual	Awareness	1n	enterprises.

Questions	Percentage
	(%)
I know the 1st Industrial Revolution.	44
I know the 2nd Industrial Revolution.	44
I know the 3rd Industrial Revolution.	53
I know the 4th Industrial Revolution.	58
I know mechanization in production.	78
I know what serial production is.	87
I know about electrically and computerization in production.	80
I know the concept of the internet of objects.	47
I know intelligent production technologies.	64
I know 3D printers.	78
I know smart factories.	80
I know artificial intelligence.	71
I know the concept of wearable internet.	31
I know the concept of Industry 4.0.	36
When production is mentioned, the mind first comes to work with factories and physical power	73
I understand the production of information from the concept of production and the work done with brainpower.	51
When I say Industry 4.0, I understand that machine power manages the production processes instead of human power.	38
3D printers are used only for 3-D books and similar prints.	33
Machines can connect and communicate with each other in the virtual environment and manage the process.	60
Industry 4.0 is a process that creates intelligent factories that can be developed and transformed by production technology, cyber-physical systems, Internet of objects and cloud computing.	36

A significant difference between the distribution of the respondents' enterprises and the level of awareness was not determined. It has found that the awareness levels of the enterprises are around 50%. A significant relationship found between the production sites and awareness levels of the enterprises. It is determined that as the technical infrastructure of the production sites improves, there is an increase in awareness level. While the awareness level of the enterprises in which producing between neighborhoods in the inner-city is around 38%, the awareness of those produced in the organized industrial zones is 74%.



Figure 1. Awareness levels according to the place of production.

A meaningful relationship found between the title of the respondents and awareness levels. It determines that the level of awareness of employer or employee is well below the level of awareness of professional managers or engineers/ architects/technicians in the enterprises. The employees' awareness level is 37%, that of employers is 49%, that of engineers/ architects/technicians is 72%, and also that of professional managers is 80%.



Figure 2. Awareness levels according to the qualitative of respondents.

When the respondents' awareness level to the questionnaire examined according to the age criterion, it seemed that there was a significant difference between the age group of 19-25 and the other age groups. As a person's age grows older, the awareness level has been reduced. Awareness levels of people aged between 19 and 25 determined as 78%.

There is also a significant relationship between the levels of education and awareness of respondents in the enterprises. As education levels of respondents increased, awareness levels increased too. It seems that the awareness level of primary graduates is 35%, that of secondary graduates is 27.5%, that of high school graduates is 37%, that of vocational high school graduates is 45%, that of bachelor's degree is 77.6%, and that of the master's or doctoral graduates is 95%.



Figure 3. Survey respondents' awareness levels by age.



Figure 4. Awareness levels of survey respondents according to their education level.

Awareness Based on Enterprise: The answers given in 20 questions, which prepared for awareness based on enterprise, are shown in the table below. It has been calculated that the mean score of the responses to the questions is 3.11, the standard deviation is 1.086, and the standard error is 0.036.

Table 3. Awareness	based	on	enterprise.
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Questions	Percentage
	(%)
I use a computer effectively in my enterprise.	49
I make use of the internet in production.	60
I follow technological developments closely.	78
I use computerized tools and machines in production.	40
I use CNC machines effectively on my production line.	31
Workers in the production line usually work using physical force.	80
The effective use of computers and the internet in my enterprise provides me with administrative convenience.	47
The usage of computerized tools and machines in production reduces the effectiveness of my employees.	18
Usage of many computers or more internet leads to security weaknesses in my enterprise.	18
It is unnecessary to use a 3D printer in my enterprise.	11
As the number of computerized tools and machines in production increases, the need for employees decreases	42
Technological developments make it difficult to train and find qualified workers	58
Industry 4.0 is a threat to the future of my enterprise	13
It is not affected by my enterprise that hig enterprises have adopted Industry 4.0	16
I have to combine and grow with my opponents for Industry 4.0.	13
Smartphone usage at my enterprise is sufficient for the transition to Industry 4.0.	13
Industry 4.0 is a great advantage for the growth of my enterprise and competition with my opponents.	36
The roadmaps that governments will set for Industry 4.0 do not adversely affect my enterprise.	31
The transition to Industry 4.0 will increase production capacity in my enterprise.	44
I do not have any difficulty in finding a qualified employee to work in my enterprise.	22

There was no relationship between the distribution according to provinces of enterprises responding to the survey and awareness level. Although awareness levels of enterprises in Ordu are higher than enterprises in Giresun, the awareness level of the enterprises is very low. The level of awareness of enterprises in Ordu was 40% while the level of awareness of enterprises in Giresun was 25%.

There was also no significant relationship between the foundation year of the enterprises and awareness levels. However, it has seen that the enterprises established between 1991 and 2000 have a higher awareness than the enterprises established in the other years.

It is founded that there was a meaningful relationship between respondents and awareness levels in the enterprises. It has been determined that awareness levels of the enterprises' employers or employees are lower than awareness levels of professional managers or engineers/architects/technicians in the enterprises. The level of awareness is 18% in employees, 28% in employers, 55% professional managers, 58% in in engineering/architect/technicians.

When the level of awareness of respondents to the questionnaire was examined according to the age criterion, it was seen that there was a significant difference between the age group of 19-25 and the other age groups. As a person's age grows older, the awareness level has been reduced. Awareness levels of people aged between 19 and 25 were determined as 78%.



Figure 5. Survey respondents' awareness levels by age.

Table 4. Awareness on the basis of country in the enterprises.

Questions	Percentage
With the 1st Industrial Revolution, people working in agricultural areas have	(70)
been fronted to the factories.	44
With the 4th Industrial Revolution, there will be lived comeback from	40
agricultural areas to factories.	
With Industry 4.0, there will be entrepreneurship in the country and sectoral growth in SMFs	40
Integrating new systems with Industry 4.0 increases data security issues.	18
Industry 4.0 is reduced human efficiency in protecting the integrity of the	16
production process.	16
The inclusion of robots and robotic systems in the production process will lead	42
to the mobility of the workforce.	
Due to technical problems that may be experienced in new technological systems, there may be big losses.	29
For Industry 4.0, our country has sufficient R&D infrastructure and an	22
accumulation of knowledge.	22
For Industry 4.0, the qualification of the University and Public Research	16
Current legislation is appropriate and sufficient for the transition to Industry 4.0	24
The R&D experience and infrastructure of the enterprises across the country are	24
sufficient.	24
It is sufficient for enterprises across the country to integrate Intelligent	33
Automation Systems and related technologies into production lines.	
Industry 3.0.	38
The transition to Industry 4.0 leads to an increase in unemployment across the	20
country.	20
In our country, new work fields have been developed for the manpower that will	11
be made redundant with industry 4.0.	
to reduce production costs.	33
Our country is prepared for the cyber-security problems that may arise after the	11
Industrial 4.0 transformation.	11
Public financing resources and incentives directed at SMEs for Industry 4.0	9
There are training and qualification programs in schools and universities in our	
country to educate people with sufficient qualifications for Industry 4.0.	22
SMEs should be supported separately as they can deal with financial, technological and personnel problems less than large enterprises	71
teennoiogical and personnel problems less than large enterprises.	

There is no significant relationship between the respondents' levels of education in enterprises and awareness levels. As the education levels of people increase, the awareness level increases. But this increase remains at a pretty low level. Even the people having a postgraduate degree's whose conceptual awareness level about Industry 4.0 is 95%, their enterprise-based awareness level remained at about 45%.

It seemed that there was no significant relationship between awareness levels and production sites when enterprises analyzed according to their production places. As the technical infrastructure of the production sites improves, awareness levels increase slightly. While enterprises in organized industrial zones' awareness are 55%, the awareness of those in small industrial sites is around 30%.

Awareness Based on Country: The answers given for 20 questions, which prepared for country-based awareness, are shown in the table below. It has been calculated that the mean score of the responses to the questions is 3.17, the standard deviation is 0.76, and the standard error is 0.025.

