

ISSN 0535-8418

SERİ		CİLT		SAYI		
SERIES	A	VOLUME	58	NUMBER	2	2008
SERIE		BAND		HEFT		
SÉRIE		TOME		FASCICULE		

İSTANBUL ÜNİVERSİTESİ  
**ORMAN FAKÜLTESİ**  
D E R G İ S İ

REVIEW OF THE FACULTY OF FORESTRY,  
UNIVERSITY OF ISTANBUL

ZEITSCHRIFT DER FORSTLICHEN FAKULTÄT  
DER UNIVERSITÄT ISTANBUL

REVUE DE LA FACULTÉ FORESTIÈRE  
DEL 'UNIVERSITÉ D'ISTANBUL



## BU SAYININ HAKEM LİSTESİ (REFEREE LIST FOR THIS ISSUE)

We thank the referees, who contributed their expertise, time and effort for the journal.

Bu sayıda yer alan makalelerin hakemliği için zamanını, uzmanlığını ve emeğini  
Harcayan hakemlerimize teşekkür ederiz.

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Orman Fakültesi Dergisi Cilt 58, Seri A/2

ISSN 0535-8418 2009 basımı 500 adet basılmıştır.

İstanbul Üniversitesi  
Basım ve Yayınevi Müdürlüğü  
Tel: (0212) 631 35 04 - 05

**İSTANBUL ÜNİVERSİTESİ**  
**ORMAN FAKÜLTESİ DERGİSİ**

Review of the Faculty of Forestry, University of Istanbul  
Zeitschrift der Forstlichen Fakultät der Universität Istanbul  
Revue de la Faculté Forestière de l'Université d'Istanbul

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# Accumulation of Heavy Metals as Related to Cation Exchange in Some Forest and Pasture Soils of Stara Planina (Serbia)

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## Abstract

Heavy metal accumulation in the components of terrestrial ecosystems is conditioned by the deposition intensity, soil characteristics, mineral composition, and vegetation type. The exchangeable ionic reactions between the adsorptive complex and the soil solution are important sources of nutrients and also a significant mechanism for heavy metal retardation in the soil. The aim of this study is to determine the effect of cation exchange capacity on heavy metal accumulation in the soils under different vegetation (forest and pasture). The differences in the behaviour of individual elements depending on CEC in forest and pasture soils result primarily from different ratios of base and acid cations in the soil adsorptive complex and their reactions with heavy metals.

**Keywords:** Heavy metal, cation exchange capacity, forest soil, pasture soil

## 1. Introduction

Heavy metal accumulation in the components of terrestrial ecosystems is conditioned by the deposition intensity, soil characteristics, mineral composition, and vegetation type. Soil, as a natural resource, has the main ecological and production function in the terrestrial ecosystems. It is significant for the retention of heavy metals, primarily thanks to its buffer and filter role. In the soil adsorptive complex, base and acid cations in different ratios are in dynamic balance with the corresponding cations in the soil solution. The exchangeable ionic reactions between the adsorptive complex and

the soil solution are important sources of nutrients and also a significant mechanism for heavy metal retardation in the soil.

The distribution of exchangeable cations in the soil profiles of the same soil type depends on the type of vegetation (pasture, forest), as well as on the properties of the element and its cycling in the ecosystem (Jobbágy and Jackson, 2004). This research is especially significant in hilly and mountainous areas, where forest and agroecosystems are combined. The study of soil properties in the conditions of different vegetation covers points to the potentials of the sustainable use of this strategic resource in different silvopastoral systems.

The research of the soil chemical properties under different land uses is especially significant for the organisation of the territory in the aim of sustainable management. Ruark and Schoeneberger (2003) report that the cultivation of woody plants and other crops together (silvopastorally) improves the soil properties. The same authors report that the research during the past two decades emphasised three main indirect processes of soil property improvement: 1) increased N input through biological nitrogen fixation by nitrogen-fixing trees, 2) enhanced availability of nutrients resulting from decomposition of tree biomass, 3) greater uptake of nutrients from deeper layers by the roots of tree species.

The content of mineral nutrients in the soil solution is defined by soil type, climate and vegetation type, i.e., nutrient cycling in the ecosystem. Different processes and chemical reactions in the soil, especially the transformation of organic matter, affect the cation release from the adsorptive complex. Thus, according to Jobbágy and Jackson (2004), in base saturated soils (75 – 100%), surface layers contain higher concentrations of Ca, Mg and Na under forest, while the content K ions is higher under pasture. However, according to Krishnaswamy and Richter (2002), the content of base cations is in direct relation with soil acidity. The above authors claim that base cation content in the top 30 cm of soils is 73.9 % under pasture and 22.8 % under forest, where the observed soil pH-value was lower. According to the same authors, carbon content is higher in forest soils, whereas the lower content in pasture soils is attributed to the low quantity of organic residue and frequent wildfires. Under such conditions, with small losses of carbon quantity, pH-value and the content of exchangeable cations increase, and Al ions are exchanged by Ca<sup>2+</sup> ions.

Based on the previous research in this area and based on the literature, higher contents of base cations can be expected in pasture soils, and of aluminium in forest soils. The differences in base cation contents in the adsorptive complex will have a significant impact on the heavy metal accumulation in the soils.

The aim of this study is to determine the effect of cation exchange capacity on heavy metal accumulation in the soils under different vegetation (forest and pasture).

## 2. Material and Method

The study area is Stara Planina, the localities Babin Zub - N= 43°22'35.7'' E= 022°37'38.3'' altitude 1547 ± 4m, Široke Luke - N= 43°14'24.7'' E= 022° 51'36.8''

altitude  $1288 \pm 6\text{m}$ , and Prelesje - N=  $43^{\circ}10'42.5''$  E=  $022^{\circ}56'20.0''$  altitude  $1287 \pm 7\text{m}$ .

As for the climate, Stara Planina region is the transition zone between temperate-continental climate in the north and montane climate of the Balkan mountainous system in the southeast. Annual temperature amplitude declines from the north towards the south and southeast, so the difference in air temperature between Zaječar and Dimitrovgrad is more than  $2^{\circ}\text{C}$ . The distribution of annual precipitation sum is different in the area of Stara Planina, in the Timok valley annual precipitation is 700 mm, while in Pirotska Dolina (valley), it is often below 600 mm. Based on Popović's (2007) study and the analyses of meteorological data for the area of Stara Planina (Đorđević-Milošević, 1996), it can be concluded that the change in the direction of arid climate is underway.

Stara Planina vegetation, in general, is characterised by a considerable diversity of forest, shrub, meadow, pasture and peat bog communities and, as such, it is one of the centres of flora diversity of the Balkan Peninsula, with 147 identified threatened and endemic species (Mišić et al., 1978; Randelović, Randelović, 2002).

Pasture areas of the study sites are occupied by plant community *Agrostietum vulgaris (capillaris)* Z. Pavlović 1955. Meadows of *Agrostietum vulgaris* are of secondary anthropogenic origin, as they are the result of two anthropogenic factors: reduced area under forest on the one hand, and mowing, on the other hand. The association *Agrostietum vulgaris* covers a huge area in the hilly region. On Stara Planina, this community is the dominant meadow type and it is widely distributed. In this area, it develops on quite different sites.

During the plant community research in beech forest ecosystems, the community of subalpine beech forest (*Fagetum moesiacaе subalpinum* Greb. 1950) was identified at the site Babin Zub: canopy 0.6, diameter of mean stand tree  $d_s - 36\text{cm}$ , height 25.4 m and age 110 years.

The community of montane beech forest (*Fagetum moesiacaе montanum* B. Jov. 1976, subassociation *typicum facies asperulosum*) was identified in beech forest ecosystems at the site Javor: canopy 0.6 – 0.8, diameter of mean stand tree  $d_s - 34\text{cm}$ , height 24.0 m and age 130 years.

The community of montane beech forest (*Fagetum moesiacaе montanum* B. Jov. 1976, subassociation *typicum facies nudum*) was identified in beech forest ecosystems at the site Prelesje: canopy 0.7, diameter of mean stand tree  $d_s - 19\text{cm}$ , height 17.3 m and age 65 years.

Four soil profiles were opened in each of the pasture areas. Based on morphological and basic physico-chemical soil characteristics after FAO (1985) classification, two types of pasture soils were determined:

Babin Zub:

Dystric leptosol on sandstone

Javor:

Dystric leptosol on chlorite schist

Dystric cambisol on chlorite schist

## Prelesje:

Eutric cambisol on sandstone

Dystric leptosol on sandstone.

Four soil profiles were opened in each of the forest areas. Based on morphological and basic physico-chemical soil characteristics after FAO (1988) classification, two types of pasture soils were determined:

## Babin Zub:

Dystric cambisol on sandstone

Javor:

Dystric cambisol on chlorite schist and phyllite

## Prelesje:

Dystric cambisol on sandstone

Eutric Cambisol on sandstone.

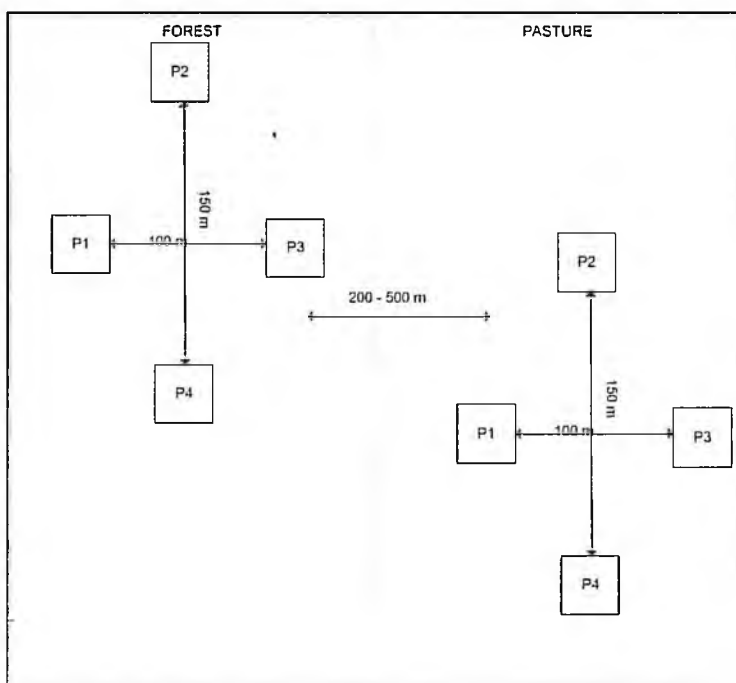


Figure 1. Profile opening scheme in pasture and forest areas

The soils were sampled at the depth of: 0-5, 5-10, 10-20 and 20-40 cm. The main physical and chemical soil characteristics were determined by ICP Methodology (IPC Forest Manual, Part III, Soil Sampling and Analysis, Hamburg, updated 2006). Total heavy metal content was determined by atomic adsorption spectrophotometer (AAS), and the conservation and preparation of samples by the extraction with HCl, HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> (3:1:2). Cation exchange capacity was calculated as the sum of acid (Al, H, Fe and Mn) and base (Ca, Mg, K, and Na) cations. Exchangeable cations were extracted with



0.1M BaCl<sub>2</sub>, and measured (ISO11260 & ISO 14254). The significance of differences between total heavy metal contents, as well as other characteristics of forest soil and pasture soil, was tested by the analysis of variance. Indicator values of heavy metal sorption were calculated according to equation:

$$I_{SO} = I_{Ad} / RI_1 \text{ (range 0- 5)}$$

$I_{Ad}$  – Indicator value of adsorption

The retardation indicator (*Helhveg, 2000*) serves for the classification of heavy metal mobility in soils. The retardation indicator is determined in function of the pH value and supplements (clay content, organic content, Fe-oxide), and calculated according to equation:

$$RI_1 = RI_{pH} + RI_{Organic\ content} + RI_{clay\ content}$$

Indicator values of heavy metal adsorption (*Belanovič, 2006*), were calculated according to equation:

$$I_{Ad} = \sum_i^0 \left[ \left( \frac{TM}{Ca + Mg + K} \right)_c \times \omega_i \right]; i - \text{layer (0-5; 5-10 and 10-20)}$$

$I_{Ad}$  – indicator of adsorption (rank 0 - 10);  $(TM/(Ca+Mg+K))_c$  – class of indicator of adsorption (class 1-5),

TM – content of heavy metals (Zn, Cu, Pb and Cd); (Ca+Mg+K) exchangeable ionic Ca+Mg+K;

### 3. Results and Discussion

Cation exchange capacity is higher in forest soils than in pasture soils. Forest ecosystems are characterised by a higher degree of heavy metal retardation than grass ecosystems or aquatic ecosystems, thanks to their larger relative area and greater roughness. The processes such as dry and wet deposition, washing down from the surface of plant photosynthetic organs, canopy drop and stem flow and the deposition in forest litter on the soil surface, as a rule, result in the increase in heavy metal content on the soil surface (*Vanmechelen et al., 1997*).

The results of the analysis of variance (F-test) (Table 1) show that there are no statistically significant differences between total concentrations of Zn, Pb, Cd in forest soils and pasture soils at the significance level 95%.

Table 1. Results of the analysis of variance

LSD 95%															
	Cu <sup>2+</sup>	Zn <sup>2+</sup>	Pb <sup>2+</sup>	Cd <sup>2+</sup>	orgC	Humus	totalN	CEC	Al <sup>+++</sup>	Ca <sup>2+</sup>	Al/ (Ca+Mg)	Al/Ca	AxE	Fe	Mn
F calculate	7.90	0.01	0.67	1.46	5.78	5.55	7.30	10.0	6.64	0.08	7.29	6.86	7.03	2.69	16.89
F table	3.84	3.84	3.84	3.84	3.94	3.94	3.84	3.96	3.96	3.96	3.96	3.96	3.96	3.96	3.96
Average value Forest	24.21	66.22	26.07	0.81	3.55	6.19	0.39	30.19	22.52	5.78	8.67	10.66	23.14	0.016	0.41
Average value Pasture	31.52	66.65	24.62	0.69	5.22	8.97	0.51	21.92	14.61	5.53	3.82	4.65	15.02	0.009	0.22
Group homogene	a b	a a	a a	a a	a b	a b	a b	a b	a b	a a	a b	a b	a b	a b	a b

The results of the analysis of variance (F-test) show that there are statistically significant differences, at the significance level 95%, between the contents of total Cu, organic C, humus, total N, exchangeable cations, contents of exchangeable Al, Fe and Mn, Al/Ca ratio, total acid cations (AxEc), in forest soils and pasture soils. According to LSD test, at the significance threshold 5%, two groups of factors separate according to the significance of the contents of total Cu, organic C, humus, total N, exchangeable cations, contents of exchangeable Al, Fe and Mn, Al/Ca ratio, total acid cations (AxEc).

Heavy metals occur in the soil as exchangeable - adsorbed on soil colloids, specifically-adsorbed, bound in various chemical compounds (oxides, carbonates, phosphates, sulphides) and structurally bound in the silicates (primary and secondary minerals) (Adriano, 1986). Different factors affect the bonding of heavy metals to the soil, and the main problem is the estimation of the heavy metal load in the soil. The load of heavy metals in the soil can be indicated to some extent by the monitoring of their content in the soil.

The assessment of the soil sorptive capacity renders significant information on the soil susceptibility to heavy metal loads (Sastre et al., 2006). According to Sastre et al. (2006) the concentration Ca+Mg in the soil solution increases with the increase in heavy metal concentration, which points to the cation exchange process between these macroelements and heavy metals. By the increase in heavy metal content, the cation exchange process increases and leads to the release of Ca, Mg and K from the soil adsorptive complex. It is a fact that cation exchange is the leading force in metal sorption.

Sparling et al., (2001) conclude that soil pH is related to land use. According to Krishnaswamy and Richter (2002), the content of base cations is in direct correlation with soil acidity. They report that base cation content in the study conditions, in the top 30 cm under pasture was considerably higher than in forest soils where the observed soil pH-value was lower.

Soil acidification has a significant impact on the soil chemical and biological processes. Higher acidification of forests and pastures by atmospheric deposition causes an irreversible decline of cation exchange capacity (CEC) and the mobilisation of potentially toxic elements Al, Fe and Mn (Blake and Goulding, 2002). Soil acidification reduces the number of plant species in natural pastures.

Soil characteristics positively correlated with cadmium retention were pH, organic matter content and cation exchange capacity (CEC), specific surface area, while free iron oxides were negatively correlated (De Matos, et al., 2001). The buffer characteristics of the soil solid phase are conditioned by the quantity of colloids and the type of adsorbed cations. A high significance is assigned to the energy of hydrogen ion adsorption by the colloids and the degree of colloid dissociation. The soil organic matter mainly consists of weak acids so, accordingly, hydrogen ions are weakly dissociated, and for this reason organic matter increases the buffering capacity of the soil. Adsorption, surface deposition and polymerisation are the examples of sorption, the basic term for the retention mechanism on the soil surface. The sorption of metal cations depends primarily on pH value, and it is characterised by a narrow pH rank when adsorption increases up to almost 100%. De Matos et al. (2000) showed the strong influence of the cation exchange phenomenon on the retardation and mobility of Zn and Cd. Zinc retention was positively correlated with pH, CEC and specific surface area (de

Matos, et al., 2001). Copper retention has positive correlation with pH, sum of bases or exchangeable calcium, organic matter content and to CEC (De Matos et al., 2001). Lead retention was better correlated to clay content, pH and SB or exchangeable Ca (De Matos, et al., 2001).

Table 2 presents the relation between heavy metals and cation exchange of the adsorptive complex. Zn and Pb concentrations increase with the CEC increase in forest and pasture soils. In forest and pasture soils, Cu contents decrease with the increase of CEC. Cd concentration increases with the CEC increase in forest soil, while Cd contents decrease with CEC increase in pasture soils. The differences in the behaviour of individual elements depending on CEC in forest and pasture soils result primarily from different ratios of base and acid cations in the soil adsorptive complex and their reactions with heavy metals.

