https://doi.org/10.22320/s0718221x/2025.18

ARTÍCULO

Physical and mechanical aging of wood-plastic composites. Non-destructive methods for quality control♣

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♣ This article is an extended and peer-reviewed version of the paper presented first at the III Congreso Ibero-Latinoamericano de la Madera en la Construcción (CIMAD), held in Madrid-Spain, june 2024. Associate-Editor: Eva Hermoso

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Abstract:

Wood-plastic composites have emerged as important new wood-based materials due to their environmental benefits, economic advantages, and recyclability. However, when used outdoors, they are exposed to fluctuating moisture and temperature conditions. Understanding the effects of climate aging on these composites is essential.

This study investigates the relationship between laboratory aging, physical and mechanical changes, and the outcomes of non-destructive testing. A total of 45 composite specimens containing 60 % wood fiber, 35 % low-density polyethylene, and 5 % additives were tested. A laboratory aging process, consisting of water immersion at 20 ºC and 50 ºC for a total of 56 days, was applied to the specimens. During the aging process, several batches of specimens were extracted and tested to measure their physical (density) and mechanical properties (bending strength and modulus of elasticity). Non-destructive testing, including ultrasound and stress wave devices, a screw withdrawal resistance meter, and a penetration tester, were employed. Results indicated that temperature had a greater influence on the WPC deterioration than humidity. A decrease in density (2 % - 4 %) and a significant reduction in mechanical properties (20 % - 60 %) were observed. The non-destructive methods used proved to be reliable estimators of composite properties, especially ultrasound wave propagation, confirming previous findings on other materials.

Keywords: Penetration tester, screw withdrawal force, stress wave, ultrasound, wood-plastic composites, non-destructive testing, aging tests, mechanical and physical aging, mechanical degradation

Received: 06.09.2024
Accepted: 31.01.2025

Introduction

Wood-polymer composites (WPCs) are used in a wide variety of applications, primarily for outdoor use, such as decking, railing, and fencing (Smith and Wolcott 2006). When used outdoors, WPCs are subjected to fluctuating moisture and temperature conditions, as well as decay fungi and solar (UV + IR) radiation. WPCs are a combination of wood in the form of fibers or particles and a thermoplastic matrix, typically polyethylene (PE), polyvinyl chloride (PVC), or polypropylene (PP). Since the wood component is hydrophilic and the thermoplastic is hydrophobic, moisture sorption in these composites primarily occurs in the wood phase (Smith and Wolcott 2006).

The initial advantage in terms of dimensional stability and moisture resistance may be less significant when considering the impact of sunlight, as well as the thermal expansion and softening of the material at high temperatures. However, a study on the effects of moisture and heat on the mechanical properties of wood and wood-plastic composites indicated that WPCs are a significantly improved product compared to wood derivatives (Mubarak and Idriss 1993). Given the fast-growing market for current and new WPC products, the extended warranties on existing products, and the critical need for long-term performance in certain applications, more research is required to quantify the long-term performance of WPCs under outdoor weather conditions.

Static methods are currently used to evaluate the properties of wood-plastic composites. These methods generally depend on wood filler/fibre content, and use standards for wood-based composites (when the wood filler content exceeds 50 %) or for plastics and reinforced plastics (when the wood filler content is below 50 %) to assess the properties of WPCs. ASTM D7031-11 (2019) and the EN 15534 (2018) were developed for the physical and mechanical properties of wood-plastic composite products. Although these static methods are precise, they are costly and destructive, in contrast to non-destructive, fast, easy, and flexible techniques. Non-destructive evaluation (NDE) techniques, which minimize defects and ensure quality control of the final product, can be effective methods for assessing WPC properties (Najafi *et al.* 2008).

There are various methods to evaluate materials or components, including non-destructive methods, which have many applications. The field of non-destructive evaluation (NDE) or non-destructive testing (NDT) involves identifying and characterizing damage on the surface and interior of materials without altering them (Lockard 2015). In other words, NDT refers to the process of evaluating and inspecting materials or components to characterize them or identify defects and flaws, comparing them to certain standards, without altering their original properties or harming the object being tested. NDT techniques offer a cost-effective means of testing individual samples or can be applied to entire materials as part of a production quality control system (Gholizadeh 2016). Numerous studies have been conducted to verify the effectiveness of non-destructive methods for estimating the physical and mechanical properties of materials. When applied to wood materials, such as various types of wood based pannels, these studies generally yield successful results, as demonstrated by Chung and Wang (2019) with particleboard, Bobadilla *et al.* (2012) with particleboard and fibreboard, Mirbolouk and Roohnia (2015) with dry process fibreboard (MDF), Haseli *et al.* (2020) with heterogeneous sandwich panels (blockboard and battenboard), and Zhang *et al.* (2021) with cross-laminated timber exposed to harsh environments, among others.

