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ARTÍCULO

Impact of nano-aluminum-oxide impregnation, densification, and vapor pre-treatment on the physical and mechanical properties of white willow wood

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Abstract:

Fast-growing wood species are often characterized by low density, unsatisfactory mechanical properties, and poor biological durability. Therefore, various modification techniques have been tested to improve these disadvantages. The aim of this research work was to investigate the effects of densification of *Salix alba* (white willow) under hot pressing for 15 and 30 minutes on its physical and mechanical properties. Vapor pre-treatment for four and six hours was also applied to mitigate the negative effects of cracks and checks caused by breakage in wood cell wall under pressure. Markedly, separate sets of specimens were impregnated with aluminum oxide nano-suspension to evaluate if an increase in thermal conductivity would improve the properties of wood. The results indicated that densification significantly enhanced both the physical and mechanical properties of the wood. The four-hour vapor pre-treatment demonstrated the optimal improving results in both hot pressing durations. Though impregnation of specimens with the nano-suspension improved some properties (including spring back, hardness, and physical properties), most of the studied mechanical properties did not show any statistically significant improvement. Therefore, it was concluded that densifying willow wood for 15 minutes with a four-hour vapor pre-treatment yields optimal results. The enhancement in mechanical properties due to nano-aluminum oxide was not substantial enough to justify the associated costs, and thus, its use is not recommended for industrial applications.

Keywords: Aluminum oxide nanoparticles, fast-growing wood species, physical and mechanical properties, white willow (*Salix alba*), wood impregnation.

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Introduction

Wood has written the history of human civilization. It is a renewable material with numerous applications and versatile properties (Sandberg *et al.* 2017). The price is also low in comparison to other constructional materials (Luan *et al.* 2022). However, since the introduction of mass production techniques, natural forests have experienced great pressure to meet the ever-increasing demand for wood, wood-based products, and biomass for renewable energy purposes. As a result, authorities in many countries have become interested in planting fast-growing trees and low-density species (Thomas *et al.* 2021, Barbu *et al.* 2022).

However, these trees typically produce low-quality wood with poor mechanical properties (Moya and Muñoz 2010, Istikowati *et al.* 2014, Bektas *et al.* 2020). As a result, researchers have studied these species from various perspectives in order to improve their properties so that they can be used for industrial applications (Gaitán-Alvarez *et al.* 2021). Various modification methods have been developed, including chemical, thermal, and impregnation techniques (Sandberg *et al.* 2013, Gérardin 2016, Sandberg and Kutnar 2016, Mantanis 2017, Papadopoulos *et al.* 2019, Militz 2020, Čabalová*et al.* 2022, Jiang *et al.* 2022, Kozakiewicz*et al.* 2022).

Thermal modification (sometimes referred to as heat treatment) of wood in different media is considered the most commercially used modification technique (Sandberg *et al.* 2013, Gérardin 2016, Sandberg and Kutnar 2016, Mantanis 2017, Papadopoulos *et al.* 2019, Militz 2020, Čabalová*et al.* 2022, Jiang *et al.* 2022, Kozakiewicz*et al.* 2022). It improves the biological durability against wood-decay fungi and dimensional stability of wood. Though it alters the chemical composition of the main polymers in wood (cellulose, hemicellulose, and lignin), it is not considered a chemical process, as no chemicals are used in the process, thus it is considered an environmentally friendly modification technique.

Acetylation and furfurylation of wood are well-known commercial chemical processes by which many properties of wood can be improved (Gaitán-Alvarez *et al.* 2021, Mantanis 2017). In the recent two decades, nanomaterials have also been studied to improve a variety of different properties of wood and wood-based products and composites, and to enhance the production processes as well (Gaitán-Alvarez *et al.* 2021, Papadopoulos *et al.* 2019). As the chemical compositions and the proportions of the main cell-wall polymers are different among softwood and hardwood species (and even differences are common the same wood genera and species), thermal modification may affect differently on different species (Feng *et al.* 2012, Bektas*et al.* 2020, Ali *et al.* 2022, Čabalová*et al.* 2022, Kozakiewicz*et al.* 2022). Therefore, the most practical and effective modification method must be carefully selected on a case-by-case basis.

One of the modification techniques that have been introduced used to improve the mechanical properties of low-density wood species is densification (Luan *et al.* 2022, Welzbacher *et al.* 2008, Feng *et al.* 2012, Sadatnezhad *et al.* 2017, Cabral *et al.* 2022, Scharf *et al.* 2023), though it is considered a fairly expensive niche product due to the rather costly processing equipment (Scharf *et al.* 2023). In the densification process, wood is pressed so that the final thickness would decrease by decreasing the voids in wood through various techniques, such as compression, application of steam and heat, impregnation of chemicals into the lumen, or a combination of these methods (Kutnar and Sernek 2007, Sotayo *et al.* 2020). These treatments increase the density of wood and enhance its mechanical properties, e.g., modulus of elasticity (MOE), modulus of rupture (MOR), hardness, and stiffness.

White willow (*Salix alba*) is a fast-growing tree species indigenous to Europe, western and central Asia. Apart from its fast-growing characteristics, white willow is a favourable species owing to several advantages such as its usage for environmental restoration purposes, medical applications of tree bark, e.g. antipyretic, anti-inflammatory, and analgesic effects, as well as its wood that can be used for energy applications, and in the wood industry (Ledin 1996, Karp 2014, Gryc *et al.* 2017). However, white willow is a hardwood species that produces lightweight wood which is extremely soft with very low mechanical strength. In addition, white willow wood is classified as non-durable and very susceptible to insect attacks (David 2022). Therefore, appropriate modification is required in order to enhance its physical and mechanical properties.

Wood has a low thermal conductivity (Bergman *et al.* 2011, Çavuş *et al.* 2019, Taghiyari*et al.* 2020a, Pásztory*et al.*2020). This low thermal conductivity delays the transfer of heat to the inner parts of wood modified by applying any kind of modification methods that uses heat in the process, like thermal modification (heat treatment), or hot-pressing densification. The low heat transfer would eventually increase the number and possibly gravity of the previously mentioned micro-checks. Moreover, it can result in non-uniform densification in the inner and outer layers of the modified timber.

Over the last years, various metal and mineral materials were used to increase thermal conductivity in wood and wood-based composites. For instance, the positive effects of silver and copper nanoparticles, enhancing the physical and mechanical properties and decreasing the hot press time in thermally-modified solid wood and wood-based composites, were reported (Taghiyari *et al.* 2016).The positive effects were attributed to two main reasons, i.e. the enhanced thermal conductivity and the formation of new bonds between the metal nanoparticles and the cell wall polymers.

The use of metal nanoparticles in wood modification has been shown to improve the treated wood's resistance to fungi decay (Oliva *et al.* 2015, Lykidis*et al.* 2016). According to Bi *et al.* (2021), considering the latest technological advancements, the application of metal nanoparticles for enhancing various wood properties will become more popular in the near future. The majority of the studies, however, have been focused on the application of copper, silver, boron, and zinc nanoparticles. Other metal nanoparticles have received little attention.

Nano-titanium-dioxide (TiO_2), for instance, has shown promising characteristics to serve as a wood preservative (Bi *et al.* 2021). In the meantime, one study reported improved properties of poplar wood impregnated with nano-aluminum-oxide (NA) (Taghiyari *et al.* 2017). Markedly, the improvement was minor, and research on the use of NA on other wood species is still rather limited. Therefore, in the present research work, nano-aluminum oxide was used. It was hypothesized that nano-aluminum oxide, as a metal-based chemical with higher thermal conductivity compared to wood, could potentially improve the physical and mechanical properties of densified white willow. In this study, density functional theory (DFT) analysis was employed (Argaman *et al.* 2000) to explore the reason for improvements that were achieved as a result of impregnation with aluminium oxide nanoparticles.

Materials and methods

Specimen preparation

Three white willow (*Salix alba* L.) trees were cut from the fields near Shalamzar city (Chaharmahal and Bakhtiari Province, Iran) in autumn. Trees were 12 years old, with a diameter of 30 cm at breast height. The geographical coordinates of the site of tree harvesting were 31° 30' North, and 50° 20' East, at an altitude of 2000 m above sea level. Boards of 1,5 to 2 m long with a thickness of 5 cm and width of about 15 cm were cut from the logs. They were kept in a warehouse (25 ± 3°C, and 20±4% relative humidity) for two months before specimens were cut. The mean moisture content (MC) of the conditioned wood was 12 %.

