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A FACTORIAL DESIGN APPROACH IN THE OPTIMIZATION OF WOOD THERMAL MODIFICATION PARAMETERS FOR FLOORING

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Abstract:
The thermal modification is a method to improve the dimensional stability of wood and enhance its decay resistance. Thermal modification process also contributes to uniform brown color to the wood. The improved properties make the thermally-treated wood a suitable product for flooring applications. This study focused on the optimization of the process parameters by applying a factorial design in order to determine the performance of thermally-treated wood for flooring indoors. Therefore, the ash (Fraxinus excelsior) and the silver birch (Betula Pendula) wood samples were exposed to thermal treatment at three different temperatures (170, 190, 210°C) and two different exposure times (2, 6h) and then, the dimensional stability, color change and hardness (Brinell hardness) which are important properties for use of wood in flooring indoors were determined. As a result, there are some advantages to the use of thermally-treated wood for flooring, as its lower swelling and more aesthetic appearance. The better results were also achieved with ash than with birch samples for flooring indoors. The thermal treatment results showed that the temperature exhibited the highest effect, sequentially followed by duration and wood species.

Key words: *color; dimensional stability; factorial design; flooring; hardness; thermally-treated wood.*

INTRODUCTION

The thermal modification is a process, which improves dimensional stability and decay resistance of wood (Hakkou *et al*. 2006, Ates *et al*. 2009, Şahin Kol and Kokten 2016). Thermal treatment of wood also contributes to uniform color change to darker, brownish tones (Mitsui *et al*. 2001, Hill 2006, Srinivas and Pandey 2012). Accordingly, thermally-treated wood is attractive to use for floorings.

The color, dimensional stability, hardness, brightness and surface roughness are important properties for use of wood in flooring indoors (Živković *et al*. 2008, Nejad *et al*. 2013, Gurleyen *et al*. 2017). Lower swelling and more aesthetic appearance of thermally-treated wood make the treated wood a suitable product for flooring applications. The higher value of wood hardness is also important in flooring in order to withstand impacts and scratches (Todaro 2012).

As previously reported, the hardness changes of the thermally-treated wood depend on treatment intensity (treatment temperature and duration), wood species and direction of the test (Shi *et al*. 2007, Sivrikaya *et al*. 2015). The higher treatment intensities result in a significant decrease in hardness, while changes in lower treatment conditions are insignificant (Poncsak *et al*. 2006, Korkut *et al*. 2008, Korkut and Guller 2008, Salca and Hiziroglu 2014).

This study focused on the optimization of the process parameters in order to determine the performance of thermally-treated wood for flooring indoors. Therefore, the ash (*Fraxinus excelsior*) and the silver birch (*Betula Pendula*) wood samples were exposed to thermal treatment at three different temperatures (170, 190, 210°C) and two different exposure times (2, 6h). Subsequently, the dimensional stability, color change and hardness (Brinell Hardness) which are important properties for use of wood in flooring indoors were determined.

Factorial experiments/designs are the suitable method used to determine the effects of experimental parameters and the interactions between those parameters (Tarley *et al*. 2009, Bingol *et al*. 2010). In this work, a full factorial design was employed in order to achieve the best overall optimization of the system. For this purpose, three factors (wood species, treatment temperature and duration) and three dependent variables (the dimensional stability, color change and hardness) were considered in the experiments.

MATERIAL AND METHODS

Materials

The ash (*Fraxinus excelsior*) and Silver birch (*Betula Pendula*) wood samples provided from Yenice (Karabük) location, Turkey were used as test materials. Special attention was taken for the selection of the

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test materials to avoid errors (non-deficient, knotless, without zone line, without reaction wood and without decay, insect, mushroom damages). The moisture content of the test samples before they were oven-dried was 11-13%. A total of seven variations per wood species including untreated (reference) and treated samples were prepared for each experiment.

Thermal modification process

The test samples were thermally-modified in an oven in which the temperature could be controlled (±1°C). The treatments were performed at three different temperatures (170, 190 and 210°C) and for two different period of times (2 and 6h) at atmospheric pressure in the presence of air. Once thermal treatment was finished, the samples (treated and untreated) were conditioned in a climate controlled room at 65±5% relative humidity and 20±2°C for 15 days to attain the equilibrium moisture content (EMC).