However, there are very few studies on NDT in composites, and the results obtained are inconclusive. Ultrasound testing has produced inconclusive results in estimating composite properties (Nesvijski 2000). Stress wave testing was used to determine the modulus of elasticity (MOE) of composites, but the authors concluded that this method was not suitable for estimating MOE (Nzokou *et al.* 2006). Wood flour and glass fibre content influenced a 16 kHz wave velocity in polypropylene wood composites (Najafi *et al.* 2008), and ultrasonic longitudinal and shear wave velocity measurements enabled the determination of Poisson's ratio for an orthotropic composite produced with bagasse fibre and polypropylene (Bader *et al.* 2016). The aim of this study is to establish whether it is possible to estimate the physical and mechanical properties of WPCs using non-destructive methods and to determine the behavior of the composite after applying a laboratory aging test.

Materials and methods

This study aims to analyze the influence of artificial aging on the basic properties of the material, as well as on certain secondary properties determined through non-destructive methodologies. The goal is to correlate these indirect and non-destructive measurements with the actual characteristics of the material, whose determination would require standardized destructive tests.

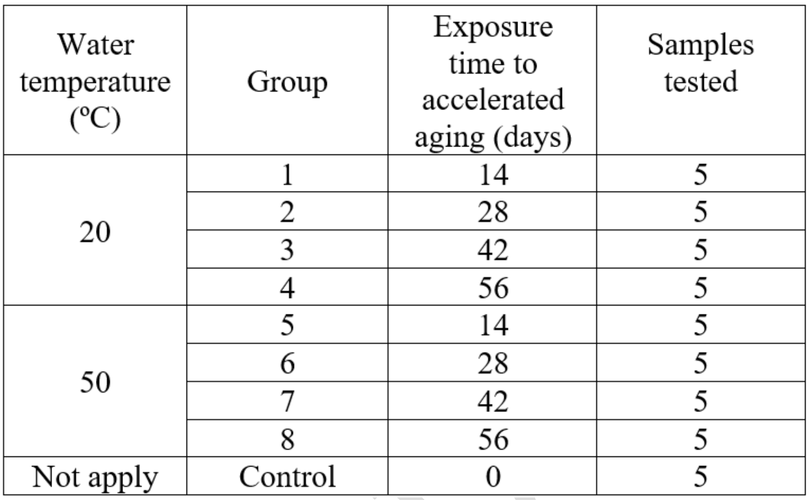
Specimens

Forty-five 210 mm x 50 mm x 8 mm longitudinal orientation pieces of composite with 60 % wood fibre, 35 % low-density polyethylene (LDPE) and 5 % additives from a commercial source were tested. All the test pieces were obtained from the same manufacturing batch.

Before the aging process, the samples were conditioned in a climate chamber at a temperature of 20 ºC ± 2 °C and 65 % ± 5 % relative humidity (RH) until constant mass.

The accelerated aging procedure consists of water immersion, in which 20 samples were immersed in water at 20 ºC, and another 20 samples at 50 ºC, divided into eight groups of five samples. For each bath temperature, one group was extracted every 14 days, and non-destructive methods and standard tests were performed (Table 1). Specimens were tested after removal from the bath and conditioned until constant mass in a climate chamber (20 ºC ± 2 °C and 65 % ± 5 % RH). Finally, five unaged samples also conditioned until constant mass in a climate chamber (same conditions) were tested as a control group.

Table 1: Summary of treatments, exposure time and number of specimens tested in the overall test process.



Water temperature (ºC)	Group	Exposure time to accelerated aging (days)	Samples tested
20	1	14	5
	2	28	5
	3	42	5
	4	56	5
50	5	14	5
	6	28	5
	7	42	5
	8	56	5
Not apply	Control	0	5

Standard testing

Since the WPC tested is composed of 60 % lignocellulosic material (> 50 %), following the guidelines mentioned in the introduction, and to be able to compare the results obtained with those studies conducted on other wood-based products, and given that the testing methodology differs very little, it has been decided to use the European standards for testing wood-based panels. Density was thus determined according to European Standard UNE EN 323 (1993); moisture content was determined according to UNE EN 322 (1994); and MOR and MOE were determined according to UNE EN 310 (1994).