Aqueous nanosuspension impregnation

A 400-ppm nano-aluminum oxide suspension was prepared to be used for impregnation. Ammonium polymethacrylic acid was used as a dispersant based on M_w of 13000 g/mol, and with a concentration of 3 % (dry weight of nano-particles). Nano-particles had a size range of 50-70 nm based on the provider (Noor Technological Materials, Tehran, Iran). The Lowry method was used to impregnate specimens, as there was no need for the nano-suspension to remain in cell cavity once the impregnation was done. The dimensions of the pressure vessel were 700 mm in length and 500 mm in diameter. The load of specimens was placed inside the vessel with an overhead perforated plate to prevent the specimens from floating. Then, nano-suspension was pumped into the vessel without any initial pressurization or vacuum. This way, the air already inside cell cavities was trapped. The suspension temperature at the time of impregnation was 28 °C. Once the vessel was filled with the nano-suspension, a pressure was gradually applied. It took 30 seconds to reach the target pressure of 300 kPa inside the vessel. The specimens remained under the pressure for 30 minutes. The pressure was released slowly to avoid as little shock as possible to the wood micro structures. It took 60 seconds for the pressure inside the vessel to reach the atmospheric level. The dry and wet weights of specimens just before and after the impregnation process were measured to be further used to calculate the nano-suspension uptake. Three extra specimens were prepared to be simultaneously impregnated along with other specimens. Once impregnation process was carried out, these extra specimens were cut in half to make sure that the nano-aluminum suspension penetrated all throughout and to the core of specimens. After the impregnation process, all specimens were kept in a chamber (20 ± 3 °C, and 30 ±4% relative humidity) along with the control specimens for three months to avoid any possible confounding effects caused by different moisture contents. The moisture content (MC) of specimens was 7,5±0,5% when the physical and mechanical tests were performed. This MC was chosen to comply with the general MC levels of wood objects in Tehran.

Densification process

Samples with dimensions of 50 mm × 50 mm × 500 mm (t × w × l) were used. Densification was carried out uniformly with a densification ratio of 33 % (based on the target size) in the radial direction. A HT/MLM-220 hot press (Mehrabadi Mfg. Co., Tehran, Iran) was used. The nominal pressure of the hot press was 22 MPa, with a max. heat setting of 250 °C. Steel spacers were put on both sides of specimens under the hot press to avoid over-densification. The thickness of the spacers was the same as the target thickness. Based on a previous research work in which the optimum temperature was studied (Rassam *et al.* 2012), the temperature of the hot press was set at 150 °C, and the process continued for 15 and 30 minutes. Once the time was up, the heating system of the hotpress was turned off, but the specimens were kept under pressure for 1,5 h more to cool off. The temperature of the specimens was 80±5 °C when they were removed from the hot press. In order to avoid thermal shock and occurrence of micro-cracks and sudden spring-back in the specimens, they were kept in a closed container (1 m^3 internal capacity) so that they cooled down gradually. It took about 3 hours for the specimens to get to the room temperature. Then, they were kept in a chamber (20±3 °C, and 30±4 % relative humidity), which are the average indoor conditions in Tehran, for three months along with other specimens.

Physical and mechanical properties

Specimens were prepared and tested under the terms and conditions of the ASTM D143-22 (2023) standard specifications. Specimen size for MOR and MOE tests was 25 mm × 25 mm × 410 mm, using a center-point loading bar. Specimen size for compression strength parallel to grain was 25 mm × 25 mm × 100 mm. Loading span for impact bending test was 710 mm, and specimen size was 25 mm ×25 mm ×370 mm.

Specimen size for water absorption (WA) and thickness swelling (TS) was 25 mm × 25 mm × 100 mm. Weight and dimensions of specimens were measured after 2 and 24 hours of immersion in distilled water. Figure 1 schematically shows the densification process and direction of pressure applied on the specimens.

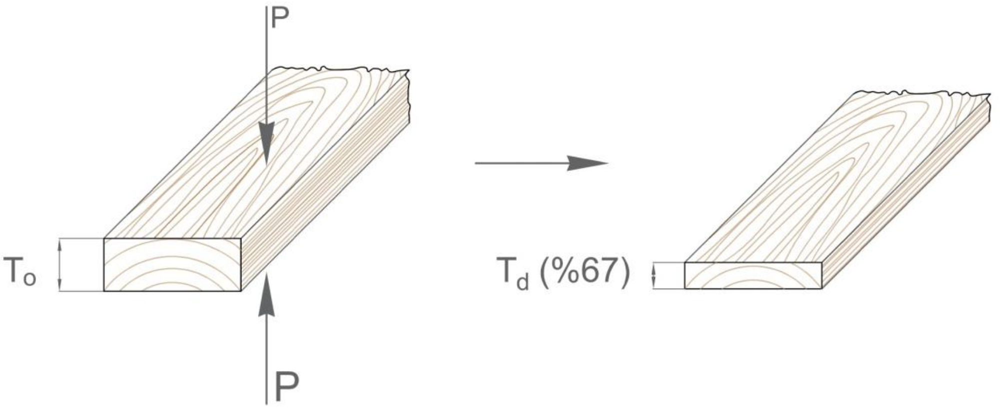

Figure 1: Schematic drawing of willow wood before and after densification (T_0 = thickness before densification; T_d = thickness after densification).

Spring-back (SpB%) was measured based on dimension measurement of specimens that were densified in each treatment. Two SpB values were calculated; the first one was measured immediately after the specimens were removed from the hot press; and second SpB was calculated based on the measurement after 15 days. SpB% was determined based on Equation 1.

$$Spring\ back\ (\%) = \frac{T_2 - T_1}{T_1} x100 \quad (1)$$

Where T_1 was the thickness under hot press (target thickness), T_2 was the thickness after densification (based on the two thicknesses as explained above).

For each test and treatment, three replicate specimens were cut and prepared; all specimens were free from any checks, fissures, knots, or other visual defects.

Water vapor pre-treatment

Separate sets of densified willow wood samples were cut and prepared to be pre-treated with vapor for four and six hours, respectively, at a target temperature of 120 °C. Vapor pre-treatment was carried out in a cylindrical vessel. The length and diameter of the vessel for vapor pre-treatment were 600 mm and 450 mm, respectively. At the bottom side of the vessel, there was a perforated plate upon which the specimens were placed. The sheet was located 100 mm above the lowest point of the vessel. The purpose of the sheet was to avoid direct contact of the specimens with water. For steam generation, the lower half of the cylindrical vessel was equipped with electrical heating elements to turn the reserved water below the perforated plate into vapor. It took 15 minutes for the interior of the vessel to reach the target temperature and the heating time was not included in the total duration of vapor pre-treatment.

Impregnation uptake and density of densified wood

After impregnation treatment, the uptake of nano-aluminum-oxide(NA) suspension by the wood was determined. The wood samples were weighed before and after NA impregnation. The suspension uptake was calculated using Equation 2.

$$Suspension\ uptake\ \left(\frac{g}{cm^3}\right)=\left(\frac{NAUV}{V}\ x\ \frac{concentration\ of\ \ NA\ suspension}{100}\right) \quad (2)$$

Where NAU was nano-aluminum-oxide suspension uptake (g) and V was volume of wood (cm^3). For density measurement, the mass of the samples was divided by its respective volume and the density value was expressed in kg/m^3. The moisture content of specimens was 7,5±0,5 % when the density of specimens was measured.

Theoretical modeling based on DFT

The adsorption behavior of alumina (Al_2O_3) nanoparticles on plant cell wall polymers was simulated theoretically. The nanoparticles were modeled as $(Al_2O_3)_n$ clusters, where $n = 1$ to 3. Interaction of these nanoparticles with the key structural polymers in the plant cell wall, cellulose and hemicellulose, was studied. Theoretical modeling in this study was carried out based on density functional theory (DFT), a computational quantum mechanical method widely used to investigate the electronic structure of atoms, molecules, and materials. DFT calculations allow us to predict key properties such as adsorption energies, bond lengths, and electronic interactions at the atomic level, providing a fundamental understanding of the adsorption process between the Al_2O_3 clusters and the cell wall polymers that is difficult to obtain through experimentation alone. All DFT calculations were done by Open MX3,8 package (Ozaki *et al.* 2018). The generalized gradient approximation (GGA) method proposed by Perdew-Burke-Ernzerhof (PBE) was applied to deal with the exchange-correlation functional (Perdew *et al.* 1996). The van der Waals correction (DFT-D2) within the PBE functional was employed, proposed by Grimme (2006). The plane wave cut-off energy was fixed at 100 Ry.