It is determined that there is no relationship between the location of enterprises responding to the survey and the awareness level. The awareness level of the enterprises in Giresun is ultra-low. While awareness level in Ordu is 33%, the awareness level of enterprises in Giresun determined as 19%.

There aren't any links between the establishment date of the enterprises and the awareness level. The awareness level of enterprises established between 1991 and 2000 is higher than that of other enterprises. Still, this level is around 38%. It has found that as well as there is no significant relationship between the type of enterprises and awareness levels and that the employers' awareness is an ultra-low level. This level is approximately 18%.

In enterprises, there is a relationship between the respondents' qualifications and the awareness level. If the respondents are employers or employees, the level of awareness is low, whereas if they are professional managers or engineers/architects/technicians, awareness levels are relatively higher. Even so, awareness levels are ultra-low. While the awareness of employers is 20%, the awareness level of engineers/architects/technicians is 48%.



Figure 6. Awareness levels of respondents in the survey by age.

There is an inverse relationship between the age of the respondents and their level of awareness. Awareness levels decrease as people age. For example, those aged between 19 and 25 have a level of awareness of 73%, while those aged between 40 and 46 have a level of awareness of 19%. There was no significant relationship between the education level of the respondents and the awareness level. On the other hand, awareness levels decreased as the education levels of the persons decreased. For example, postgraduate alumni awareness is 45%, while the secondary graduates' awareness level is 7.5%.



**Figure 7.** Level of awareness of respondents according to the education level in the enterprises.

# CONCLUSIONS

The questionnaire applied to 45 enterprises in total. 30 of enterprises are in Ordu, and the others are in Giresun. Of the 45 respondents, 25 are employers, 10 are professional managers, 5 are engineers/architects/technicians, and 5 are employees. When the data obtained is analyzed, it seems that the awareness levels increased in the questions concerning the business in the 3 sections of the questionnaire. It also seems that the awareness levels decreased in the questions concerning the concept of Industry 4.0 and the general situation. In the question of the conceptual and general situation, it determined that the respondents generally gave moderate answers as Undecided. It determined that the awareness level of employers or employees who responded to the survey are lower than those of the professional manager or engineer/architect/technician. Also, there is an inverse correlation between the education level and awareness of the persons.

The conclusion of the research shows that the SMEtype furniture enterprises heard about the Industry 4.0 concept and the new technological concepts emerging with this concept, but do not know what these concepts mean. Moreover, when the responses to the demographic questions examined, it is seen that the enterprises still use old technologies in production and cannot accomplish technology renewal due to financial insufficiencies. At the same time, it could be said that this situation also reflects our country's realities. It is to say that technological maturity in our country is between Industry 2.0 and Industry 3.0.

# DISCUSSION

In summary, enterprises have adopted the concept of Industry 4.0 depending on the size of the enterprises. Big enterprises have used new technologies in their production lines and have adapted quickly. However, for especially small-scale enterprises, the same mobility is out of the question. It is quite difficult to capture Industry 4.0 for those enterprises that have not even reached the digital maturity.

The SMEs' resistance to the transition to the Industry 4.0 concept is entirely based on financial insufficiencies and unawareness. For this reason, at first, it is necessary to raise awareness for employees, -especiallyemployers, about the issue. In order to have well-trained employees in terms of Industry 4.0 and related new concepts, at the point of educating qualified people, training and proficiency programs should organize by the Ministry of National Education and by the universities.

New technological investments should be made to increase the technological maturity level in our country; especially SMEs- should be encouraged with public financial resources. It is necessary to catch up with the great technological transformations expected to take place by 2025 and to seize the opportunities of developing technologies. Therefore, SMEs should support, since it is more difficult for them to cope with economic and technological and personnel qualifications than bigger enterprises. Since SMEs are suppliers of large-scale enterprises, the gap between large enterprises and SMEs should not increase on Industry 4.0 journey.

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### ARAŞTIRMA MAKALESİ

6

### **RESEARCH PAPER**

# Gel Permeative Chromatography (GPC) Analysis of Polycaprolactone (PCL) Based Biodegradable Composites through Laboratory Soil Test

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**Abstract:** Biodegradability is an important asset for a polymer for their acceptance by the society due to the increased environmental awareness of people over the years. In this study, Polycaprolactone (PCL) based biocomposites were produced using PCL, polyvinyl alcohol (PVA)/Starch mixture and polyethylene (PE) as a polymeric matrix and Turkish Red Pine Pruning waste flours (RPWF) a lignocellulosic filler. Biocomposites were manufactured through combination of extruder and injection molding processes. Forest soil was collected from arid regions of Konya and transferred to Laboratory. During 18 Months of soil test, degradation of PCL was monitored using Gel Permeative Chromatography (GPC). Results showed that major factor on GPC results were RPWF and degradation time. Especially at higher level of lignocellulosic filler use, there is a linear relation between filler and degradation time.

Keywords: Gel permeative chromatograph (GPC), polycaprolactone (PCL), polyvinyl alcohol (PVA), biodegradable, mechanical and physical properties.

# Laboratuar Toprak Testi Uygulanmış Polikaprolakton Esaslı Biyobozunur Kompozitlerin Jel Geçirgenlik Kromatografi Analizleri

Öz: Yıllar içerisinde toplumun çevresel duyarlılığındaki artış dolayısıyla, polimerlerin biyolojik olarak bozunma özelliği onların toplum tarafından kabul edilmesinde önemli bir parametre haline gelmiştir. Bu çalışmada, polpolikaprolakton (PCL), polivinil alkol (PVA)/nişasta karışımı polimer matrisi olarak ve atık Kızılçam budama atık unları ise lignoselülozik dolgu maddesi olarak kullanılarak PCL esaslı biyolojik esaslı kompozitler üretilmiştir. Biyokompozitlerin üretimi ekstruder ve enjeksiyon kalıplama yöntemlerinin kombinasyonu ile üretilmiştir. Orman toprakları Konya'nın kurak alanlarından alınarak laboratuarlarımıza getirilmiştir. Jel Geçirgenlik Kromatografisi (GPC) yardımıyla belirlenmiştir. 18 ay boyunca gerçekleştirilen toprak testleri süresince, PCL'nin topraktaki bozunma oranı Jel Geçirgenlik Kromotografi (GPC) yardımıyla ölçülmüştür. Sonuçlar GPC değerleri üzerinde en etkili faktörlerin kızılçam odun unu miktarı ve bozunma süresi olduğunu göstermiştir. Özellikle yüksek dolgu maddesi oranına sahip örneklerde dolgu maddesi ile bozunma süresi arasında doğrusal bir ilişki tespit edilmiştir.

Anahtar sözcükler: Jel geçirgenlik kromatografisi (GPC), polikaprolakton (PCL), polivinil alkol (PVA), biyobozunma, mekanik ve fiziksel özellikler.

# INTRODUCTION

Petrochemicals driven non-biodegradable plastics cause serious environmental problems since they produce greenhouse gas during their manufacturing and wastes after their disposal. Even though they seem to be used for a long time due to newly add application areas, reliable and repeatable product quality and cost advantages, research towards finding alternative resources and using them continue.

That's why past 30 years, biodegradable plastics and new manufacturing methods have been studied intensively (Van de velde & Kiekens, 2002; Kolybaba et al., 2003; Yu & Chen, 2009; Leja & Lewandowicz, 2010; Averous & Pollet, 2012;). Biodegradation is the decomposition of organic material to the carbon, hydrogen and oxygen by microorganisms. Most common biodegradable plastics are starch (Walenta et al., 2001a & 2001b; Laohakunjit & Noomhorm, 2004; Zhang & Han, 2006; Hanna & Xu, 2009; Inman, 2010), protein, polylactic asit (PLA) (Viera et al., 2011; Gorrasi & Pantani, 2013) and polyhydroxyalkonates (PHA), driven from renewable resource and polycaprolactone (PCL) (Viera et al., 2011), petroleum driven from petrochemicals. Among them, the most commonly used one is starch since it can be used as plastic matrix and also a plastic filler. However, its drawbacks prevent them from being good alternative to other synthetic plastics. That's why they need to be combined with other biodegradable plastics for many application areas.