Non-destructive testing

The non-destructive techniques used for estimating the physical and mechanical properties were divided into two groups:

Method of longitudinal propagation waves, through the time-of-flight of an ultrasonic wave with the Sylvatest Duo ultrasound device by CBS-CBT equipped with conical 22 kHz transducers and the time-of-flight of stress waves with the microsecond timer (MST) impact stress wave device by Fakopp Enterprise (Figure 1a, Figure 1b). The wave propagation speed has been measured in the longitudinal direction of the samples.

Probing methods for gauging pullout resistance were performed using a screw withdrawal force meter (SWFM) by Fakopp Enterprise. The device was equipped with a yellow zinc-plated 4x70 mm standard Heco-Fix plus screw, inserted to a penetration depth of 20 mm, allowing the screw to pass through the 8 mm thick specimen without pre-drilling. Penetration resistance was tested using the Pilodyn 6J penetrometer by Proceq, which consists of a calibrated spring that propels a 2.5 mm diameter steel needle with a constant energy of 6J. The depth of the needle's penetration into the material was then measured for analysis (Figure 1c and Figure 1d).

Since some tests could interfere with others, the order of the tests was as follows: density, time of flight of ultrasonic wave with the Sylvatest Duo; time of flight of stress waves with MST; flexural strength modulus (MOR); flexural modulus of elasticity (MOE); pullout resistance; penetration resistance; and moisture content.

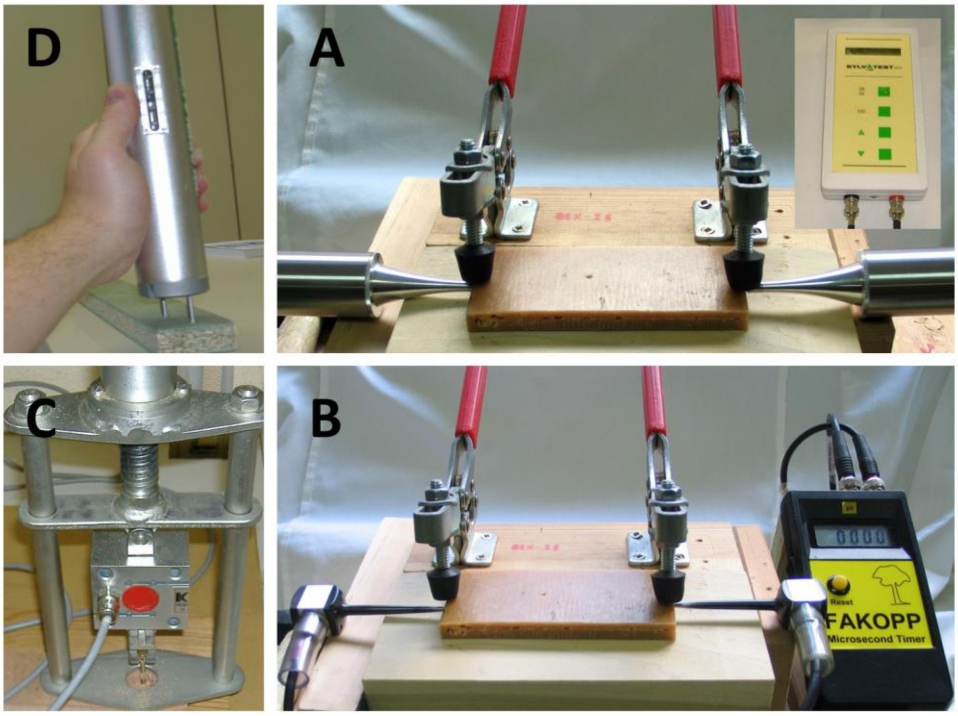


Figure 1: Detail of devices and test method for: A 22 kHz ultrasound, B stress wave, C screw withdrawal and D penetrometer.

Statistical analysis

Using Statgraphics Centurion 18 (Version 18.1.14) software, a statistical analysis was carried out on the results of the destructive and non-destructive tests. First, evolution graphs were plotted and an ANOVA test between the different treatment levels was analysed. Finally, regression models were proposed to estimate the physical and mechanical properties of the boards using the results of the non-destructive tests as estimators. The normality of the data populations was verified previously.