The atomic models of cell wall polymers (cellulose and hemicellulose) studied here were previously presented by Nishiyama *et al.* (2002) and Kaith *et al.* (2011). Atomic models of cellulose and hemicellulose are shown in Figure 2. In the models, red spheres represent oxygen atoms, gray spheres represent carbon atoms, and white spheres represent hydrogen atoms. The polymeric molecules are repeated in the direction of arrows. It is to be noted that cellulose and hemicellulose have various structures and are differentiated between hardwoods and softwoods. Therefore, further studies should be carried out to investigate on the differences of absorption distances and energies of Al_2O_3 nanoparticles on each and every polymer structure based on wood species.

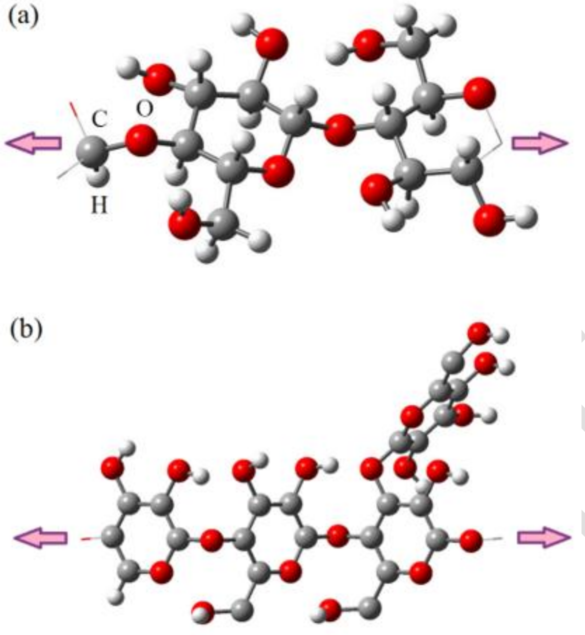

Figure 2: Atomic models of (a) cellulose and (b) hemicellulose.

To simulate adsorption of Al_2O_3 nanoparticles on cellulose and hemicellulose, $(Al_2O_3)_n$ clusters with n=1-3 were considered. The atomic models of $(Al_2O_3)_n$ clusters with n=1-3 are presented in Figure 3. In the models, red spheres represent oxygen atoms and pink spheres represent aluminum atoms. To find the adsorption configurations, the clusters with different orientations were placed on different sites on cellulose and hemicellulose chains (Figure 4). The distance of the cluster to the chain was then gradually increased to determine the optimal adsorption distance.

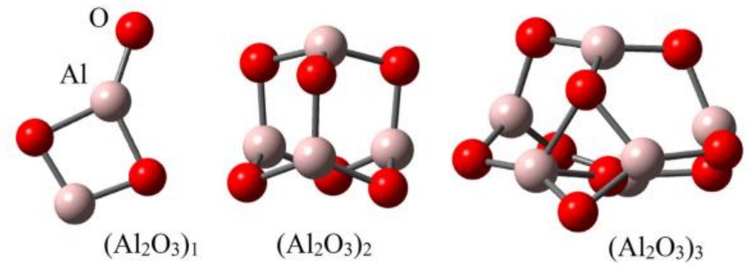

Figure 3: Atomic models of $(Al_2O_3)_n$ clusters with n=1-3.

To determine the most stable configurations, adsorption energy of different configurations was calculated according to Equation 3.

$$E_{ads} = \frac{E_{cellulose/hemicellulose+NA\ cluster} - \left(E_{cellulose/hemicellulose} + E_{NA\ cluster}\right)}{n} \quad (3)$$

Where $E_{cellulose/hemicellulose+NA\ cluster}$ denotes the total energy of the adsorbed system, $E_{cellulose/hemicellulose}$ Was the total energy of the cellulose or hemicellulose. $E_{NA\ cluster}$ was the total energy of $(Al_2O_3)_n$ cluster, and n was the number of Al_2O_3. The total energies required for this equation were obtained using DFT. DFT determines the energy of a molecular system by solving for its electron density, which uniquely defines its properties. It should be noted that the negative adsorption energy indicates the adsorption process is exothermic and the adsorbed models are stable. Multiple initial configurations of the $(Al_2O_3)_n$ clusters on the cellulose and hemicellulose polymers were considered.

These configurations differed in the adsorption site, cluster orientation, and initial distance from the polymer surface. All structures were subsequently subjected to full geometry optimization until the forces converged, and their adsorption energies were computed. The configuration with the largest negative adsorption energy was chosen as the most stable one. As an example, atomic models of $(Al_2O_3)_2$ cluster adsorbed on cellulose and hemicellulose are shown in Figure 4.

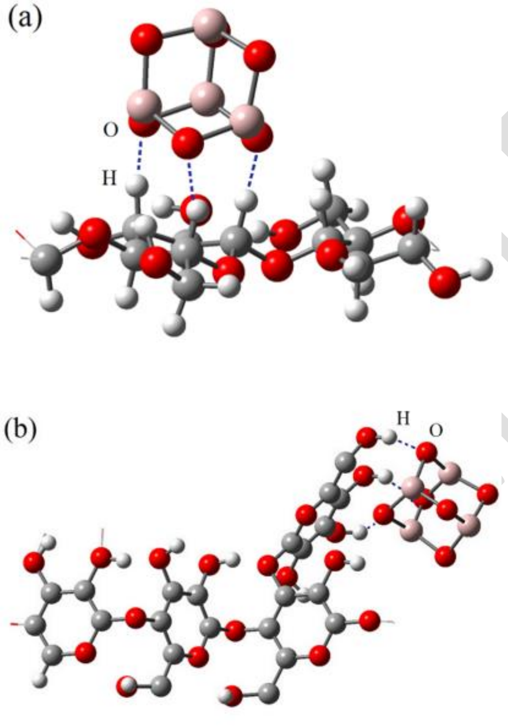

Figure 4: Atomic models of $(Al_2O_3)_2$ clusters adsorbed on (a) cellulose and (b) hemicellulose.

Adsorption distance is defined as the nearest distance between cluster and cellulose or hemicellulose. As shown in Figure 4 by dot lines, the nearest distance corresponds to the O atoms of cluster, and H atoms of cellulose and hemicellulose.

Statistical analyses

Statistical analyses were carried out by SPSS/18 (IBM, USA, 2009) in terms of analysis of variance (ANOVA) to discern significant difference at 95 % level of confidence. Groupings were then determined between fourteen treatments, using the Duncan Multiple Range test. Hierarchical cluster analyses were carried out to categorize the treatments, and find our similarities, based on all physical and mechanical properties, measured in this study. The treatments that were connected together by vertical bars nearer to zero on the top scale bar are more similar to one another than those which are connected by vertical bars nearer to 25 on the scale bar. Fitted-line, contour, and surface plots were designed using Minitab software, version 16.2.2 (Minitab Inc., State College, PA, USA, 2010).

Results and discussion

Density of the densified wood

To calculate the amount of uptake of nano-aluminum-oxide (NA) suspension, all specimens were weighed before and after being impregnated with NA-suspension. The determined mean uptake of nano-suspension (in liquid form) was 0,17 $g.cm^{-3}$; that is, 0,068$g.cm^{-3}$ retention of dry nano-aluminum. Density measurements of specimens for various treatments revealed a general increasing trend as the duration of both variables of press time and vapour pre-treatment increased, though the increase was not statistically significant in some cases (Figure 5). The greatest increase in density (90 %) was found in NA-impregnated specimens pressed for 30 minutes and pre-treated with vapour for 6 hours, while the smallest density increase (32 %) was found in specimens without NA-impregnation pressed for 15 minutes with no vapour pre-treatment.

