Optimization of Hardness Values *via* Taguchi Method for Chestnut Wood Etched or Impregnated with Sodium Bicarbonate after Artificial Aging

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In today's wood industry, research is being conducted to increase material strength, ensure long-term use, and increase its hardness against many harmful external factors. With the studies on the protection of wood, new protection materials and methods are introduced. In this study, wood was etched with solid sodium bicarbonate ($NaHCO_3$) and 1, 2, and 3 bars of air pressure after treatment with $NaHCO_3$ solution and drying. The change in hardness values of impregnated and surface-treated (paint, varnish) chestnut wood after artificial aging was examined. Etching, impregnation, and surface treatment factors were optimized using the Taguchi design of experiments (DoE) after artificial aging for 3, 6, and 9 months. L16 orthogonal array was used to determine the optimum conditions for determining hardness values and their percentage changes. The results showed that the abrasive factor prevails over the effect of surface treatments. It has been understood that the most effective factor in the hardness value changes during the artificial aging period (3-6-9 months) is abrasion, and the factor with the least effect is the surface parameter. The percentage accuracy of the model used in estimating the wear factor average R^2 across all dependent variables was 95%.

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INTRODUCTION

Although wooden material is resistant to external factors for a certain period, it cannot remain durable for a long time, as its natural structure is destroyed by chemical, physical, and biological changes (Williams 2005; Budakci 2006; Kilic and Hafizoglu 2009). To protect wooden materials from external effects and increase their usage performance, some surface treatments (such as paint and varnish) and impregnation processes are used (Kartal 2016). Impregnation of the *Castanea sativa* Mill. tree with a natural extract such as acorn tannin provided both an increase in mechanical properties and a method of protection against biological pests (Güneş and Altunok 2022). Thus, the interactions of natural decomposition factors of wood material are somewhat restricted. All surfaces of the wood must be covered against variable outdoor weather conditions (Hill *et al*. 2022). Surface treatments extend the lifespan of wooden materials by protecting them from external factors and giving them an aesthetic appearance. Compared to organic solvents as liquids, the use of water-based paints and varnishes is becoming more widespread with the passage of time because they contain low amounts of volatile organic compounds, are environmentally friendly, evaporate slowly, are easy to penetrate, provide permanent color, and are easy to apply (Blanchet and Pepin 2021). In particular, water-based varnishes that do not cause changes in the texture and color of the wood material generally protect against chemical reactions and can form an irreversible layer (Johnson 1997). The wood was first treated with sodium bicarbonate and then with sodium silicate. Then, by impregnating it using sodium silicate and sodium bicarbonate, a method for protection from fungi, insects and other pests was obtained (Thougaard and Hayden 2014).

Establishing the most adequate interactions between wood materials and outdoor conditions requires years of work. However, these tests are quite costly and time-consuming (Brischke *et al.* 2006). Especially when considering the restoration of historical wooden structures, aging processes must be conducted in addition to abrasion to obtain the deteriorations that may occur in the wooden materials to be used in the fastest and closest way to reality (Peng *et al*. 2024). These processes are performed by various methods, including natural, accelerated, and artificial procedures. However, it should be taken into consideration that there may be differences between test results and actual results. It is important to perform tests using standards most appropriate to the intensity and shape of the impact of all factors affecting the wood material to obtain the most reliable results (Uluata 2011). It has been stated that the accelerated aging effect of approximately 240 h is equivalent to 5 years of outdoor conditions (Aytin *et al*. 2021).

Time-consuming mechanical treatments of surfaces for restoration, and the lack of a safe environment have increased the importance of durable environmental abrasives, such as $NaHCO_3$, which has antacid properties, in making wooden surfaces porous. Etching processes create slight cracks or gaps in the material surface, allowing impregnation or protective coating agents to penetrate more effectively. This can increase the durability and lifespan of the material. When chemicals such as $NaHCO_3$ are used, grains of these substances tend to accumulate on the surface and pores of the material. This may affect the effectiveness of the etching process and the suitability of the material for subsequent processing (Bayraktar and Kesik 2022). Therefore, it is important to take into account the particles that may form in the pore spaces of the material during the etching process and to clean these particles if necessary. This can ensure that the material has the desired properties and the machining process is completed successfully. In addition, the use of water-soluble $NaHCO_3$ for impregnation purposes is rapidly increasing, and it is claimed that it is effective in protecting treated wood material against damage caused by external conditions and biologically harmful organisms (Johnson 1997). If the moisture content of the wood surface increases as a result of contact with water, the buffering capacity of sodium bicarbonate applied by dry etching may be affected, which may reduce the ability of sodium bicarbonate to actively neutralize acids present in the wood.

