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Comparing Some Hardness Methods for Estimating Thermo-Vacuum Treatment Efficiency

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Research Article

Öz − Durometer hardness (Shore D) is generally used for measuring hardness of the thermoplastic elastomers, vulcanized rubber, some plastics etc. In this study, availability of the Shore D hardness method for wood hardness was investigated. In addition, it was investigated whether the values obtained from thermo-vacuum treated material differed from those obtained from untreated material. Because the method has potential for in-situ measurements as a non-destructive evaluation method with its relatively smaller measurement needle. Untreated and thermo-vacuum (Termovuoto®) treated at 200ºC treated Scots pine (*Pinus sylvestris*) samples were used in this study. Due to the needle size, method principle, and heterogeneity caused by annual ring structure, full radial and radial specimens were prepared, and earlywood and latewood hardness values were measured in each group. In addition, the Shore D hardness method was compared with the static Janka hardness method to evaluate efficiency. The results showed that the thermal treatment can be predicted using a durometer hardness tester with taking anatomical features into account. In terms of cutting method, the data obtained from fully radial and radially cut samples were similar. Based on annual rings, different results were obtained in early- and latewood. Furthermore, higher results were obtained in both untreated and treated latewood layers, and in both fully radial and radially cut samples. However, a review of the literature reveals that the results obtained using the Shore D method in Scots pine show significant variations. In light of the results of this study, it is suggested that anatomical differences should also be taken into account in order to eliminate variation dependent on macroscopic variables. Otherwise, although the standards related to the Shore D method recommend the application of standard weights in hardness measurements of other materials, in this study, low variations were obtained even though the tests were performed heterogeneously using human force.

***Keywords** – Durometer, janka, Pinus sylvestris, shore D, thermal modification*

Termo-Vakum İşlem Verimliliğini Tahmin Etmek İçin Bazı Sertlik Yöntemlerinin Karşılaştırılması

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Abstract – Durometre sertliği (Shore D), genellikle termoplastik elastomerler, vulkanize kauçuklar ve bazı plastiklerin sertliğini ölçmek için kullanılmaktadır. Bu çalışmada ise Shore D sertlik ölçüm yönteminin, ahşap sertliğinin belirlenmesinde uygulanabilirliği araştırılmıştır. Ayrıca termo-vakum işlem görmüş malzemede elde edilen değerlerin, işlem görmemiş malzemeye göre farklı olup olmadığı da incelenmiştir. Çünkü yöntem, nispeten daha küçük olan ölçüm iğnesi ile bir tahribatsız değerlendirme yöntemi olarak yerinde ölçüm yapma potansiyeline sahiptir. Bu çalışmada, işlem görmemiş (kontrol) ve 200ºC'de termo-vakum (Termovuoto®) işlemine tabi tutulmuş sarıçam (*Pinus sylvestris*) örnekleri kullanılmıştır. İğne boyutu, yöntem prensibi ve yıllık halka yapısının getirdiği heterojenlik nedeniyle, tam radyal ve radyal numuneler hazırlanmış ve her grupta ilkbahar ve yaz odunu sertlikleri ölçülmüştür. Ayrıca verimliliği değerlendirmek amacıyla Shore D sertlik yöntemi, statik Janka sertlik yöntemiyle de karşılaştırılmıştır. Sonuçlar, anatomik özellikler dikkate alınarak durometre sertlik ölçüm cihazı ile ısıl işlem görme durumunun tahmin edilebileceğini göstermiştir. Kesiş yöntemi açısından, tam radyal ve radyal kesilmiş numunelerde elde edilen veriler benzer çıkmıştır. Yıllık halkalar esas alındığında; ilkbahar ve yaz odununda farklı sonuçlar elde edilmiştir. Ayrıca yaz odunu tabakası hem işlem görmemiş hem de işlem görmüş hem tam radyal ve hem de radyal kesilmiş numunelerde daha yüksek sonuçlar elde edilmiştir. Literatür incelendiğinde ise sarıçamda Shore D yöntemiyle elde edilen sonuçların geniş varyasyonda olduğu, bu çalışmadaki sonuçlarla anatomik farklılıkların dikkate alınmasıyla varyasyonun azalabileceği tespit edilmiştir. Diğer yandan Shore D yöntemiyle ilgili standartlarda diğer malzemelerin sertlik ölçümlerinde belirli ağırlıklar önerilmesine karşın, bu çalışmada testler insan kuvvetiyle heterojen bir şekilde gerçekleştirilse de düşük varyasyonlar elde edilmiştir.