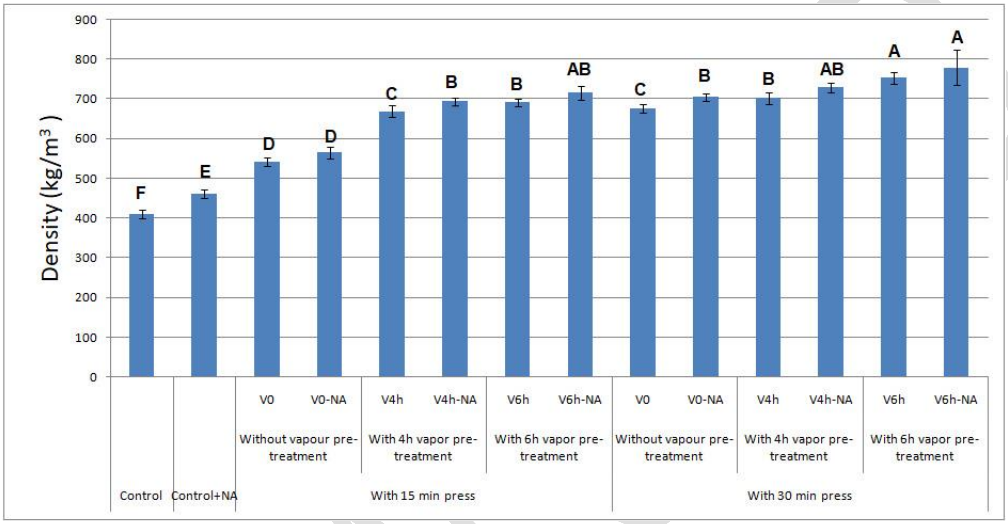

Figure 5: Mean density of the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence).

Mechanical properties of the densified wood

MOR values in all densified willow wood specimens were significantly higher than MOR in control specimens, regardless of NA-impregnation, hot press duration, or vapour pre-treatment (Figure 6). Hot pressing (at both 15- and 30-minute durations) resulted in a significant increase in

MOR values when compared to the control specimens. The increased density, caused by the densification treatment, resulted in significantly increased woody mass, and subsequently in greater MOR values. When compared to control specimens, vapour pre-treatment significantly improved (increased) the MOR values. This was primarily due to the improved effect of vapour on the viscoelasticity of cell walls, which is hypothesized to have resulted in lower internal stress and micro-cracks as a result of hot pressing. Still, in order to come to a final conclusion, comprehensive imaging of specimens with different pre-treatments should be carried out in further studies. When compared to specimens subjected to six hours of vapour pre-treatment, four hours of vapour pre-treatment had a greater increasing impact on MOR in both hot press durations (15 and 30 minutes). MOE, impact bending, compression parallel to grain, and hardness (both radial and tangential directions), showed nearly the same trend in maximum and minimum values in treatments as MOR values (Figure 7, Figure 8, Figure 9, Figure 10), though NA-impregnation had a significant effect in some of the treatments.

The extension of the vapour pre-treatment time from four to six hours resulted in a significant decrease in nearly all mechanical properties at both hot press times (15 and 30 minutes). The decrease in mechanical strength could be attributed to the thermal degradation of polymers (mostly cellulose and hemicellulose) (Taghiyari *et al.* 2020a). The decrease in mechanical properties corresponded to a decrease in WA and TS values, as discussed further in this section. It has been previously reported that degradation of cell wall polymers would eventually result in a decreased equilibrium moisture content, resulting in a significant decrease in TS values (Goli *et al.* 2014, Lee *et al.* 2015).

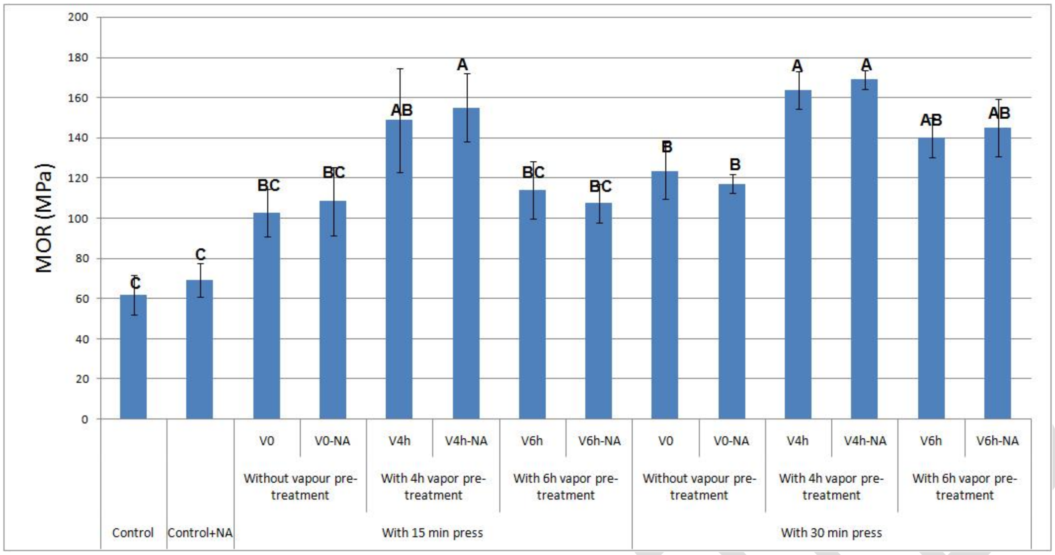

Figure 6: Modulus of rupture in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence).

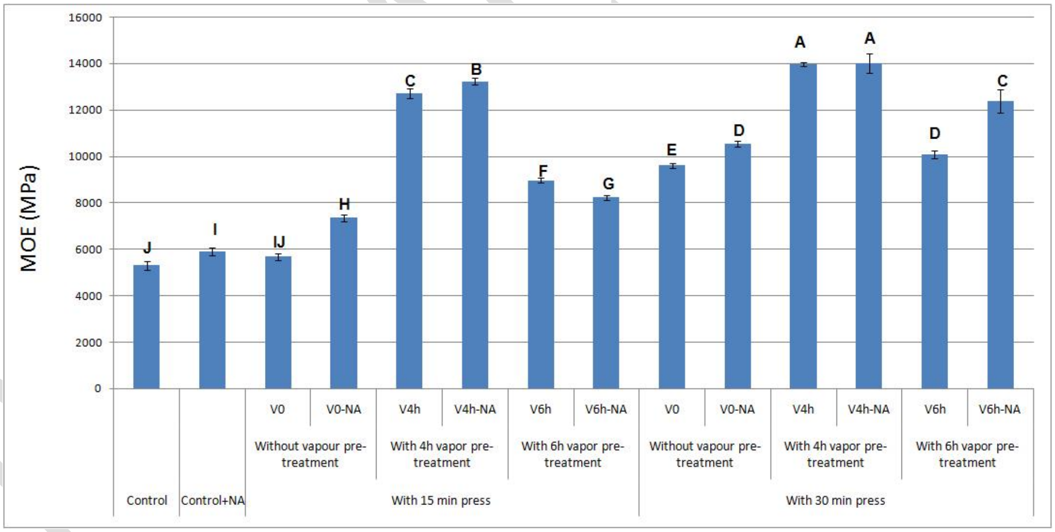

Figure 7: Modulus of elasticity in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence.).

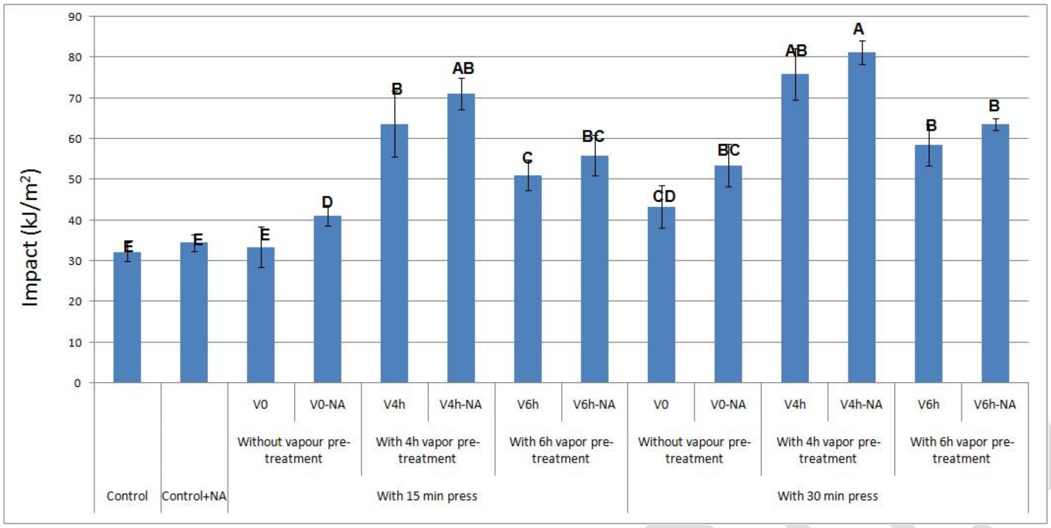

Figure 8: Impact bending in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence).