Dimensional stability test

Based on the Turkish Standards (TS 4084), fifteen swelling samples with dimensions of 20mm×20mm×30mm were tested for each group of treatment condition. For swelling tests, the samples were oven dried at 103±2°C until the oven-dry weight was reached. After the oven-dry dimensions were measured, the samples were placed in distilled water (20±2°C) until stable dimensions were obtained. Swelling of the samples (tangential and radial directions) was calculated according to equation (1):

$$\alpha = \left[\left(Wm - Dm \right) / Dm \right] \times 100 \quad [\%] \tag{1}$$

where: α: Swelling of the sample (%), Wm: Wet measurement of the sample (mm), Dm: Dry measurement of the same sample (mm).

Determination of color change

Color test samples (five pieces for each group of treatment condition) were prepared with dimensions of 100mmx50mmx20mm in axial, tangential and radial directions, respectively. Color tests were performed for untreated and treated samples. The three-dimensional (the lightness, the red-green share and the blue-yellow share) color space was used for the quantitative color assessment. The values measured after thermal treatment were compared with the values measured before treatment for each set of treatment condition. The total color change of the samples was calculated according to equation (2):

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \tag{2}$$

where: ΔE^*: The total color difference, ΔL^*: The lightness change on the same sample, Δa^*: The red-green share change on the same sample, Δb^*: The blue-yellow share change on the same sample.

Determination of hardness

Fourteen groups (treated and untreated) each of ten samples with dimensions of 50mm×50mm×50mm were prepared to examine the hardness. Hardness performance of the samples (tangential and radial directions) was determined in accordance to TS 2479 (Brinell Hardness) (TS 2479, 1976).

Factorial experiments/designs

The experimental factors and levels for thermal treatment process are shown in Table 1. The design was performed on a full factorial design with three factors. The main effects and interactions between factors were analyzed with the Minitab 16 software.

Table 1

Factors and levels

Factors	Levels		
Wood Species	Ash	Silver Birch	-
Duration (h)	2	6	-
Temperature (°C)	170	190	210

RESULTS AND DISCUSSION

Table 2 displays the descriptive statistics of the swelling, the color change and the hardness properties of the samples from two species as function of different treatment conditions. The swelling (tangential and radial directions) and the hardness (tangential and radial hardness) of the samples decreased with increasing thermal treatment intensity, which is a product of treatment temperature and duration. The total color change (ΔE) of the samples increased with increasing thermal treatment intensity. For this reason, the samples became darker with increasing treatment temperature and duration.

The dimensional stability of ash wood samples after thermal modification increased from 6% to 60% and its value for birch wood samples increased from 7% to 51%. Similar results were observed by Korkut and Guller (2008). They reported that tangential swelling of red-bud maple (*Acer trautvetteri* Medw.) samples decreased about 34% as a result of heat treatment with 180°C for 10h treatment.

The total color change of samples increased from 11.27 to 48.69 for ash wood and from 4.16 to 46.9 for birch wood as a function of treatment intensity. These findings were similar to the incidences reported in previous studies. Şahin Kol and Kokten (2016) found that the total color change of Oak (*Quercus robur*) wood had 42 when samples were treated at 230°C for 3h. They also reported that the dimensional stability of the samples increased about 52% at the same treatment conditions. In another study, Sivrikaya *et al*. (2015) determined that the total color change of ash (*Fraxinus* spp.) wood samples at 180°C for 1.5h was about 18 and its value at 210°C for 2h was about 34. Korkut *et al*. (2013) reported that when wild cherry (*Prunus avium*) wood samples were heat-treated having a temperature of 212°C for 2.5h, their ΔE value had about 36.

The hardness values after treatment decreased between 3% and 35% for ash wood, 7% and 36% for birch wood. Ates *et al*. (2009) found that calabrian pine (*Pinus brutia* Ten.) samples had 34% reduction in their tangential hardness values with exposure of 230°C for 8h. This finding is in good agreement with this study results.