Results and discussion

Table 2 and Figure 2 show the trends of the variables measured throughout the two processes of aging immersion and after a week's conditioning; and whether or not there are statistical differences between the data groups.

The results indicate that the influence of the aging immersion at the higher temperature is much more marked, and the variations in the physical and mechanical properties are clearer in the case of the highest immersion temperature. Thus, the immersion process at 20 °C produced losses in density of less than 2 %, MOR less than 20 %, and MOE slightly higher than 40 %, but not always with statistical differences between treatment phases, as can be seen on Table 2; however, at 50 °C these losses are practically double, specifically about 4 % for density, over 50 % for MOR, and 60 % for MOE, this time with clear statistical differences.

In the context of a Wood-Plastic Composite (WPC), the term "matrix" refers to the plastic or polymeric component that binds the wood fibers or particles together, forming the composite material. The decrease in mechanical strength could be due to the degradation of the fibre and fibre-matrix interfacial bonding (Beg and Pickering 2008). The artificial aging of the wood/plastic composites caused matrix cracking and a wood-matrix delamination, having a negative effect on their physical and mechanical properties (Segerholm *et al.* 2012).

The results of this work are also consistent with many experimental studies on the effect of temperature on the mechanical properties of wood composites, which showed a significant decrease in mechanical strength at temperatures higher than 50 °C, which its authors attribute to the thermal softening of the polymer matrix and the weaken of interfacial bonding due to creep deformation (Pulngern *et al.* 2016).

Table 2: General mean results for density, modulus of rupture (MOR), modulus of elasticity (MOE), stress wave velocity (MST), ultrasonic 22 kHz velocity (US22), Pilodyn penetration (PIL) and screw withdrawal resistance (SWRM).

Water treatment	Days	Density (kg/m^3)	MOR (MPa)	MOE (MPa)	Vel US22 (m/s)	Vel MST (m/s)	SWRM (N/mm)	PIL (mm)
No Treatment	0	1422 aA	39 aA	4529 aA	2724 aA	2070 aA	201 aA	2,45 aA
20 ºC	14	1417 a	35 b	3935 b	2712 a	2081 a	177 b	2,50 a
	28	1414 a	34 bc	3540 c	2678 a	1934 b	193 c	3,05 b
	42	1410 a	33 cd	3330 c	2608 b	1986 c	188 c	2,80 bc
	56	1411 a	32 d	2654 d	2457 c	1988 c	187 bc	2,65 ac
50 ºC	14	1396 B	31 B	3081 B	2549 B	2087 A	180 B	2,75 A
	28	1392 C	25 C	2920 B	2433 C	1861 B	170 C	3,55 B
	42	1383 CD	22 D	2432 C	2213 D	1648 C	160 D	3,85 B
	56	1371 D	19 E	1716 D	2006 E	1596 C	145 E	4,75 C

The letters next to the numbers indicate statistical differences between groups. Different letters imply statistical differences. Lowercase letters are used for the first group (20 ºC) and uppercase letters for the second (50 ºC). The untreated samples belong to both test groups, so they are assigned both letters, upper and lower case.