***Anahtar Kelimeler** − Durometre, janka, Pinus sylvestris, shore D, termal modifikasyon*

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1. Introduction

Thermal treatment of wood is a modification method that the treatment temperature generally varies between 160ºC and 230ºC, and its effect on wood generally increases depending on the intensity of the process (Sandak et al. 2015). In recent years, thermo-vacuum treatment (TVT or Termovuoto®) has been developed as a new method (Mai and Militz 2023). In this method, the oxygen in the reactor is replaced by a partial vacuum and heating is provided by forced airflow. In studies conducted on poplar (*Populus deltoides* Bartr. cv. "Lux" ex I-69/55) (Gao et al. 2016), black pine (*Pinus nigra*) (Bal 2018), Scots pine (*Pinus sylvestris*) (Jebrane et al. 2018), Siberian Larch (*Larix sibirica* L.) (Giudice et al. 2023) and similar species, improvements in physical properties were reported as in other thermal treatment methods. However, it is also stated that due to the application principle, there is a lesser wood material content loss in this method (Allegretti et al. 2012) and the decrease in mechanical strength can be minimized (Giudice et al. 2023). According to Hill (2006), this difference is due to the continuous operation of the vacuum pump during the process, and all volatile compounds that accelerate the degradation of polysaccharides in the cell wall are continuously removed from the kiln. Bal (2018) compared thermal treatment methods performed in air, nitrogen and vacuum atmospheres and determined that the TVT method had the least effect on the mechanical properties.

There are some methods to determine the treatment efficiencies, for example TSE CEN/TS 15679 (2010) standard has reference tables which presents according to physical, mechanical and biological test results in direct proportion to temperature. Because, especially in applications carried out at 180ºC and above, the significant decrease in mechanical strength (Esteves and Pereira 2009) is an indication that can be evaluated in the detection of thermally treated wood. However, the standard is related to 'conventional' thermal treatments and it may give different results for different methods such a TVT.

As stated in the TSE CEN/TS 15679 (2010), hardness one of the indicators of thermal treatment efficiency. Todorović and Popović (2011) stated that the (Brinell) hardness values on the tangential surfaces of samples taken from oak heartwood and sapwood and subjected to (conventional) thermal treatment at different temperatures showed a statistically significant decrease after the treatment. Salca and Hiziroglu (2014) examined the (Janka) hardness, surface quality and cellular structure changes after thermal treatment in four different wood species. They stated that increasing the time and temperature in the treatment caused a decrease in the hardness values, which could be associated with the deterioration of the cell wall structure after the treatment. Esteves *et al.* (2021) determined that the shore D hardness values decreased in all studied 14 species growing in tropical regions after thermal treatment at 212ºC according to the Finnish method (Anonymous 2003). The finding of a significant relationship with high correlation coefficients between density and classical (Holmberg 2000) or durometer hardness (Esteves *et al.* 2021) could be an important indicator for hardness methods using as a non-destructive evaluation method.

Methods such as Janka, Brinell, and Monnin are generally used to measure wood hardness (Koczan *et al.* 2021). However, the durometer which is a method based on measuring the hardness of a material according to its resistance to being notched, is also among the researches. Although the D method (Shore D) in the ASTM D 2240 (2010) standard is frequently preferred for measurements is recommended for testing hard rubber, thermoplastic elastomers, harder plastics and hard thermoplastics, it has also been tried for wood in recent years due to its practical use and fast results. Sofuoğlu and Yeşil (2016) found that approximate results were obtained in hardness measurements made with a pendulum and durometer (Shore D). Sofuoğlu (2022) concluded that both methods were suitable for use in hardness measurements of wooden materials. However, it can be said that anatomical properties should also be taken into account since the measuring head is relatively small. For example, Hirata et al. (2001) measured different hardness values in early- and latewood layers on the three softwood species.