Table 2

Descriptive statistics of swelling, color change and hardness properties of thermally-treated and untreated samples

		Ash							**Silver Birch**						
Duration (h)		**C**	**2**			**6**			**C**	**2**			**6**		
Temperature (°C)		**C**	**170**	**190**	**210**	**170**	**190**	**210**	**C**	**170**	**190**	**210**	**170**	**190**	**210**
Tangential Swelling (%) *α-T*	**M**	12.87	11.41	10.32	7.62	9.89	8.54	5.13	11.48	10.65	9.47	6.42	10.11	8.21	5.59
	SD	1.09	1.10	1.14	1.51	1.09	0.93	0.65	1.54	1.51	1.36	0.63	1.90	0.55	0.71
Radial Swelling (%) *α-R*	**M**	6.46	6.04	5.58	4.32	5.60	5.91	3.16	7.26	6.56	6.12	4.17	5.99	5.43	3.51
	SD	1.11	1.09	0.99	0.66	0.53	1.28	0.67	0.99	0.90	0.65	0.48	0.61	0.85	0.64
Color Change Δ*E**	**M**	-	11.27	14.18	42.48	29.74	34.2	48.69	-	4.16	14.66	37.8	8.55	26.06	46.9
	SD	-	1.92	1.09	2.35	1.49	2.72	1.55	-	1.01	1.72	1.36	2.16	2.96	1.25
Tangential Hardness (Kg/mm^2) *H-T*	**M**	3.35	3.23	3.01	2.85	3.15	2.94	2.26	3.40	3.15	2.91	2.84	2.79	2.95	2.26
	SD	0.61	0.57	0.21	0.39	0.40	0.27	0.16	0.43	0.39	0.20	0.39	0.54	0.27	0.17
Radial Hardness (Kg/mm^2) *H-R*	**M**	2.90	2.64	2.66	2.22	2.60	2.38	1.87	2.96	2.66	2.46	2.18	2.47	2.47	1.87
	SD	0.29	0.27	0.28	0.23	0.41	0.38	0.20	0.22	0.30	0.45	0.28	0.48	0.37	0.19

C: Control group, M: Mean, SD: Standard deviation

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The effects of thermal modification parameters on swelling of wood samples

Fig. 1 and Fig. 2 show the main effects of wood species, duration and temperature on swelling (tangential and radial directions) of thermally-treated samples. By evaluating main effects plot, it can be determined which level of related factor is optimum.

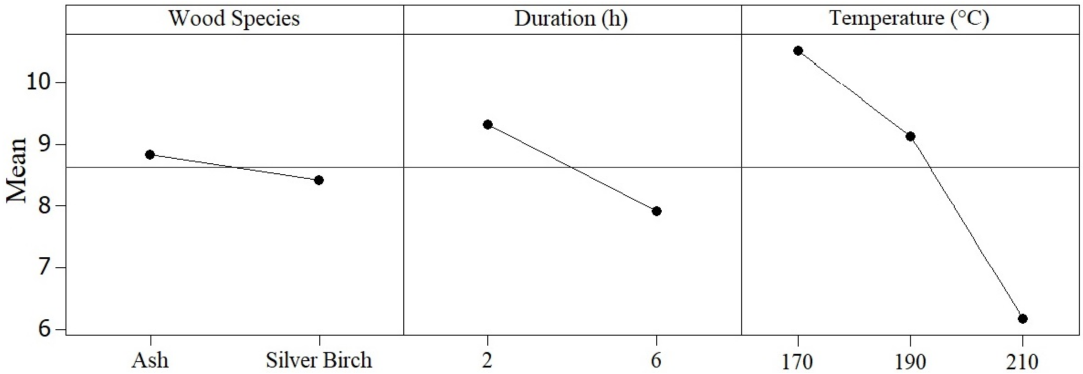

Fig. 1.
Main effects plot for tangential swelling (%).

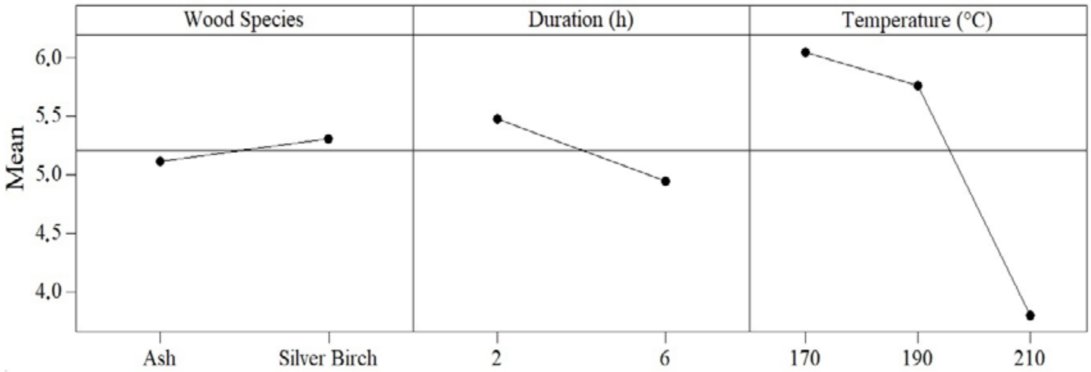

Fig. 2.
Main effects plot for radial swelling (%).