The heavy metal solubility is mainly caused by relatively constant soil characteristics, such as the contents of organic matter and clay, pH value, but also the parameters such as the concentration of available Ca and the concentration of soluble organic carbon (De Vries and Bakker, 1998). The assessment of pollutant sorption in the soil is based on the solid – liquid distribution coefficient, which is the relation between the quantity of heavy metals sorbed on the solid phase and the total concentration in the solution in contact with the soil (Sparks, 1995; Sastre et al., 2006).

Some authors suggest the ratios of elements as the criterion for the soil chemical status from the aspect of root development, i.e. Ca/Al, Mg/Al, base cations/Al (Rehefuess and Prietzel, 1998; Alveteg, 1998; Shaodong and Min, 2004). Al/BCE ratio in pasture soils is on the average lower than in forest soils in the area of Stara Planina (Belanović and Košanin, 2004). Al/Ca ratio does not define the critical values from the aspect of root growth (Kulhavy, 1998), but the wide ratio of Al/Ca results in the low seed development of some mountainous plants (Van der Berg et al., 2003). Also, the ratio Al/base cations indicates the changes in critical loads occurring due to the increased or decreased deposition (Posch and Hettelingh, 2001). Several studies report the unfavourable ratio Mg/Al in the soil solution as the main problem of increment in European forests (Joki-Heiskala et al., 2003). Rehefuess and Prietzel (1998) claim that tree vitality and increment depend on the supply of nutrients Mg, K and Ca in the soil solution and their interaction with Al. Soil acidification has a direct or indirect effect on the increase of heavy metal concentration in the soil solution.

The sorption indicator (Table 3) represents the retardation capacity of the soil for heavy metals, and is obtained from the ratio of adsorption indicators and retardation indicators. The adsorption indicator points to the equilibrium state between macro elements Ca, Mg and K and heavy metal contents. If the sorption indicator is higher, the soil capacity of accumulating and retaining heavy metals is lower, and this increases the potential pollution of surface and ground waters and the environmental quality in general.

Accumulation of Heavy Metals as Related to Cation Exchange in Some Forest...

Table 2. Results of regression analysis of total heavy metal contents and CEC

Element	Regression equation	R	R <sup>2</sup> (%)	P-value	F	F -tab	t <sub>a</sub> p- value	t <sub>b</sub> p- value
FOREST								
	$Zn = \frac{1}{(0,0086 + 0,183 \cdot CEC^{-1})}$	0.55	30.20	0.0001	19.90	4.04	4.554 0.000	4.461 0.0001
	$Cu = 33,565 \cdot CEC^{0,109}$	-0.15	2.18	0.3215	1.00	4.06	9.78 0.000	-1.002 0.3215
	$Pb = \frac{1}{(0,0269 + 0,3888 \cdot CEC^{-1})}$	0.43	18.51	0.0023	10.45	4.04	4.855 0.000	3.232 0.0023
	$Cd = 1,1133 - \frac{7,441}{CEC}$	-0.24	5.95	0.0947	2.91	4.04	5.549 0.0000	-1.706 0.0947
PASTURE								
	$Zn = \frac{1}{(-0,00299 + 0,4487 \cdot CEC^{-1})}$	0.69	48.14	0.0000	39.91	4.07	-0.786 0.4360	6.317 0.000
	$Cu = 3,1169 + 586,779 \cdot CEC^{-1}$	0.59	34.79	0.000	22.94	4.07	0.475 0.6370	4.789 0.000
	$Pb = -27,58 + 16,96 \cdot \ln(CEC)$	0.59	34.61	0.000	22.76	4.07	-2.54 0.0148	4.771 0.000
	$Cd = 0,981 - 0,015 \cdot CEC$	-0.19	3.62	0.2104	1.62	4.07	3.709 0.0006	-1.271 0.2104

Function parameters: S<sub>r</sub> - standard error of regression; t<sub>a</sub> - t - test; t<sub>b</sub> - t - test; p - confidence level; F - calculated; F value; F<sub>i</sub> - F value from tables of F distribution; R<sup>2</sup> - determination coefficient F (0.05; k-1 and N-k); R - correlation coefficient

Table 3. The indicator value for heavy metal sorption in studied soils

Locality	Profile	Is Zn	Is Cu	Is Pb	Is Cd
		FOREST			
Javor	9	4.24	2.22	1.40	0.95
	10	4.17	2.27	1.69	4.55
	11	3.10	1.53	1.16	3.06
	12	2.63	1.72	1.32	2.78
Babin Zub	17	4.35	2.28	1.79	4.06
	18	4.91	2.46	1.89	4.91
	19	3.89	1.97	1.58	3.94
	20	4.67	1.83	1.58	4.00
Prelesje	26	1.52	1.09	0.80	1.52
	27	4.33	2.17	1.70	4.00
	28	1.67	1.28	0.97	1.84
	29	2.62	1.67	1.16	3.06
		PASTURE			
Javor	1	2.22	1.31	0.91	0.71
	2	2.80	1.70	1.11	3.33
	3	3.46	1.99	1.29	3.08
	4	2.76	1.50	0.94	2.72
Babin Zub	13	2.35	1.24	1.16	0.63
	14	3.33	2.00	1.67	3.20
	15	3.70	1.99	1.61	3.08
	16	3.70	2.13	1.61	3.33
Prelesje	22	2.86	2.08	1.16	3.33
	23	2.62	1.67	1.27	3.33
	24	2.56	2.03	1.20	2.90
	25	3.33	2.17	1.42	1.74

The indicators of heavy metal adsorption are in relation with their accumulation in individual layers of the above soils and range within the classes of very low to very high for Cd and medium to very high for Zn, Cu and Pb. The sorption indicator ranges between 0-5, depending on  $I_{Ab}$  and RI, i.e. heavy metal load and retention capacity for heavy metals in the study soils. The differences are conditioned by the state of nutrient elements in the soil, edaphic conditions, and type of vegetation.

## 4. Conclusion

This research deals with forest soils and pasture soils on Mt. Stara Planina. Heavy metal accumulation in the components of the terrestrial ecosystems is conditioned by the deposition intensity, soil characteristics, mineral composition of the substrate, and vegetation type. In the soil adsorptive complex, base and acid cations in different ratios are in dynamic balance with the corresponding cations in the soil solution. The exchangeable ionic reactions between the adsorptive complex and soil solution are important sources of nutrients and also a significant mechanism for heavy metal retardation in the soil.

Zn and Pb concentrations increase with the CEC increase in forest and pasture soils. In forest and pasture soils, Cu contents decrease with the CEC increase. Cd concentration increases with the CEC increase in forest soil, while Cd contents decrease with the CEC increase in pasture soils. The differences in the behaviour of individual elements depending on CEC in forest and pasture soils result primarily from different ratios of base and acid cations in the soil adsorptive complex and their reactions with heavy metals.

The assessment of the soil sorptive capacity renders significant information on the soil susceptibility to heavy metal loads. The sorption indicator represents the soil retardation capacity for heavy metals. The adsorption indicator points to the equilibrium state between macroelements Ca, Mg and K and the content of heavy metals. If the sorption indicator is higher, the soil capacity of accumulating and retaining heavy metals is lower, and this increases the potential pollution of surface and ground waters and environmental quality in general.

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# Evaluation of Decay, Oxalic Acid Production and Strength Loss in Wood by the Dry Rot Fungus, *Serpula lacrymans*

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## Abstract

*Serpula lacrymans* (Wulfen:Fr.) Schroeter, the dry rot fungus, is generally accepted as one of the most economically important wood degrading fungi in some temperate regions of the world. This study evaluated decay capacity of one isolate of *S. lacrymans* at four different wood species by the two different decay tests by using wood blocks and stakes. Besides mass losses in the specimens, strength losses and oxalic acid (OA) production in wood by the fungus during decay process were also measured. Higher mass losses were observed in the wood blocks in soil block tests when compared to the stakes in a modified soil bed tests. Losses in modulus of rupture (MOR) in bending were more distinctive in the stakes than mass losses. In the specimens subjected to decay tests, there was a good correlation between both mass losses and OA production and MOR losses and OA production. Further studies are in progress for treated wood specimens to understand copper tolerance ability of *S. lacrymans* to copper-based wood preservatives.

**Keywords:** *Serpula lacrymans*, oxalic acid, mass loss, MOR loss, decay

## 1. Introduction

Decay caused by brown-rot fungi is the most prevalent and destructive type of wood deterioration because it can cause rapid structural failure. The dry rot fungus, *Serpula lacrymans* (Wulfen:Fr.) Schroeter is one of the most destructive and important decay fungi in buildings in Northern and Central Europe and it may cause decay and

structural damage in both timber and masonry (Hastrup et al., 2006). Depolymerization of cellulose in wood by brown-rot fungi has been explained by different pathways; by the oxidative radical reactions, such as the Fenton reaction, initiated by the production of extracellular hydrogen peroxide (Koenigs, 1974; Highley, 1987; Ritschkoff and Viikari, 1991) by one-electron oxidation (Enoki et al., 1990, 1991) and by production of oxalic acid (OA) (Schmidt et al., 1981; Bech-Anderson, 1987; Green et al., 1991). Brown-rot fungi contain at least two different OA producing enzymes, glyoxylate oxidase (dehydrogenase) and oxalate hydrolase, and the production of OA is connected to the tricarboxylic acid (TCA) cycle (Shimada et al., 1991). OA plays an important role in both brown and white rot decay. It is also assumed to be a metabolic byproduct of incomplete glucose oxidation either via malate in the TCA cycle or glyoxylate in the glyox cycle (Gadd, 1999; Munir et al., 2001). It is secreted by the majority of brown-rot fungi, including *S. lacrymans*, but only in limited amounts by white-rot fungi because of the presence of the OA-degrading enzyme oxalate decarboxylase (Akamatsu et al., 1992).

There might be no visible damage to the wood although brown rot decay fungi initiate colonization and start to release enzymes and organic acids such as OA. During early decay, color or texture of wood slightly changes, but decay may not be yet obvious (Clausen and Kartal, 2003; Clausen et al., 2001; Zabel and Morrell, 1992; Köse 2006). However, due to chemical changes during initial colonization, considerable losses in strength before measurable mass losses can occur (Clausen and Kartal 2003; Kim et al., 1996; Imamura 1993, Schmidt et al., 1978; Wilcox 1978).

It is of great importance to be able to prevent damage by *S. lacrymans* since this fungus is able to cause considerable failure in wooden structures. Since a relationship between copper tolerance and OA production has been implicated (Hastrup et al., 2005; Murphy and Levy, 1983), it is beneficial to know the resistance of *S. lacrymans* to wood treated with copper-containing wood preservatives. However, before tolerance tests, it is important to know the ability of the dry-wood fungus, *S. lacrymans* to produce OA during its decay process considering mass and strength losses in untreated wood. The aim of this study was to evaluate OA production and decay capacity in untreated wood blocks and stakes from various softwood and hardwood species exposed to the dry rot fungus, *S. lacrymans*. In the study, wood specimens were subjected to the fungus in a soil block test and a modified soil-bed test and strength and mass losses in the specimens were determined after a 3, 6 and 12 weeks incubation period together with pH and OA production.

## 2. Materials and Methods

### 2.1. Fungal culture

One isolate of *Serpula lacrymans* (Wulfen:Fr.) Schroeter (ATTC 36335) provided by the Center for Forest Mycology Research, Forest Products Laboratory, Madison, WI, USA was maintained on 2% malt extract agar.

## 2.2. Decay test

Wood blocks (19 x 19 x 19 mm) were prepared from sapwood portions of two softwood (*Pinus sylvestris* L., *Abies bornmülleriana* Mattf.) and two hardwood (*Fagus orientalis* Lipsky, *Populus x euramericana* I 214) logs. The blocks were free of knots, and visible concentration of resins, and showed no visible evidence of infection by mold, stain on wood destroying fungi. All blocks were conditioned at room temperature prior to steam sterilization for 30 minutes. Blocks were then subjected to *S. lacrymans* in soil block test (ASTM 1998). Test bottles were incubated at 27°C and 70% relative humidity (RH) for 3, 6 and 12 weeks. Mass loss in the blocks caused by fungus was then calculated from the weights before and after decay tests. Six replicate blocks for each wood species and duration were used in the tests.

Wood stakes from the sapwood portions of the four wood species (250 mm (L) by 25 mm (T) by 10 mm (R)) were exposed to *S. lacrymans* in a modified cake pan test. One liter of a 1:1 soil and vermiculite mixture was placed in an aluminum cake pan. The surface of the mixture was covered with rows of southern pine feeders (42 mm by 29 mm by 3 mm). The moisture content of the soil/vermiculite mixture was adjusted to 50% of the water-holding capacity, and the test apparatus was autoclaved at 103 kPa and 121°C for 45 min. When the pans were cooled, the feeders were inoculated with the fungus by pipetting 100 ml/pan of a macerated 3-week-old liquid culture of *S. lacrymans* evenly over the feeders. The test pans were sealed in a plastic bag to prevent drying and incubated at 27°C and 70% RH for 3 weeks until the feeders were completely covered by fungal growth. Steam-sterilized test specimens were then placed on the top of the feeders and the pans were incubated at 27°C and 70% RH for 3, 6 and 12 weeks. Mass loss in the stakes caused by fungus was then calculated from the weights before and after decay tests. Six replicate stakes for each wood species and duration were used in the tests.

## 2.3. Determination of modulus of rupture (MOR)

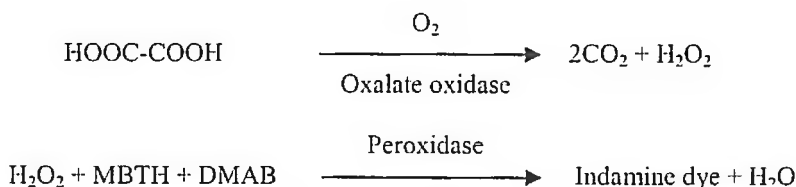
Stakes were conditioned at 20°C and 65% RH prior to modulus of rupture (MOR) in bending determinations. MOR was conducted according to ASTM D4761 (2005). Wood stakes were tested on a Losenhausen Universal Testing System equipped with a load cell with a capacity of 10,000 N.

## 2.4. Determination of pH value and oxalic acid production

The stakes approximately 25 mm-long by full cross-section were cut from the decay zone near the mechanical failure of specimen. The pH of wood was determined by an extraction method. Wood blocks and stakes samples were ground into sawdust. Sawdust of 2.0 g was then immediately added to 50 ml of boiling de-ionized water and stirred for 5 min. in an Erlenmeyer flask with reflux. The mixture was standing in the closed flask for 30 minutes and was then rapidly cooled to room temperature. The

extract was then filtered and pH of the solution was measured with a glass electrode. The experiment was performed in three replicates.

Soluble OA was measured in the same sawdust samples for pH determinations. Sawdust samples were extracted in 3.0 ml 0.1 M phosphate buffer, pH 7.0, for 2 h with shaking. For each extracted sample, OA was determined by microassay with a diagnostic kit (Trinity Biotech Plc Ida Business Park Bray, Co., Wicklow, Ireland). Amount of OA was expressed as micromoles OA per gram of final dry weight of wood. The enzymatic reactions involved in the assay procedure are as follows:



Oxalate is oxidized to carbon dioxide and hydrogen peroxide by oxalate oxidase. The hydrogen peroxide reacts with 3-methyl-2-benzothiazolinone hydrazone (MBTH) and 3-(dimethylamino) benzoic acid (DMAB) in the presence of peroxidase to yield an indamine dye which has an absorbance maximum at 590 nm. The intensity of the color produced is directly proportional to the concentration of oxalate in the sample.

### 3. Results and Discussion

Mass losses in wood blocks exposed to *S. lacrymans* for 3, 6 and 12 weeks are given in Figure 1. Average mass losses in the wood blocks ranged from 40% to 60% for 12 week incubation period. Highest mass loss was seen in *P. euramericana* specimens whilst the study revealed mass loss hierarchies of *P. euramericana* > *P. sylvestris* > *A. bornmülleriana* > *F. orientalis*. Hastrup et al. (2006) tested southern yellow pine wood blocks (10 x 10 x 10 mm) against various strains of *S. lacrymans* and the strain ATTC 36335 in soil block tests for 10 weeks. In their study, mass losses ranged from 28% and 53%.

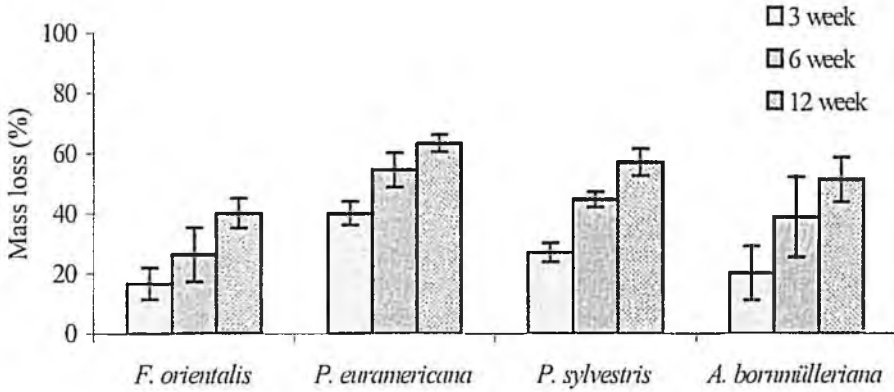


Figure 1. Mass losses occurred in the wood blocks.

Şekil 1. Odun bloklarında meydana gelen ağırlık kayıpları.