Taguchi's design of the experiment has been used successfully in solving optimization problems. It ensures that result is obtained with the least number of experiments and supports the development of high-quality processes and products (Aktaş and Sofuoğlu 2022). Many variables are used to determine some properties of wood material. This restricts effective and efficient use in feature determination (Çabuk *et al.* 2014; Kurt and Can 2021). In addition to their statistical significance, experimental design methods are complementary and supportive. They can be used in various research and development applications, minimizing costs, and increasing quality, thus strengthening the reliability of the outputs obtained. Dr. Taguchi has introduced an innovative solution that will increase efficiency in performing multi-factor experiments and evaluating applications (Ross 1988; Taguchi *et al*. 1988). Apart from being an experimental design method, the

Taguchi method is a useful application for high-quality system design by reducing the number of experiments.

Test outputs were evaluated using the approach of Köksoy and Muluk (2004). Among the factors used, the following equations are used to determine those that affect the result and those that have less impact. The lowest and best factor was quantified using Eq. 1.

$$\frac{S}{N} = -10x \log \left(\frac{1}{n}\sum_{i:1}^{n} y_i^2\right) \tag{1}$$

In such problems, the target value of y is zero. The smallest value represents the highest signal/noise (S/N) ratio for the best case. For example, the quantity of interest may be surface roughness, tire wear process failure, *etc*. Where appropriate, the highest and best value can be determined as:

$$\frac{S}{N} = -10x \log \left(\frac{1}{n}\sum_{i:1}^{n} \frac{1}{y_i^2}\right) \tag{2}$$

In this case, the value of y is a non-negative measurable property with an ideal target of infinity. The largest value represents the S/N ratio for the best case. For example, power and efficiency, *etc*. Nominal best is when:

$$\frac{S}{N} = -10x \log \left(\frac{\bar{y}}{S^2{}_y}\right) \tag{3}$$

In this case, when there is a characteristic dual tolerance, the nominal value is the target. Therefore, if all parts are brought to this value, the variation will be zero and it is the best. It refers to the S/N ratio for the target value best case scenario.

Through estimating the effects of the factors considered in the experiments on the quality value, the quality value that can be obtained as a result of the optimum experimental parameters can be found. While determining the best and optimal levels of the factors, the hardness values should be the highest to improve the efficiency of the process. When determining hardness values, the largest, best function was used to calculate S/N ratios.

In this study, abrasives that do not harm human health and environmentally friendly paints were used. Chestnut wood is abraded with the natural abrasive $NaHCO_3$ under 1 bar, 2 bar, and 3 bar pressure, impregnated with $NaHCO_3$ at 5%, 7%, and 9% solutions and after the top surface treatment is applied with water-based varnish and paints for 3-6-9 months. The change in hardness values as a result of exposure to artificial outdoor conditions was investigated.

EXPERIMENTAL

Materials

In this study, Anatolian chestnut (*Castanea sativa* Mill.), one of the broad-leaved tree species, was used. The tree used were freshly cut from the Forest Management Directorate in Turkey. The trunk diameter was 1.30 m, the circumference of the crown area, and the tree lengths of the trees with the north direction marked were determined. Then, 2-m-long logs were cut from 2 to 4 m in line with the height of the trunk from the root. These logs were turned into planks by paying attention to the radial and tangential cross-section directions. In this study, $NaHCO_3$ compound, which is harmless to human health and environmentally friendly, was used both as an impregnation and abrasion agent.