Hardness is a simple, fast and effective method (Peker *et al.* 2023). Due to its features, it has the potential to be used as a non-destructive evaluation method alone or in combination with other methods. Because, the method can be used to estimate some properties without significantly changing the end-use performance (Pellerin and Ross 2002) in many usage areas. However, they can be combined with other methods to increase accuracy rates in practice (Imposa et al. 2014; Todoroki et al. 2021). Martins et al. (2022) stated that the dynamic hardness measurement (sclerometric) method can be used as a non-destructive evaluation method in the studied hardwood species. Sydor *et al.* (2022) observed that hardness tests could be evaluated as non-destructive, especially on radial or tangential surfaces. They stated that experiments performed in cross-section (longitudinal direction to the grain) are destructive and irreversible on a microscopic scale due to the bending of the cell walls and the breaking of their ends. In addition, due to the relatively low-scale damage caused by conventional (static) hardness methods to the material, studies have been conducted on the microhardness which gives similar results to these methods (Oral 2023).

In this study, the possibility of the durometer hardness method to evaluate the thermal treatment efficiency was investigated. In addition, it was aimed to investigate the usability of portable models produced according to the Shore D method as a non-destructive evaluation method, since it allows in-situ measuring and the measurement area is relatively small.

2. Materials and Methods

Untreated (control, with 23 x 130 x 880 mm sizes) and thermo-vacuum treated (with 18 x 110 x 880 mm sizes) Scots pine (*Pinus sylvestris*) lumbers were commercially obtained. The treatment was realized at 200ºC temperature and -760 mmHg pressure during 4 h. The lumbers were sawn to sample sizes for related tests. Density and equilibrium moisture content samples were sawn according to the TSE CEN/TS 15679 (2010). Hardness samples were sawn to 50 x 50 mm (width and length) with current thicknesses.

50-samples for each group were tested according to ASTM D 2240 (2010) for Shore D and TS ISO 13061-12 (2021) for Janka hardness. Both hardness tests (firstly Shore D) were applied on the same sample. Because the measurement needle was relatively thin, the method can be considered minimally destructive. The Shore D procedure was repeated three times and the average was taken.

Since the ASTM D 2240 (2021) is valid for different materials, mainly for plastics, the D version was chosen to compare the other studies in the literature. However, factors that could affect the results due to the diameter of the needle, anatomical properties of the wood were considered. Earlywood and latewood layers which were parallel to the Janka hole were individually measured (Figure 2). Additionally, the samples were separated as quarter and rift-sawn (90° - 60° and 60° - 30°, respectively, for the majority of the angle between the growth rings and the face) according to TS EN 844 (2019). They were respectively described as quartered and rift. Plain-sawn (tangential) samples weren't used, because when the needle was inserted into the tangential surface, there was a possibility of reaching both early- and latewood (Figure 1)

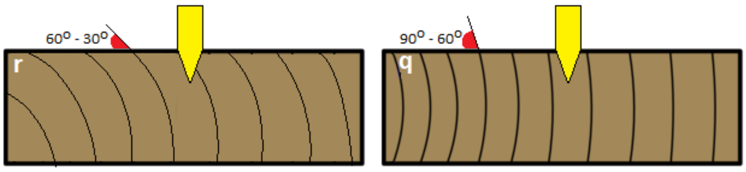

Figure 1. The measurement needle situation related to annual rings on the rift (r) and quartered (q) samples (Yellow area: Needle)

Şekil 1. Radyal (r) ve çeyrek kesilmiş (q) örneklerde yıllık halkalara göre ölçüm iğnesinin yerleşimi (Sarı bölge: Ölçüm iğnesi)

The maximum value on the Shore D durometer hardness device (Chuan Lu Durometer LX-D, China) was recorded when the needle was manually spiked to the sample. The gauge readings ranged from 0 to 100 (unitless) and were converted to Newtons using the following equation:

$$Force\ (N) = Gauge\ value\ (unitless) \times 0.4445 \qquad (2.1)$$

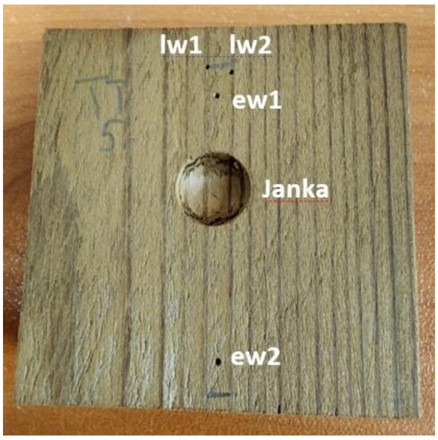

Figure 2. Hardness measurements on the thermo-vacuum-treated sample (ew: earlywood, lw: latewood, 1-2: first and second measurement for ShoreD)
Şekil 2. Termo-vakum işlemi uygulanmış örnekte sertlik ölçüm noktaları (ew: ilkbahar odunu, lw: yaz odunu, 1–2: Shore D sertlik ölçümü için birinci ve ikinci ölçüm).