Relationship between mass losses and OA production in wood blocks exposed to *S. lacrymans* for 3, 6 and 12 weeks are given in Figure 2. The highest amount of OA was measured in *P. sylvestris* wood blocks; however, when compared to 6-week results, amount of OA decreased after 12-week-exposure period. Even though higher mass losses occurred in *P. euramericana* wood blocks than the other wood species, less OA production was seen in those blocks. The OA concentration and the percentage mass loss increased in blocks from week 3 to week 6. In *F. orientalis* and *P. euramericana* wood blocks, amount of OA increased from week 6 to week 12; however, no increases were seen in *P. sylvestris* and *A. bornmülleriana* blocks. *S. lacrymans* continued producing mass losses in *P. sylvestris* and *A. bornmülleriana* blocks even though those blocks showed no more OA production by the fungus.

Relationship between pH and oxalic acid accumulation in the wood blocks is given in Figure 3. There was a clear relation between pH and OA production in the blocks prepared from *F. orientalis*. In those blocks, as amount of OA increased, pH of wood decreased as expected. Some fluctuations were observed in *P. euramericana* and *A. bornmülleriana* blocks, even though OA production by the fungus continued. In *P. sylvestris* wood blocks, after 12 week exposure period, pH decreased when compared to week 6; however, OA production also decreased in the respective wood blocks for the same exposure periods.

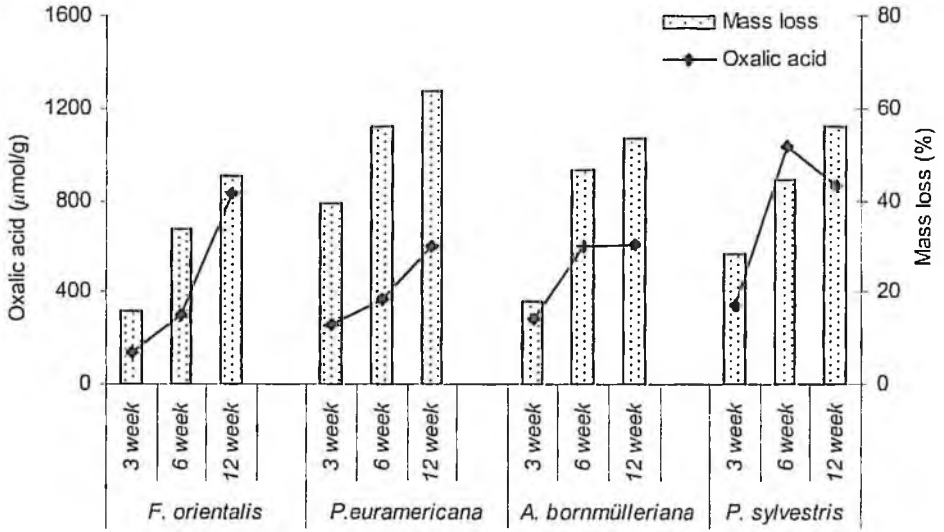


Figure 2. Relationship between mass loss and oxalic acid accumulation in the wood blocks.

Şekil 2. Odun bloklarında oksalik asit üretimi ile ağırlık kaybı arasındaki ilişki.

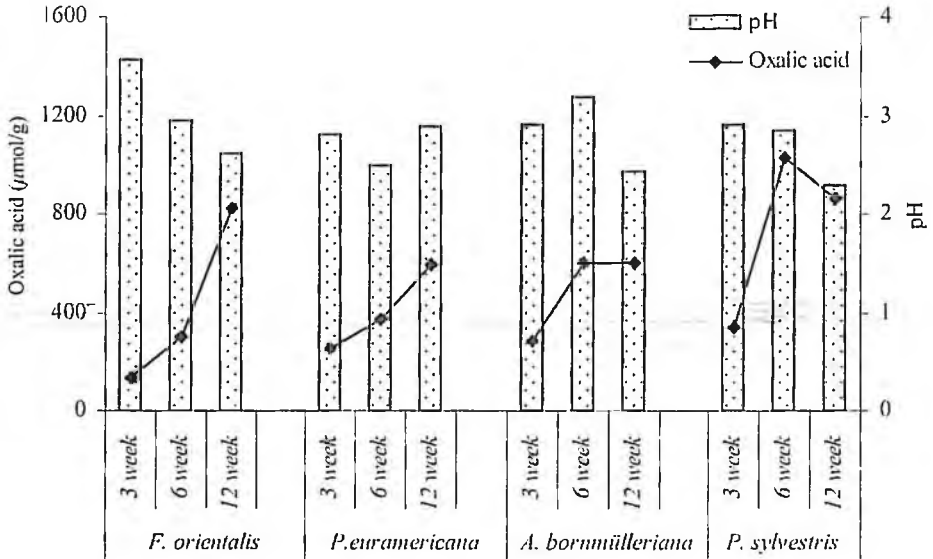


Figure 3. Relationship between pH and oxalic acid accumulation in the wood blocks.

Şekil 3. Odun bloklarında oksalik asit üretimi ile pH arasındaki ilişki.



Hastrup et al. (2006) studied correlations between the amount of soluble OA and the degree of wood decay in untreated southern yellow pine wood blocks. They found that OA levels declined in heavily degraded wood where average weight reduction of about 45–50% occurred. They stated that the amount of soluble OA were almost undetectable in wood blocks that were highly to completely degraded (Espejo and Agosin 1991; Itakura et al. 1994). In a study by Green III and Clausen (2003), two different *S. lacrymans* strains were tested to evaluate decay capacities and OA production in wood blocks. One strain caused mass loss of about 53% with OA production of about 169 mM; however, the other strain resulted in mass loss of 15% with OA production of 27 mM in southern yellow pine blocks. They state that OA production and a rapid lowering of pH by decay fungi are important in the initial stages of brown rot (Bech-Andersen, 1987; Green et al., 1991; Shimada et al., 1994). Micales and Highley (1988) state that OA production is not always directly related to the ability of fungi to decay wood.

Figure 4 illustrates MOR loss in the stakes exposed to *S. lacrymans* for 3, 6 and 12 weeks. Higher MOR losses were occurred in *P. euramericana*, *A. bormülleriana*, and *F. orientalis* stakes when compared to *P. sylvestris*. After 12-week exposure, MOR losses in the wood stakes in *F. orientalis*, *P. euramericana*, and *A. bormülleriana* reached nearly 90%; however, *P. sylvestris* wood stakes had MOR losses of about 60%. In *P. euramericana*, *A. bormülleriana*, and *F. orientalis* stakes, MOR losses after 6 weeks were as high as those after 12 weeks. MOR losses in *F. orientalis*, *A. bormülleriana*, and *P. sylvestris* wood stakes after 3-week-decay tests were around 40% while *P. euramericana* wood stakes showed MOR losses of nearly 70% in the same exposure period.

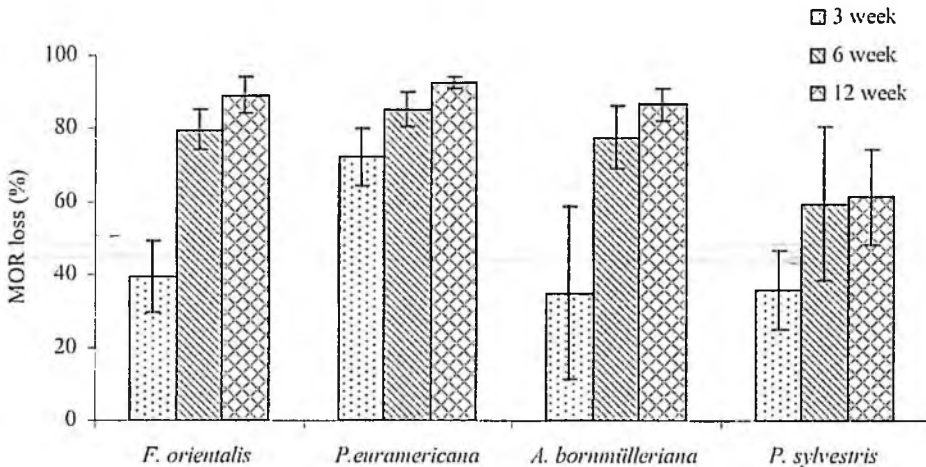


Figure 4. MOR loss in the stakes after decay tests.

Şekil 4. Çürüklük testleri sonrasında meydana gelen eğilme direnci kayıpları.

Figure 5 shows relationship among MOR, mass loss, and oxalic acid accumulation in the stakes after decay tests. The mass losses in wood blocks were two times greater than wood stakes. Measuring mass loss in the stakes in the modified cake pan method might be an inaccurate method compared soil block test method where considerably higher mass losses occurred. The size, shape, and greater volume of stakes might have slowed the progression of fungal colonization (Clausen and Kartal, 2003). In the study, MOR was reduced 72% to 93% by the time mass losses of 10% to 33% occurred. In general, OA production was correlated directly with decreases in pH of the substrates. *S. lacrymans* rapidly lowered the pH to 2.9 after 3 to 12-week decay process in *P. sylvestris*, *A. bormülleriana*, and *P. euramericana* wood stakes. Highest OA production was observed in the stakes from *P. euramericana* where highest MOR and mass losses were observed. *F. orientalis* and *A. bormülleriana* stakes also showed MOR losses more than 80%; however, lower OA and mass losses were found in the respective stakes in comparison with *P. euramericana* wood stakes.

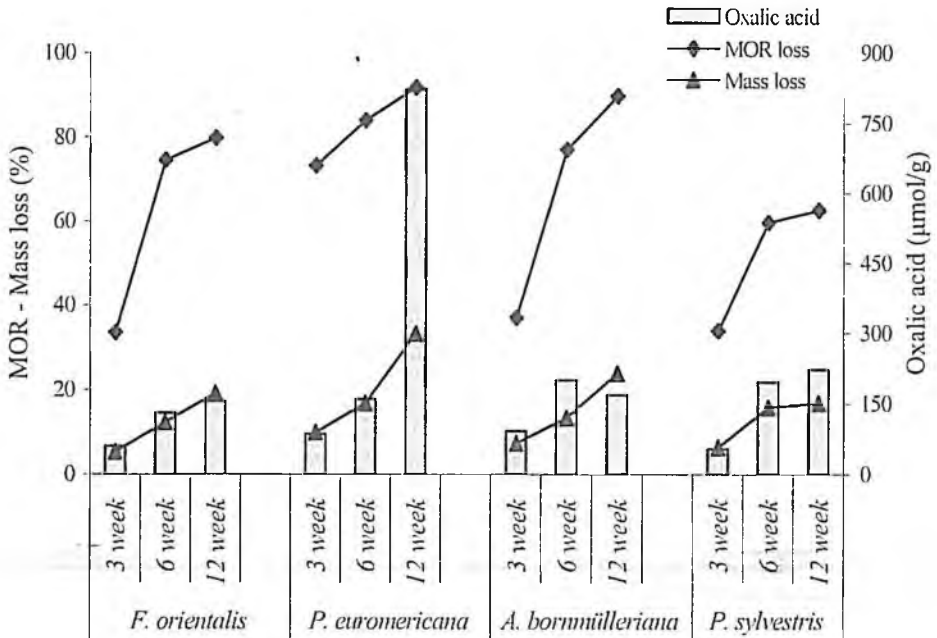


Figure 5. Relationship among MOR, mass loss, and oxalic acid accumulation in the stakes after decay tests.

Şekil 5. Çürüklük testleri sonrasında eğilme direnci örneklerinde direnç kaybı, ağırlık kaybı ve oksalik asit üretimi arasındaki ilişki.

In a study by Clausen and Kartal (2003) MOR showed the most rapid decline of mechanical properties tested in cake pan method using wood stakes. In their study, stakes showed a 6:1 ratio of strength loss to weight loss after 4 weeks incubation and MOR was reduced 19% by the time 3% weight loss had occurred. In a study by Curling et al. (2001), the effect of hemicellulose degradation on strength properties of wood was studied by exposing southern yellow pine stakes to *Gloeophyllum trabeum*. Their results showed a ratio of strength to weight loss of approximately 40:1.

Figures 6, 7 and 8 represent relationships between mass loss and oxalic acid accumulation, MOR and mass loss, and MOR loss and oxalic acid accumulation in the stakes after decay tests. In general good correlations were observed between mass loss and oxalic acid accumulation and MOR and mass losses in the stakes. However weaker correlation was found for MOR loss and oxalic acid production.

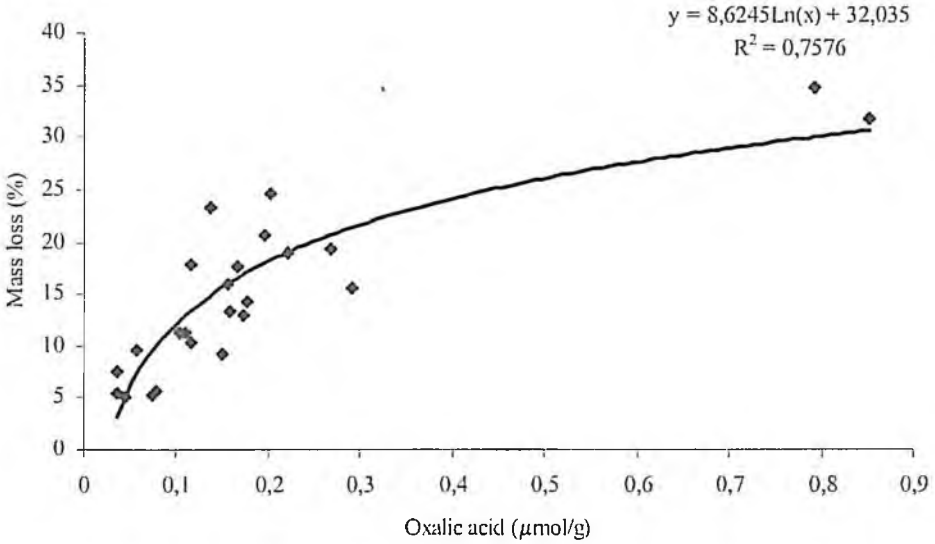


Figure 6. Relationship between mass loss and oxalic acid accumulation in the stakes after decay tests.

Şekil 6. Çürüklük testleri sonrasında eğilme direnci örneklerinde ağırlık kaybı ve oksalik asit üretimi arasındaki ilişki.

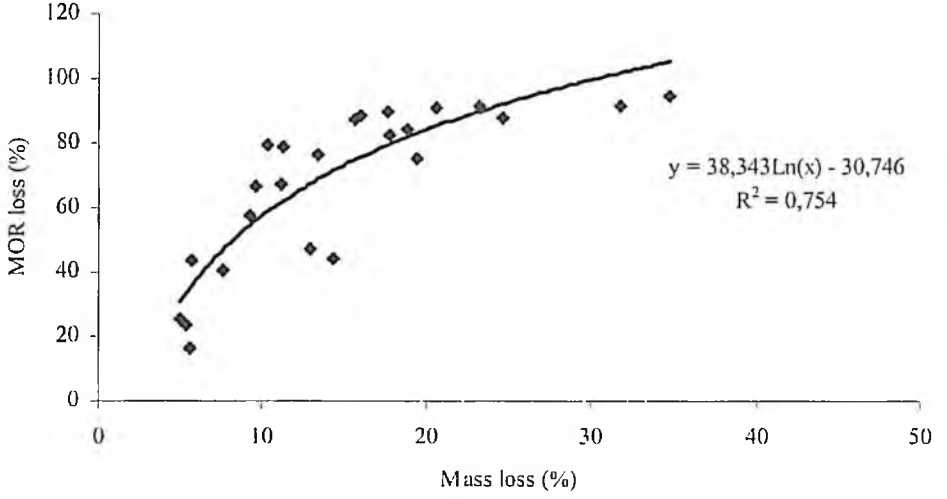


Figure 7. Relationship between MOR and mass loss in the stakes after decay tests.

Şekil 7. Çürüklük testleri sonrasında eğilme direnci örneklerinde direnç ve ağırlık kayıpları arasındaki ilişki.

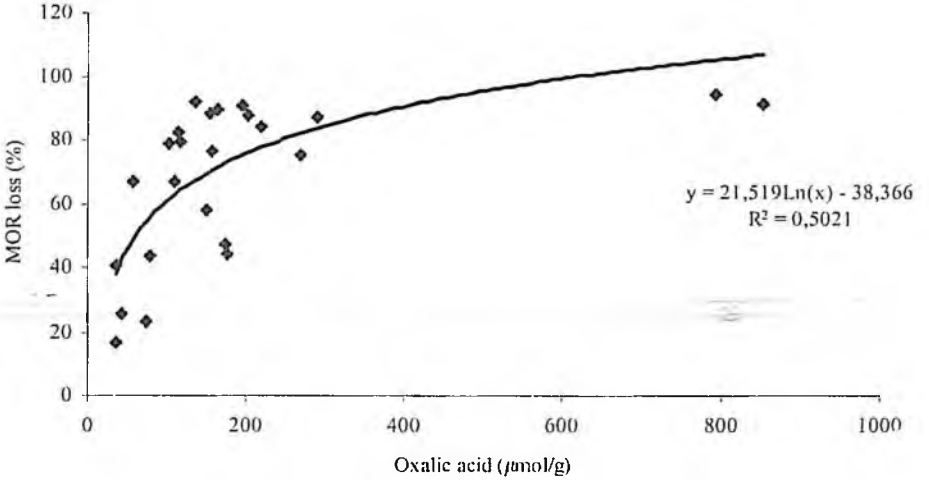


Figure 8. Relationship between MOR and oxalic acid accumulation in the stakes after decay tests.

Şekil 8. Çürüklük testleri sonrasında eğilme direnci örneklerinde direnç kayıpları v. oksalik asit üretimi arasındaki ilişki.