Biodegradable plastics are commonly used in the areas of packaging, agricultural mulch films and medical applications (Davis & Song, 2006). The usage of low density polyethylene (LDPE) based agricultural films are increasing in Turkey and in the World. After usage, these materials produce great amount of waste on the agricultural land. The use of biodegradable plastic instead, would be a better option.

Biodegradable plastics are known for being environmental friendly and being used as an alternative to petroleum driven plastics. They are also known for medical applications like tissue engineering, surgery ropes and drug delivery systems. In those applications, they are taken advantage of biodegradability of plastics. There is a need to increase application areas of biodegradability of plastics beyond the packaging, agricultural mulch films and medical applications.

In our previous study, biodegradable composite materials using starch and wood flour were manufactured. However, this type of material still prone to hydrolyses in short time when in contact with water. That's why the use of starch in combination with other biodegradable plastic is advised in many applications. The objective of this study is to manufacture biocomposites using combination of biodegradable polymers and monitor their degradation under soil with GPC analysis.

# **MATERIAL and METHODS**

Summary of the experimental design and description of the manufactured samples were presented in Table 1 and Table 2, respectively. PCL, PVA-Starch mixture and PE were used as thermoplastic matrix. Turkish red pine pruning waste (RPW) was used as lignocellulosic filler. Depending on the formulation given PCL, dried PVA-starch pellets, PE and RPW were dry-mixed in a high-intensity mixer to produce a homogeneous blend. These blends were compounded in a single-screw extruder at 40 rpm screw speed in the temperatures (barrel to die) of 170-175-180-185-190°C. Extruded samples were cooled in water pool and subsequently granulated into pellets. The pellets were dried in oven at 103±2°C for 24 hours. Dried pellets were injection molded using an HDX-88 Injection Molding Machine to produce standard test samples.

**Table 1.** Summary of the experimental design.

Analysis Type:	Response Surface Method Quadratic		Varian	ce (nm)
Desing Model:			Desing Model: Quadratic Lev	
	Factors	Type	Low Actual	High Actual
Α	RPW(%)	Numeric	4.39	25.61
В	PE (%)	Numeric	1.46	8.54

Table 2. Description of the manufactured samples.

	•		-			
ID	Neat PCL (%)	RPWF (%)	PE(%)	PVA-Starch (%)		
PCL0	55.00	15.00	0.00	30		
PCL1	64.15	4.39	1.46	30		
PCL2	42.93	25.61	1.46	30		
PCL3	65.00	0.00	5.00	30		
PCL4	50.00	15.00	5.00	30		
PCL5	35.00	30.00	5.00	30		
PCL6	57.07	4.39	8.54	30		
PCL7	35.85	25.61	8.54	30		
PCL8	45.00	15.00	10.00	30		

Forest soil was collected from arid regions of Konya and transferred to Laboratory. Samples were put into the flowerpots and covered by forest soil. They were watered up to field capacity (Figure 1).



Figure 1. Samples were put under soil (a) and watered up to the field capacity (b).

For GPC Analysis; samples were taken out of soil, cleaned and weighed before testing. Before running test GPC was cleaned with 2-propanol for 120 min. Flow was 1ml/h. Later eluent THF was run for 180 min. GPC was calibrated. 0,5g composite powder was mixed with 50 mL for 60 min. Mixture was filtered with 45  $\mu$ m Agilent brand nylon filter. Samples passing 45  $\mu$ m filter was injected 20 mL volume of the system. Analysis was accomplished at 25°C column temperature, 33-35 bar colon pressure and 35°C RID detector

temp. One agilent brand Polargel-M guard 50 x 7.5 mm column and 2 agilent brand Polargel-M 300 x 7.5 mm column were used for analysis (Figure 2).



**Figure 2.** Samples were cleaned from dirt (a), granulated with IKA (b), diluted in THF (c) and tested in GPC (d).

#### **RESULTS and DISCUSSION**

After soil burial test, maximum molecular weight loss of the samples were determined and results were analyzed. Statistical analysis showed that both RPWF loading and degradation time had significant effect on molecular weight of the PCL matrix. Interaction graph of the result is presented in Figure 3. In first 6 month period, samples with low and high RPWF loading have provided similar molecular weight degradation (10%). After 6 months, on the other hand, high RPWF loading samples lost more molecular weight. After 18 months of burial test, samples with high RPWF loading lost around 40% of its molecular weight. However, it was still around 10% for samples having low RPWF loading. Arvanitoyannis et al. (1998) reported that there is close relation between degradation and excess of the microorganism to lignocellulosic materials. We believe that at high RPWF loading, water and microorganism had a better chance of excessing the lignocellulosic materials resulting in increased molecular weight reduction of the PCL matrix.



Figure 3. GPC- Maximum Weight Lost (GPC-MWL).

Produced composite materials were buried under soil which is collected from Konya region. Tensile, flexural and impact samples were kept under soil over 18 months of period. Samples were taken out every six months and weight losses were measured. Weight loss percentage interaction graphs of tensile, flexural and impact samples were shown in Figure 4, Figure 5 and Figure 6, respectively. Based on the statistical analysis, RPWF loading had no significant effect on weight loss for tensile and flexural samples (P=0.1268, P=0.2108, respectively). In the case of impact samples, it had significant effect on weight loss (P=0.0039). Even though PE amount had some effect on weight loss, degradation time has the most important effect on weight loss for all samples (P<0.0001). Among them, impact samples had the highest weight loss compared to tensile and impact samples. It is believed that higher surface area of the impact samples (being in almost half the size of other samples) increased the wear of the samples under soil.



C: Time (Month)

Figure 4. Weight loss of tensile samples (TWL).



C: Time (Month)

Figure 5. Weight loss of flexural samples (FWL)



Figure 6. Weight loss of impact samples (IWL).

# CONCLUSION

Biodegradable composites were successfully manufactured and 18 months of soil burial test were conducted. Based on the GPC analyses following conclusions were reached;

RPWF concentration and degradation time have a significant effect on GPC molecular weight.

Especially at high RPWF loading, there is a linear correlation between degradation amount and filler loading.

GPC can be used to monitor degradation of polymers but needs further studies to compare and correlate them to standard weight loss methods.

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# ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Utilization of Mass of Industrial Hemp in the Production of Medium-density Fibreboards (MDF)

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**Abstract:** In the near past, industrial hemp (*Cannabis sativa* L. subsp. *sativa*) found main application in the production of ropes and fabrics. At the present moment, this crop finds increasingly big application for pharmaceutical purposes and in the cosmetics industry. As a result of which not only hurds, but whole stalks remain as waste from this production. On the other hand, MDF, which on a world scale are the second production of wood-based boards in terms of volume after that of veneer plywood, allow at least partial inclusion of non-woody lignocellulosic raw materials in their composition. That is why, in this paper, an investigation about the possibility for inclusion of mass of hemp stalks in the MDF composition is presented.

Under laboratory conditions, previously washed hemp stalks were defibred. The defibration was performed in a laboratory crusher defibrator for 2 min. Under laboratory conditions, MDF with participation of mass of hemp stalks form 0% to 100% in the MDF composition were produced. The content of mass of hemp stalks was increased by a step of 10%. The boards were produced at a temperature of hot pressing of 185 °C, with 10% participation of urea-formaldehyde resin (UFR) and have a density of 850 kg/m<sup>3</sup>.

The effect of the content of mass of hemp stalks on the physicomechanical indicators of MDF was established. Regression equations for this effect on the individual physicomechanical indicators were also derived. It has been established in case of increase of what share in the composition of boards, most significant deterioration of the MDF indicators is observed. On this basis, a recommendation for the maximum justified content of mass of hemp stalks in the MDF composition was derived.

Keywords: MDF, Non-woody lignocellulosic raw material, Stalks of industrial hemp.