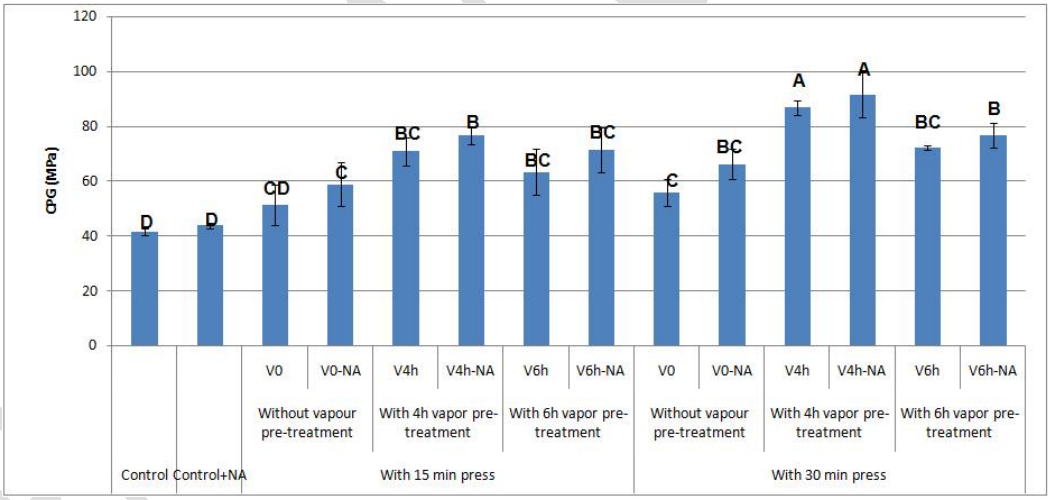

Figure 9: Compression strength parallel to grain in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence.).

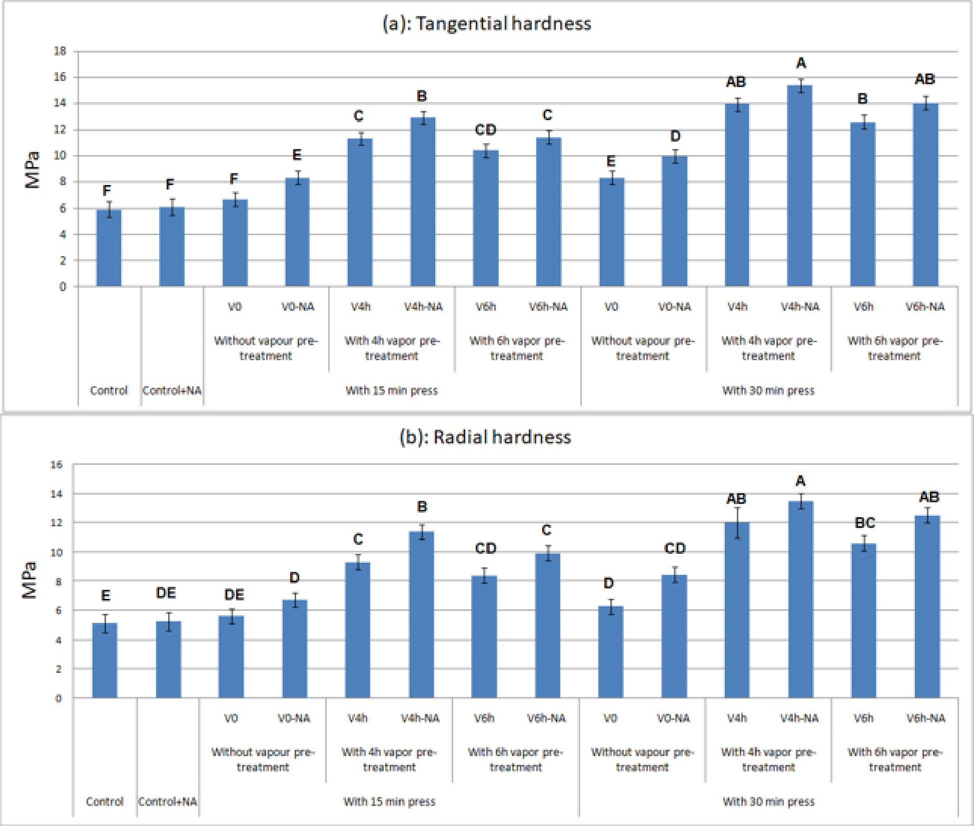

Figure 10: Hardness values of white willow wood specimens in two anatomical directions: (a) Tangential and (b) Radial, in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on Duncan's multiple range test, with 95% level of confidence.).

Except for hardness, impregnating specimens with aluminium oxide nano-suspension had a negligible and statistically-not-significant effect on nearly all mechanical properties. The cause was attributed to two major factors. The first one was more pronounced in the specimens that were not vapour pre-treated. Previous research has shown that two metal nano-particles (silver and copper nano-particles) have a significant impact in both solid wood species (thermally modified) and wood-based composites (Taghiyari *et al.* 2016 and Taghiyari *et al.* 2020b). When the thermal conductivity coefficients of silver, copper, and aluminium oxide were compared, aluminium oxide had significantly lower thermal conductivity than silver and copper (Bergman *et al.* 2011). The cited authors reported thermal conductivity of silver, copper, and aluminium oxide to be 429, 401, and 36 W/m.K at 300 K, respectively. As a result, the low thermal conductivity of aluminium oxide contributed to the nano-relatively suspension's low and mostly insignificant impact on mechanical properties. However, it should be noted that the effects of silver and copper nano-suspensions on densification have not been investigated yet. Therefore, further studies should cover these areas to be able to come to a final firm conclusion on the comparison between impregnation of solid wood species with different nano-metal suspensions.

The second reason was relevant to the vapor-treated specimens prior to densification. In this regard, it should be noted that water and vapour generally facilitate heat transfer to such an extent that the effect of nano-suspension in the current study was put into perspective; ultimately, no significant difference was observed between the specimens impregnated with NA and their counterparts with no NA-impregnation. As a result, it was concluded that impregnation with nano-aluminum oxide would not improve the mechanical properties of densified white willow wood, and thus it is not recommended to the industry in terms of mechanical properties because it could not compensate the extra costs of the nanomaterial used and the impregnation process.

Density functional theory (DFT) analysis of the densified wood

The measurement of SpB revealed a significant decreasing effect of NA-suspension impregnation on instantaneous SpB values (immediately after hot press) (Figure 11). All NA-impregnated treatments had significantly lower instantaneous SpB values than their non-NA-impregnated counterparts. In this regard, DFT calculations revealed the formation of new hydrogen bonds between aluminum oxide nanoclusters and both cell wall polymers (cellulose and hemicellulose), as well as the adsorption of Al_2O_3 nanoparticles on them (Table 1). The primary interaction between the Al_2O_3 clusters and the wood polymers is hydrogen bonding when the oxygen atoms of aluminum oxide nanoparticles are close to the hydroxyl groups of cellulose and hemicellulose. Analysis of the optimized structures and electron density (Figure 4) confirms the formation of O···H bonds, where the oxygen atoms of the alumina cluster act as hydrogen bond acceptors, and the hydroxyl (O–H) groups of the cellulose and hemicellulose polymers act as hydrogen bond donors. The bonds formed between oxygen atoms of nanoparticles and hydrogen atoms of cellulose and hemicellulose are shown in Figure 2, demonstrated by dashed lines. The hydrogen bond lengths were between 1,55 to 1,70 Å (Table 1).

The adsorption energy values for aluminum oxide clusters adsorbed on cellulose and hemicellulose are also listed in Table 1. It is hypothesized that these hydrogen bonds contributed to holding the nanoparticles on the cell wall polymers. When specimens were removed from the hot press, the new bonds helped reduce the amount of spring back due to the entanglement of hydroxyl groups in cellulose and hemicellulose. However, as the dimensions of NA-particles are larger than cellular polymers (cellulose and hemicellulose) at molecular scales, it is to be noted that the above-mentioned bonds may have been formed only on the surface polymers of cell wall. Therefore, further studies should not only evaluate formation of the bonds in more details, they should also consider potential positive effects in combination of "cooling belt" (Cabral *et al.* 2022, Scharf *et al.* 2023) with the impregnated specimens.