## 5. Conclusions

This study evaluated decay capacity of the dry wood fungus, *S. lacrymans* in various wood species measuring mass losses, OA production and strength losses in wood specimens. In the study, untreated wood specimens were exposed to the fungus in both soil block tests and modified soil bed tests. In the soil block tests, more mass losses in the wood blocks were observed in the stakes subjected to soil bed tests. Amount of OA production by the fungus over the wood blocks was greater than the stakes. MOR losses in the stakes were much more apparent than mass losses to determine initial steps of the decay by the fungus. Further studies are in progress to evaluate copper tolerance of *S. lacrymans* considering OA production and different wood species treated with copper-containing wood preservatives.

## Acknowledgements

This work was supported by Istanbul University Research fund, project number: T-176 / 06032003 and The Scientific and Technical Research Council of Turkey (TUBITAK), Project No: TOGTAG 3395.

# Kuru Çürüklük Mantarı *Serpula lacrymans* Tarafından Odunda Meydana Getirilen Çürüklük, Oksalik Asit Üretimi ve Direnç Kaybının İncelenmesi

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## Özet

Kuru çürüklük mantarı *Serpula lacrymans* (Wulfen:Fr.) Schroeter, dünyanın ılıman bölgelerinde önemli ekonomik kayıplara neden olan odun tahripçisi mantarlardan biri olarak kabul edilmektedir. Bu çalışma, dört farklı ağaç odunlarından 2 farklı boyutta hazırlanan örneklerde uygulanan 2 farklı çürüklük testinde *S. lacrymans*'ın bir izolasyonunun oluşturduğu çürüklük kapasitesini incelemektedir. Çürüklük sürecinde *S. lacrymans* tarafından örneklerde oluşturulan ağırlık kayıplarının yanı sıra, direnç kayıpları ve oluşan oksalik asit miktarı da belirlenmiştir. Modifiye edilmiş toprak yatak (soil bed) denemeleriyle karşılaştırıldığında, toprak blok (soil block) denemelerinde odun örneklerinde daha yüksek ağırlık kayıpları meydana geldiği görülmüştür. Eğilme direnci deneme örneklerinde meydana gelen direnç kayıpları, ağırlık kayıplarına göre çok daha ayırt edicidir. Çürüklük denemelerinde kullanılan örneklerde hem ağırlık kaybı ile oksalik asit üretimi hem de eğilme direnci kaybı ile oksalik asit üretimi arasında iyi bir ilişki olduğu belirlenmiştir. Bakır esash emprenye maddelerine karşı *S. lacrymans* mantarının emprenyeli odun örneklerinde bakır toleransı kapasitesinin belirlenmesine yönelik çalışmalar sürdürülmektedir.

**Anahtar Kelimeler:** *Serpula lacrymans*, oksalik asit, ağırlık kaybı, eğilme direnci kaybı, çürüklük

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# The Need for Performance Criteria in Evaluating the Durability of Wood Products

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## Abstract

Data generated from wood-product durability evaluations can be difficult to interpret. Standard methods used to evaluate the potential long-term durability of wood products often provide little guidance on interpretation of test results. Decisions on acceptable performance for standardization and code compliance are based on the judgment of reviewers or committees. This decision-making process has potential pitfalls, especially when there is pressure to minimize the time needed for evaluation. This paper discusses some of the pitfalls encountered in interpretation of in-ground and above-ground durability test data and suggests areas where more prescriptive performance criteria may be warranted.

**Keywords:** Durability, performance criteria, evaluation methods, wood products

## 1. Introduction

The evolution in durable wood products continues to accelerate. Safety and environmental concerns with traditional and second generation wood preservatives have led to the evaluation of less toxic preservatives and alternatives to preservative treatment. In the United States the effects of withdrawal of chromated copper arsenate (CCA) from most lumber applications in 2004 continues to ripple through the industry. The last few years have seen the introduction and rapid acceptance of a micronized copper formulation of alkaline copper quat (ACQ) as well as the introduction and commercialization of two metal-free organic preservative systems. Numerous other types of preservative formulations are rumored to be on the near horizon. A barrier wrap system has gained acceptance for use in combination with lower preservative retentions,

and there is increasing interest in the use of naturally durable tropical hardwoods. Non-preservative approaches to durability such as thermal treatments and modified wood have been commercialized in European countries, and their potential is being explored in the United States.

This trend of rapid changes in types of preservative treatments appears likely to continue. In the United States one of the drivers in this process has been the formation of the ICC-ES (International Code Council- Evaluation Service (ICC-ES) in 2003. The ICC-ES provides an additional route for proponents of potential new preservative treatments to demonstrate compliance with building codes. Prior to the formation of the ICC-ES, building code acceptance was typically achieved through standardization in the American Wood Protection Association (AWPA, formerly American Wood-Preservers' Association). Although most of the recent activity has targeted residential applications for treated wood, changes in preservative treatments for industrial applications are also a possibility. Creosote, pentachlorophenol and CCA are currently undergoing review by the U.S. EPA, with decisions on future allowable uses expected in 2008. In many European countries the movement away from traditional preservative treatments occurred earlier than in the United States, but change continues in these countries as well. Preservatives that were considered benign a decade ago face increasing scrutiny from regulators and the public.

The rapid evolution of durable wood products has further highlighted an old problem in wood preservation... how do we evaluate long term durability with short term tests? There is no shortage of test methods. Over the last century numerous laboratory and field test methods have been developed to evaluate durability, and many of these methods have gained broad acceptance in Europe, Australia, Asia and the United States. In the United States the AWPA has over 20 preservative evaluation standard methods, and other organizations, such as ASTM International, have applicable methods as well. The AWPA and ICC-ES both provide lists of tests that must be conducted before a durable product can expect to gain acceptance. The ICC-ES typically prescribes that AWPA test methods be used to evaluate a preservative. These methods detail the testing procedures, and in some cases suggest or prescribe the manner of presenting the results. However, the methods generally provide little guidance on how to interpret the results in terms of expected service life or in terms of "pass/fail" criteria. In other words, what is the significance of an average stake rating of 9.0 after 3 years exposure? Does this rating demonstrate efficacy of a preservative in ground-contact? A similar problem in data interpretation exists in European countries, where there is a need for a harmonized system for durability classification (Brischke and Rapp, 2007). Standards used in some countries do provide more guidance than those used in the United States, and some countries have attempted to address this concern by incorporating approval criteria into their testing protocols. For example, acceptance of a candidate preservative may be based on its equivalent performance to reference preservative once specimens treated with the reference preservative have degraded to below 70% mean soundness (AWPC, 1997).

In the United States the interpretation of test results has traditionally been handled through debates and votes within the committee structure of the AWPA. AWPA subcommittees are composed of representatives from industry, academia and government agencies who have some familiarity with conducting and interpreting durability evaluations. The durability results of test products are compared with those of established durable products and non-durable controls. Ultimately, however, the

decision of acceptable performance and standardization remains semi-qualitative. This process has the advantage of flexibility; it allows subcommittee members to consider a wide range of factors that may affect interpretation of test results. However, it is also vulnerable to subjectivity, and potentially the tendency toward a lowering of the bar in judging acceptable performance. Although the process of considering data packets differs somewhat in the ICC-ES, the potential problem of subjective interpretation remains. In this paper we discuss some of the pitfalls encountered in interpretation of durability test data, and suggest areas where more prescriptive performance criteria may be warranted.

## 2. Ground Contact Durability Evaluations

Ground-contact field exposures have been used to evaluate durability for over a century, and stake and post tests continue to be the primary test method for products intended for use in ground contact. However, there are several factors that can interact to affect the results of these tests. Perhaps the most important of these factors are site conditions and duration of the test. It has long been recognized that deterioration is more rapid in warm, moist climates than in cool or dry climates. The AWPAs standards recognize that climate affects the rate of deterioration, stating that while the minimum exposure time is 3 years in high decay hazard areas such as southern Mississippi, longer exposure times are required for lower decay hazard test sites such as Wisconsin. It is left up to the discretion of the subcommittee evaluating the proposal to determine whether the length of the exposure is adequate, but in the past 3 - 5 years of data have generally been considered to be sufficient. However, a comparison of matched sets of stakes (Figure 1), demonstrates that the results derived from northern climates are potentially misleading, even with longer exposures. In this case the test preservative had an average rating of 9 after 5 years of exposure in Wisconsin. Based on these data one might have the impression that the formulation is a promising candidate for protecting wood in ground contact applications. It is apparent from the Mississippi data, however, that this formulation will not adequately protect wood used in the southeastern U.S.

Similar challenges in interpreting data from different sites are encountered in European countries (Edlund et al, 2006; Brischke and Rapp, 2007). The performance of untreated controls does provide some indication of the severity of a test site, but controls may fail so rapidly that the data is difficult to use in developing adjustment factors. It has been proposed that the differences in sites be can be partially accounted for by creating adjustment factors based on the relative performance of reference materials at various sites (Brischke and Rapp, 2007). While this approach would remove some of the subjectivity in determining the required length of exposure, it is not a perfect solution because the effect of test site on preservative performance is a function of the formulation (or type of product) being evaluated. Thus, we cannot always assume that exposure for a certain number of years in a moderately severe site is equivalent to exposure for a certain number of years in a more severe location. Perhaps the most practical solution is to require data from at least one test site that has demonstrated a severe deterioration hazard.

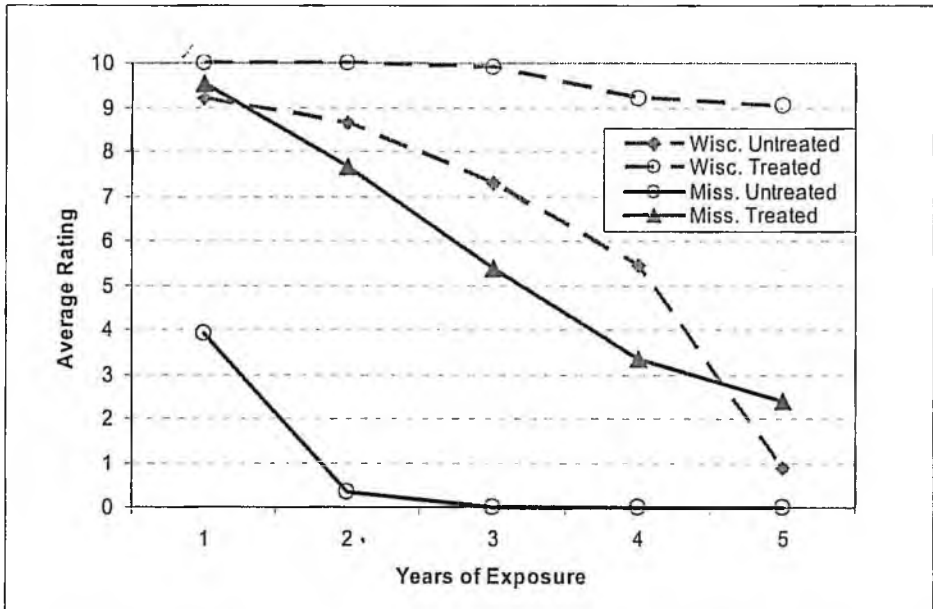


Figure 1. Example of difference in ratings obtained for stakes exposed in Wisconsin (moderate hazard) and Mississippi (severe hazard).

Length of exposure is also a concern even within high decay hazard areas. As mentioned above, a minimum of three years is specified in AWPA guidelines for preservative evaluation. However, it is far from clear that three years is sufficient. For example, consider the ratings of stakes in one of the USDA, Forest Products Laboratory's plots in southern Mississippi. This plot contains over 100 treatment groups (preservative/wood species/retention combinations), each of which was replicated with 20 stakes (19 by 19 by 457 mm). In Figure 2 we graphically compare how well the average stake ratings at three years correspond to their ratings after 11 years of exposure (the most recent rating of this plot). Treatment groups with an average rating of less than 9.4 after three years all performed poorly, with average ratings falling below 3 after 11 years. Treatment groups with an average rating of 10.0 (all 20 stakes rated as perfect) after three years fared better, but only half of these groups appeared to perform as well as the reference preservative (ground-contact retention of chromated copper arsenate, CCA Type C) after 11 years. And, 4 of the treatment groups with an average rating of 10.0 after 3 years had declined to an average rating of 7 or below after 11 years. Thus, perfect ratings or equivalent performance to CCA after three years does not provide a high degree of confidence that a test system will be performing similarly to the reference preservative over the long term. Increasing the duration of the test to 5 years improved the prediction of performance at 11 years. Eleven of the 18 groups performing as well as CCA after 5 years (average rating of 9.85 or above) continued to perform at least as well as CCA after 11 years. However, ratings of 2 of these groups did drop

substantially between 5 and 11 years. None of the 15 treatment groups with average ratings between 9.0 and 9.8 after 5 years were performing as well as CCA after 11 years, and average ratings of 6 of these groups dropped to 7 or below. This indicates that even small differences in average ratings in the 9 – 10 range are important in predicting future performance. It is worthwhile noting that all of the systems evaluated in this plot are considered to be relatively resistant to leaching. These data indicate that when evaluating preservatives intended for use in ground contact in high hazard areas, a minimum of at least 5 years of exposure data is needed, and that the average rating of the test preservative should be at least as high as that of the reference preservative. Even slight evidence of vulnerability after 5 years appears to be a strong indicator of poor future performance. Studies are needed to explore these temporal relationships in greater detail while accounting for the possible underlying dependencies.

The European Standard EN 252 for ground contact exposure does require a minimum of 5 years of testing before results can be interpreted (CEN, 1998). However, similar concerns have been expressed about the use of 5 year data from Nordic test plots to predict long term performance (Edlund et al., 2006). Edlund et al compared the average ratings of over 700 treatment groups (approximately 10,000 total stakes) at 5 and 10 years to their median life and concluded that even treatment groups with no signs of decay after 5 or even 10 years may have a relatively short median life (Edlund et al., 2006). One treatment group with no evidence of decay after 10 years had a median life of only 14 years.

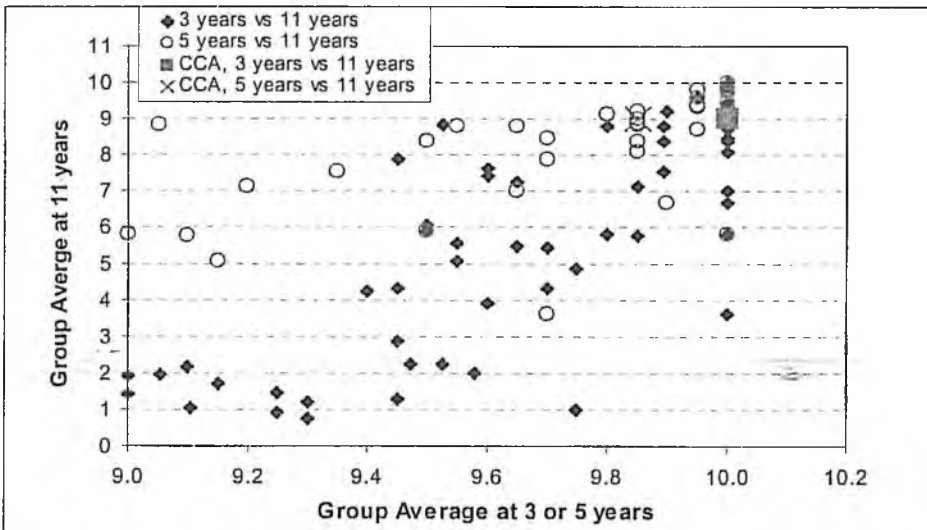


Figure 2. Comparison of average ratings at 3 years or 5 years to average ratings after 11 years. Each point represents the average of 20 replicates.

### 3. Above-Ground Durability Evaluations

Evaluation of preservatives intended for wood used above-ground has proven even more difficult than ground contact evaluations. Although it is recognized that the decay environment presented by stake tests is very severe for products intended for use above-ground, the selection of an appropriate above-ground test method has been problematic. The greatest source of difficulty appears to be the wide variations in severity of exposure for wood used above-ground. The severity of above ground exposure does vary with climate (Lebow and Highley, 2008; Rapp et al., 2006; Zahora, 2002), but it also varies greatly with construction practices and localized site conditions that influence moisture, temperature and UV exposure. In areas where organic debris can collect in connections, the above-ground decay hazard can be high (Figure 3).

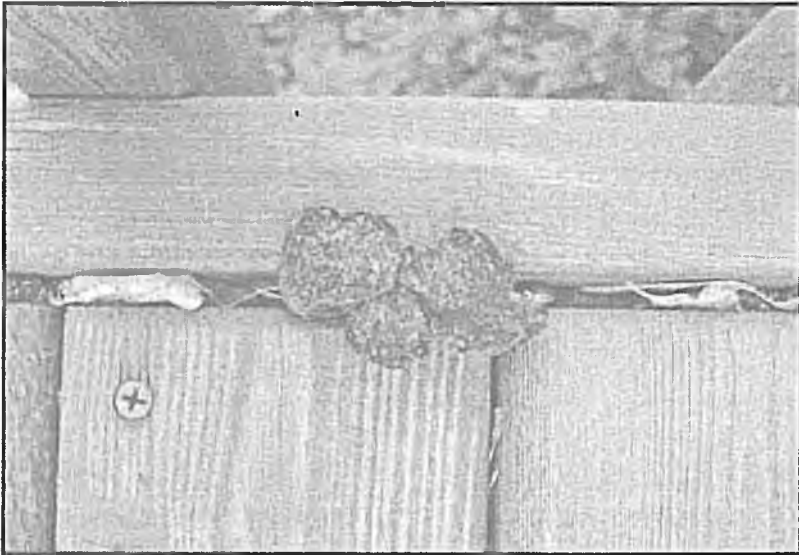


Figure 3. Accumulated organic debris helped to promote decay in the end-grain of this cedar decking.