From the Fig. 1 and Fig. 2 it can be revealed that increase in duration and temperature resulted in the decrease in swelling.

The analysis of variance (ANOVA) results for tangential and radial swelling are shown in Table 3 and Table 4, respectively. The results showed that temperature exhibited the highest effect, sequentially followed by duration and wood species. Also, the effects of the factors on swelling were found to be statistically significant ($p \leq 0.05$).

According to factorial design results in the present study, the better swelling results were obtained by choosing the second level of duration (6h) and the third level of temperature (210°C) in minimizing swelling of samples.

Table 3

The analysis of variance for tangential swelling

Source	DF	Seq SS	Adj SS	Adj MS	F	P*
Wood Species	1	7.541	7.541	7.541	4.89	0.028
Duration (h)	1	88.434	88.434	88.434	57.29	0.000
Temperature (°C)	2	585.612	585.612	292.806	189.68	0.000
Wood Species*Duration	1	12.443	12.443	12.443	8.06	0.005
Wood Species*Temperature	2	0.831	0.831	0.416	0.27	0.764
Duration*Temperature	2	3.268	3.268	1.634	1.06	0.349
Wood Species*Duration* Temperature	2	2.432	2.432	1.216	0.79	0.456
Error	168	259.340	259.340	1.544		
Total	179	959.903				

*At least 95% confidence

Table 4

Analysis of variance for radial swelling

Source	DF	Seq SS	Adj SS	Adj MS	F	P*
Wood Species	1	1.688	1.688	1.688	2.52	0.114
Duration (h)	1	12.767	12.767	12.767	19.08	0.000
Temperature (°C)	2	181.530	181.530	90.765	135.67	0.000
Wood Species*Duration	1	0.520	0.520	0.520	0.78	0.379
Wood Species*Temperature	2	1.554	1.554	0.777	1.16	0.316
Duration*Temperature	2	4.005	4.005	2.002	2.99	0.053
Wood Species*Duration* Temperature	2	4.339	4.339	2.170	3.24	0.041
Error	168	112.392	112.392	0.669		
Total	179	318.795				

*At least 95% confidence

The effects of thermal modification parameters on color properties

Fig. 3 presents the main effects of wood species, duration and temperature on color change of thermally-treated wood.

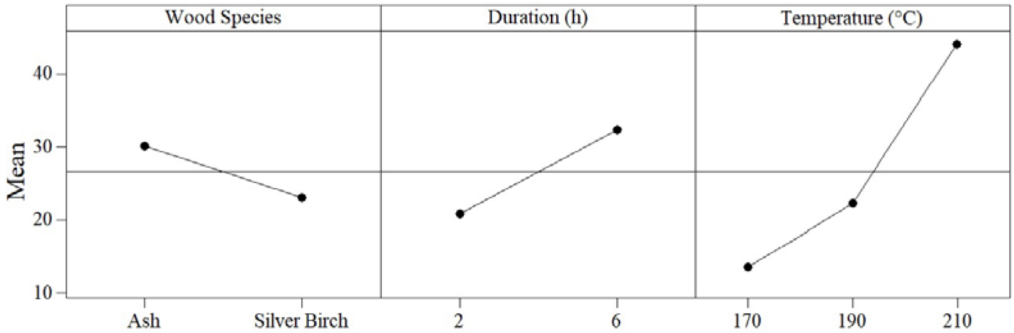

Fig. 3.
Main effects plot for color change.

It can be concluded that the increase in thermal treatment intensity resulted in the increase in color change (Fig. 3).

The analysis of variance (ANOVA) results for color change are shown in Table 5. The results showed that the most effective thermal treatment parameter is the temperature, sequentially followed by duration and wood species. In addition, the effects of the factors and its interactions on color change were found to be statistically significant.