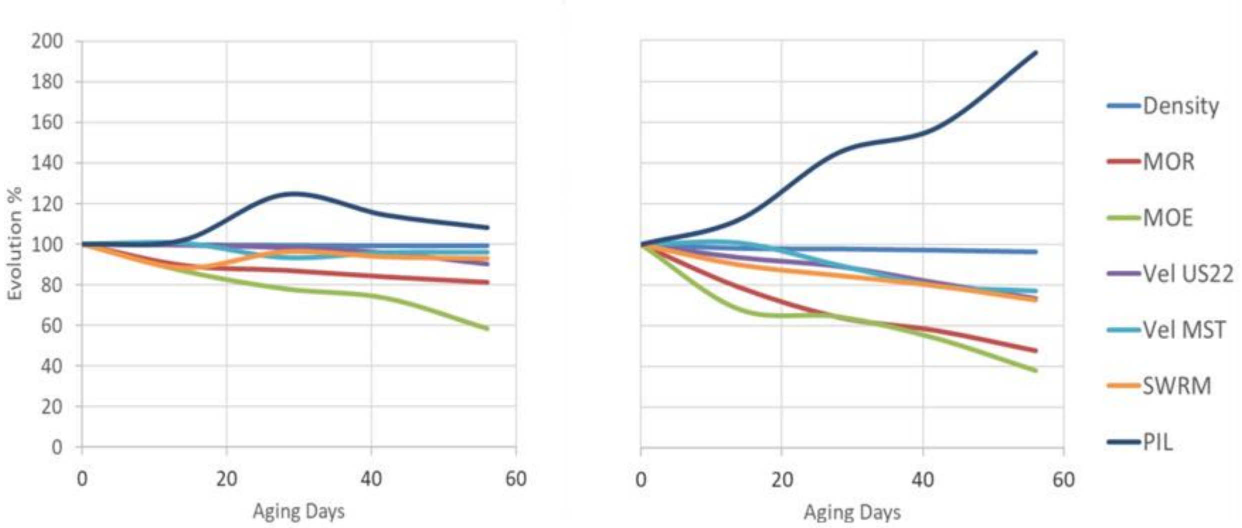


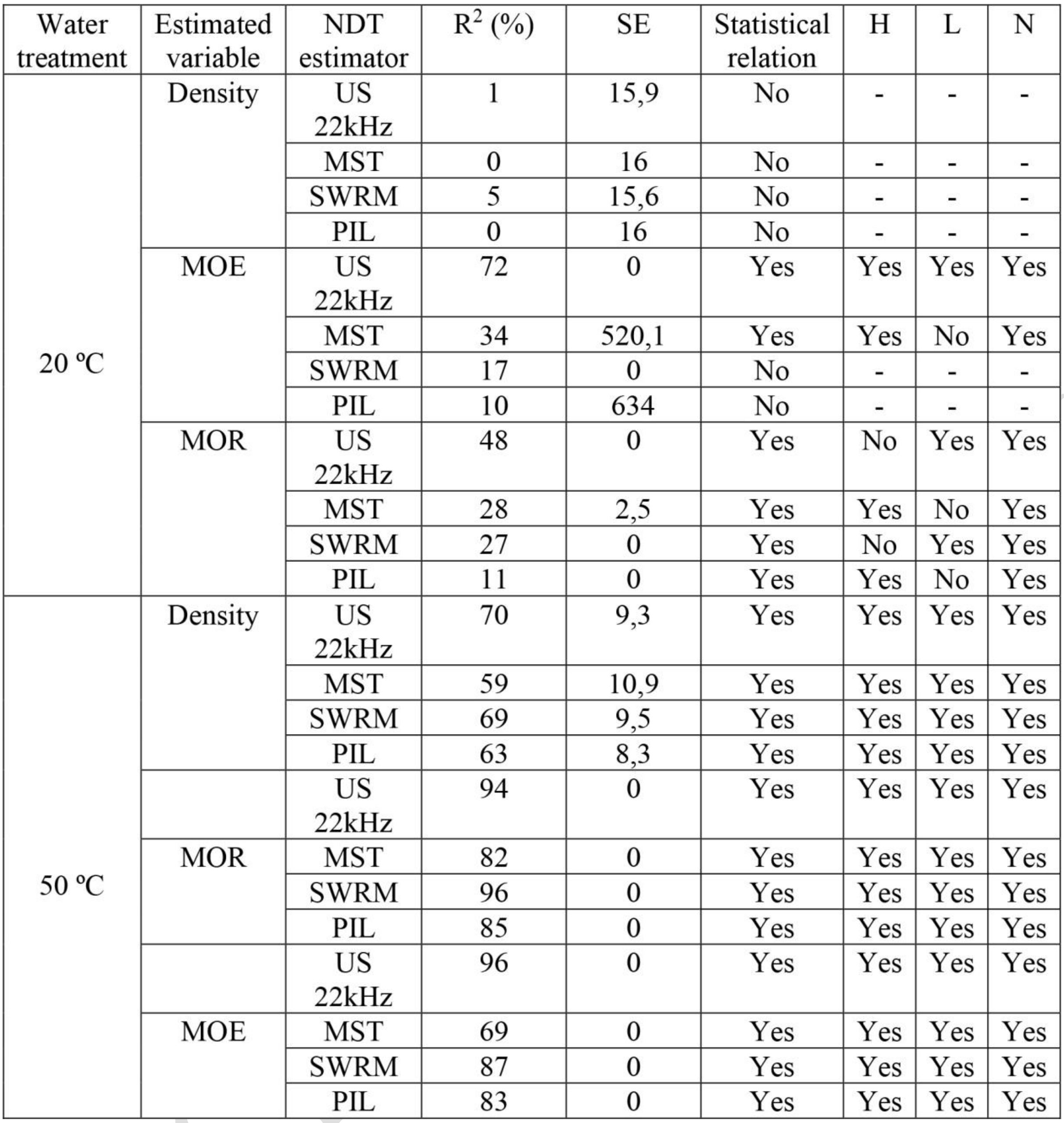
Figure 2: Evolution of the measurements taken during aging caused by immersion, calculated in percentage terms (variation in % relative to the reference value without aging). On the left, the evolution during aging at 20 °C is shown, on the right at 50 °C. Physical (density), mechanical (MOR and MOE) and non-destructive measurements (ultrasound wave velocity VelUS22, stress wave velocity VelMST, screw withdrawal force meter SWFM and penetrometer PIL).