A wide range of test methods has been used to evaluate above-ground decay (Blom and Bergstrom, 2006; Clausen et al., 2006; De Groot and Highley, 1995; Highley, 1995; Lindegaard and Morsing, 2003; Van Acker and Stevens, 2003; Zahora, 2002). Substantial research on above-ground evaluations continues to take place in Europe, where the transition to use of "above-ground only" preservatives has preceded that in the United States. Despite extensive research however, it remains unclear how well above-ground tests characterize the hazard, or if they actually accelerate the rate of decay relative to in-service applications. Much of the difficulty is derived from creating test arrangements that simulate the moisture-trapping conditions present in actual structures. Most methods utilize some type of joint, connection or layering in an effort

to trap moisture (Figure 4), but this effect can be undermined by the use of specimens with small dimensions. Although the smaller dimensions do allow more rapid detection of decay once it is present, smaller specimens dry more rapidly than dimension lumber. Smaller specimens also may be less susceptible to the formation of the checks that allow penetration and trapping of moisture in larger material (De Groot and Highley, 1995). Thus, although we associate the use of small specimens with accelerated testing for wood placed in ground contact, this assumption may be misleading for above-ground evaluations. Some studies suggest that common test arrangements may actually slow the time needed for decay to develop. In a comparison of tests units of untreated southern pine sapwood exposed above-ground in southern Mississippi, the most rapid visually evident failure (6 years) was achieved by simply using 102 mm thick planks (De Groot and Highley 1995; Eslyn et al., 1985) (Figure 5). An earlier study reported that initial decay was not observed in untreated pine cross-brace units (20 x 75 x 15 mm) until after 6 years of exposure in southern Mississippi (Eslyn et al., 1985). In contrast, the authors of this report often observe fruiting bodies of the brown rot fungus *Gloeophyllum sepiarium* after only three years of exposure of southern pine decking specimens (38 by 140 x 914 mm) in the lower decay hazard climate of southern Wisconsin. It is worth noting that visual evidence of decay is often a delayed indicator of fungal colonization. It is possible to culture the white-rot fungus *Irpelex lacteus* from both lap joints and L-joints after as little as 4 months of exposure in Wisconsin. In addition to the effects of specimen dimensions, none of the commonly used test methods simulate the accumulation of decaying organic debris that often occurs in connections of treated wood used above-ground. Specimens are typically exposed in open areas to remove variability associated with natural shading, and when organic debris does accumulate it is removed during periodic inspections. The role of shading in promoting above-ground decay was reported by Augusta and Rapp (2003) and Rapp et al. (2006), who attributed the effect to the increased wood moisture content.

Above-ground evaluations are further complicated when the effects of wood species and composite products are considered. Although wood species do have some affect on ground-contact durability, the relative species effect becomes much greater in the slower deterioration that occurs above-ground. In evaluations of sapwood much of the species effects may be attributed to permeability, or resistance to moisture absorption. Several studies have reported substantial differences in wood moisture content for wood species exposed under identical test conditions, (Blom and Bergstrom, 2006; Lindegaard and Morsing, 2003; Miltz et al., 1998). Because moisture content is the primary limiting factor in above-ground decay, even small differences in moisture content can affect durability evaluations. Moisture absorption and retention also differ greatly for composite products. The quantity and properties of the adhesive, and the shape and orientation of the furnish greatly influence moisture absorption. In some cases initial moisture absorption is low, but increases over time as irreversible swelling occurs (Laks and Larkin, 2007). The wood fibers within wood-plastic composites products are particularly slow to acquire moisture, but fibers near the surface eventually do gain and maintain sufficient moisture to sustain decay (Clemons and Ibach, 2004; Wang and Morrell, 2004). Specialized test methods may be required to evaluate durability in these products.

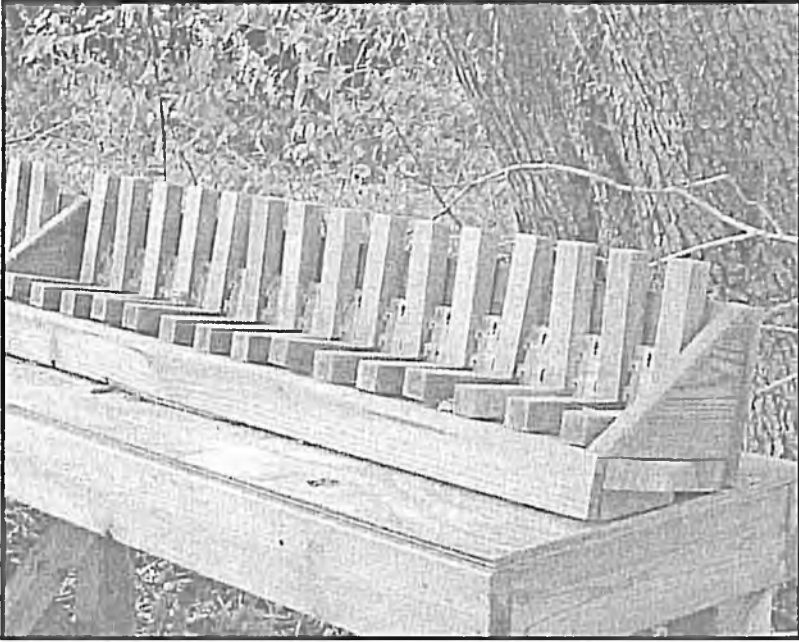


Figure 4. L-joint specimens are configured to trap moisture with the intent of accelerating fungal colonization.

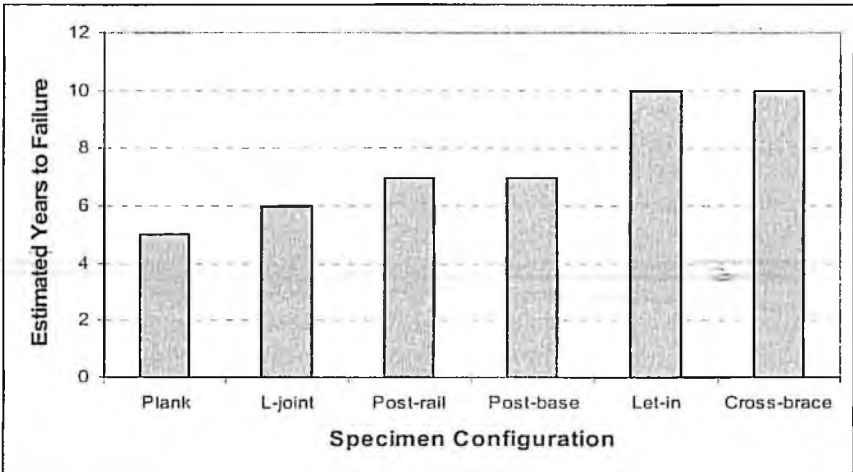


Figure 5. Estimated years to failure for various specimen configurations of untreated southern pine sapwood exposed above ground in a high decay hazard climate.



In the United States the AWPAs currently lists three standard field test methods for evaluating above-ground decay: the L-joint method (Standard E9), lap-joint method (Standard E16) and the ground-proximity method (Standard E18). The lap joint method attempts to address the shortcomings of small specimen dimensions and debris accumulation by providing a larger joint area that is conducive to moisture development. The ground-proximity test is a newer method that is intended to provide a more severe exposure because the specimens are placed directly on cinder blocks and covered with shade cloth (Zahora, 2002). However, it uses small (19 by 50 by 125 mm) specimens without any overlap or joint areas, and its relative severity appears to vary with location. A fourth method, utilizing decking specimens, is in the final stages of the standardization process. Under current AWPAs guidelines any of these standardized methods can be used to evaluate above-ground durability for any intended above-ground use.

The European standards also incorporate two above-ground testing methods. Standard ENV 12037 is a lap-joint method for general evaluation of wood to be used above-ground, while Standard EN330 is an L-joint method primarily intended for evaluation of wood that will be coated in service (CEN, 1993; CEN, 1996). Standard ENV 12037 does provide guidance, on test duration, requiring that the test be continued until the untreated controls reach a mean rating of 3 (severe decay). Standard EN 330 recommends that the test be continued for a minimum of 5 years, and preferably until failure.

Both the United States and European methods do point out that meaningful results are not obtained until the untreated specimens reach a certain level of deterioration, but AWPAs guidelines for preservative evaluation also state that a minimum of only three years of data may be needed in high hazard climates. This relatively short test duration may be based on the optimistic assumption that the above-ground test arrangements provide for accelerated testing. The standard methods do not provide criteria for ratings that would be considered acceptable or "passing" for the preservative-treated specimens, and given our uncertainty about the relationship between the results of these tests and in-service performance such criteria may be difficult to develop. It is also worth noting that although we may associate above-ground treatments with decking, the same use category also applies to structurally critical support members used above-ground. Given the ramifications of failure in some of these members (e.g., second story balcony supports) some consideration should be given to providing more conservative durability estimates. There may be value in returning to the use of stake tests to provide truly accelerated evaluations for preservatives intended for use above-ground.

#### 4. Reporting Average Ratings

It has become common practice to report only average ratings in preservative evaluation data packets submitted to AWPAs, and we have routinely referred to average ratings in this paper as well. While averages are perhaps the single most descriptive statistic, they do not always accurately characterize the performance of a preservative system (De Groot and Evans, 1998, 1999; Link and De Groot, 1989). Variability in

performance and the occurrence of early failures can provide important information in evaluating a preservative. Figure 6 provides an example of how average ratings may not fully capture the performance of copper based preservatives. In this case 20 replicates were used, and the treatment group had respectable average ratings of 8.9 after four years and 7.7 after 5 years of exposure in a severe decay environment. However, within 3 years 2 of the stakes had failed completely, and after 5 years a total of 4 of the 20 stakes had failed. All of the remaining stakes had ratings of either 9 or 10, with 10 remaining the most common (mode) rating. This pattern of high ratings for most stakes and complete failures of others is frequently observed in tests of copper-based preservatives in plots where copper tolerant fungi are present. Likewise, termite distribution is inherently non-uniform within a test plot and this may also affect the performance of individual stakes. In other situations, such as with some naturally durable species, there may be a wide distribution of durability ratings between specimens that is not adequately characterized by simply presenting the average rating. Link and De Groot (1989) discuss the problem of relying too heavily on average ratings, and suggest the use of box plots to characterize the "time to failure" for stakes within each treatment group. Prior to 2003, the AWPAs standards did include a method for more detailed data analysis and presentation. However, this "Standard Procedure for the Calculation of the Performance Index of Preservatives in Stakes and Posts" was mathematically complex and the standard was eventually removed for lack of use. For key durability tests it may be worthwhile to again provide additional guidance on the type of data presented in summarizing the performance of a candidate preservative system. This guidance could be as simple as prescribing the type of data presented (i.e. box plots or number of stakes in each rating category) or a more thorough statistical analysis.

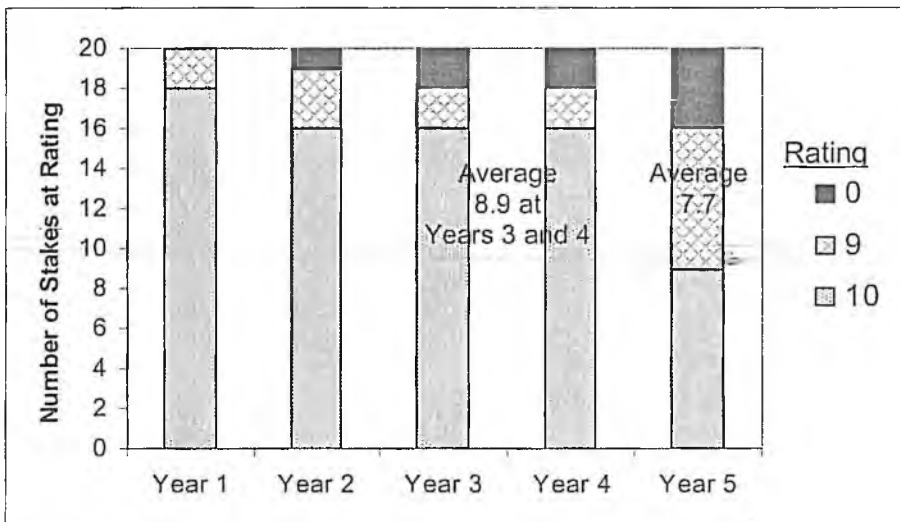


Figure 6. Example of average ratings masking the occurrence of early failures.

## 5. Conclusions and Recommendations

The increasing pressure to rapidly evaluate and commercialize durable wood products is challenging our ability to interpret the results of short-term durability tests. The current process of subjectively interpreting data packets is vulnerable to the pressure for rapid commercialization, and there is potential for a loss of conservatism in our performance criteria. In the United States data packets may be reviewed by organizations whose members are not familiar with the intricacies of wood product durability evaluations.

For evaluation of products intended for use in contact with the ground, it appears that even extended durability evaluations conducted in less severe (northern) climates may not be adequate for estimation of durability in more severe climates. A practical solution would be to require data from at least one test site that has demonstrated a severe deterioration hazard. Even in severe decay hazard climates, excellent performance of stakes after only three years is not a reliable indicator of long term durability. Basing test duration or performance criteria on the durability of untreated controls also does not appear to be sufficient for ground contact evaluations. The approach used in Australia, where test duration is based on the performance of low concentrations of an established reference preservative, does appear to have some merit.

Our current methods of assessing above-ground durability may not accelerate decay in comparison to some conditions encountered for durable wood products in service, suggesting that much longer evaluation periods or more severe tests should be considered. Alternatively, above-ground uses could be further divided, with more stringent test methods utilized for products intended as above-ground structural supports. Ground-contact testing of products used in structurally critical above-ground members may be necessary until appropriate above-ground test methods are developed.

Interpretation of test data also remains problematic. A return to more prescriptive data presentation may be warranted, as average ratings do not always adequately characterize the performance of a durable product. In addition, methods should provide more specific guidance on the distribution of ratings that is considered to represent adequate performance.

## Acknowledgement

The authors gratefully acknowledge the assistance of Apolonia Bocanegra in preparing data used in this publication.

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# The Prediction of Mechanical Properties of Wood-Based Composites with Vibration NDE Method

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## Abstract

In this study, dynamic modulus of elasticity ( $MOE_d$ ) of plywood, particleboard and medium density fiberboard (MDF) were determined by using a Fast Fourier Transform (FFT) analyzer and compared to their mechanical properties obtained from the same specimens. It was revealed that strong correlations between  $MOE_d$  and static bending properties ( $MOE_s$ , MOR) were found in both longitudinal and bending vibration for particleboard. There were also good correlations between  $MOE_d$  and bending properties of MDF. The  $MOE_d$  in longitudinal and bending vibration is acceptable to predict the bending properties of plywood in parallel direction to long axis. However poor correlations were found between  $MOE_d$  and IB in particleboard and MDF.

**Keywords:** NDE, vibration frequency, dynamic MOE, strength properties, wood-based composites.

## 1. Introduction

The physical and mechanical properties of wood-based composite products have wide variations and many factors can contribute to those such as; raw material parameters, species, resin types, manufacturing parameters including pressure, press time, temperature, machines and equipments. On the other hand, quality requirements for wood based panels have to be fulfilled as specified in standards and by customers.

Systems of quality control in the wood based panel industry are using different methods to ensure continuous production of guaranteed quality products. In order to

evaluate the quality of wood composites, generally, full-sized panels are selected randomly from production line and small diameter specimens sawn from these panels are tested destructively. The mechanical properties of selected panels can be determined conclusively by destructive test methods. However, by the effect of numerous uncontrolled variables those values may not reflect the other panels properties, manufactured under the same conditions. The Turkish Standard, TS EN 326-2 (2006) allows alternative techniques instead of standardized tests for quality control of wood based panels if a significant correlation between measurement and standard test can be proven. Therefore, quick and accurate non-destructive evaluation (NDE) methods seem as an effective alternative to predict mechanical properties of panel products and to ensure quality requirements.

NDE have been used for many years to evaluate properties of wood products. Among the different techniques that have been used for predicting the quality of wood and wood based composite materials, one of the most commonly used methods is the stress wave NDE. It uses low stress molecular motions to measure two fundamental material properties: energy storage and dissipation. Energy storage is manifested as the speed at which a wave travels in a material. In contrast, the rate at which a wave attenuates is an indication of energy dissipation. Jayne argued that energy storage and dissipation properties are controlled by the same mechanisms that determine a material's mechanical behavior. Thus, useful mathematical relationships between stress wave and static mechanical properties should be attainable through statistical regression analysis techniques (Ross and Pellerin, 1988).

In past several years, a number of research were conducted by using NDE methods including stress wave technique in order to predict the quality of wood based composite. These studies focused on relationships between wave velocity - dynamic modulus of elasticity and static elasticity - strength properties. However, some reserchers found useful correlations among these parameters. Pellerin and Morschauer (1974) reported a good correlation between square of longitudinal wave velocity and static MOE with  $r$ -values of 0.93-0.95 and MOR with  $r$ -values of 0.87-0.93. Another research about the using of stress wave technique in wood-based composites also showed that there were strong correlations between square of longitudinal wave velocity and MOE in tensile with an  $r$ -value of 0.98, ultimate tensile strength with an  $r$ -value of 0.91, flexural MOE with an  $r$ -value of 0.97, and MOR with an  $r$ -value of 0.93. In addition, there were good correlations between dynamic MOE and static MOE in tension with an  $r$ -value of 0.98, ultimate tensile strength with an  $r$ -value of 0.93, flexural MOE with an  $r$ -value of 0.96, and MOR with an  $r$ -value of 0.92 (Ross and Pellerin, 1988).