The molecular mass of $NaHCO_3$ is 84.01 g/mol, its density is 2.22 g/cm^3 at 20 °C, and its solubility in water is approximately 95.5 g/L at 20 °C. A water-based varnish called Exterior was used. This varnish is a water-repellent, breathable, UV-resistant, and transparent product. Its density is 1.05 ± 0.02 g/cm^3 and it contains 35.6 ± 1% of its weight in solid matter. Its viscosity is 45 ± 5 at 20 °C. As for the paint, Hickson paint produced by Hemel company and decor Aqua wood water solvent paint called Stain were used. The paint used was water-repellent, breathable, resistant to UV rays, and translucent; it contains 38.8 ± 1% of its weight in solid matter. Its viscosity is 45 ± 5 at 20 °C.

Preparation of test samples

Test samples prepared in air-dry humidity according to the principles of ISO 3129 (2019) and TS 53 (1981) were kept in an air-conditioning cabinet at 20 ± 2 °C and a relative humidity of 65 ± 5% until they reached a constant weight. Samples for surface abrasion tests were of dimensions 300 × 75 × 10 ± 1 mm^3. For mechanical processing tests, samples were prepared from sapwood sections with net dimensions of 20 × 20 × 30 ± 1 mm^3 and 20 × 20 × 360 ± 1 mm^3, with annual rings perpendicular and tangent to the surfaces. Chestnut tree sapwood was preferred because it is easy to process and can be used in wooden building protection areas. In addition, the elastic structure of chestnut sapwood has been preferred in applications requiring structural strength and durability. Thanks to these properties, chestnut sapwood is frequently used in traditional wooden construction techniques and restoration projects. Finally, the samples were sanded with 240-grit sandpaper and made ready for testing.

Methods

Surface abrasion processes

The $NaHCO_3$-based dry media at different pressures (1 bar, 2 bar, and 3 bar) on each test sample prepared from chestnut wood in a private enterprise in Ankara Siteler furniture industry site, making the surface rough, is shown in Fig. 1.

Surface abrasion processes were carried out by spraying each test sample prepared from chestnut wood at different pressures (1 bar, 2 bar, 3 bar) with the help of a $NaHCO_3$ based dry median pressure gun in a private enterprise in Ankara Siteler furniture industry site, creating a rough surface as shown in Fig. 1.

Fig. 1. Weathered chestnut wood samples

Impregnation processes

The vacuum impregnation process impregnates the protective $NaHCO_3$ into the wood, making it last longer. This process helps prevent wood rot and fungal or insect damage. Even if sodium bicarbonate is used to provide superficial corrosion resistance, it may not need to be thoroughly impregnated. Instead, it may be recommended to apply it directly to the surface of the wood.

For the natural outdoor environment test, the air-dried test samples with abraded surfaces were impregnated with $NaHCO_3$ solutions prepared at 5%, 7%, and 9% concentrations using the vacuum pressure method, as shown in Fig. 2. After a pre-vacuum of 750 mm Hg was applied to the wood samples for 30 min, the impregnation material was taken into the cylinder and a pressure of 5 bar was applied for 60 min.

Fig. 2. Impregnation of test samples

Varnishing and painting of test samples

Water solvent wood paint and varnish (Hemel Varnish for Exterior VA1030) were applied to the front surfaces of the samples measuring 300 × 75 × 10 mm^3. The samples were prepared for the test in artificial outdoor conditions, in two layers using the brush application method, and by the company's recommendations. Approximately 250 g of paint was applied to 1.0 m^2 area in two layers. Then, the samples were lightly sanded by hand using 240-grit sandpaper, and a final coat of top surface material was applied. There was a 24-h drying time between each coat.

Artificial aging test

Accelerated aging tests were performed in a laboratory environment on test samples with abraded surfaces according to the principles of ASTM G151 (2019) and ASTM G154 (2023). After the tests, the results were compared with the control samples. The aging test was conducted with artificial light with an average irradiance level of 0.85 W/m^2 at 340 nm with the Atlas UV2000 Fluorescent UV test device to ensure the material's resistance to solar ultraviolet radiation. Test samples were exposed to accelerated aging for 120 h and 240 h with a cycle program consisting of UV light, 15 min of water spray, and 3 h and 45 min of concentration at an ambient temperature of 60 ± 2 °C.

Hardness resistance

As shown in Fig. 3, Shore A (Series 811) hardness values on samples were measured using a device from Mitutoyo company.