After the treatment in water at 20 °C, the prediction models obtained to estimate the physical or mechanical properties using non-destructive parameters rarely meet the statistical criteria; and when they do, they are not very reliable, with poor coefficients of determination and in many cases with no significant statistical relationship. Only ultrasound waves give relatively good results in the MOE estimation, since as with other materials such as particle- or fibreboards, the propagation velocity of this type of waves is usually a good estimator of the elastic properties of the material (Bobadilla *et al.* 2009). Moreover, none of the non-destructive methods tested allows the estimation of the three control parameters, namely density, MOR and MOE (Table 3).

However, the data obtained after the hot water treatment indicate that most non-destructive methods allow a reasonably reliable estimation of the control parameters, with coefficients of determination ranging from 59 % to 96 %. Table 4 briefly summarizes these models, in which, as in previous works with other materials such as particle-, fibre- and oriented strand boards, all under artificial aging, the ultrasound wave velocity presents the best behaviour (Bobadilla *et al.* 2009, Bobadilla *et al.* 2011), closely followed by stress wave and screw withdrawal methods. These previous works on different boards produced similar results to the current study with the difference that in the last, stress waves had more modest results in the estimation compared to ultrasonic waves and even probing methods.

Figure 3 shows the graphs of the estimation models for the control parameters with ultrasound velocity. The proposed models for estimating MOR, MOE, and density using the ultrasonic equipment are shown. As can be observed, the point cloud fitting is stronger in the MOR and MOE models, resulting in narrower confidence intervals. This is logical given the very high coefficients of determination and negligible standard errors (Table 4). In contrast, the density estimation model is more modest, displaying a more dispersed point cloud and wider confidence intervals. It is important to emphasize that these models, even the most modest one, enable the estimation of the material's basic properties and its characterization in a quick, simple, and cost-effective manner.

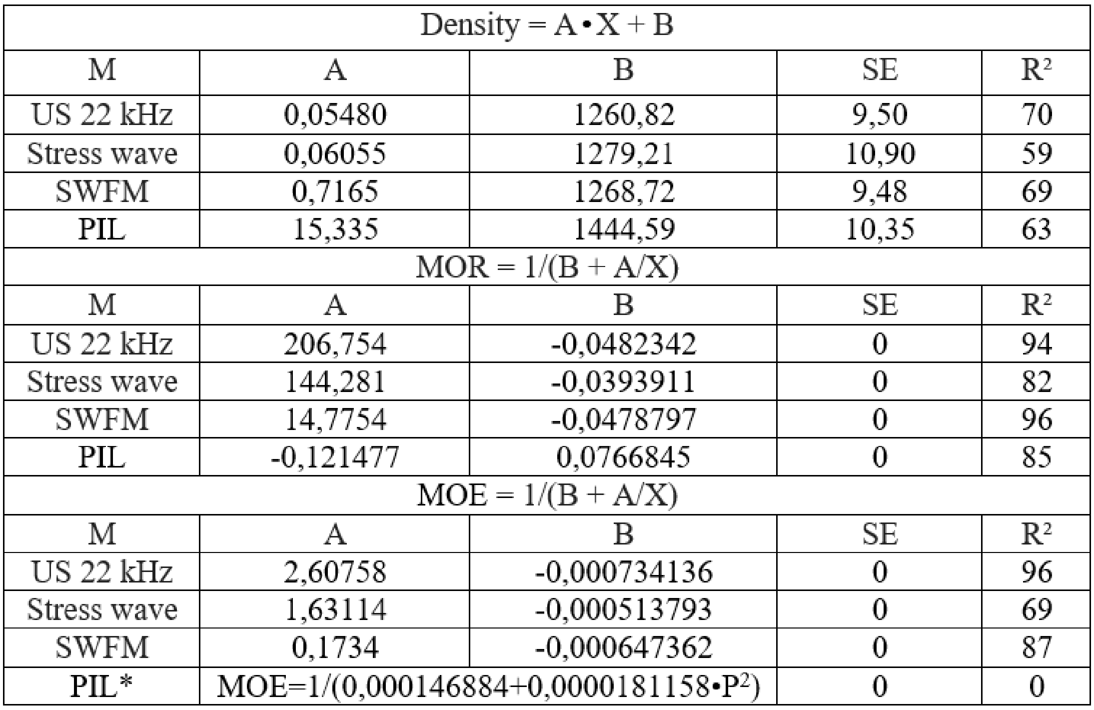
Table 3: Behaviour of estimation models performed with aging data at 20 ºC and 50 ºC.