In brief, the wave velocity and attenuation could be used successfully to predict the static tensile and flexural properties of wood based composite materials. Ross and Pellerin (1988) predicted a useful relationship between stress wave velocity and attenuation and internal bond (IB). Similarly, Vogt (1985) studied on MDF and it was found a good relationship between square of wave velocity and static MOE in tension with an  $r$ -value of 0.90, static MOE in bending with an  $r$ -value of 0.76, ultimate tensile strength with an  $r$ -value of 0.81, and MOR with an  $r$ -value of 0.96. Furthermore good correlations were achieved between dynamic MOE and static MOE in tensile with an  $r$ -value of 0.88, static MOE in bending with an  $r$ -value of 0.72, ultimate tensile strength



with an  $r$ -value of 0.88, and MOR with an  $r$ -value of 0.92. It was also investigated the possibilities of using stress wave to predict internal bond strength of particleboard and structural panel products by Vogt (1986). It was reported that there were relationships between the square of wave velocity and internal bond with a correlation coefficient of 0.70-0.72 and between dynamic MOE and internal bond with a correlation coefficient of 0.80-0.99. Han et al. (2006) found that both MOE and MOR of wood based panels (plywood, oriented strandboard and particleboard) with related to moisture effect and they at different moisture contents can be estimated by observing the longitudinal wave velocity. Sotomayor (2003) found a strong relationship between static MOE and dynamic MOE based on stress wave velocity.

Some other NDE methods except stress wave technique have been also employed to estimate mechanical properties of wood-based panels. Vibration technique and ultrasound velocity, parallel and perpendicular to panel surface have been used for this purpose and good correlations were found (Bektha et al., 2000; Dunlop, 1980; Kruse, 2000; Schweitzer and Niemz, 1990; Sotomayor, 2003; Sun and Arima, 1999; Vun et al., 2000; Yang et al., 2005).

The objective of this study is to evaluate stress wave velocity and dynamic MOE of the wood based composites such as plywood, particleboard, and medium density fiberboard determined by using of longitudinal and bending vibration frequency measured by Fast Fourier Transform (FFT) analyzer and investigated the possibilities of predicting the static mechanical properties of wood based panels.

## 2. Material and Methods

Commercially manufactured 3 particleboards with a dimension of 18×2100×2800 mm, 3 fiberboards (MDF) with a dimension of 18×2100×2800 mm and 1 plywood with a dimension of 20×2100×2800 mm were obtained randomly from several manufacturers. The particleboards and fiberboards were manufactured from mixed species both softwood and hardwoods using urea-formaldehyde resin and the plywoods were manufactured from okoume (*Aucoumea klaineana* Pierre) veneer using phenol-formaldehyde resin. Thirty-two (16 parallel and 16 perpendicular to the sending direction) 50×500 mm specimens were cut from each particleboard and MDF panel and forty 50×500 mm specimens were cut from plywood along two grain orientation (20 parallel and 20 perpendicular direction). Totally, 40 specimens of plywood, 96 specimens of particleboard, and 96 specimens of MDF were conditioned in a room with a relative humidity of 65% ±5 and a temperature of 20 ±2°C.

In order to determine  $MOE_d$ , vibration frequencies in longitudinal and bending vibration were used. A Fast Fourier Transform (FFT) analyzer calculated the frequency of the longitudinal or bending resonance induced by hitting the end of the specimen (for longitudinal vibration) or the center of the specimen (for bending vibration), which was supported by a porous and an elastic material. The experimental setup is shown in Figure 1.

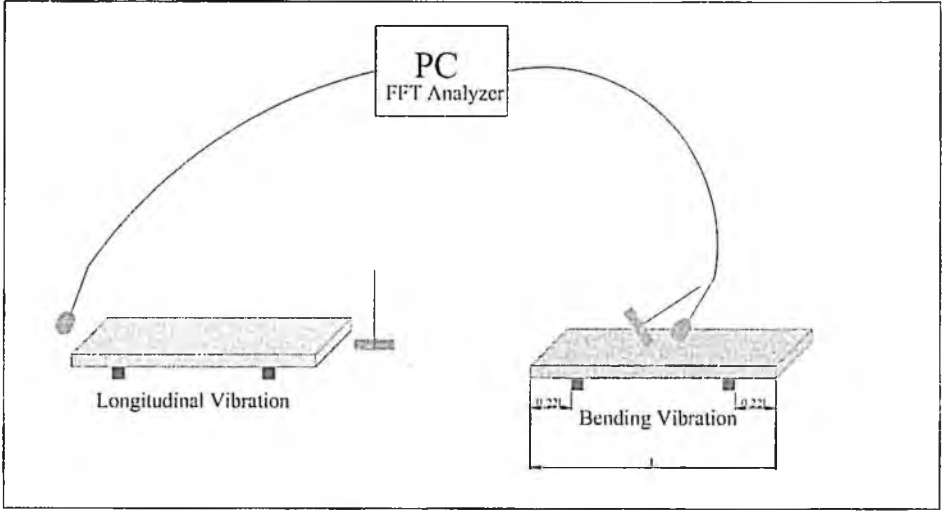


Figure 1. The experimental setup of NDE.

Şekil 1. Tahribatsız değerlendirme deney düzeneği.

The wave velocity in longitudinal vibration was determined by using of the following equation (Hearmon, 1966):

$$V = 2fl \quad (1)$$

where  $V$  is the velocity of the longitudinal stress wave,  $l$  is the length of the specimen and  $f$  is the frequency of the first longitudinal resonance mode. The dynamic modulus of elasticity in longitudinal vibration ( $MOE_{dl}$ ) was calculated by using the longitudinal velocity ( $V$ ) and mass density of the specimen ( $\rho$ ) by the following equation (Hearmon, 1966):

$$MOE_{dl} = \rho V^2 \quad (2)$$

The dynamic modulus of elasticity in free bending vibration ( $MOE_{db}$ ) was also determined by using of the Euler beam theory described by Timoshenko (1954):

$$MOE_{db} = \left( \frac{f}{\gamma} \right)^2 \frac{mL^3}{I} \quad (3)$$

where  $f$  is the frequency of bending vibration in mode no. 1,  $m$  is the mass of specimen,  $L$  is the length of specimen,  $I$  is the moment of inertia and  $\gamma$  is the 3.561 for free support condition mode no. 1 (Timoshenko, 1954). No shear correction was made because the effect of shear was negligible if the span to thickness ratio of the specimen was above 15 (Divos et al., 1994).

After completing the determination of the NDE parameters, the specimens for the static tests were prepared from each NDE specimens. Static modulus of elasticity

( $MOE_s$ ) and modulus of rupture (MOR) were conducted according to EN 310 (1993) in particleboard, MDF and plywood. The internal bond strength was also conducted according to EN 319 (1993) in particleboard and MDF. Totally, 40 specimens of plywood, 96 specimens of particleboard, and 96 specimens of MDF were tested for  $MOE_s$ , MOR and IB.

A simple linear regression analysis ( $y = ax + b$ ) was performed to establish relationships between  $MOE_d$  and static stiffness and strength properties ( $MOE_s$ , MOR and IB) at a confidence level of 0.95. The correlation coefficient ( $r$ ) and the standard error of regression ( $S_{y,x}$ ) were also calculated to evaluate the benefits of the NDE technique. As stated in the standards, the static test results of particleboard and MDF obtained from two directions that differs based on the manufacturing direction were evaluated together and the static test results of plywood were evaluated separately in two directions.

### 3. Results and Discussion

The results of linear regression analysis were summarized in Table 1 for particleboard, in Table 2 for MDF and in Table 3 for plywood. As shown in Tables 1-3, the strongest correlations between  $MOE_d$  that is determined nondestructively by using of both longitudinal and bending vibration frequency and bending properties ( $MOE_s$  and MOR) were obtained from particleboards.

The correlation coefficients calculated for particleboard indicated that there were strong correlations between  $MOE_d$  and  $MOE_s$  (Figure 2) and MOR (Figure 3) while it was found that a poor relationship between  $MOE_d$  and IB (Figure 4) in both longitudinal and bending vibration existed. The  $MOE_d$ - $MOE_s$  correlation was stronger than  $MOE_d$ -MOR correlation in either longitudinal or bending vibration. In addition, the dynamic MOE determined by using of bending vibration presented slightly a better correlation than the longitudinal vibration. Consequently, the dynamic MOE determined by both longitudinal and bending frequency can be used successfully to predict the bending properties (static MOE and MOR) of particleboards. However, they are poor predictors of IB.

Table 1. The results of linear regression analysis for particleboard.  
Tablo 1. Yongalevhalar için doğrusal regresyon analizi sonuçları.

	a	b	R <sup>1</sup>	S <sub>yx</sub> <sup>2</sup>
Dynamic modulus of elasticity based on longitudinal vibration Boyuna vibrasyonda dinamik elastikiyet modülü				
Flexural modulus of elasticity Eğilmede elastikiyet modülü	1.12 (1.022, 1.218) <sup>3</sup>	-455.9 (-740.6, -171.2)	0.92	76,68
Modulus of rupture Eğilme direnci	0.07 (0.065, 0.088)	-53.71 (-86.75, -20.68)	0.81	8.89
Internal Bond Yapışma direnci	1.05×10 <sup>-4</sup> (-6.21×10 <sup>-6</sup> , 2.16×10 <sup>-4</sup> )	0.1762 (-0.14, 0.49)	0.19	0.08
Dynamic modulus of elasticity based on bending vibration Eğilme vibrasyonda dinamik elastikiyet modülü				
Flexural modulus of elasticity Eğilmede elastikiyet modülü	0.81 (0.74, 0.88)	-288.6 (-565.6, -11.61)	0.92	78.33
Modulus of rupture Eğilme direnci	0.06 (0.05, 0.06)	-51.42 (-80.38, -22.46)	0.84	8.19
Internal Bond Yapışma direnci	7.21×10 <sup>-5</sup> (-9.15×10 <sup>-6</sup> , 1.53×10 <sup>-4</sup> )	0.2075 (-0.10, 0.51)	0.18	0.08

<sup>1</sup> Correlation coefficient.

<sup>2</sup> Standard error of the regression.

<sup>3</sup> The values in parentheses are confidence bounds (p=0.05)

Table 2. The results of linear regression analysis for MDF.  
Tablo 2. MDF için doğrusal regresyon analizi sonuçları.

	a	b	R <sup>1</sup>	S <sub>yx</sub> <sup>2</sup>
Dynamic modulus of elasticity based on longitudinal vibration Boyuna vibrasyonda dinamik elastikiyet modülü				
Flexural modulus of elasticity Eğilmede elastikiyet modülü	1.29 (1.11, 1.47) <sup>3</sup>	-818.15 (-1387, -249.4)	0.83	101.38
Modulus of rupture Eğilme direnci	0.17 (0.15, 0.19)	-207.21 (-282.2, -132.5)	0.83	13.34
Internal Bond Yapışma direnci	0.1×10 <sup>-3</sup> (-1.5×10 <sup>-4</sup> , 3.9×10 <sup>-4</sup> )	0.26 (-0.59, 1.10)	0.09	0.15
Dynamic modulus of elasticity based on bending vibration Eğilme vibrasyonda dinamik elastikiyet modülü				
Flexural modulus of elasticity Eğilmede elastikiyet modülü	0.76 (6.68×10 <sup>-2</sup> , 8.60×10 <sup>-2</sup> )	190.77 (-197.1, 578.7)	0.85	94.42
Modulus of rupture Eğilme direnci	0.10 (8.75×10 <sup>-2</sup> , 1.13×10 <sup>-1</sup> )	-66.03 (-119.1, -12.99)	0.84	12.91
Internal Bond Yapışma direnci	1.6×10 <sup>-5</sup> (-1.3×10 <sup>-4</sup> , 1.7×10 <sup>-4</sup> )	0.5761 (-4.83×10 <sup>-2</sup> , 1.2)	0.02	0.15

<sup>1</sup> Correlation coefficient.

<sup>2</sup> Standard error of the regression.

<sup>3</sup> The values in parentheses are confidence bounds (p=0.05)

Table 3. The results of linear regression analysis for plywood.  
Tablo 3. Kontrplaklar için doğrusal regresyon analizi sonuçları.

	a		b		R <sup>1</sup>		S <sub>yx</sub> <sup>2</sup>	
	Par. <sup>3</sup>	Perp. <sup>4</sup>	Par.	Perp.	Par.	Perp.	Par.	Perp.
Dynamic modulus of elasticity based on longitudinal vibration Boyuna vibrasyonda dinamik elastikiyet modülü								
Flexural modulus of elasticity Eğilmede elastikiyet modülü	0.74 (0.416, 1.062) <sup>5</sup>	0.54 (0.25, 0.83)	-87.12 (-1664, 1490)	209.92 (-1221, 1641)	0.75	0.68	76.01	257.73
Modulus of rupture Eğilme direnci	0.014 (8.6×10 <sup>-3</sup> , 2.04×10 <sup>-2</sup> )	5.1×10 <sup>-3</sup> (2.1×10 <sup>-3</sup> , 8.0×10 <sup>-3</sup> )	-40.99 (-69.86, -12.13)	16.66 (2.07, 31.24)	0.77	0.64	1.39	2.63
Dynamic modulus of elasticity based on bending vibration Eğilme vibrasyonda dinamik elastikiyet modülü								
Flexural modulus of elasticity Eğilmede elastikiyet modülü	0.65 (0.50, 0.81)	0.56 (0.28, 0.83)	768.74 (122.1, 1415)	164.63 (-1157, 1487)	0.90	0.71	49.25	246.09
Modulus of rupture Eğilme direnci	0.009 (4.5×10 <sup>-3</sup> , 1.44×10 <sup>-2</sup> )	0.008 (3.8×10 <sup>-3</sup> , 0.012)	-10.05 (-30.95, 10.85)	3.415 (-16, 22.83)	0.68	0.69	1.59	3.61

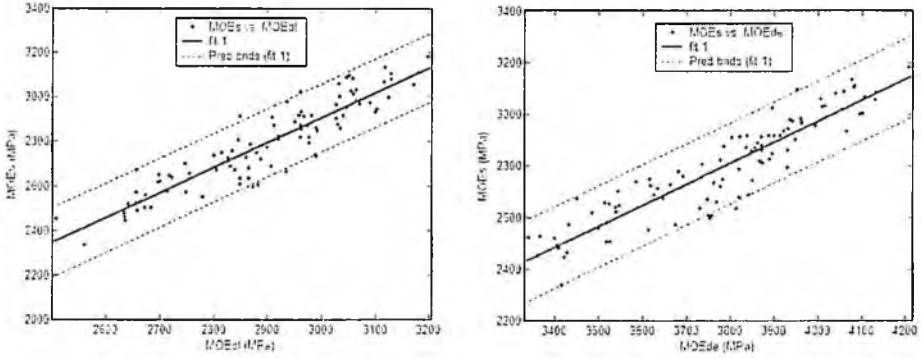
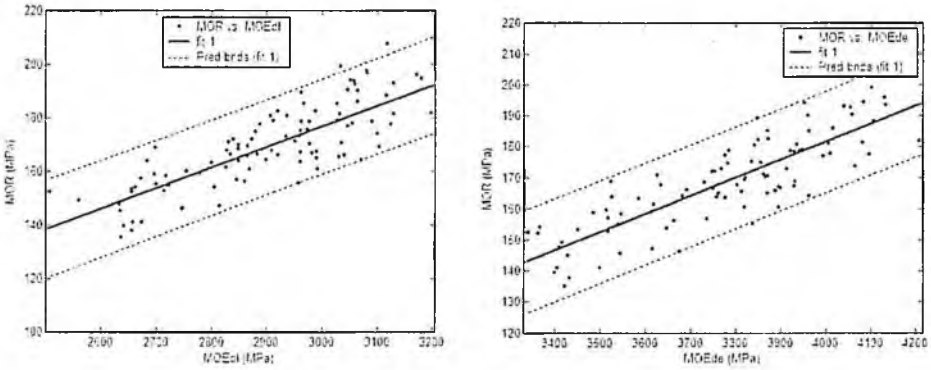
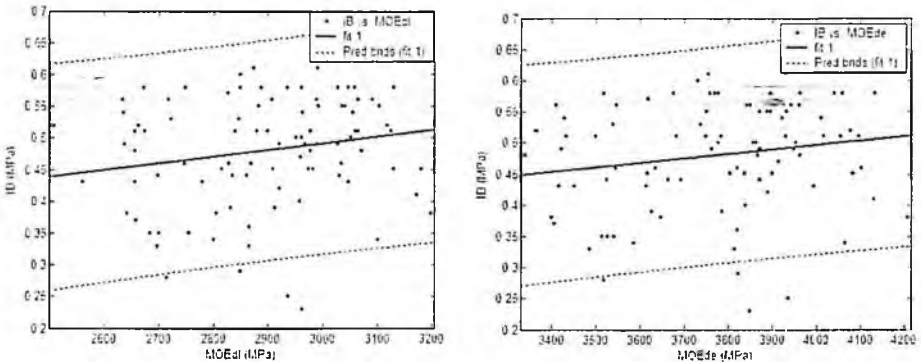
<sup>1</sup> Correlation coefficient.

<sup>2</sup> Standard error of regression.

<sup>3</sup> The grain direction of surface veneer is parallel to long axis.

<sup>4</sup> The grain direction of surface veneer is perpendicular to long axis.

<sup>5</sup> The values in parentheses are confidence bounds p=0.05).

Figure 2. The relationship between MOE<sub>d</sub> and MOE<sub>s</sub> in particleboard.Şekil 2. Yongalevhalarda MOE<sub>d</sub> ve MOE<sub>s</sub> arasındaki ilişki.Figure 3. The relationship between MOE<sub>d</sub> and MOR in particleboard.Şekil 3. Yongalevhalarda MOE<sub>d</sub> ve MOR arasındaki ilişki.Figure 4. The relationship between MOE<sub>d</sub> and IB in particleboard.Şekil 4. Yongalevhalarda MOE<sub>d</sub> ve IB arasındaki ilişki.