Janka method was preferred as a conventional hardness measurement, although the TSE CEN/TS 15679 (2010) suggests the Brinell method for thermal treatment efficiency. Because Brinell method has difficulty measuring diameter, while the Janka method gives more reliable data from a universal test machine. Janka hardness measurements were realized according to the TS ISO 13061-12 (2021) on the universal test machine (Lloyd Instruments, United Kingdom). Janka and durometer hardness values were compared on the same samples at the actual moisture content (MC). However, hardness values for both methods were adjusted to air-dry 12% MC. The standard suggests an equation for adjusting with indicating that it is valid 12% ± 5% MC interval. In this study, the equation was applied in the descriptive statistics, although the MC of thermo-vacuum treated samples were found 5.46% on average to contribute to the literature data. In the equation, while the W shows the MC (%), hardness value at air-dry MC (H12, N) and different MC (HW, N) of the samples, α is an adjusting factor. TS ISO standard suggests taking the value from the national standard for α. The last valid standard for Janka hardness (TS 2479 1976) suggested using 0.025 for measurements on the radial (wide) surfaces (Figure 2).

$$H_{12} = H_w[1 + \alpha(W - 12)] \qquad (2.2)$$

IBM Statistics and MS Office Excel software were used for data visualizations and statistical analysis. In the statistical analysis, p-values of 0.05 were considered to determine the significance level.

3. Results and Discussion

The moisture contents of untreated samples were 15.42% and 5.46% of treated samples on average. The 5% difference might show the efficient thermal treatment. Because TSE CEN/TS 15679 (2010) indicates that, the equilibrium moisture content difference between untreated and thermally modified pine for exterior conditions should be 6-7% at 20℃ temperature and 65% relative humidity. Moreover, the difference can increase to 10-11% in more humid conditions.

The values in Table 1 adjusted to 12% MC were given for the literature. Because the treated wood is generally used at lower moisture contents (5-7%) in the usage area. Additionally, the moisture content of the treated wood is out of the limit for hardness value adjustment. There are significant differences between the untreated and treated samples. The density decrement may have affect the results, because densities were 0.45g/cm3 and 0.38 g/cm3 of untreated and treated samples on average, respectively.

Table 1. Descriptive statistics of the samples*
*Tablo 1. Örneklere ilişkin tanımlayıcı istatistikler**

Treatment	Sample	Annual ring		Shore-D hardness				Janka hardness	
				Unitless		N		N	Adj. 12%
		Layer	Thickness (mm)	Sections	Mean	Normal	Adj. 12%		
Untreated	Quartered	Early wood	2.49 (0.38)	34.55 (4.33)	46.49 (3.59)	20.66 (1.60)	22.34 (1.71)	1546.52 (165.76)	1671.88 (175.84)
		Late wood		58.42 (5.34)					
	Rift	Early wood	2.41 (0.43)	55.26 (4.19)	45.71 (3.19)	20.32 (1.42)	22.14 (1.52)	1492.20 (133.83)	1626.06 (144.50)
		Late wood		36.16 (5.37)					
Thermo-Vacuum	Quartered	Early wood	2.96 (0.77)	39.55 (5.93)	49.16 (3.35)	21.85 (1.49)	18.41 (1.29)	1482.60 (138.17)	1249.30 (119.39)
		Late wood		58.77 (4.69)					
	Rift	Early wood	3.71 (1.15)	39.11 (8.34)	48.52 (4.10)	21.57 (1.82)	17.91 (1.55)	1379.50 (251.71)	1145.08 (209.21)
		Late wood		57.92 (5.48)					

* Standard deviation in brackets. Adj. - values adjusted to 12% MC. The mean is the average of two measurements taken from each layer. N: Newton

In the literature and this study, the average ShoreD hardness values obtained in untreated samples from studies conducted on untreated Scots Pine (*Pinus sylvestris*) were shown in Table 2. It can be said that the data varies in a wide range. A similar situation exists in studies realized on other tree species in which lumber is commercially preferred (Ayata and Dilik, 2020; Esteves et al., 2021). It can be said that the lack of sufficient consideration of anatomical features was effective in this wide variation. If the durometer test will continue to be used, it will be useful to determine a standardized methodology by taking into account anatomical features such as radial-tangential surface, as in other test methods, for healthy comparisons.