Fig. 3. Hardness measurement device

The scale range used was 0 to 100 Shore A in the device and the measurement range was 10 to 90 Shore A. Ten measurements were made according to ASTM D2240-15 (2021) with a 5 kg load applied on the hardness measuring device. The average of these measurements was taken and used in the Taguchi orthogonal array.

Taguchi experiment design

Artificial weathering conditions were used to determine the effect of abraded chestnut wood, impregnated with $NaHCO_3$, and surface treated (paint, varnish) for obtaining the hardness values. Data on the parameters and their response levels are shown in Table 1.

Table 1. Factors and Values at Different Levels

Factors		Response			
		1	2	3	4
A	Abrasion	Control	1 bar	2 bar	3 bar
B	Impregnation	0%	5%	7%	9%
C	Surface	Varnish	Paint	-	-

An orthogonal array was used to determine optimum conditions. Table 2 shows the hardness values measured before and after the experiment and the percentage changes in the values of the wood subjected to artificially aged conditions. Optimization was done based on these change percentage values.

Statistical Analysis

Minitab 21 software (Minitab LLC, State College, PA, USA) program was used for analyzing statistical data, evaluation data, creating equations, estimation, and for drawing graphs. L16 (4^2 x 2^1) order was used to create the sequences. Table 2 shows the experimental conditions, hardness values measured before and after weathering conditions, and the percent change in values. The optimization process was carried out by determining the hardness values and Brinell hardness number (H_B) percentage changes.

Table 2. Percentage Changes of H_B Values After Artificial Aging (3, 6, and 9 Months) Shown in L16 (4^2 x 2^1) Taguchi Sequence

Experiment	Factors			*H*B	Hardness Change (%)		
					3 Months	6 Months	9 Months
1	1	1	1	57.25	4.17	2.81	0.32
2	1	2	1	56.29	5.87	3.44	1.50
3	1	3	2	54.54	7.82	4.72	2.47
4	1	4	2	55.71	8.03	5.84	2.79
5	2	1	1	55.29	5.03	3.22	0.61
6	2	2	1	57.42	8.19	4.38	1.26
7	2	3	2	55.42	8.25	7.55	3.20
8	2	4	2	54.29	8.74	7.97	3.67
9	3	1	2	59.08	9.69	4.91	1.91
10	3	2	2	54.29	10.65	8.95	2.18
11	3	3	1	56.79	12.47	9.62	7.63
12	3	4	1	54.33	18.59	14.94	11.42
13	4	1	2	54.71	9.88	7.23	4.80
14	4	2	2	55.29	10.23	7.85	6.16
15	4	3	1	49.83	13.25	10.22	8.41
16	4	4	1	53.67	19.67	15.66	12.04

RESULTS AND DISCUSSION

To determine the effective and ineffective factors among the factors applied to the wooden material, the results obtained by calculating the average S/N ratios are shown on the graph in Fig. 4.