Figures 5, 6, 7 show plots of regression between  $MOE_d$  and  $MOE_s$ , between  $MOE_d$  and MOR, and between  $MOE_d$  and IB of medium density fiberboards, respectively. The dynamic MOE of MDF in both longitudinal and bending vibration also showed good correlations with static MOE and MOR (Table 2). These correlations were weaker than particleboard, but stronger than plywood. However, a poor relationship was found between  $MOE_d$  in longitudinal and bending vibration and IB like particleboards (Table 2). According to regression analyses, the correlations between  $MOE_{dc}$  and  $MOE_s$  and MOR were slightly higher than  $MOE_{dl}$ - $MOE_s$  and  $MOE_{dl}$ -MOR relationships. Thus, it seems that the dynamic MOE is reliable to predict the bending properties, but it is not suitable to predict IB of MDF as experienced in particleboard.

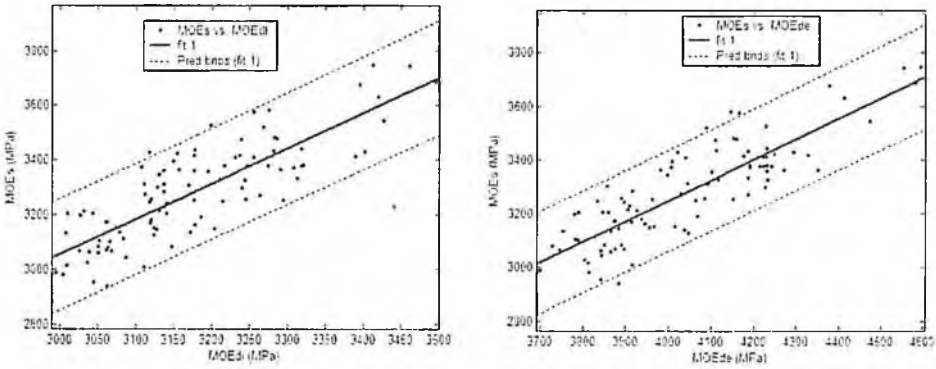


Figure 5. The relationship between  $MOE_d$  and  $MOE_s$  in MDF.

Şekil 5. MDF'lerde  $MOE_d$  ve  $MOE_s$  arasındaki ilişki.

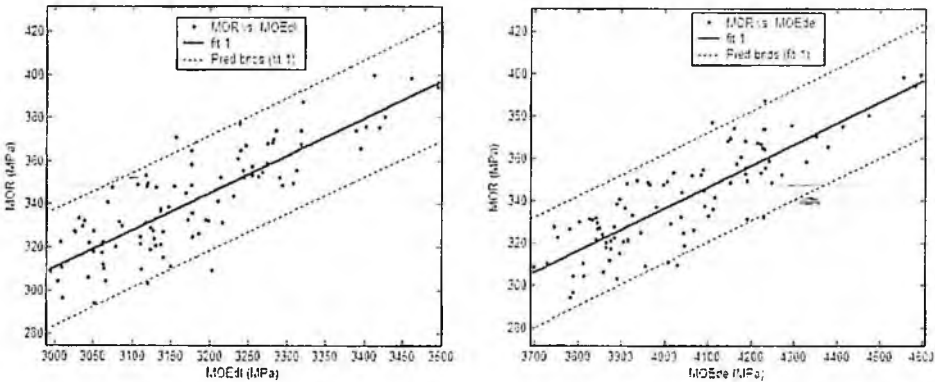


Figure 6. The relationship between  $MOE_d$  and MOR in MDF.

Şekil 6. MDF'lerde  $MOE_d$  ve MOR arasındaki ilişki.

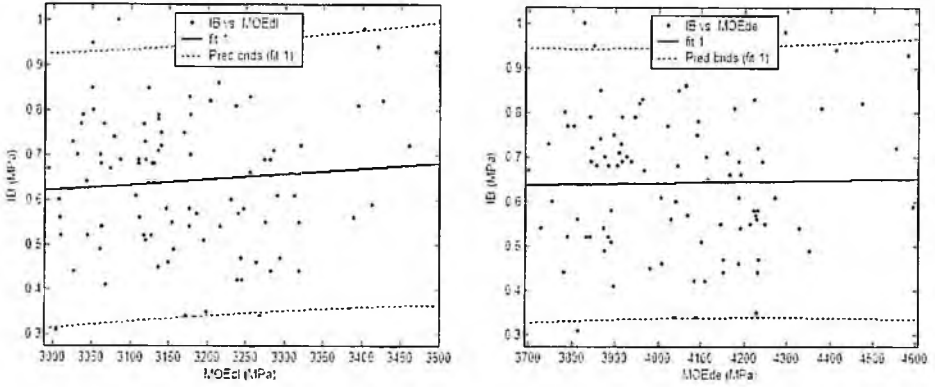


Figure 7. The relationship between  $MOE_d$  and IB in MDF.  
Şekil 7. MDF'lerde  $MOE_d$  ve IB arasındaki ilişki.

Figure 8 and 9 shows plots of  $MOE_d$ - $MOE_s$  and  $MOE_d$ -MOR relationship in parallel direction and Figure 10 and 11 shows the relationship in perpendicular direction, respectively.

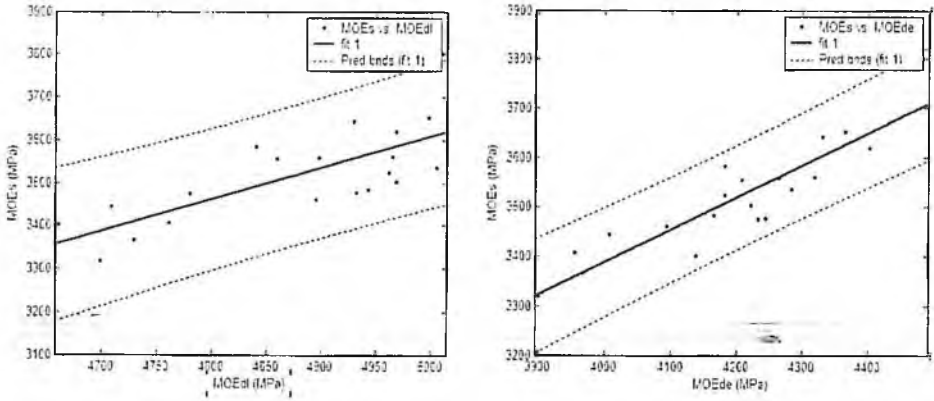


Figure 8. The  $MOE_d$  and  $MOE_s$  relationship in plywood in parallel direction.  
Şekil 8. Kontrplaklarda paralel doğrultuda  $MOE_d$  ve  $MOE_s$  arasındaki ilişki.



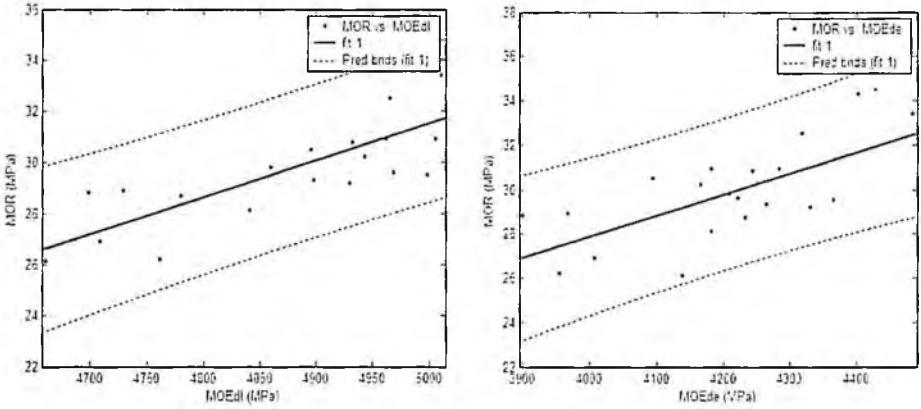


Figure 9. The MOE<sub>d</sub>-MOR relationship in plywood in parallel direction.  
 Şekil 9. Kontrplaklarda paralel doğrultuda MOE<sub>d</sub> ve MOR arasındaki ilişki.

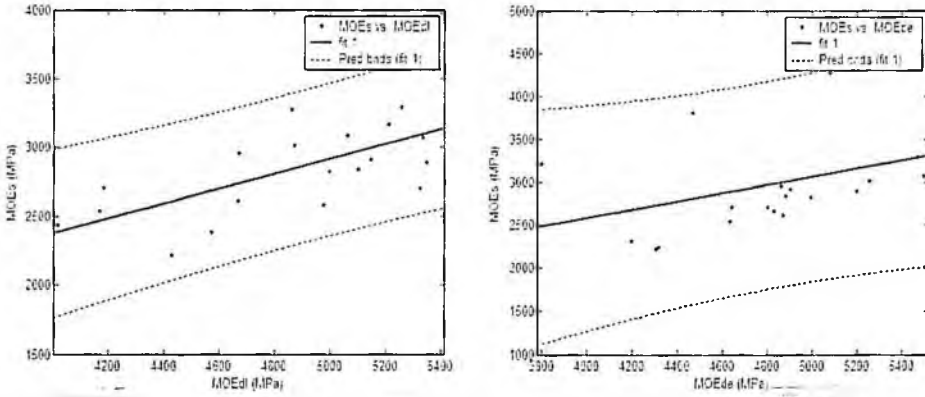


Figure 10. The MOE<sub>d</sub>-MOE<sub>s</sub> relationship in plywood in perpendicular direction.  
 Şekil 10. Kontrplaklarda dik doğrultuda MOE<sub>d</sub> ve MOE<sub>s</sub> arasındaki ilişki.

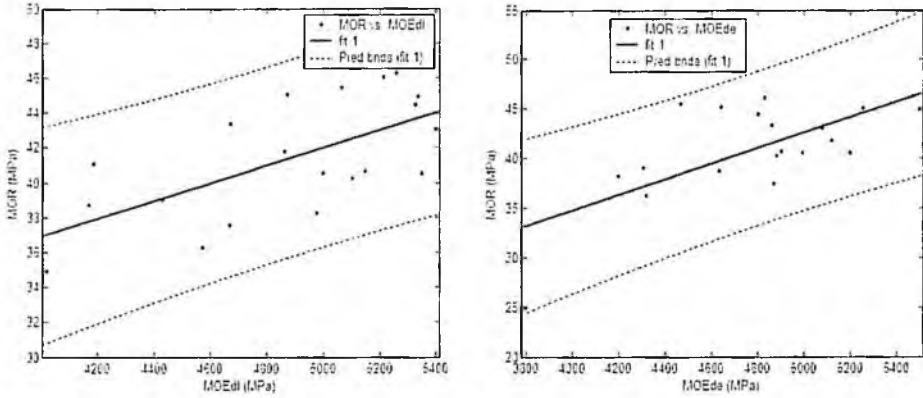


Figure 11. The  $MOE_d$ -MOR relationship in plywood in perpendicular direction.  
Şekil 11. Kontrplaklarda dik doğrultuda  $MOE_d$  ve MOR arasındaki ilişki.

As shown in Table 3 and Fig. 8-11, especially  $MOE_{de}$ - $MOE_s$  relationship was remarkable in parallel direction of plywood when compared to the others. The correlation coefficient between dynamic MOE in bending vibration and static MOE was 0.90 in parallel direction while the others varied between 0.64 and 0.77. In general, the relationships between dynamic MOE and static bending properties in parallel direction were better than perpendicular direction. Consequently, dynamic MOE in longitudinal and bending vibration is acceptable to predict the bending properties of plywood in parallel direction to long axis. However, using of dynamic MOE obtained from perpendicular direction as a predictor of static MOE and MOR may give inaccurate results when compared to the parallel direction.

#### 4. Conclusion

Based on the findings obtained from present study, we concluded as follows:

Dynamic MOE determined nondestructively by using of both longitudinal and bending vibration frequency technique is a good predictor to estimate static bending properties (MOE and MOR) of particleboards and medium density fiberboards.

Dynamic MOE determined both longitudinal and bending vibration frequency is a poor predictor of IB of particleboard and MDF.

Dynamic MOE based on both longitudinal and bending frequency is acceptable to predict the bending properties of plywood when the grain direction of surface veneer is parallel to manufacturing direction, but it may give inaccurate results in perpendicular direction.

In general, dynamic MOE in bending vibration shows slightly better correlation with static bending properties (MOE, MOR) than longitudinal vibration.

## **Acknowledgements**

The authors would like to thanks Kastamonu Entegre Industry and Trade Inc. and Ekol Plywood Industry and Trade Inc. for the composite material support and N. Köse for the Matlap statistical program support.

# Odun Esaslı Levhalarda Mekanik Özelliklerin Vibrasyon Tahribatsız Değerlendirme Yöntemi ile Belirlenmesi

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## Kısa Özet

Bu çalışmada kontrplak, yongalevha ve orta yoğunlukta liflevha (MDF) örneklerinde vibrasyon frekansı yöntemi ile tahribatsız olarak tespit edilen dinamik elastikiyet modülü ( $MOE_d$ ) değerleri ile tahribatlı testlerden elde edilen mekanik özellikler arasındaki ilişkiler araştırılmıştır. Sonuç olarak yongalevhelerde hem boyuna vibrasyon hem de eğilme vibrasyonunda  $MOE_d$  ile statik eğilme direnci (MOR) ve statik eğilmede elastikiyet modülü ( $MOE_s$ ) değerleri arasında kuvvetli korelasyonlar tespit edilmiştir. Aynı şekilde MDF örneklerinde de  $MOE_d$  ile  $MOE_s$  ve MOR arasında iyi korelasyonlar bulunmuştur. Kontrplaklarda ise uzun aksenal paralel yönde  $MOE_s$  ve MOR ile  $MOE_d$  arasındaki ilişkiler kabul edilebilir niteliktedir. Ancak uzun aksenal dik yönde elde edilen sonuçlar tatminkâr değildir. Son olarak yongalevha ve MDF'lerin yapılaşma direnci ile  $MOE_d$  arasında bir korelasyon tespit edilememiştir.

**Anahtar Kelimeler:** Tahribatsız değerlendirme, vibrasyon frekansı, dinamik MOE, direnç özellikleri, odun esaslı levhalar.

## 1. Giriş

Odun esaslı levhaların mekanik özellikleri üretimde kullanılan hammaddelerin özelliklerinden üretim parametrelerine kadar bir dizi faktörün etkisiyle geniş bir aralıkta değişim gösterebilmektedir. Buna karşın ürünlerin sahip olması gereken asgari nitelikler ilgili standartlar tarafından belirlenmiş bulunmaktadır ve üretimin bu standartları sağlayıp sağlamadığı düzenli olarak kontrol edilmektedir. Üretim kontrolü genel olarak

bir partide üretilen levhaların rasgele olarak örneklenmesi yöntemi ile yapılmaktadır. Kalite kontrol için partiden rasgele ve belli sayıda seçilen tam boyutlu levhalardan ilgili standartların öngördüğü ölçülerde küçük deney örnekleri kesilerek bunlar üzerinde klasik tahribatlı testler uygulanmakta ve elde edilen sonuçların partideki bütün levhaları temsil ettiği varsayılmaktadır. Fakat oldukça kompleks bir üretim sürecinden geçen levhalarda kontrol edilemeyen değişkenlerin etkisiyle test edilen levhalar diğer levhaları tam olarak temsil edemeyebilmektedir. Ayrıca bu şekilde test edilen levhalar artık tahrip olduğundan ekonomik bir kayıptır.

TS EN 326-2 (2006) standardı üretimde kalite kontrolü için güvenilirliği istatistik olarak ispat edilmiş alternatif yöntemlerin kullanımına izin vermektedir. Bu durumda güvenilir, pratik ve hızlı sonuç veren tahribatsız test yöntemleri üretimde kalite kontrolü için dikkate değer bir alternatif olarak karşınıza çıkmaktadır.

Geçmiş yıllarda birçok araştırmacı odun esaslı levhaların kalitesinin belirlenmesi için değişik tahribatsız test yöntemlerinin kullanılabilirliği üzerine araştırmalar yapmışlardır. Bu çalışmalarda genel olarak dalga yayılma hızı- dinamik elastikiyet modülü ve statik elastikiyet-direnç ilişkileri üzerine odaklanmışlardır. Birçok araştırmacı bu parametreler arasında kullanışlı ilişkiler bulmuşlardır (Pellerin ve Morschauer, 1974; Dunlop, 1980; Vogt, 1985, 1986; Ross ve Pelerin, 1988; Schweitzer ve Niemez, 1990; Sun ve Arima, 1999; Bektha ve diğ., 2000; Kruse, 2000; Vun ve diğ., 2000; Sotomayor, 2003; Yang ve diğ., 2005; Han ve diğ., 2006).

Bu araştırmanın amacı yongalevha, orta yoğunlukta lif levha (MDF) ve kontrplak gibi odun esaslı levha ürünlerinde Fast Fourier Transform (FFT) analizi ile stres dalgalarının oluşturduğu boyuna ve eğilme vibrasyon frekansını tespit ederek dinamik elastikiyet modülünü hesaplamak ve bunların odun esaslı levhaların statik mekanik özellikleri ile ilişkilerini ortaya koyarak, bu yolla odun esaslı levha ürünlerinin kalite özelliklerinin belirlenme olanaklarını araştırmaktır.