Table 2. Shore D hardness values measured on Scots pine (*Pinus sylvestris*) in the literature
Tablo 2. Literatürde yer alan sarıçam (Pinus sylvestris) odununun Shore D sertlik değerleri

Study	Shore D hardness
Han et al. (2017)	38.60
Akçay (2020)	39.20
Akçay et al. (2022)	43.16
This study – Rift	*45.71*
This study – Quartered	*46.49*
Özkan (2022)	49.60
Budakçı and Karamanoğlu (2014)	54.50
Şahin et al. (2020)	57.00 ~

ASTM D 2240 (2010) indicates that manual operation (handheld) of a durometer will cause variations in the results attained, therefore standard weights (5 kg for D type) are suggested. In this study, manual measurements were done due to the portable usage possibility of the device. The results (Table 3) showed that coefficients of variation (CoV) were at a low level, while only CoV values of latewoods from the treated rift samples were higher than 10%. In the box-plot graph (Figure 3), the distribution of the values and statistical differences of all layers in both untreated and treated samples was found similarly. It was thought that this was due to the possibility that the needle might not show enough resistance on the treated samples and might hit the summerwood according to the annual ring angle in the rift samples. Because the CoV values of latewoods untreated rift samples were lower than 10%. As proof of this, the CoV values of earlywoods of all samples were lower.

Table 3. Some descriptive durometer hardness measurements on the layers
Tablo 3. Katmanlara ilişkin durometre sertlik ölçümlerine ait tanımlayıcı istatistikler

	Quartered – Treated		**Quartered – Untreated**		**Rift – Treated**		**Rift – Untreated**	
	EW	LW	EW	LW	EW	LW	EW	LW
Mean	40	59	35	58	39	58	36	55
StdDev	1.9	4.1	2.2	1.4	2.6	6.6	2.3	8.4
CoV (%)	4.8	7.0	6.3	2.4	6.7	11.0	6.5	15.0

*StdDev: standard Deviation, CoV: coefficient of variation, EW: earlywood, LW: latewood.