#### **INTRODUCTION**

Industrial hemp (*Cannabis sativa* L.) has many different applications, mainly in the production of paper, fabrics, biodegradable plastics, building materials, healthy food, fuels and another approximately 50,000 types of applications besides industry, in medicine and potentially in almost any other sphere.

Hemp is an annual plant that reaches 5 m in height and 6 to 60 mm stalk thickness for a period of growth of about 3 months depending on the plantation density (Mohanty et al, 2000).

This is one of the fastest growing biomasses known to man and one of the plant crops most early known and

home-grown by man. Hemp does not require pesticides for its cultivation, does not lead to erosion of soil, just the contrary, it aerates it by producing oxygen (https://www.konop.bg).

Hemp stalk consists of lignified heartwood with wide pith, covered by a cambial layer, bast, bark and epidermis (Fig. 1).

Bast represents about 1/4 of the stalk and plays a main role for the tensile strength and, respectively, bending strength of the stalk. Bast is covered by epidermis covered with great number of cuticles that protect the plant from parasites by means of the strong resins they secrete. Xylem (so called lignified part) of the stalk represents about 3/4 of the volume and plays a main role for the plant firmness.



**Figure 1.** Structure of hemp stalk. (1- epidermis; 2 - collenchyma; 3 – cortical parenchyma; 4 – endodermis; 5 – primary bast fibres; 6 – bast parenchyma; 7 – vascular tissue; 8 – cambium; 9 – wood; 10 – pith)

There are considerable differences between the chemical composition of the xylem and the bast of the hemp stalk. The bast fibres have cellulose content of about 60-70%, hemicelluloses – 15-20%, lignin – 2-4%, pectins – 2-4%, fats and waxes – 1-2%. Xylem has rather content analogous to broad-leaved tree species: cellulose about 40%, hemicelluloses about 25%, lignin about 20% and extractives – 4% (Garcia-Jaldon et al., 1998). This difference determines also the serious difference in the strength of bast fibres and those of xylem, that is why whole stalks comprising both types of fibres were used for the investigation.

In the past, hemp was mainly grown to use the bark fibres for ropes, paper and textile, but nowadays these productions are supplemented by the extraction of highquality seed oil and resins irreplaceable for medicine, the plant value being increased many times in this way and its cultivation becoming low-risk and promising business. In most of those productions, waste are exactly hemp stalks that, in the countries developing this branch, are abundant and acquire industrial significance. In older references, directions are given for the use of hemp as additive to wood raw material at most up to 30% of the total mass, but in recent investigations results meeting some basic standards at hemp content of 60% of the raw material's total mass were achieved (Radosavljevic et al., 2008). Characteristic of hemp, as well as of wheat lignocellulosic waste is the need for treatment with weak solution of alkali (NaOH 5-8%) and 1 to 3% of oligomeric siloxane in alcohol solution because they

separate hemicelluloses, lignin and waxes from the fibres and thus assist the adhesion at a later stage, and, hence, increase the strength and performance characteristics (Kabir et al., 2012).

It should be emphasized that there is a considerable amount of investigations about utilization of hemp stalks in particleboards (PB) composition (Mahieu et al., 2015; Shöpper et al., 2008; Selinger et al., 2015), while investigations about their utilization in MDF composition are considerably fewer (Fajrin, et. al. 2018).

#### **MATERIAL and METHODS**

For the purposes of the investigation, MDF with set thickness of 8 mm and density of 850 kg/m<sup>3</sup> were produced under laboratory conditions. Urea-formaldehyde resin at content of 10% relative to the oven-dry mass was used as binding agent.

The wood-fibre mass that was used to produce the boards was produced in the fibreboards (FB) factory of VELDE Bulgaria AD – the town of Troyan. It is composed of wood of Turkey oak and beech to the ratio of approximately 2:1 and has water content of 10%.

The hemp mass was produced in the Pressing Laboratory of the Chair of Mechanical Wood Technology at the University of Forestry. The hemp stalks with diameter of 6 to 32 mm were previously cut to a length of 200 mm and were immersed in water for 5 days at room temperature. Defibration was performed with laboratory defibrator in amounts of 100 to 150 g with addition of additional 200 ml of water and cycle duration of 150 s, whereupon they were dried at a temperature of 100 °C to water content of 6%. The fibres obtained in this way were sifted through a sieve with mesh of 2x2 mm and are up to 5 mm long.

Urea-formaldehyde resin produced by KASTAMONU Bulgaria AD was used as binding agent. The resin is with initial concentration of 68% and working concentration of 55%.

For the purpose of the investigation, 11 boards with various share of hemp mass within the range of 0 to 100%, at a step of 10%, were produced.

Pressing was performed at a platen temperature of  $185 \pm 5$  °C and duration of 1 min/mm, in three stages, at specific pressure as follows:  $1^{st}$  stage – 2.6 MPa;  $2^{nd}$  stage – 1.3 MPa;  $3^{rd}$  stage – 0.6 MPa. The duration of the individual stages is the following:  $1^{st}$  stage – 12.5% of the complete cycle;  $2^{nd}$  stage – 37.5% of the cycle;  $3^{rd}$  stage – 50% of the pressing cycle.

Determination of the performance indicators of the boards was performed in conformity with the valid European norms EN 310; EN 316; EN 317; EN 323.

The results for the performance indicators of MDF, depending on the content of mass of hemp stalks, were processed after the methods of variation statistics and regression analysis, with approximation functions being derived for the examined relationships. The coefficient of determination was used as a measure for definiteness.

# **RESULTS and DISCUSSION**

The summarized results for the performance indicators of MDF, depending on the content of mass of hemp stalks, are presented in Table 1.

**Table 1.** Performance indicators of MDF, depending on the content of mass of hemp stalks.

No	Content of mass of hemp stalks <i>Px</i> , %	Density $\rho$ , kg/m <sup>3</sup>	Water absorption A, %	Swelling in thickness <i>Gt</i> , %	Bending strength fm, N/mm <sup>2</sup>
1	0	848	81.44	28.53	44.02
2	10	854	85.78	29.53	43.99
3	20	845	86.54	31.50	43.52
4	30	858	89.13	32.96	42.96
5	40	846	89.40	36.03	39.49
6	50	850	95.61	40.41	34.19
7	60	844	94.54	40.44	34.03
8	70	860	96.70	41.18	34.97
9	80	859	100.2	41.47	31.21
10	90	850	102.2	41.04	22.32
11	100	846	119.2	50.43	23.30

In graphic form, the variation of the water absorption of MDF, depending on the content of mass of hemp stalks, is presented in Fig. 2.

The relationship between the water absorption and the content of mass of hemp stalks is described with regression equation of the type:

$$\widehat{A} = 84.18 + 0.026.P_x + 0.003.P_x^2, \tag{1}$$

where A is the predicted value for water absorption of the boards, %;

Px – the content of mass of hemp stalks, %.

Equation (1) is characterized with coefficient of determination,  $R^2$ , of 0.95.

From the data presented is seen that with addition of mass of hemp stalks in the MDF composition, the water absorption of the boards increases, i.e. deteriorates. Under the conditions of the experiment, the water absorption varies from 81.44 to 119.2%. Lowest is the water absorption of the board obtained from 100% wood raw material, and highest in that obtained from 100% hemp stalks. Deterioration of the indicator is by 37.76%. Significant deterioration of the water absorption is observed already with the addition of the first 10% of mass of hemp stalks - by 4.34%. Increase of water absorption is also recorded in case of further increase of the content of mass of hemp stalks, with the deterioration being more significant in case of increase of its content from 20 to 30% (by 2.59%), from 40 to 50% (by 6.21%), from 70 to 80% (by 3.50%). Most significant deterioration of this indicator is observed in case of increase of the content of mass of hemp stalks from 90 to 100%.



**Figure 2.** Variation of the water absorption of FB, depending on the content of mass of hemp stalks.

The dependence of the swelling in thickness of MDF on the content of mass of hemp stalks in their composition is presented in Fig. 3.