## 2. Malzeme ve Yöntem

Denemeler, ticari olarak üretilmiş 18 mm kalınlığında 3 yongalevha ve 3 MDF levha ile 20 mm kalınlığında 1 kontrplak levha üzerinde yürütülmüştür. Yongalevha ve liflevhalar iğne yapraklı ve yapraklı ağaç türlerinin karışımından üretilen formaldehit tutkalı kullanılarak üretilmiştir. Kontrplak ise fenol formaldehit tutkalı ile okume kaplamalarından üretilmiştir. Yongalevha ve MDF'lerin her bir panelinden 16 tanesi zımparalama yönüne paralel, 16 tanesi dik olmak üzere 32 adet ve kontrplaktan ise 20 tanesi yüzey kaplamasının lif yönü uzun eksene paralel, 20 tanesi dik olmak üzere 40 adet 50×500×levha kalınlığı mm boyutlarında örnekler hazırlanmıştır. Bu örnekler %65±5 bağıl nem ve 20±2°C sıcaklık koşullarında denge rutubetine ulaşmaya kadar iklimlendirilmişlerdir.

Dinamik elastikiyet modülü ( $MOE_d$ )'nün tespiti için iki ucundan serbest olarak desteklenmiş örneğin uç kısmındaki enine kesite vurmak suretiyle oluşturulan boyuna yönde vibrasyonun ve yine aynı örnekte üst yüzeyin merkezine vurmak suretiyle oluşturulan eğilme yönünde oluşturulan vibrasyonun frekansları kullanılmıştır. Frekansını belirlemek için bir Fast Fourier Transform (FFT) analiz programından faydalanılmıştır.

Deney düzeneği Şekil 1'de görülmektedir. Frekans yardımıyla MOEd'nin hesaplanma yöntemleri Formül 1, 2 ve 3'te verilmiştir.

Örnekler tahribatsız değerlendirilmenin ardından EN 310 (1993) standardına göre eğilme direnci (MOR) ve eğilmeye elastikiyet modülü ( $MOE_s$ ) deneylerine tabi tutulmuştur. Ayrıca yongalevha ve MDF örneklerinde EN 319 (1993) standardına göre yapışma direnci (IB) denemeleri yapılmıştır.

Tahribatsız ve tahribatlı deneylerden elde edilen veriler kullanılarak basit doğrusal regresyon analizi yapılarak  $p=0.05$  güven düzeyinde  $MOE_d$  ile  $MOE_s$ , MOR ve IB arasındaki ilişkiler araştırılmıştır.

### 3. Sonuç ve Tartışma

Regresyon analizinden elde edilen sonuçlar yongalevha için Tablo 1, MDF için Tablo 2 ve kontrplak için Tablo 3'te verilmiştir.  $MOE_d$  ile  $MOE_s$  ve MOR arasındaki en güçlü korelasyonlar yongalevhalarda elde edilmiştir. Bunu MDF ve kontrplaklar takip etmiştir. Kontrplaklarda yüzey kaplaması uzun eksene paralel olan örneklerde dik olan örneklerle nazaran daha kuvvetli ilişkiler bulunmuştur. Bununla birlikte, yongalevha ve MDF'lerde  $MOE_d$  ile IB arasında bir ilişki kurulamamıştır. Regresyon analizi sonucunda elde edilen regresyon grafikleri Şekil 2-11'de görülmektedir.

Sonuç olarak boyuna ve eğilme vibrasyonu frekansı yardımıyla tespit edilen MOEd değeri yongalevhalarda ve MDF'lerde statik eğilme özelliklerinin ( $MOE_s$  ve MOR) kestirilmesinde iyi bir veri sağlamaktadır. Ancak bu yolla yapışma direncinin kestirilmesi mümkün olmamaktadır. Kontrplaklarda yüzey kaplaması üretim yönüne paralel olarak tespit edilen  $MOE_d$  değeri eğilme özelliklerinin tespit için tatminkâr sonuçlar verebilir. Ancak dik yönde yeterli güvenilirliğe sahip olmadığı düşünülmektedir. Son olarak eğilme vibrasyonundan elde edilen MOEd ile statik eğilme özellikleri arasındaki ilişkiler, boyuna vibrasyonla elde edilenlere nazaran bir miktar daha iyi bulunmuştur.

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**İSTANBUL ÜNİVERSİTESİ ORMAN FAKÜLTESİ DERGİSİ A SERİSİ**  
**MAKALE HAZIRLAMA VE YAZIM KURALLARI**

**1. MAKALENİN HAZIRLANMASI**

Makaleler İngilizce ve Türkçe olmak üzere iki dilde yazılabilir. Yazar veya yazarlar ana metin için bu dillerden birisini, özet metin içinde diğerini tercihi edebilirler.

Makaleler aşağıdaki yazım kurallarına göre hazırlanmalıdır.

**a) Makale Başlığı**

Sayfa üstünden 3 satır boşluk bırakılarak, satır ortasına Times New Roman Tur 14 punto ve bütün kelimelerin ilk harfi büyük olacak şekilde koyu (bold) harflerle yazılmalıdır.

**b) Yazar Adları**

Makale başlığından sonra 2 aralık boşluk bırakılarak sayfaya ortalanmalı, unvan belirtilmeden baş harfleri hariç ad ve soyad küçük harflerle 10 punto koyu (bold) olarak yazılmalıdır. Yazar adlarının altına 1 satır boşluk bırakılarak 10 punto büyüklükte açık adresler belirtilmelidir. Yazarların adresleri; her bir yazarın soyadını sonunda ve adresinin başında aynı rakam (1., 2., 3. şeklinde) kullanılarak üst simge şeklinde belirtilmelidir. Ayrıca makalenin yazışmalarından sorumlu yazar, isminin üzerine bir yıldız işareti (adres belirtmek amacıyla yazılan rakamından sonra, \* işareti) konularak belirtilmeli ve adreslerden sonra 1 satır boşluk bırakılarak sorumlu yazarın telefon ve faks numaraları ile e-posta adresi yazılmalıdır.

**c) Kısa Özet**

Kısa Özet başlığından sonra 1 satır aralık verilerek 100 kelimeyi aşmayacak şekilde koyu (bold) harflerle 10 punto ve normal yazım marjında sola dayalı yazılmalı, paragraf başları normal yazım marjına göre 1 cm içeriden başlanmalıdır.

**d) Anahtar Kelimeler**

Kısa özetten sonra 1 satır boşluk bırakılarak; **Anahtar Kelimeler:** den sonra en az 3, en çok 5 kelime; virgille ayrılarak, sadece ilk anahtar kelimenin ilk harfi büyük harfle başlayacak, diğerleri tümü küçük harflerle 10 punto yazılmalıdır.

**e) Yayın Komisyonuna Sunulduğu Tarih**

Düzeltilmiş makalelerin ilk sayfasında sola dayalı olarak dip not şeklinde makalenin yayına sunulduğu ve kabul edildiği tarihler Times New Roman Tur tipinde 8 punto koyu (bold) harflerle şu şekilde yazılmalıdır: Received: 25/03/2008; accepted: 12/01/2009.

**f) Metin Bölümleri**

Özgün araştırma makaleleri "Giriş", "Materyal ve Yöntem" ve "Bulgular", "Tartışma" bölümlerine göre yazılmalıdır. Sentez ve kaynak incelemesine dayalı özgün makalelerin başlık ve alt başlıkları yazar ya da yazarların yaklaşımlarına göre belirlenebilir.

İlk başlık anahtar kelimelerden sonra 2 satır boşluk bırakılarak başlanmalı ve (Referanslar ana başlığı hariç) 1'den başlayarak (References ana başlığı hariç) numaralandırılmalı (örnek: 1. Giriş, 2. Materyal ve Yöntemler, .... şeklinde), diğer ana başlıklar bunu takip etmelidir. Ana ve alt başlıklar küçük harflerle koyu (bold) 12 punto yazılmalı, ana başlıklarda her kelime büyük harfle başlamalı alt başlıklarda sadece ilk harfler büyük olmalı ve alt başlıklar 1.1., 1.2., 1.2.1., 2.1. şeklinde numaralandırılmalıdır. Ana başlıklarla bir üst satır arasında 2, bir sonraki satır arasında da 1 satır boşluk bırakılmalıdır. Tüm metin iki yana hizalı olmalı; Kısa Özet ve Abstract başlıkları da dahil olmak üzere ana ve alt başlıklar sola dayalı paragraf başı olmaksızın normal yazım marjından başlanmalıdır. Ana metinlerde ise paragraf başlangıçları normal yazım marjına göre 1 cm içeriden başlanmalıdır.

**g) Makale Metninin Yazım Biçimi**

Makaleler 2 satır aralıkla, sayfa ve satırlara numaralar verilerek A4 kağıda, üstten ve alttan 5,85 cm, sağ ve sol kenardan 4,25 cm bırakılarak 12,5 x 18,0 cm lik yazı alanı içine yazılmalıdır. Makaleler MS Word programında Times New Roman Tur yazı tipinde, 10 punto, çift aralıklı, tüm metinde (kaynaklar ve anahtar kelimeler dahil) her sayfa 1'den başlayarak numaralandırılmış ve ilk sayfadan itibaren sayfa numarası verilmiş olarak toplamı 30 sayfayı geçmeyecek şekilde hazırlanmalıdır. Sadece doktora tez özetleri 35 sayfa yazılabilir. Makalenin başlığı, yazar adı/adları, kısa özet, yayım komisyonuna sunulduğu tarih, tüm bölüm ve alt bölüm başlıkları ile "References" bölümündeki yazar isimleri ve yayım tarihleri koyu (bold) yazılacaktır. Ayrıca, sadece metin içerisindeki bilimsel isimler (bitki ve hayvan isimleri gibi) ile "References" bölümündeki dergi isimleri italik yazılacaktır.

Makale içerisinde aynı veriler hem tabloda hem de grafikte yer almamalı, tablo ve grafiklerde standart hataların gösterilmesine özen gösterilmelidir (aritmetik ortalama  $\pm$  standart hata). Ortalamalar karşılaştırılırken önemlilik derecesi sadece yıldızla (\*) veya sadece rakamla ( $P < 0.021$  gibi) gösterilmeli, her ikisi birlikte kullanılmamalıdır ( $P < 0.5$  için \*,  $P < 0.01$  için \*\* ve  $P < 0.001$  için \*\*\*). Tablolarda yer alan ortalamalar veya işlemlerin etkisi karşılaştırılırken, karşılaştırmalar hem bitişiklerine yazılan küçük harflerle üst simge olarak belirtilmeli ve açıklanması tablo altına 10 punto büyüklüğünde yazı ile yapılmalıdır (Örneğin: 12\* gibi).

**h) Şekil ve Tablolar**

Şekil, tablo, grafik ve resimler belirtilen yazı alanı içerisinde sayfa ortalanarak konmalı, her şekil, tablo, grafik ve resime metin içinde atıf yapılmalı ve atıf yapılan paragrafın hemen sonra yerleştirilmelidir. Tablo ve grafik içerikleri ile başlıkları 10 punto büyüklükte olmalı, başlıklar numaralandırılarak tabloların üstüne; şekil, resim ve grafiklerin altına sola dayalı olarak yazılmalıdır. Şekil, tablo, grafik ve resimler Türkçe ve yabancı dilde başlık ve içerikleri ile birlikte makalenin ana metni kısmında yer almalı, başlık kelimelerinin ilk harfi büyük olmalıdır.

Buna ilişkin örnek aşağıda yer almaktadır.

Örnek:

Şekil 1. İstranca neşesinde liflere paralel basınç direnci ile yoğunluk arasındaki ilişki.

Figure 1. The relation between the compression strength parallel to grain and the density in Istranca oak

Tablo 1. Liflere paralel basınç direnci değerleri.

Table 1. The values of compression strength parallel to grain.

Metin içerisinde şekil ve tablolara (Şekil 1) (Figure 1), (Tablo 1) (Table 1) şeklinde atıf yapılmalıdır. Fotoğraf ve şekiller fotoğraf alınabilecek kalitede olmalıdır (Fotoğraflar siyah-beyaz olarak parlak karta basılmış, şekiller aydınlar üzerine çini mürekkeple veya bilgisayarla çizilmiş, yazı ve rakamlar da çini mürekkep veya bilgisayar yazılmış olmalıdır). Fotoğraf ve şekiller, JPEG formatında taranmış olarak metin içinde verilebilir.



#### 1-) Yabancı Dilde Yazılan Bölümün Başlığı ve Yazar/Yazarların Adları

Makalenin İngilizce başlığı, makalenin Türkçe kısmının bitiminden sonra yeni sayfaya geçilerek, satır ortasına Times New Roman Tur 14 punto bütün kelimelerin ilk harfi büyük olacak şekilde ve koyu (bold) harflerle yazılmalıdır.

Yazar ismi/isimleri ve adresleri makale başlığından sonra 2 aralık boşluk bırakılarak sayfaya ortalanarak, unvan belirtilmeden baş harfleri hariç ad ve soyad küçük harflerle 10 punto koyu (bold) olarak yazılmalıdır. Yazar adlarının altına 1 satır boşluk bırakılarak 10 punto büyüklükte açık adresler belirtilmelidir. Ayrıca makalenin yazışmalarından sorumlu yazar isminin üzerine bir yıldız işareti (\*) konularak belirtilmeli ve adreslerden sonra 1 satır boşluk bırakılarak sorumlu yazarın telefon ve faks numaraları ile e-posta adresi yazılmalıdır.

#### j) Abstract

Yabancı dilde yazılan başlık, yazar ismi/isimleri ve adreslerinden sonra 1 satır boşluk bırakılıp 100 kelimeyi geçmeyecek şekilde koyu (bold) harflerle 10 punto ve normal yazım marjında sola dayalı yazılmalıdır. Sayfa düzeni ana metinle aynı olmalıdır. Sadece paragraf başlangıçları normal yazım marjına göre 1 cm içeriden başlamalıdır.

#### k-)Keywords

Abstract'ın sonra 1 satır boşluk bırakılarak, en az 3, en çok 5 kelime olacak şekilde virgülle ayrılarak, (tümü küçük harflerle 10 punto yazılmalıdır.

#### l)- Yabancı Dilde Özet

Yabancı dilde özet, Keywords'ten sonra 2 satır boşluk bırakılarak başlamalıdır. Yabancı dildeki özet İngilizce, Almanca ve Fransızca olabileceği gibi İngilizce olması daha çok tercih edilmektedir. Makalenin yabancı dildeki özeti; Abstract, Introduction, Material and Methods, Results and Discussion, Conclusion, References bölümlerinden oluşmalıdır (Fransızca ve Almanca özetlerde benzer bölümlerden oluşmalıdır). Yabancı dildeki özet konunun anlaşılmasına yardımcı olacak uzunlukta ve açıklayıcı olmalıdır.

#### m- Kaynakların Metin İçerisinde Gösterimi

Kaynaklar metin içerisinde parantez içerisinde; tek kaynak için (Bozkurt, 1992) ve (FAO, 2006), birden fazla kaynak için tarihsel olarak sıralanmış şekilde (Taşvanoglu, 1973; Özçelik, 1984; Heede, 1991), ortak yayımlar için (Kurtoglu ve Koç, 1997) şeklinde yapılmalıdır. İkiden fazla yazarın olan kaynaklar metin içinde kısaca (Aykut ve ark., 1997) şeklinde verilmelidir. Aynı yazarın aynı tarihte yapılmış iki eseri olduğu takdirde bu eserler yılların sonuna a ve b harfleri konarak belirtilmelidir. Aynı yazarın bireysel ve ortak yayımları olduğunda önce bireysel yayımlar sıralanmalı, ortak yayımlar bireysel yayımlardan sonra verilmelidir. Kaynak listesinde bütün yazarlar açık olarak gösterilmelidir. Kaynaklar, yabancı dildeki özetten sonra References başlığı altında, alfabetik sıraya göre aşağıdaki şekilde verilmelidir.

Sempozyumlardan ve dergilerden alınan makalelerin isimleri yazılırken sadece ismin ilk harfi büyük, diğerleri küçük harflerle yazılmalıdır. Kitap isimlerinde ise her kelime büyük harfle başlanmalıdır. References bölümündeki yazar isimleri koyu (bold) yazılmalı, internet kaynakları olarak sadece resmi kurum isimleri yer verilmelidir.

#### n- References / Kaynaklar

Atf yapılan makalelerin References kısmında gösterilmesine ilişkin örnekler:

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#### 2. MAKALENİN TESLİMİ VE DEĞERLENDİRME SÜRECİ

Yukarıda kurallara uygun yazılan makaleler, 4 nüsha basılmış olarak başvuru dilekçesi ile birlikte Yayın Kurulu'na gönderilir ve ön elemeye tabi tutulan makalelerin hakemlere gönderilip gönderilmeyeceğine karar verilir. Hakemler tarafından yayınlanması uygun bulunmayan makaleler, yazarlarına iade edilmez. Yayına uygun bulunmakla birlikte düzeltilmesi veya değiştirilmesi istenen hususlarla ilgili hakem eleştirileri yazarlara gönderilecek düzeltilmesi yada düzeltilme isteklerine açıklamalar yapılması istenir. Yazar/yazarların savunmaları yeniden ilgili hakemlerin görüşlerine sunular ve tatmin edici bulunması halinde yayımlanmasına karar verilir.

Yayımlanması uygun bulunan makaleler, son düzeltmeleri yapıldıktan sonra tek satır aralıklı olacak ve satır numaraları silinmiş şekilde 2 adet CD içerisinde MS Word programında yazılmış olarak (Yazar ve makale adları CD üzerinde yazılmamalıdır) başvuru dilekçesi ile birlikte Yayın Kuruluna gönderilir.

Yayın Kuruluna verilecek dilekçe aşağıdaki bilgileri içermelidir;

- Makalenin daha önce herhangi bir yerde yayımlanmamış olduğu ve telif ücreti alınmadığı,

- Toplam 5 kelimeyi geçmeyen kısa makale başlığı,

- Toplam klışe alanı (cm<sup>2</sup>) (basılması istenen boyutlara göre hesaplanacak),

- Düzeltilmelerin kimler tarafından yapılacağı (en az bir isim),

- Yazarların yazışma adresi, telefon numaraları ve e-mailleri.

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