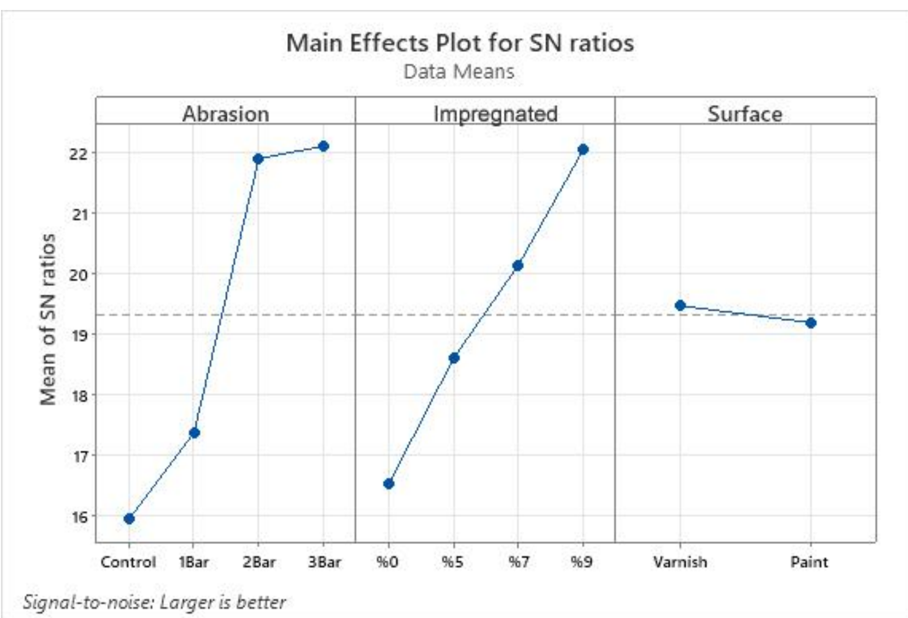

Fig. 4. Effect chart of chestnut wood S/N ratios

From these results, it was determined that the most effective set of conditions, relative to wear, involved a pressure of 3 bar and an abrasive level of 9% $NaHCO_3$. It was observed that artificially aged and impregnation solution applied surface treatments affected the hardness values of varnish and paint at the lowest level. After determining the parameters affecting the hardness values according to S/N ratios, their effects on the response values were investigated. For this purpose, an analysis of variance (ANOVA) was performed for assessing 3, 6, and 9 month hardness change rates on abrasion, impregnation, and surface treatment.

While the surface hardness of wooden material increases on impregnated and varnish-coated surfaces, a significant decrease in the hardness values of impregnated and varnish-coated test samples was observed due to the aging process in an accelerated ultraviolet environment (Gündüz *et al*. 2020). What makes varnish UV resistant are usually the chemicals present in it. These chemicals are designed to increase resistance to degradation caused by UV rays. UV rays can lead to deterioration of the material by weakening the bonds on the varnish surface and negatively affecting the color, texture, and durability of the material. The chemicals used in UV-resistant varnishes reduce these effects, prolong the life of the varnish and help protect the surface. These chemicals may include UV absorbers, UV stabilizers, and antioxidants. These ingredients increase the UV resistance of the varnish by reducing degradation caused by UV rays. Additionally, it was determined that the impregnation process caused an increase in the hardness values of the test samples. The hardness values of the test samples were low before the aging process. It was determined that the values increased as a result of the aging process because of the continuous hardening effect of the varnish layers (Ayata *et al.* 2021). Based on other studies, it is understood that impregnation and surface treatments generally increase the hardness values, and this is in parallel with the data obtained in this study.