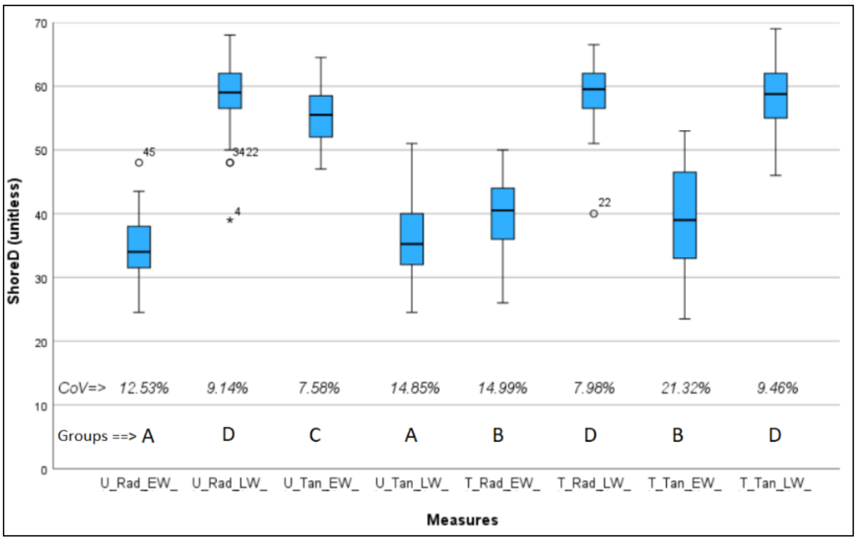

Figure 3. Distribution of ShoreD measurement values. CoV: coefficient of variation, U: untreated, T: treated, Rad: quartered, Tan: rift, EW: earlywood, LW: latewood. Letters show statistically different homogeneous groups at a 95% confidence level ($p < 0.05$)
Şekil 3. Shore D ölçüm değerlerinin dağılımı. CoV: varyasyon katsayısı, U: işlem uygulanmamış, T: işlem uygulanmış, Rad: çeyrek kesim, Tan: radyal kesim (rift), EW: ilkbahar odunu, LW: yaz odunu. Harfler, %95 güven düzeyinde (p < 0,05) istatistiksel olarak farklı homojen grupları göstermektedir

Additionally, the possible relationship between annual ring width and hardness was investigated. Because of annual ring width and density (Karlman et al. 2005), density and treatment effect especially at the higher temperature according to mass loss (Allegretti et al. 2021), density and hardness (Grekin and Verkasalo 2013) have a positive relationship. However, no significant relationship was found both within and between groups in this study (Figure 4, Table 4). R^2 is low, indicating annual ring width is not a strong predictor of hardness.

Table 4. Simple regression analyses result for durometer hardness.
Tablo 4. Durometre sertliğine ilişkin basit doğrusal regresyon analizi sonuçları

	Quartered		**Rift**	
	Untreated	Treated	Untreated	Treated
Coefficient of determination (R^2)	0.0084	0.0256	0.0291	0.2427

Jebrane et al. (2018) found that thermo-vacuum-treated samples had lower hardness than Scots pine samples. However, they only specified perpendicular to the grain for hardness measurement surface without specifying whether it is a radial or tangent surface. Similar results were found although the differentiation was made in this study. Statistically significant decrements were found in Janka hardness before and after the treatment on both surfaces. When all methods and all measurement areas were considered, only shore D hardness values on the radial surface at the latewood layer didn't change after the treatment (Table 5).

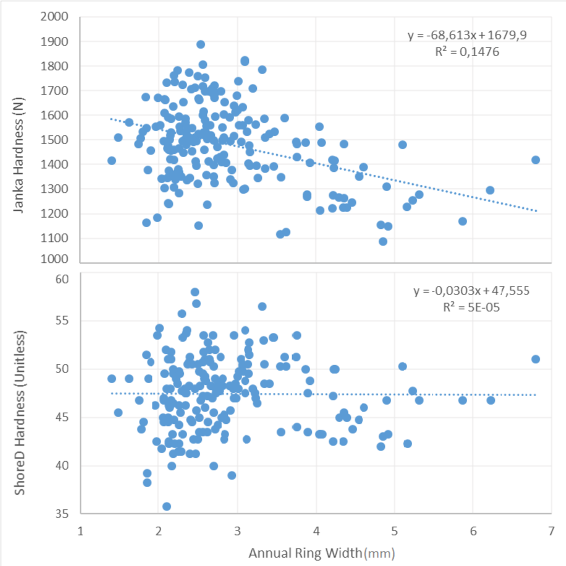

Figure 4. Annual ring width and hardness relations
Şekil 4. Yıllık halka genişliği ile sertlik arasındaki ilişkiler

Table 5. The hardness difference analysis after the treatment*
*Tablo 5. İşlem sonrasında sertlikte meydana gelen farklılıkların analizi**

Surface	Hardness	Area	Significance (2-tailed)
Quartered	ShoreD	Earlywood	< 0.001
		Latewood	0.676*
		Annual ring	< 0.001
	Janka	Surface	0.041
Rift	ShoreD	Earlywood	< 0.001
		Latewood	0.045
		Annual ring	< 0.001
	Janka	Surface	0.003

*No difference at 95% level (p<0.050).

When quartered and rift samples are taken into consideration, statistically significant differences were found for both untreated and treated samples. However, although the shore D hardness measurements on the early- and latewood layers were individually found different. The minimal or statistically insignificant change in latewood after processing may be due to it being more affected by heat treatment than earlywood. This could be because latewood contains more cell wall material. The mean of all layer (area) measurements was found similar on the untreated samples (Table 6).