Figure 3. Variation of the swelling in thickness of FB, depending on the content of mass of hemp stalks.

The relationship between the content of mass of hemp stalks and the swelling in thickness of MDF is of the type:

$$Gt = 28.22 + 0.19.P_x$$
, (2)

where Gt is the predicted value for the swelling in thickness of boards, %;

Px – the content of mass of hemp stalks, %.

The equation is characterized with coefficient of determination  $R^2 = 0.86$ .

Under the conditions of the experiment, the swelling in thickness of MDF varies from 28.53 to 50.43%, i.e. here also we observe deterioration of the indicator with increase of the content of mass of hemp stalks in the composition of boards, with this deterioration being by whole 21.9%. Lowest is the swelling in thickness of FB entirely composed of wood raw material, and highest in those composed entirely of mass of hemp stalks. Most significant deterioration of the indicator is observed in case of increase of the content of mass of hemp stalks from 90 to 100%, where the increase is by 9.39%. More significant deterioration of the indicator is also observed in case of increase of the content of mass of hemp stalks from 30 to 40%, as well as in case of increase of the content of mass of hemp stalks from 40 to 50% (by 4.38%).

In graphic form, the variation of the bending strength of MDF, depending on the content of mass of hemp stalks in their composition, is presented in Fig. 4.



Figure 4. Variation of the bending strength of FB, depending on the content of mass of hemp stalks

The regression equation in the case of this indicator is:

$$\widehat{fm} = 44.70 - 0.065 \cdot P_x - 0.002 \cdot P_x^2$$
, (3)

where fm is the predicted value for the bending strength of boards, N/mm<sup>2</sup>;

Px – the content of mass of hemp stalks, %.

The equation is characterized with coefficient of determination  $R^2 = 0.96$ .

As a whole, with increase of the content of mass of hemp stalks, the bending strength of the obtained MDF deteriorates.

Under the conditions of the investigation, the bending strength of the boards varies from 44.02 to 23.30 N/mm<sup>2</sup>. The total deterioration is by 20.72 N/mm<sup>2</sup>. Most significant deterioration of the indicator is recorded in case of increase of the content of mass of hemp stalks from 80 to 90% (by 8.89 N/mm<sup>2</sup>), from 70 to 80% (by 3.76 N/mm<sup>2</sup>) and from 30 to 40% (by 3.47 N/mm<sup>2</sup>).

In spite of the deterioration of the indicator as a whole, the boards with content of up to 80% mass of hemp stalks meet the requirements for bending strength for boards of general purpose, for use in dry environment (23 N/mm<sup>2</sup>) and for use in humid environment (27 N/mm<sup>2</sup> for type MDF.LA). The boards with content of up to 80% mass of hemp stalks also meet the requirements for bending strength to boards for bearing structures, used in humid environment (34 N/mm<sup>2</sup> for type MDF.HLS).

# CONCLUSIONS

As a result of the investigation performed about the effect of participation of mass of hemp stalks on the performance indicators of MDF, the following conclusions may be drawn and the following recommendations may be made: • The industrial hemp stalks may be successfully used as a substitute for the wood raw material in the MDF composition;

• It is recommended that the investigations about the utilization of hemp stalks as part of the MDF composition are continued by looking for possibilities for improvement of the hydrophobic properties of the boards.

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# ARAŞTIRMA MAKALESİ

## **RESEARCH PAPER**

# The Effect of Cryogenic Application on Parallel Fiber Pressure Resistance of Thermowood-Treated Sorbus Torminalis

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**Abstract:** This study aimed to analyze the possible changes in the pressure resistance values parallel to the fibers(CS) by subjecting heat-treated wood to sub-zero temperatures. Within the scope of the study, test specimens were first prepared by applying thermowood heat treatment at 190 and 212 °C for 1 h to samples of wild service tree wood (Sorbus torminalis). Both the heat-treated samples along with untreated control samples were then kept at -80 °C for 6. 18 and 54 h to obtain the test specimens. When compared to the control samples. Test results showed a difference in the parallel fiber pressure resistance of the heat-treated test specimens held at -80 °C and the values were seen as very positive, especially with the 6-h and 18-h applications. The CS resistance values of the control samples and the heat treated samples as N/mm<sup>2</sup> at 190 and 212 °C for 1 h were respectively 56.12, 69.28 and 62.12 for initial; 51.38, 73.25 and 80.55 for 6 h; 50.31, 72.82 and 75.66 for 18 h; 55.85, 60.19 and 62.14 for 54 h.

Keywords: Cryogenic treatment, compression strength, heat treatment, Sorbus torminalis.

# Kriyojenik İşlemin Thermowood Akçaağaç Yapraklı Üvez Odununda Liflere Paralel Basınç Direnci Üzerine Etkisi

Öz: Bu çalışmadaki amaç, ısıl işleme tabi tutulmuş odunda kriyojenik işlem uygulandıktan sonar liflere parallel basınç direncindeki olası değişimleri analiz etmektir. Bu kapsamda Akçaağaç yapraklı üvez odunu test örnekleri ilk olarak 190 ve 212°C sıcaklıklarda ısıl işleme tabi tutularak Thermowood paneller üretildi. Her iki gruptaki örnekler kontrol örnekleri ile birlikte sıfırın altında 80°C sıcaklıkta 6, 18 ve 54 saat süre işleme tabi tutuldu. Daha sonra gruplara liflere paralel basınç direnci testi uygulandı. testler uygulandıarasında karşılaştırma yapıldı. Test sonuçları özellikle 6 ve 18 saatlik kriyojenik uygulama yapılan test örneklerinde farklılıkların olduğunu göstermiştir. Liflere paralel basınç direnci kontrol, 190 ve 212°C Thermowood örneklerde sırası ile N/mm<sup>2</sup> olmak üzere; 56.12, 69.28 ve 62.12 (başlangıç); 6 saat için 51.38, 73.25 ve 80.55; 18 saat için 50.31, 72.82 ve 75.6; 54 saat için 55.85, 60.19 ve 62.14 olarak belirlenmiştir.

Anahtar sözcükler: Akçaağaç yapraklı üvez, basınç direnci, ısıl işlem, kriyojenik işlem.

# INTRODUCTION

Although wood is a widely utilized natural raw material, in a number of applications it exhibits undesirable properties such as dimensional and color changes as well as biological degradation. These features shorten its service life in those areas of application and lead to losses in value (Sahin et al., 2017; Aytin, 2013).

Today, various methods and techniques can be employed to improve these stated negative properties of wood materials in those application areas. One example of these is heat treatment (HT), which is a modification technique that can improve the properties of wood material by increasing dimensional stability and resistance to biological degradation in addition to achieving color homogeneity (Srinivas & Pandey, 2012; Johansson, 2005).

In contrast to the improvements observed in the physical properties of heat-treated wood materials, the values of mechanical properties are generally lower, except for the elasticity modulus and the compression strength parallel to the fiber (CS). There is also an increase in the modulus of elasticity and CS, up to a certain temperature range, and then the values decrease after the heat treatment temperature reaches 205°C (Anonim, 2003). However, in some studies it has been reported that CS decreases with HT (Caliova, 2011)

Although HT offers significant opportunities for users in the applications of wood materials, mechanical resistance losses are seen as an obstacle to these prospects. It is obvious that allowable safe stresses should be seriously taken into account, especially when a load carrying element is being considered. Hence, in such areas of usage, there is uncertainty in the evaluation of HT wood products compared to natural wood materials. From this point of view, the assessment of potential qualities may actually be quite important for HT wood materials in places where mechanical properties are in the forefront, with preferences for those providing more extensive improvements in physical characteristics.

Cryogenic (Cr) treatment is an application used to increase hardness in steel. In the process for hardened steel, the material is cooled to sub-zero temperatures and held for a certain period of time. It is reported that with cryogenic hardening, steel has higher wear ability and a longer service life (Kam et al., 2016). The application phases of Cr treatment for steel are shown below (Figure 1).