Table 1: Adsorption distance and adsorption energy for $(Al_2O_3)_n$ clusters with n=1-3 adsorbed on cellulose and hemicellulose.

Cell wall polymer	cellulose		hemicellulose	
n	$d_{O\cdots H}$(Å)	E_{ads} (eV)	$d_{O\cdots H}$(Å)	E_{ads} (eV)
1	1,70	-1,10	1,60	-1,41
2	1,65	-0,99	1,58	-1,37
3	1,63	-0,87	1,55	-1,25

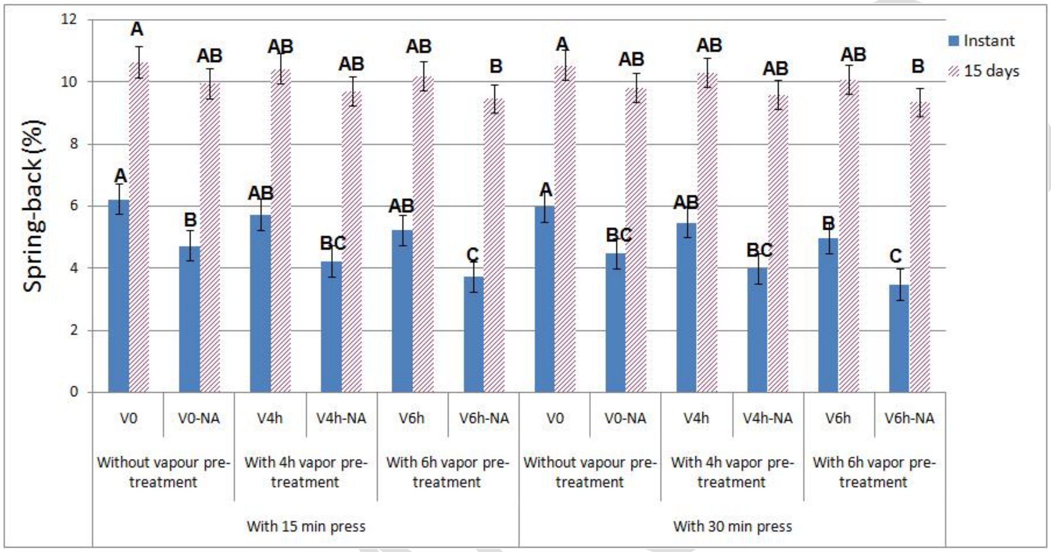

Figure 11: Spring-back in the densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on two separate Duncan's multiple range tests for instant and 15-day values, with 95 % level of confidence).

Water absorption and thickness swelling of the densified wood

Similar significant effect was observed in terms of WA and TS values (Figure 12 and Figure 13). All NA-impregnated specimens demonstrated significantly lower WA values after 2 hours of immersion in water. The difference between each pair (NA-impregnated and its counterpart treatment with no NA) decreased as immersion time extended to 24 hours. It is hypothesized that due to the abundance of water molecules, the newly formed bonds between aluminum oxide and the cell wall polymers (cellulose and hemicellulose) were broken, being replaced by water molecules. In this connection, R-squared value between WA and TS after 2 hours of immersion in water was high and significant (75 %; Figure 14a).

However, the R-squared value of the two properties after 24 hours of immersion proved to be low and statistically not significant (40 %; Figure14b). This implied that the longer duration of immersion caused more numbers of the newly formed bonds to be substituted with water molecules, resulting in inconsistency in trends of WA and TS values after 24 hours of immersion. Comparison of the values between WA and TS values after 2 and 24 hours of immersion clearly illustrated different trends in the fluctuations of values (Figure 12 and Figure 13). Scatter plot between the density versus WA and TS values (both after 2 and 24 hours of immersion) demonstrated more outliers for TS fitted lines in comparison to WA fitted lines, indicating that thickness swelling is influenced by other factors than the porosity in the wood structure of specimens (Figure 14c). Bonds formed between wollastonite and hydroxyl groups of wood cell wall have previously been reported to undergo similar substitution due to an abundance of water molecules (Perdew *et al.* 1996).

When the duration of vapour pre-treatment was increased from four to six hours, both WA and TS values decreased significantly. It may be due to the degradation of cell wall polymers when the wood was subjected to a longer period of heat treatment. As previously discussed in this section (Grimme 2006), similar thermal degradation caused mechanical properties to degrade as well.

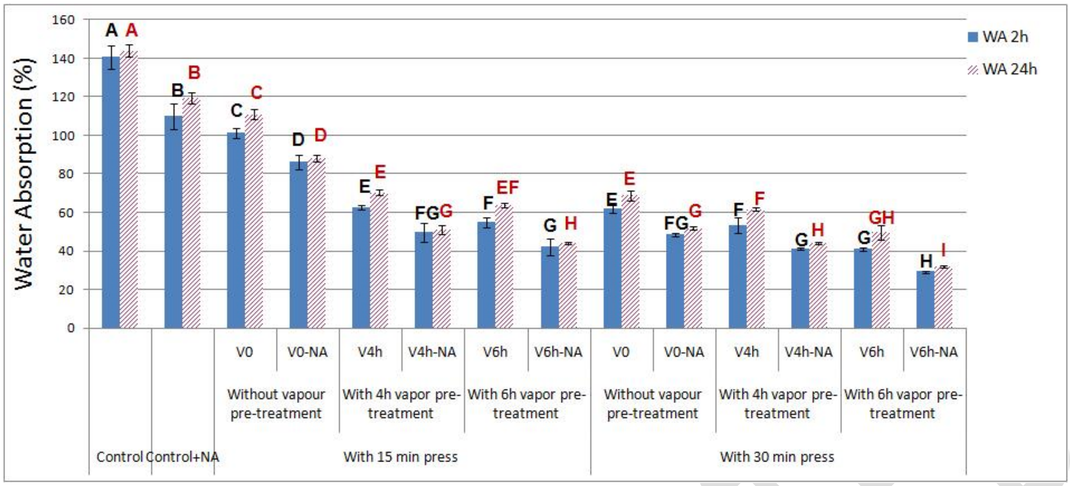

Figure 12: Water absorption in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on two separate Duncan's multiple range tests for 2h and 24h values, with 95% level of confidence).

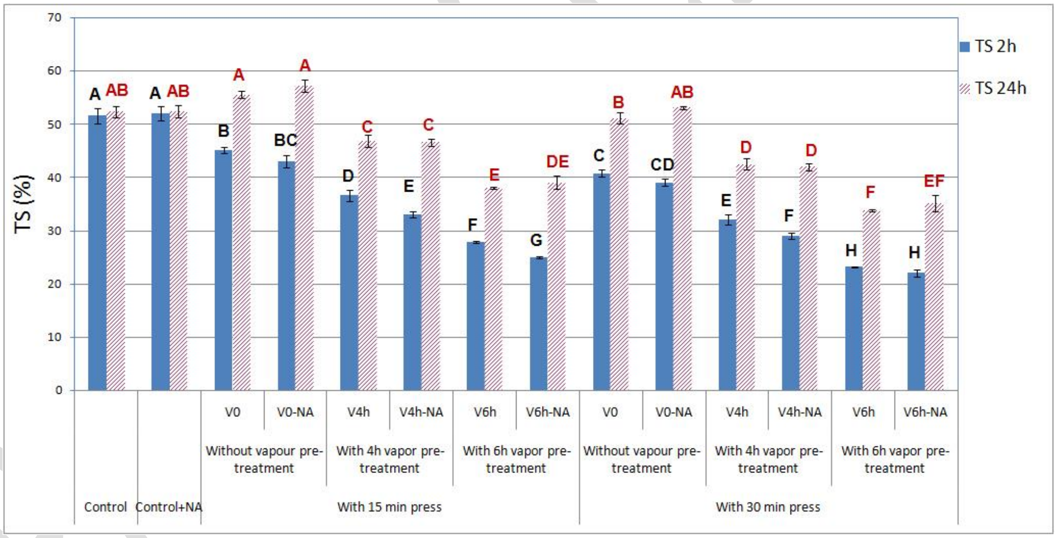

Figure 13: Thickness swelling in the control and densified white willow wood specimens hot-pressed for 15 and 30 minutes, and pre-treated with vapor (three levels of 0, 4, and 6 h) and impregnated with nano-aluminum oxide (NA).