Water treatment	Estimated variable	NDT estimator	R^2 (%)	SE	Statistical relation	H	L	N
20 ºC	Density	US 22kHz	1	15,9	No	-	-	-
		MST	0	16	No	-	-	-
		SWRM	5	15,6	No	-	-	-
		PIL	0	16	No	-	-	-
	MOE	US 22kHz	72	0	Yes	Yes	Yes	Yes
		MST	34	520,1	Yes	Yes	No	Yes
		SWRM	17	0	No	-	-	-
		PIL	10	634	No	-	-	-
	MOR	US 22kHz	48	0	Yes	No	Yes	Yes
		MST	28	2,5	Yes	Yes	No	Yes
		SWRM	27	0	Yes	No	Yes	Yes
		PIL	11	0	Yes	Yes	No	Yes
50 ºC	Density	US 22kHz	70	9,3	Yes	Yes	Yes	Yes
		MST	59	10,9	Yes	Yes	Yes	Yes
		SWRM	69	9,5	Yes	Yes	Yes	Yes
		PIL	63	8,3	Yes	Yes	Yes	Yes
		US 22kHz	94	0	Yes	Yes	Yes	Yes
	MOR	MST	82	0	Yes	Yes	Yes	Yes
		SWRM	96	0	Yes	Yes	Yes	Yes
		PIL	85	0	Yes	Yes	Yes	Yes
		US 22kHz	96	0	Yes	Yes	Yes	Yes
	MOE	MST	69	0	Yes	Yes	Yes	Yes
		SWRM	87	0	Yes	Yes	Yes	Yes
		PIL	83	0	Yes	Yes	Yes	Yes

R^2 is the coefficient of determination, SE the standard error, H the homoscedasticity, L the linearity and N the normality of the residuals of the variable. Statistically valid models are marked in bold. NDT estimators are ultrasonic device (US22 kHz), stress wave device (MST), screw withdrawal device (SWRM) and penetration device (PIL).

Table 4: Linear and double inverse models to estimate density, MOR and MOE using ultrasound, stress wave, screw withdrawal resistance and Pilodyn after aging in water at 50 ºC.



Density = A•X + B				
M	A	B	SE	R^2
US 22 kHz	0,05480	1260,82	9,50	70
Stress wave	0,06055	1279,21	10,90	59
SWFM	0,7165	1268,72	9,48	69
PIL	15,335	1444,59	10,35	63
MOR = 1/(B + A/X)				
M	A	B	SE	R^2
US 22 kHz	206,754	-0,0482342	0	94
Stress wave	144,281	-0,0393911	0	82
SWFM	14,7754	-0,0478797	0	96
PIL	-0,121477	0,0766845	0	85
MOE = 1/(B + A/X)				
M	A	B	SE	R^2
US 22 kHz	2,60758	-0,000734136	0	96
Stress wave	1,63114	-0,000513793	0	69
SWFM	0,1734	-0,000647362	0	87
PIL*	MOE=1/(0,000146884+0,0000181158•P^2)		0	0

Where: density (kg/m^3), MOE= modulus of elasticity (N/mm^2), MOR= modulus of rupture (N/mm^2), X=value obtained with the equipment used (waves m/s, SWRM N/mm, penetrometer mm) and A, B are constants. R^2 is the coefficient of determination and SE the standard error.

*The double inverse model does not meet the hypotheses of linearity and homoscedasticity, so different model is included for this device.