It is evident that the increase in hardness and wear capability which cryogenic processing has brought to bear opens up new areas of application. Reviews of the literature show that to date no study has been conducted examining HT and three versions of Cr on wood materials. This study aimed to reveal the relationship between HT and Cr and between natural wood material, HT and Cr in terms of CS.

Thus, this is considered to be an initial study of the consequences of Cr treatment for natural wood and for HT wood.



**Figure 1.** A schematic presentation of the heat treatment schedule consisting of the hardening, tempering, deep cryogenic treatment and tempering cycles of the samples (Aytin, 2016).

#### **MATERIAL and METHODS**

The *S. torminalis* trees used in the study were obtained from the Düzce-Odayeri Forest Sub-district Directorate (Duzce. Turkey). Selection of the trees was in accordance with the TS 4176 (1984) standard (7). The trunks of selected trees were cut at the height of 1.30 m from the base and divided into 2-m sections.

Panels with dimensions of 25 mm  $\times$  100 mm  $\times$  500 mm (thickness. width. length) were prepared from the *S. torminalis* trees and were subjected to heat treatment in an industrial oven using the ThermoWood method (Novawood Factory. Gerede. Turkey) in accordance with the operational production schedule. The heat treatment was conducted at temperatures of 190 °C and The panels were then subjected to Cr treatment. The HT and subsequent Cr process steps are shown in Figure 2.



Figure 2. Application stages of HT and Cr in *Sorbus torminalis* wood.

The CS test samples were then prepared according to TS 2595 and left to condition. The experimental design used in the study is shown in Table 1.

Table 1. Experimental design of materials used in the study.

Test samples(TS)	Abbreviation	Cryogenic(Cr)
		6*
Control	UT	18
		54
		6
190 °C 1 h	$TW_1$	18
		54
		6
212°C 1 h	TW <sub>2</sub>	18
		54

#### \*Time is given as hour.

After the study was performed according to the experimental design in Table 1, the results obtained were analyzed using the Windows Evaluation SPSS Version 15.0 program, with significance set at P  $\leq 0.05$ . Homogeneity groups were examined using the Duncan test.

#### FINDINGS

The results of the variance analysis of the CS values in the Cr *Sorbus torminalis* specimens are given in Table 2.

Table 2. Results of variance analysis of CS values of Sorbus torminalis specimens subjected to cryogenic treatment for different time periods.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	10944.262(a)	11	994.933	12.503	0.000	0.560
Intercept	494268.694	1	494268.694	6211.50	0.000	0.983
Tree variety	6972.133	2	3486.067	43.810	0.000	0.448
Cryogenic	1392.496	3	464.165	5.833	0.001	0.139
Tree variety * Cryogenic	2579.633	6	429.939	5.403	0.000	0.231
Error	8593.901	108	79.573		·	
Total	513806.857	120	· · · · · · · · · · · · · · · · · · ·		·	
Corrected Total	19538.163	119				

According to the results of the variance analysis in Table 2, among the Cr-applied experimental samples, statistically significant differences can be seen in both the tree species and the cryogenic variations. The Duncan test was applied to determine which groups the differences were between, and the results are given in Table 3.

 Table 3. Duncan test results of CS values of Sorbus torminalis

 specimens cryogenically treated for different time periods.

Factor	Variations	N		CS	
	( unitations		Α	В	С
	UT	40	53.4186		
TS	TW1	40		68.9963	
	TW2	40		70.1213	
	Control	30	62.5118	62.5118	
Cr	6 H	30			68.3948
CI	18 H	30		66.2666	66.2666
	54 H	30	59.5415		

The Duncan test results revealed differences in both tree species (UT + heat treatment) and cryogenic (control + 6 h + 1 h + 54 h) CS values. The highest tree species values were reached in the heat-treated specimens and the highest cryogenic values were with the 6- and 18-h applications.

In Table 4, the CS values and Duncan test results are given for each subgroup after the tree species and cryogenic applications.

**Table 4.** CS values and Duncan test results for each subgroup after tree species and cryogenic applications

TS	Cr	CS (N/mm <sup>2</sup> )	SS	HG
	Control	56.12	5.50	А
UT	6 h cryogenic	51.38	10.78	А
01	18 h cryogenic	50.31	11.06	А
	54 h cryogenic	55.86	7.13	А
	Control	69.29	5.12	AB
TW1	6 h cryogenic	73.25	12.60	В
1.0.1	18 h cryogenic	72.82	12.68	В
	54 h cryogenic	60.62	6.36	А
	Control	62.12	8.54	А
TW2	6 h cryogenic	80.55	2.35	В
	18 h cryogenic	75.66	6.76	В
	54 h cryogenic	62.14	11.15	А

#### CONCLUSIONS

The CS values obtained in all the cryogenic groups were higher than in the cryogenically untreated (control) samples. The duration of the cryogenic treatment was effective on the CS values. As the time was increased, lower CS values were obtained. For this reason, determining the optimum Cr processing time is an important issue.With the heat treatment temperature increase, the increasing CS value began to decrease as the temperature rose. Decreases in the CS occurring due to the increased HT temperature can be recovered by Cr application. In Figure 3 CS values are seen for the UT, HT and Cr variations.



Figure 3. CS values are seen for the UT, HT and Cr variations.

These results show that Cr processing of wood materials is very important after HT and that it is essential to test their threshold values to enable them to be used at the desired locations.

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#### ARAŞTIRMA MAKALESİ

#### **RESEARCH PAPER**

# Utilization of Trapa natans

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Abstract: In this study, Trapa natans nut collected from the costs of Inkumu-Bartin was analyzed according to its lignin content. Acid-insoluble lignin content was determined according to TAPPI T222om-02 as 28.31%. TGA and FTIR analyses were performed.

FTIR analysis indicated that Trapa natans contained cellulose, hemicelluloses and lignin. The peaks at 1506-1510 cm<sup>-1</sup> in both samples are characteristic peaks for the lignin components due to C = O and COO-non-symmetric stretching vibrations in the aromatic rings of the lignin structure. 1510 cm<sup>-s</sup> peak is found in the literature as lignin's fingerprint peak. The peaks at 1230-1270 cm<sup>-1</sup> band show the vibrations of guayasil ring with CO tension in lignin and hemicelluloses. Around the peaks of 900, 1025, 1030 and 1050 cm<sup>-1</sup> bands cellulose's O-H, C-H and C-O-C type bonds are seen. When TGA analyzes are examined, approximately 4.5% of the sample is lost from 30 °C to 200 °C. At these intervals, samples generally lose volatile constituents and moisture. From 200 to 271 °C 4.6% of the weight is lost. The decrease in this range may be due to the decomposition of cellulose and hemicelluloses. The DTG curve shows maxima weight-loss occurring at 349 °C. The weightloss in the range from 271-352 °C is about 50%. Pure lignin sample's TGA and DTG curve results show 10%, 50%, and 70% weight-losses at 233 °C, 489 °C, and 1167 °C respectively. According to these results, dominantly lignin and small quantities of cellulose and hemicelluloses are present in trapa samples.

Keywords: Lignin, Trapa natans, water-chestnut

# Trapa natans'tan Yararlanma

Öz: Bu çalışmada, Bartın-İnkumu sahillerinden toplanmış Trapa natans yemişi boş kabuklarının lignin miktarına bağlı analizleri yapılmıştır. Asitte çözünür lignin miktarı TAPPI T2220m-02 esas alınarak yapılmış ve %28.31 bulunmuştur. TGA ve FTIR analizleri yapılmıştır.