The color of wood changed drastically when treatment temperature reached 210°C. The attractive brownish tones of thermally-treated wood were acquired at 190°C. Similar results were obtained in previous studies. Shi *et al*. (2011) reported that heat treatment resulted in a darkening of wood tissues and the darkening accelerated when treatment temperature exceeded approximately 200°C. Bekhta and Niemz (2003) found that the color of wood changed drastically after heat treatment at 200°C. Hidayat *et al*. (2015) reported that color change in sapwood slightly increased from 160°C to 180°C, and increased drastically from 180°C to 200°C. According to results of this study, the better color was obtained by choosing the second level of duration (6h) and the second level of temperature (190°C) in optimizing the color change of samples. Also, the better color change was achieved with ash than with birch samples.

Table 5

Analysis of variance for color change

Source	DF	Seq SS	Adj SS	Adj MS	F	P*
Wood Species	1	746.9	746.9	746.9	32.40	0.000
Duration (h)	1	2018.2	2018.2	2018.2	87.54	0.000
Temperature (°C)	2	9886.1	9886.1	4943.1	214.40	0.000
Wood Species*Duration	1	163.5	163.5	163.5	7.09	0.011
Wood Species*Temperature	2	377.7	377.7	188.9	8.19	0.001
Duration*Temperature	2	162.4	162.4	81.2	3.52	0.037
Wood Species*Duration* Temperature	2	187.6	187.6	93.8	4.07	0.023
Error	48	1106.7	1106.7	23.1		
Total	59	14649.2				

*At least 95% confidence

The effects of thermal modification parameters on hardness properties of wood samples

The main effect graphs for the hardness properties (tangential and radial hardness) of thermally-treated samples are presented in Fig. 4 and Fig. 5, illustrating the effects of the process parameters under different levels. From main effects plots, it is possible to determine the optimum levels of related factors.

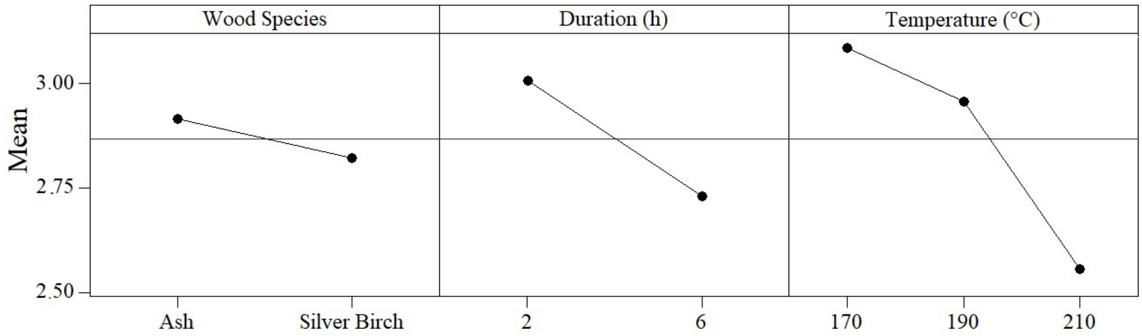

Fig. 4.
Main effects plot for tangential hardness (Kg/mm^2).

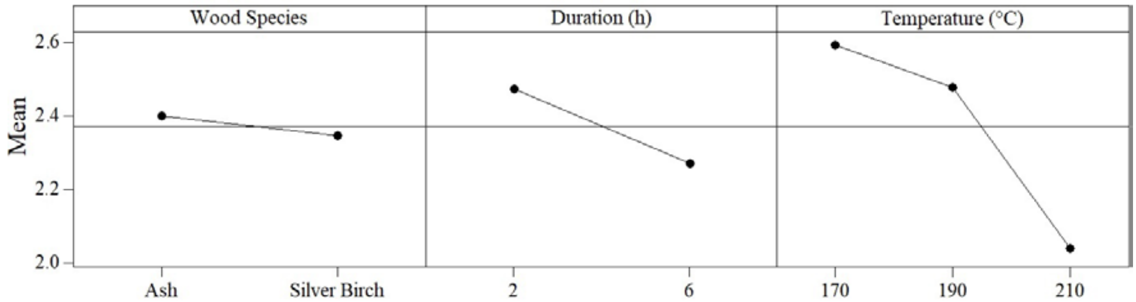

Fig. 5.
Main effects plot for radial hardness (Kg/mm^2).