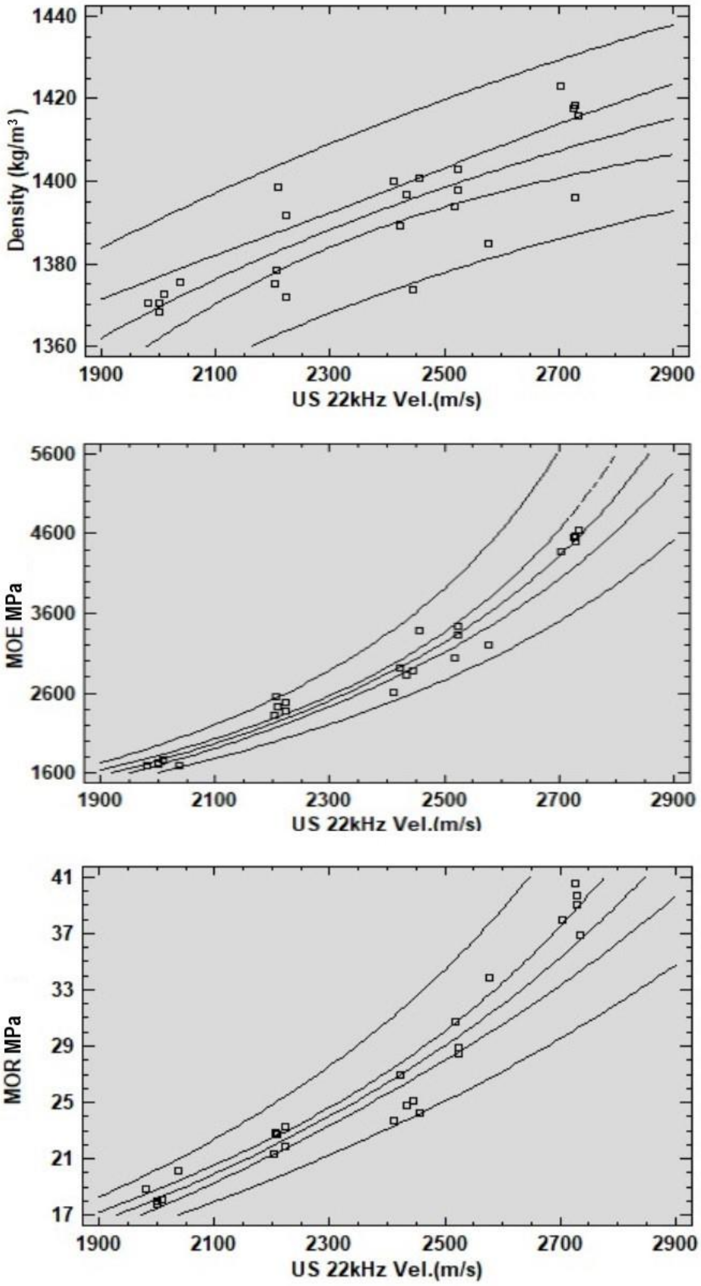


Figure 3: Estimation models of control parameters (density, MOR and MOE) with 22 kHz ultrasound device.

Conclusions

After 56 days of water immersion, the WPC exhibits a decrease in properties that is more pronounced at a temperature of 50 ºC. However, the density in both treatments is only slightly affected, likely due to the waterproofing effect of the plastic. For this reason, the remaining control parameters, such as MOR and MOE, are more significantly impacted by the immersion aging at 50 ºC, where the hot water causes much greater losses in both resistance and elasticity.

A strong relationship exists between non-destructive measurements and the decrease in properties, with better results obtained in estimating the properties after treatment at 50 ºC. In this case, all non-destructive devices are reliable estimators of the composite's properties. Ultrasound is the most reliable estimator, while probing methods, particularly screw withdrawal, also yield very good results. Stress wave testing, however, is the least reliable method in this context

Acknowledgements

We would like to thank Esteban Díez-Hernanz for his contribution and dedication, and the Association of Technical Research for Wood Industries, AITIM. Partial financial support 2018 was received from the Timber Construction Research Group, Universidad Politécnica de Madrid, Spain.

Authorship contributions

I. B-M.: Conceptualization, data curation, formal analysis, investigation, methodology, resources, writing original. R. M-L.: Investigation, resources, supervision, visualization, writing review and editing. H. M-B.: Investigation, resources, writing review and editing. E. H.: Conceptualization, data curation, formal analysis, investigation, methodology, writing review and editing.

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