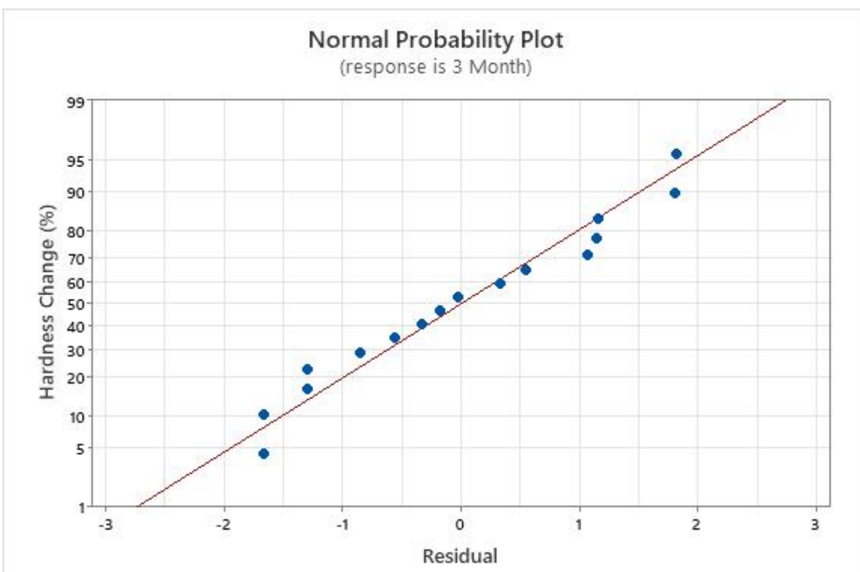

Fig. 5. Regression chart according to 3-month responses

To evaluate the 3 month hardness percentage change in terms of normality Normal Probability Plot graphs were drawn (see Fig. 5). The residual in the graph represents the average difference between the observed value and the predicted or fitted value. Residuals shown as points on the graph are normally distributed if they fall approximately along a straight line. If the residuals are not close to the straight line shown, they do not show a normal distribution (Teruo 2011). If the developed model is correct and the assumptions are met, there should be residual structures. In particular, it should not be related to any other variable, including the predicted response. Figure 5 shows that the residuals generally fell in a straight line, implying that the errors decayed normally, meaning that the experimental data came from a normal population. There was no abnormality observed as a result of the residuals. It is apparent, from Table 3, that the most effective factor in the 3-month percentage change in the hardness value was abrasion, and the factor with the lowest impact was the surface parameter.

Table 3. Taguchi Analysis of 3 Months *Versus* Abrasion; Impregnation; Surface Analysis of Variance

Source	D.F.	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Abrasion	3	148.65	53.70%	148.65	49.551	19.03	0.001
Impregnated	3	95.19	34.38%	95.19	31.729	12.18	0.002
Surface	1	12,16	4.39%	12.16	12.163	4.67	0.063
Error	8	20.83	7.53%	20.83	2.604		
Total	15	276.83	100.00%				

Table 4. Taguchi Analysis: General Linear Model: 6 Months *Versus* Abrasion; Impregnated; Surface

Source	D.F.	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Abrasion	3	103.052	46.83%	103.052	34.351	16.75	0.001
Impregnated	3	95.219	43.27%	95.219	31.740	15.48	0.001
Surface	1	5.371	2.44%	5.371	5.371	2.62	0.144
Error	8	16.406	7.46%	16.406	2.051		
Total	15	220.048	100.00%				

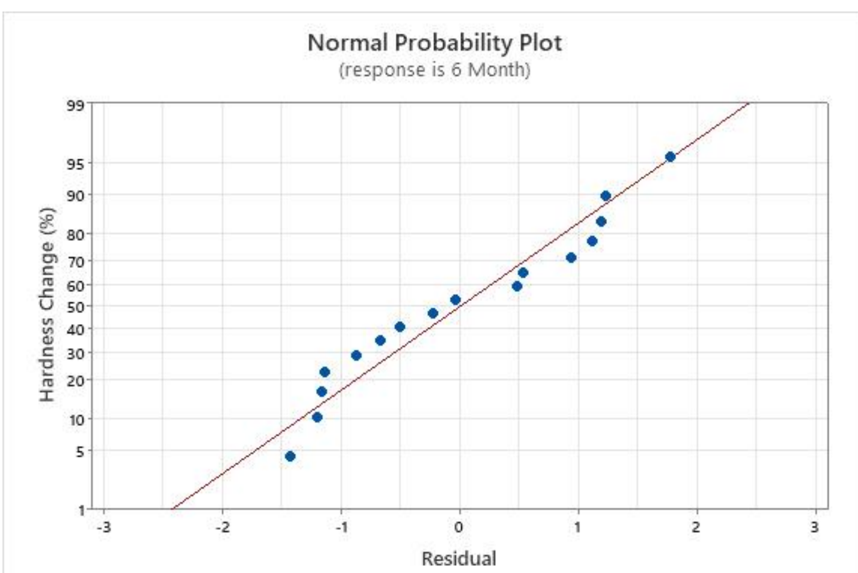

Fig. 6. Regression chart according to 6-month responses

From Table 4, it is understood that the most effective factor in the 6-month percentage change in the hardness value is the abrasion factor and the impregnation factor closest to it, and the factor with the lowest impact is the surface parameter. To evaluate the 6-month hardness change in terms of normality, a Normal Probability Plot graph was drawn (see Fig. 6). Residues were generally found to fall in a straight line. It turns out that errors decayed normally and the experimental data come from a normal population.

When Table 5 is examined, it is understood that the most effective factor in the 9-month hardness percentage change values was the abrasion factor, and the factor with the lowest effect was the surface parameter. To evaluate the 9 month hardness change in terms of normality, see Fig. 7. A Normal Probability Plot graph was drawn. The residues appeared to fall closely in a straight line. It turns out that errors decayed normally and the experimental data come from a normal population.