From the Fig. 4 and Fig. 5, it can be revealed that the increase in thermal treatment intensity resulted in the decrease in hardness. It can be also declared that the hardness values of ash wood are greater than that of birch wood.

The analysis of variance (ANOVA) results for tangential and radial hardness are tabulated in Table 6 and Table 7, respectively. As shown in Table 6 and Table 7, the temperature is found to be the most significant factor influencing the hardness of thermally-treated wood. The effect of temperature on hardness was followed by duration and wood species, respectively. The effects of temperature and duration on hardness properties of samples were found to be statistically significant.

According to factorial design results in the present study, the better hardness results were obtained by choosing the first level of wood species (the ash wood), the first level of duration (2h) and the first level of temperature (170°C) in maximizing hardness of samples.

Table 6

Analysis of variance for tangential hardness

Source	DF	Seq SS	Adj SS	Adj MS	F	P*
Wood Species	1	0.2598	0.2598	0.2598	2.00	0.160
Duration (h)	1	2.2770	2.2770	2.2770	17.56	0.000
Temperature (°C)	2	6.1238	6.1238	3.0619	23.61	0.000
Wood Species*Duration	1	0.0249	0.0249	0.0249	0.19	0.662
Wood Species*Temperature	2	0.2447	0.2447	0.1224	0.94	0.392
Duration*Temperature	2	1.6560	1.6560	0.8280	6.39	0.002
Wood Species*Duration* Temperature	2	0.2102	0.2102	0.1051	0.81	0.447
Error	108	14.0042	14.0042	0.1297		
Total	119	24.8006				

*At least 95% confidence

Table 7

Analysis of variance for radial hardness

Source	DF	Seq SS	Adj SS	Adj MS	F	P*
Wood Species	1	0.0851	0.0851	0.0851	0.75	0.390
Duration (h)	1	1.2490	1.2490	1.2490	10.96	0.001
Temperature (°C)	2	6.8490	6.8490	3.4245	30.04	0.000
Wood Species*Duration	1	0.0148	0.0148	0.0148	0.13	0.720
Wood Species*Temperature	2	0.0232	0.0232	0.0116	0.10	0.903
Duration*Temperature	2	0.2478	0.2478	0.1239	1.09	0.341
Wood Species*Duration* Temperature	2	0.1763	0.1763	0.0881	0.77	0.464
Error	108	12.3129	12.3129	0.1140		
Total	119	20.9579				

*At least 95% confidence

The optimum levels of thermal modification parameters

The optimal parameter conditions are presented in Table 8.

Table 8

Optimal levels of factors

	Factors		
	Wood species W	Duration (h) D	Temperature (°C) T
Tangential swelling	The birch	6	210
Radial swelling	The ash	6	210
Color	The ash	6	190
Tangential hardness	The ash	2	170
Radial hardness	The ash	2	170

Both wood species were found to be suitable for flooring applications, however, it should be specified that better results were acquired with ash than with birch samples.

CONCLUSIONS

This study focused on the optimization of the process parameters by applying a factorial design in order to determine the performance of thermally-treated wood for flooring indoors. The following conclusions can be obtained:

- The color, dimensional stability, hardness, brightness and surface roughness are important properties for use of wood in flooring indoors.
- There are some advantages to the use of thermally-treated wood for flooring, as its lower swelling and more aesthetic appearance.
- The better results were achieved with ash than with birch samples for flooring indoors.
- The thermal treatment results showed that the temperature exhibited the highest effect, sequentially followed by duration and wood species.
- The better swelling results were obtained by choosing the second level of duration (6h) and the third level of temperature (210°C) in minimizing swelling of samples.
- The better color was obtained by choosing the second level of duration (6h) and the second level of temperature (190°C) in optimizing the color change of samples.
- The better hardness results were acquired by choosing the first level of duration (2h) and the first level of temperature (170°C) in maximizing hardness of samples.
- Thermal treatment at 210°C for 6h is recommended in variable climatic (room) conditions in order to ensure maximum dimensional stability. Additionally, treatment at 190°C for 6h can be recommended for flooring indoors due to its aesthetic appearance and moderate hardness level.

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