Table 6. The hardness difference analysis between rift and quartered samples*
*Tablo 6. Radyal ve çeyrek kesilmiş örnekler arasındaki sertlik farklılıklarının analizi**

Treatment	Hardness	Area	Significance (2-tailed)
Untreated	ShoreD	Earlywood	< 0.001
		Latewood	< 0.001
		Annual ring	0.237*
	Janka	Surface	0.041
Treated	ShoreD	Earlywood	< 0.001
		Latewood	0.045
		Annual ring	< 0.001
	Janka	Surface	0.003

*No difference at 95% level (p<0.050).

As seen in the Figure 5, the prediction ratio of the Janka hardness increased, when considering parameters related to annual ring layers increased. The highest correlation was obtained in rift samples in both treated (R^2: 0.453) and untreated samples (R^2: 0.519). When especially untreated quartered samples were considered, coefficients of determinations gradually increased with increasing detailed measurements. Additionally, the mean of two latewood measurements had a lower relation in other groups

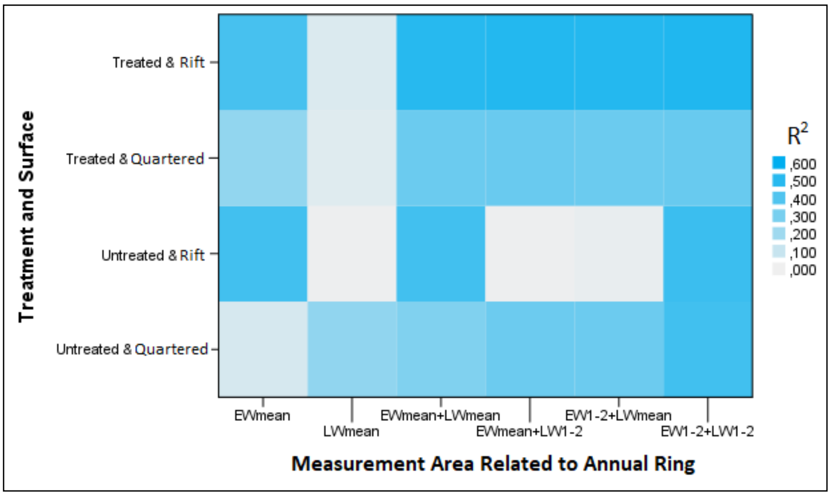

Figure 5. R^2 values distribution from regression analysis for Janka hardness prediction. EW: earlywood, LW: latewood, 1-2: including two separate measurements

Şekil 5. Janka sertliği tahmini için gerçekleştirilen regresyon analizinden elde edilen R² değerlerinin dağılımı. EW: ilkbahar odunu, LW: yaz odunu, 1–2: iki ayrı ölçümü içeren analiz

4. Conclusions

This study investigated the feasibility of using the durometer method as an alternative approach to estimate Janka hardness and to evaluate the efficiency of thermo-vacuum treatment. Unlike conventional wood hardness tests, the Shore D durometer uses a relatively small indenter needle. Therefore, anatomical features such as surface appearance (quartered and rift) and annual ring layers (early- and latewood) were considered. Results showed that: (1) The considered features significantly affected the values. It is strongly suggested that variables related to anatomic structure should be considered while using the durometer method (ShoreD) for wood hardness measurement. Other studies that did not take anatomic structure into account caused the results to vary widely, even in the same tree species. (2) The variation of the manual measurement values in this study was found at the low level, although the related standard suggests using the standardized weight. However, if Shore D hardness is used for wood, standardized weights should be included to eliminate variations in the applied force caused by the researcher. (3) Otherwise, thermo-vacuum treatment efficiency could be detected with the durometer hardness method. All tested measurement procedures showed that more measurements gave more accurate results. For example, tests can be applied on the samples having different annual ring angles, hardwoods and diffuse-porous, semi-ring-porous, ring-porous species etc. Related to needle dimensions, the measurements could be repeatable. As a result of that, it was founded that Shore D measurements in wood only yielded meaningful and comparable results with anatomical layer-based standardization. Additionally, when there is a need for on-site material hardness assessment and rapid results, the Shore D method can be used instead of the Janka method, which requires a laboratory environment for measurement.

Authors Contributions

HVG: Conceptualization, Methodology, Reviewing and Editing, Data collection and Data analysis

ÖU: Data collection, Data analysis and paper writing

Conflicts of Interest

The authors declare that there is no conflict of interest.

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