FTIR analizleri Trapa natans örneklerinin selüloz, hemiselülozlar ve lignin içerdiğini göstermektedir. Her iki örnekteki 1506-1510 cm-<sup>1</sup> pikleri lignin yapısının aromatik halkalarındaki C = O ve COO-simetrik olmayan gerilme titreşimlerine bağlı görülen, lignin bileşenleri için karakteristik piklerdir. 1510 cm<sup>-s</sup> piki literatürde ligninin parmak izi piki olarak görülmektedir. 1230-1270 cm<sup>-1</sup> bandındaki tepe noktaları, lignin ve hemiselülozlardaki CO gerilimi ile guayasil halkasının titreşimlerini göstermektedir. 900, 1025, 1030 and 1050 cm<sup>-1</sup> pikleri civarında selülozun O-H, C-H ve C-O-C tipi bağları görülmektedir. TGA analizleri incelendiğinde 30 °C'den 200 °C'ye kadar numunenin yaklaşık %4,5 kadarının kaybedildiği gözlemlenmiştir. Bu aralıklarda numune genellikle uçucu maddeleri ve nemi kaybetmektedir. 200 °C'den 271 °C'ye kadar ağırlığın %4,6'sı kaybolmuştur. Bu aralıkta gözlenen kayıp, selüloz ve hemiselülozlarda meydana gelen bozunmadan kaynaklanabilmektedir. DTG eğrisi 349 °C'de maksimum ağırlık kaybını göstermektedir. 271-352 °C'deki ağırlık kaybı yaklasık olarak %50 civarındadır. Saf lignin örneğinin TGA ve DTG eğrileri incelendiğinde, 233 °C, 489 °C ve 1167 °C'de sırasıyla %10, %50 ve %70 ağırlık kaybı görülmektedir. Bu sonuçlara göre trapa örneklerinde baskın olarak lignin ve az miktarlarda selüloz ve hemiselülozların var olduğu söylenebilir.

Anahtar sözcükler: Lignin, su kestanesi, Trapa natans

# INTRODUCTION

*Trapa natans* L. (TN); known as water chestnut or water-nut is an annual, floating-leaved plant that naturally grows in rivers, lakes, fresh-water wetlands and firths in Asia and Europe (Yasuda et al., 2014). In Turkey, it is found in trace amounds (Istanbul, Edirne and Kırklareli) (Web-1). In the last years, it was seen in the cost of North Black Sea region (TÜBİVES, 2018). It is a warm season crop and can harvest in June-September (Liu et al., 2010; Parker & Waldron, 1995).

It has 5 cm wide leaves with sharp serrate and stiffy hairs. It also has 15 cm long submersed leaves. In spring time, it has white flowers. Having an economic value, the fruit grows under water and has horned nut-like structure (Web-2).

It has an economical worth as food and raw material in different areas especially for China and India. Dried and grounded nuts can be added into flour for baking (Sturtevant & Hedrick, 1972; Web-3).Because of high water content, it is used for thirst and also used as traditional medicine. In an experimental study was showed that herbal mixture extracts which include TN decreased the pain of shingles (Hijikata et al., 2005). The outer part of nuts is utilized in pulp-paper, fish food, compost and biofuel (Hummel & Kiviat, 2004).

TN resembles a water lily at first view with its green leaves floating on the water. Floating leaves have rosette form, and leaflet is solid and triangular (5.2x6.4 cm) (Nedhuka & Kardyum, 2016). The leaves are dark green, and their bottom surface is reddish purple (Adkar et al., 2014; Zhu, 2016). The submerged stems cling to the soil by extending to 1-5 m along the roots (Adkar et al., 2014; Hummel & Kivrat, 2004).

TN, which has been used for food and therapeuthic purposes in China and India since ancient times (Zhu, 2016), is a major ingredient in many food products such as jams, beverages and biscuits in China (Sheng et al., 2006). Due to its characteristic taste and high nutritional value, it has commercial importance in the food industry (Parker & Waldron, 1995; Liu et al., 2010). The nuts has 15% protein, 7.5% fat, 52% starch, 3% sugar and 22.5% water (Zhu, 2016). TN is also an important source in terms of nutrients, protein, minerals and vitamins for fishs (Kalita et al., 2007; Kalita et al., 2008; Mukherjee, et al., 2010). It is also stated in the ancient medicine books in China that TN husks can help fight against various diseases such as diarrhea and alcohol poisoning (Sheng et al., 2006).

Its fruits and husks have a rich content consisting of starch, dietary fibre, essential amino acids and some types of phenols and minerals. Previous studies have shown that TN leaves, fruits, husks, flowers and roots have 19.5% amylose (Lertphanich et al., 2013), 1.9 mg/g phosphorus (Akao et al., 2013; Zhu, 2016), fatty acids such asnervonicacid (63.5%),  $\alpha$ -linolenic acid (6.4%), palmitolenic acid (6.4%), linoleic acid (6.3%) (Mukherjee et al., 2010; Zhu, 2016), phenols

such as caffeic acid, chlorogenic acid, ferrulic acid and 3-Omethylgallic acid (Stoicescu et al., 2012; Zhu, 2016), flavonoids such as quercetin and kaempferol (Niranjan et al., 2013; Zhu, 2016). The majority of fatty acids (69.3%) in the TN content form unsaturated fatty acids (Mukherjee et al., 2010). The fruit in the husk contains a higher amount of starch, while the husk contains higher amounts of phenolic material.

TN husks contain higher amount of C (43.4%), O (50.4%) ve H (5.7%) (Liu et al., 2010). Its husk have higher properties (anti-cancer, anti-atherogenic, bioactivity immune antimicrobial, antioxidant, hepatoprotective, enhancing, anti-imflammatory, anti-hyperglycemic, anti-skin photoaging) than fruits (Zhu, 2016). After harvesting period, pink colored TN husks change its color to dark brown in time (Huang et al., 2016). These husks contain dietary fibres and polyphenols (such as hydrolysable tannins) (Yasuda et al., 2014; Kim et al., 2014; Huang et al., 2016). The total phenolic, flavonoid and tannin amounts in TN husk extract were: 63.81% mg/g gallic acid equivalents, 21.34% mg/g of rutin equivalents and 17.11% mg/g of total tannin equivalents respectively (Malviya et al., 2010; Zhu, 2016).

Despite all of the good qualities, TN, which was exposed over large areas in North America in the 1800's, was considered as a tribulation. This is why it prevents the light permeability below the surface in the water it is spreading. With the reducing amount of light permeability, the amount of dissolved oxygen in the water reduces and that is adversely affects fish communities. It also displaces other submerged plants, making it difficult to navigate the boats, catch fish and swim (Hummel & Kiviat, 2004; Zhu, 2016).

The TN fruits must be peeled from the dark brown husks before they are cooked or canned. The peeled husks are burned or disposed of as waste. Husks thrown in this way cause water and air pollutions (Liu et al., 2010).

Lignin is the most abundant natural polymer after cellulose. It generates 10-25% of the lignocellulosic biomass. It has a complex structure composed of different types of phenol (sinapyl, p-coumaryl, and coniferyl alcohols). There are many usage areas such as emulsifiers, dyes, thermosets, dispersant agents, synthetic flooring, and paints (Watkins et al, 2015).

TN husks include 72.3 g/100 g (dry basis) of crude fiber. When this amount compared to dietary fiber contents (ranged from 10.2 to 87.9 g/100 g) of other fruits and greens (apples, citrus fruits, oat, wheat, corn), it can be said that it is a rich natural source of dietary fiber (Chau & Huang, 2004; Grigelmo-Miguel & Martin Belloso, 1999a, 1999b; Jaime et al., 2002). The previous studies have reported that the TN husks contain 6-14.6 g/100 g hemicellulose, 9-10.2 g/100 g cellulose and 35.4-48.3 g/100 g lignin (Chiang & Ciou, 2010). In the last few years, a lot of TN shells have been seen in the Bartin coasts. The reason why these husks were not utilized is that the local people were unaware of TN. In this study, the use of TN, causing coastal pollution, as a source of valuable chemicals was investigated by determining the amount of lignin.

### **MATERIAL and METHODS**

*Materials:* TN nuts were collected from the costs of Inkumu-Bartin in 2017. Identification of the plant material was performed by Prof. Dr. Z. Kaya at the Faculty of Forestry of Bartin University. Husks were dark brown, 3-5 cm wide and sclerosis and had no fruits inside. For analysis, TN husks were washed with tap water and then freezedried. Dried husks were cut with knife and grinded with Wiley Mill. Pure lignin sample was purchased from LignoWorks.