Table 5. Taguchi Analysis: 9 Months *Versus* Abrasion; Impregnated; Surface Analysis of Variance

Source	D.F.	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Abrasion	3	102.644	49.80%	102.644	34.215	27.58	0.000
Impregnated	3	77.531	37.61%	77.531	25.844	20.84	0.000
Surface	1	16.020	7.77%	16.020	16.020	12.92	0.007
Error	8	9.923	4.81%	9.923	1.240		
Total	15	206.119	100.00%				

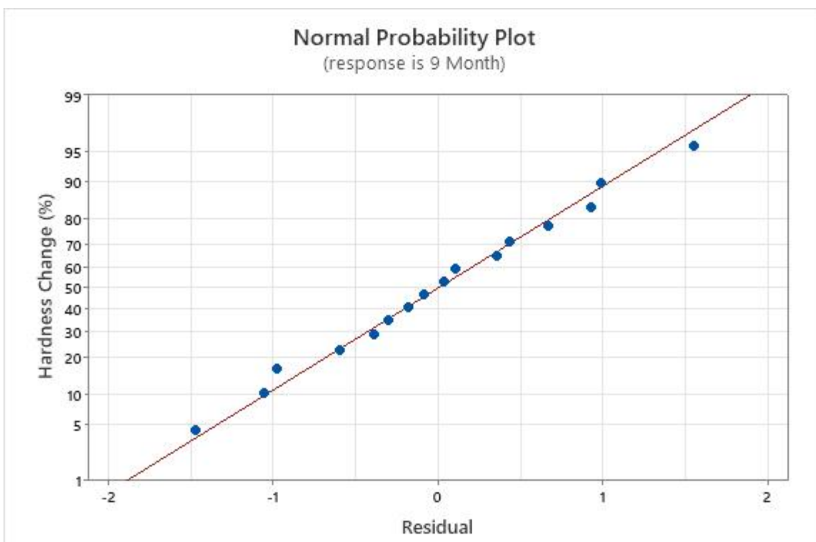

Fig. 7. Regression chart according to 9-month responses

Polynomial regression analysis

From ANOVA tests, the highest contribution percentage among the factors was calculated to be the wear parameter. Regression formulas in Table 4 were used to estimate the wear parameter using the percent hardness changes of the dependent variables at 3, 6, and 9 months, respectively.

The regression equations that best fit the data were as follows:

$$3\text{ Month} = 3.976 + 0.1780\text{ Predict 3 Month} + 0.03661\text{ Predict 3 Month}^2 \quad (4)$$

$$6\text{ Month} = 1.945 + 0.4182\text{ Predict 6 Month} + 0.03503\text{ Predict 6 Month}^2 \quad (5)$$

$$9\text{ Month} = 0.1469 + 0.9140\text{ Predict 9 Month} + 0.00731\text{ Predict 9 Month}^2 \quad (6)$$