(Error bars on each column determine the standard deviation for three replicate specimens in each treatment. Letters on each column represents the statistical groupings based on two separate Duncan's multiple range tests for 2h and 24h values, with 95 % level of confidence).

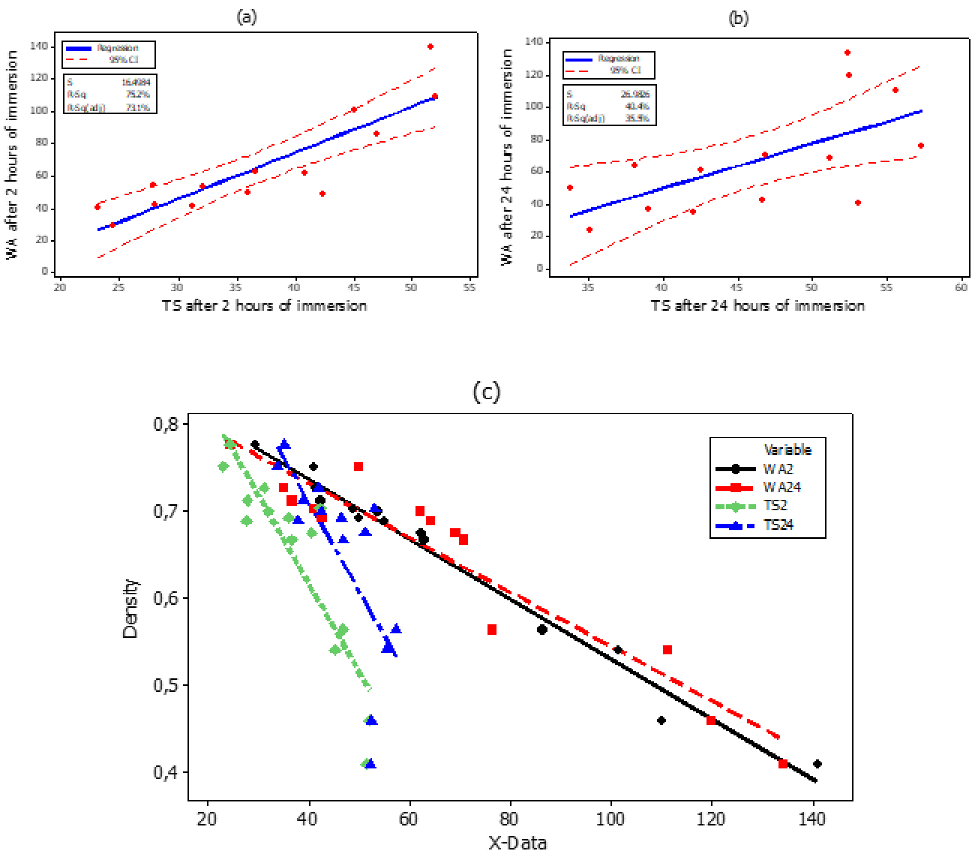

Figure 14: (a and b) Fitted-line plots between water absorption and thickness swelling after 2 hours and 24 hours of immersion in water, and (c) scatter plot of density versus water absorption and thickness swelling values (both 2 and 24 hours) (WA=water absorption after 2 and 24 hours; TS=thickness swelling after 2 and 24 hours).

Correlation between density and physical and mechanical properties of densified wood

R-squared values for fitted-line plots between density and various physical and mechanical properties were rather high and statistically significant (Figure 15a and Figure 15b). The same trends were observed in contour and surface plots of density versus physical and mechanical properties (Figure 16a and Figure 16b). Figure 16a shows that as the density increased, MOR (as one of the most important mechanical property in wood) increased, too. Moreover, as the physical properties (namely thickness swelling and water absorption)are directly dependent on the woody mass of specimens, a clear direct relationship between the density and water absorption was observed. Figure 16b also demonstrates a direct relationship between the density versus MOR and WA.

The slight discrepancies on the surface plot (Figure 16b) were partially attributed to the fluctuations in the woody substance and alterations of tree rings which are the inherent nature of wood materials. Elimination of the newly-formed bonds (as discussed above) in the presence of abundant water molecules was also partially influential in the discrepancies. According to the results of the above-mentioned analyses, there was a clear direct relationship between density and mechanical properties (in this case, hardness in tangential direction and MOR), and an inverse relationship between density and WA (after 2 hours of immersion in water). As density increased, the slope of MOR values increased while the slope of WA values decreased.

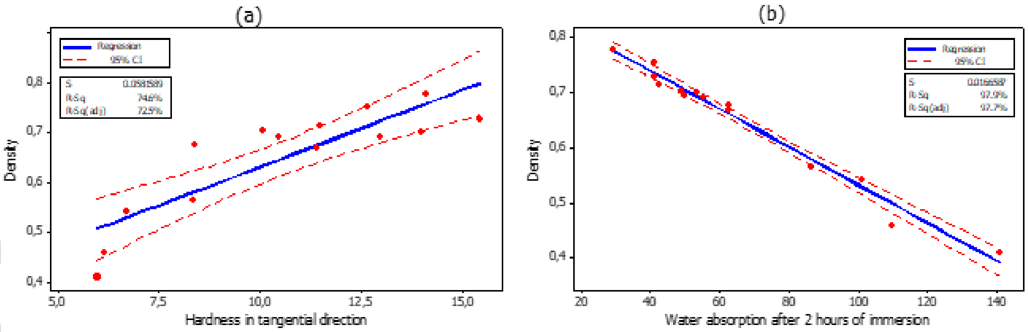

Figure 15:(a) Fitted-line plots between density values versus hardness in tangential direction showing direct correlation, and (b) versus water absorption after 2 hours of immersion in water showing inverse correlation, in the 14 treatments of white willow wood.

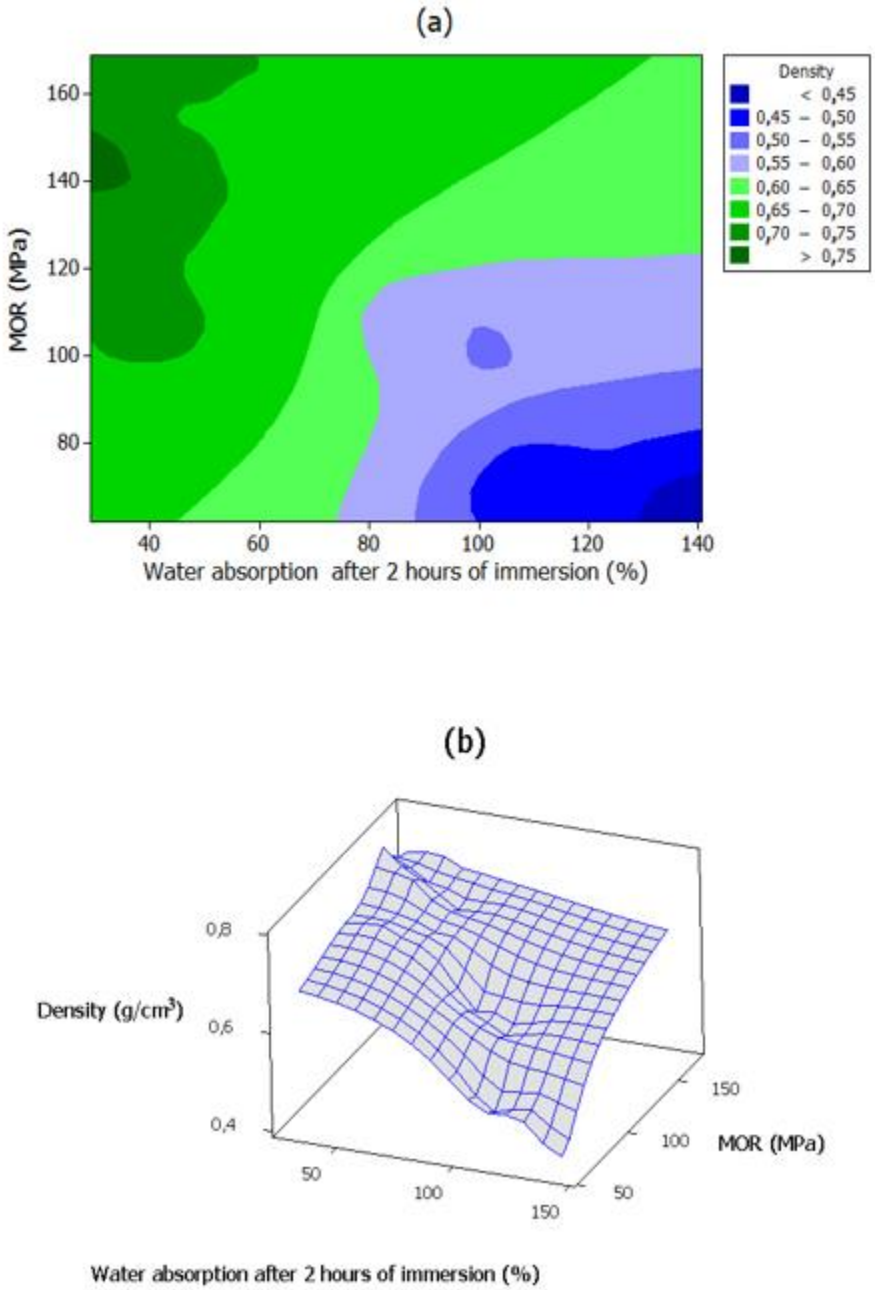

Figure 16: (a) Contour and (b) surface plots between density values versus modulus of rupture (MOR in mPa) and water absorption values (%) in the 14 treatments of white willow wood.