### Methods:

Acid-insoluble Lignin Determination: Before the lignin analysis, extractives were removed from the TN samples. Samples were extracted in a soxhlet apparatus for 6 hours with ethanol. Acid-insoluble lignin was determined according to TAPPI T2220m-02. 15 ml of 72% H<sub>2</sub>SO<sub>4</sub> was added to 1 g of extractive free TN samples and stirred for 2 hours. Then the mixture was placed in 1 L of erlenmayer with adding 560 ml of distilled water and boiled on a multiheater for 4 hours by attaching a cooler. At the end of 4 hours, the samples were filtered on glass crucibles and washed with hot distilled water.

*Fourier Transform Infrared Spectroscopy (FTIR):* FTIR analysis was carried out with Shimadzu IRAAffinity-1 spectrometer with single reflection Attenuated Total Reflectance (ATR) pike MIRacle sampling accessory (Fourier transform infrared spectra were obtained using a PerkinElmer Spectrum 100 with a universal ATR sampling accessory). Four accumulated spectra were collected in the wave number region of 700–4000 cm<sup>-1</sup>, with a spectral resolution of 4 cm<sup>-1</sup>.

Thermal gravimetric analysis (TGA) and differential thermal gravimetric (DTG) analysis: TGA/DTG

analyzes were carried out with Hitachi STA7300 Thermal Analysis System. 3 mg of dried and ground sample was heated from 30 °C up to 1200 °C. Analysis was carried out at the heating rate of 10 C min<sup>-1</sup> in nitrogen atmosphere.

# FINDINGS and DISCUSSION

*Acid-insoluble Lignin Determination:* The acidinsoluble lignin content was found to be 28.31%. As seen from Table 1, TN is a good source of lignin compared with other lignocellulosic materials.

Fourier Transform Infrared Spectroscopy (FTIR): FTIR analysis indicated that TN contained cellulose, hemicelluloses and lignin as expected. 3400-3330 cm<sup>-1</sup> OH stretching of alcohols, phenols, and acids (Tjeerdsma & Militz, 2005; Müller et al., 2009; Esteves et al., 2013; Mattos et al., 2015; Missio et al., 2015; Gonultas & Candan, 2018). 2970-2820 cm<sup>-1</sup> C-H stretching in methyl and methylene groups (Müller et al., 2009; Esteves et al., 2013; Mattos et al., 2015; Gonultas & Candan, 2018). 1650-1652 cm<sup>-1</sup> bands are absorbed water molecules of C=O stretching of the aromatic structures (Kotilainen et al., 2000; Nuopponen, 2005; Ozgenc et al., 2017). In the TN samples, it was seen that the range of 1750-1650 cm<sup>-1</sup> bands a variety of pixels unlike the pure lignin was appered (Figure 1). These peaks are derived from water and extractive substances (Zhou et al., 2015). The peaks at 1506-1510 cm<sup>-1</sup> in both samples are characteristic peaks for the lignin components due to C = Oand COO-non-symmetric stretching vibrations in the aromatic rings of the lignin structure (Özgenç, 2014; Can & Sivrikaya, 2017; Can & Sivrikaya, 2016).

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Table	1.	LIGHT	values	of some	ngnocen	uiosic	materials.

Sample	Lignin (%)	Literature
Trapanatans	28.31	Detection
Pinussylvestris	27.2	Dönmez, 2010
Larix decidua Mill.	13.42-29.54	Muhcuet al., 2015
PinusbrutiaTen.	25.9	Taş, 2017
Tectonagrandis	23.7-25	Dwumaa, 2016
QuercusroburL.	22.6-23	Krutul et al., 2010
Betula pendula Roth.	18.8-19.8	Krutulet al., 2014
Pseudocydoniasinensis	21	Qin et al., 2018
Hybrid Pennisetum	20.8	Wang et al., 2018



Figure 1. TN and Pure lignin ATR-FTIR spectrums.

The bands at 1452-1459 cm<sup>-1</sup> belong to C=C and C=H bond, O-H in plane deformation, and asymmetric bending in lignin (Nuopponen, 2005; Ishimaru et al., 2007; Ozgenc et al., 2017). 1420-1422 cm<sup>-1</sup> bands belong to aromatic skeletal vibration combined with CH inplane deformation for lignin (Muller et al., 2009; Chen et al., 2010; Herrera et al., 2014; Traore et al., 2016). 1263 cm<sup>-1</sup> band belongs to guaiacyl ring breathing, C–O stretch in lignin and C–O linkage in guaiacyl aromatic methoxyl groups (Popescu et al., 2007; Muller et al., 2009; Traore et al., 2016). 1233 cm<sup>-1</sup> and 1155 cm<sup>-1</sup> bands show the alkyl-aryl-ether bonds, lactones and cellulose C-O-C symmetric stretching

respectively (Nuopponen et al., 2003; Nuopponen, 2005; Ozgenc et al., 2017). 1110 cm<sup>-1</sup> band belongs to OH association of cellulose and this peak is seen just in the TN spectrum. At the 1033 cm<sup>-1</sup> band aromatic C-H deformation, C-O deformation, and C=O stretching in lignin are seen in both spectrum (Zhou et al., 2015).

As can be seen in the Figure 2; TN lignin and pure lignin samples have very similar FTIR spectrums. As far as we know, the pure lignin was obtained from pine species which shows that TN lignins chemical structure looks like pine lignin's.



Figure 2. TN lignin and Pure lignin ATR-FTIR spectrums.

Thermal gravimetric analysis (TGA) and differential thermal gravimetric (DTG) analysis: TN and pure lignin samples's TGA/DTG curves shown in Figure 2, are sharing similarity at some temperatures. WhenTN TGA curves were examined, approximately 4,5% of the sample is lost from 30 to 200 °C. At these intervals, samples generally lose volatile constituents and moisture (El-Sayed & Mostafa, 2014). From 200 °C to 271 °C 4,6% of the weight is lost. The decrease in this range may be due to the decomposition of cellulose and hemicelluloses. The DTG curve shows maximum weight-loss occurred at 349 °C. The weight-loss in the range from 352 °C is about 50%. According to these results, dominantly lignin and small quantities of cellulose and hemicelluloses are present in TN samples.



Figure 3. TGA and DTG diagrams of TN and Pure lignin samples.

Lignin's complex structure includes mostly, various branching aromatic rings. These chemical bands have a wide degradation temperature range from 100 °C to 800 °C (Yang et al., 2007). From 30 °C to 200 °C, volatile constituents and moisture removes from lignin sample. At the range of 180 °C–350 °C, carbohydrates are converted to volatile gasses (CO, CO<sub>2</sub>, and CH<sub>4</sub>). Following 350 °C phenolics, alcohols, and aldehyde acids are removed from lignin (Watkins et al., 2015). Pure lignin sample's TGA and DTG curve results show 10%, 50%, and 70% weight-losses at 233 °C, 489 °C, and 1167 °C respectively (Table 2). Differently from TN sample, pure lignin's 10% of degradation temperature 37,9 °C lower. It may be due to pure lignin samples contamination cause of storage conditions. 50% degradation temperature of TN has difference owing to it's cellulose and hemicelluloses content.

 Table 2. Termal degredation tempratures of TN and pure lignin samples.

	T10%	T50%	T70%	Residue (%)	DTGmax
Trapa	271,5°C	352,1°C	576,7°C	82,8	349,6°C
Lignin	233,6°C	489,2°C	1167°C	100	373,1°C

#### CONCLUSION

In many coastal strips of Bartin such as Inkumu and Guzelcehisar, TN can not only cause an environmental pollution, but also can reach the wetlands and it may be the possibility of spreading in those areas. When considering the richness of the lignin in TN husks, the evaluation of these materials will be able to be used as a new source of valuable chemicals to produce lignin and besides can help to eliminate the environmental pollution.

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