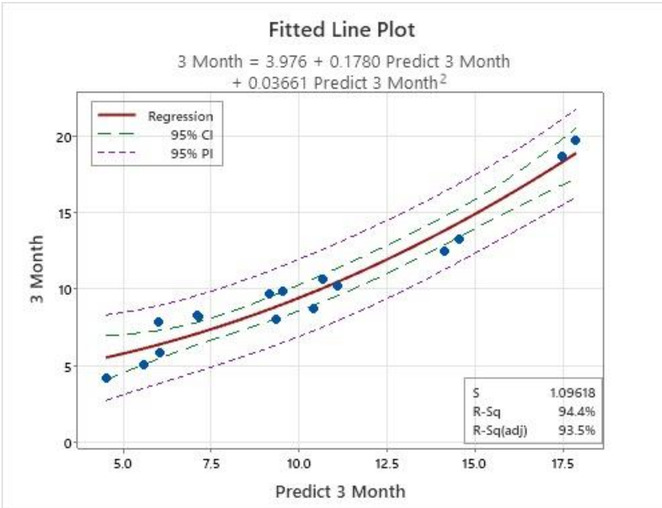

Fig. 8. Prediction of 3 months regression equation

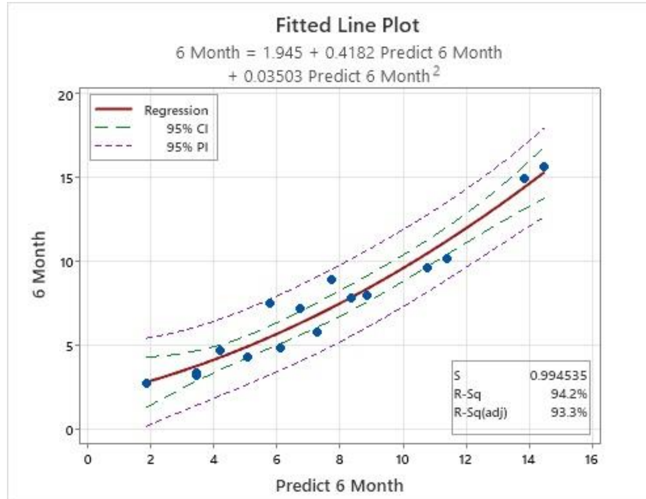

Fig. 9. Prediction of 6 months regression equation

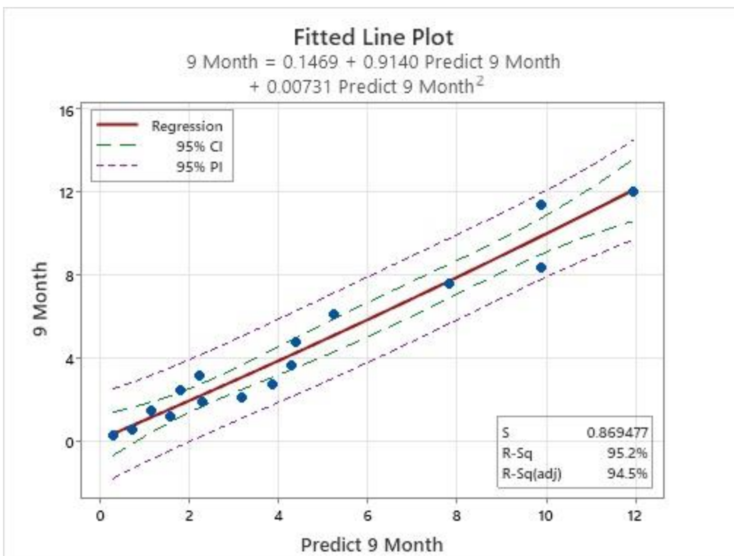

Fig. 10. Prediction of the 9-month regression equation

The statistical parameter S represent the standard deviation of the distance between the data values. S is measured in the units of the response. The value of S can be used to assess how well the model describes the response. The lower the value of S, the better the model describes the response. R-sq is the proportion of variability in the response explained by the equation. R-Sq was used to determine how well the model fit the data. The higher the R-Sq value, the better the model fits the data.

The 3-month change in surface estimate of the wear parameter was calculated as 94%. For the 6-month change it was calculated as 94% and for the 9-month change surface estimate was 95%. These rates are an indication that the model's accuracy was quite high.

CONCLUSIONS

Percentage changes in hardness values *via* abrasion, impregnation, and surface treatments after 3, 6, and 9 months of artificial aging periods were examined. The significance level of the obtained data was determined according to the Taguchi L16 orthogonal array experimental design.

1. According to the three dependent factors, 3, 6, and 9 months artificial aging hardness values, the most important factor was determined to be the abrasion parameter. Among these three factors, the lowest impact level was determined to be the surface treatment.
2. Hardness values were higher in samples without artificial aging. It is thought that the reason for this is due to the varnish and paints used on the surfaces. Varnishes create a protective layer on the surface of wooden materials and increase the hardness values.
3. It has been determined that as the artificial aging time increased, there was a decrease in hardness values due to the effect of UV rays. Regression equations were used to estimate the hardness values according to the abrasion factor, which was determined as the most effective factor. According to the regression equations obtained, it was observed that the S values of the data used in the estimation of 3-6 and 9-month hardness change rates were low and the R-sq values were above 94%. It is understood that the predictive power of the model created according to this situation was high.
4. The surface factor is one of the factors affecting hardness values; its effect and contribution levels to the model can be increased by substituting other parameters instead of paint and varnish parameters. It is recommended to remove non-significant factors and add new factors that were not included in this study as future studies.

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