Based on all physical and mechanical properties, cluster analysis of the fourteen treatments revealed that all NA-impregnated specimens clustered closely to their counterpart treatment with no NA-impregnation (Figure 17). This clearly demonstrated that impregnation with NA had no significant effect on the overall properties of densified willow wood, though some individual properties may have improved. As a result, it was determined that NA cannot be recommended to the industry in terms of physical and mechanical properties. However, more research on the biological durability of different wood species against wood decay fungi and wood boring insects is needed before concluding the effectiveness of NA impregnation for solid wood materials.

Densification of willow wood for 15 minutes and without vapor pre-treatment is not a very effective modification method, as both treatments (P15-V0 and P15-V0-NA) were closely clustered to the control specimens. However, extending the duration of hot press from 15 minutes to 30 minutes showed significant impact on the properties, as both treatments hot-pressed for 30 minutes (P30-V0 and P30-V0-NA) were clustered remotely from the control specimens.

All specimens that were pre-treated with vapor for six hours were clustered closely together, regardless of the duration of hotpress (15 or 30 minutes), and with or without NA-impregnation. Similarly, all four treatments that were pre-treated with vapor for four hours were clustered very closely. This indicated that vapor pre-treatment was more effective compared to the duration of hot press, as far as the physical and mechanical properties are concerned.

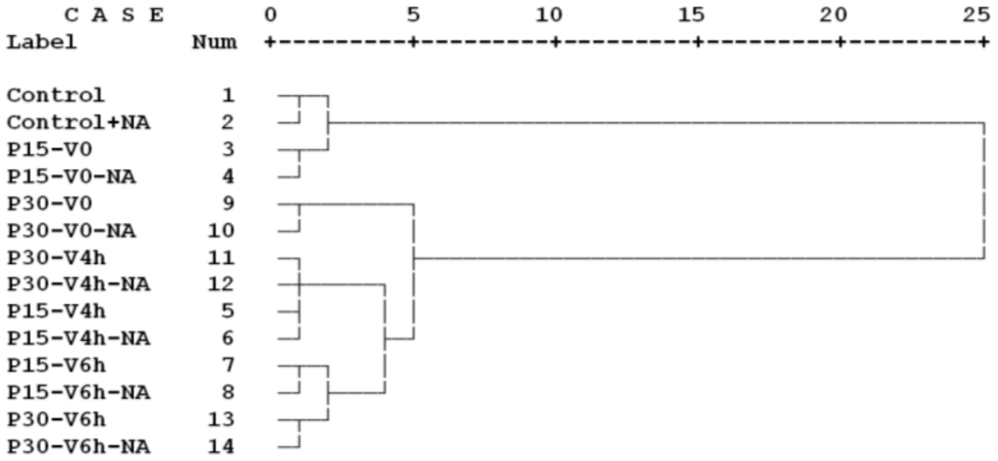

Figure 17: Cluster analysis based on all physical and mechanical properties in the fourteen treatments studied (P=duration of hot press; NA=impregnated with nano-aluminum; V0= no vapor pretreatment; V4h=vapor pretreatment for four hours; V6h=vapor pretreatment for six hours).

Based on the results of the present study, the densified white willow wood provided with promising improved properties that can be considered for structural applications if the necessary standard tests are passed. However, for the adaption of this method by the industry, additional feasibility tests on larger wood specimens might be necessary. In fact, densified wood is considered a type of engineered wood that can be used in a variety of applications in which smaller sizes can also be utilized. Low-quality and low-density wood species were previously used in furniture industry, due to the increased shortage of higher quality wood. For instance, furniture is currently produced in many countries (including Iran), using wood species with very low mechanical properties. Densified low-quality and low-density wood, even in small sizes, can satisfy this increasing demand by providing engineered wood materials with improved mechanical performance to be used in a wide variety of commercial applications, e.g., as a reinforcing or alternative connection material in timber structures (Anshari *et al.* 2012, Xu *et al.* 2021).

Cost is another important factor to consider. Some may question the logic of treating inferior willow wood instead of using plantation pine wood, which has better physical and mechanical properties and is a fast-growing species. The viewpoint is correct, as cost is an important factor in the commercialization of any product, and densification needs rather high-cost processing equipment (Scharf *et al.* 2023).

However, there is a significant difference in this case. Because of limited total forest area, high quality wood species are not available in some countries (including Iran). That is, no permit is issued by the authorities to cut trees that produce high-quality wood (like beech, oak, walnut etc.). Moreover, importation of high quality logs and lumber species are more costly. As a result, the only option left is to plant fast-growing trees that produce low-quality wood. Furthermore, high-quality wood species cannot be grown in any location (special conditions are needed, like adequate precipitation or irrigation, altitude, duration and angle of sunlight, etc.). However, fast-growing trees (such as poplar and paulownia) can be grown in most plantation areas in the region. Moreover, they are considered as a sideline of income for many farmers in villages and rural areas. As a result, despite the higher processing costs, enhancing the properties of low-quality wood represents a viable option for meeting the growing needs of the local furniture and other wood-based industries. Nonetheless, based on the study's findings, the most important point is to improve treatment efficiency so that the cost is offset by the extent of property improvement.

Conclusions

Densification is a modification method increasingly used to enhance the properties of fast-growing species to meet the rising demand for solid wood. In this study, white willow (*Salix alba* L.) was pre-treated with vapor for four and six hours, followed by hot pressing for 15 and 30 minutes, to improve its physical and mechanical properties.

Additionally, separate sets of specimens were impregnated with aluminum oxide nano-suspension to investigate whether increased thermal conductivity would enhance the wood's properties. Vapor pre-treatment significantly improved both the physical and mechanical properties of willow wood. The enhancement in viscoelasticity due to vapor pre-treatment helped mitigate the negative effects of micro-checks and cracks formed during the densification process. However, the duration of vapor pre-treatment should be limited to four hours, as longer exposure can lead to the partial breakdown and degradation of cell wall polymers, ultimately decreasing mechanical properties. To note, longer vapor pre-treatment also increases production costs. Impregnation with nano-aluminum suspension improved only spring-back and hardness values.

The improvement was attributed to the formation of new bonds between the nano-suspension and the accessible cell wall polymers (cellulose and hemicellulose) on the surface layers of cell cavities. These new bonds also contributed to the decrease in water absorption (WA) and thickness swelling (TS) in nano-aluminum-impregnated specimens. It was concluded that densifying white willow wood for 15 minutes with a four-hour vapor pre-treatment yields optimal results.

Notably, impregnation with aluminum oxide nano-suspension is not recommended for industrial use, as it does not significantly enhance most of the mechanical properties. However, further research on its effects on biological resistance against wood-deteriorating fungi and wood-boring insects is necessary to reach a definitive conclusion on its effectiveness.

Author contributions

H.R.T.: Conceptualization, methodology, software, validation, formal analysis, investigation, resources, data curation, writing-original draft preparation, writing-review and editing, visualization, supervision, project administration, funding acquisition. K.A.: Methodology, formal analysis, investigation, data curation. R.M.: Software, investigation. G.R.: Software, writing-original draft preparation, writing-review and editing. P.A.: Validation, writing-original draft preparation, writing-review and editing, visualization. S.H.L.: Conceptualization, validation, writing-original draft preparation, writing-review and editing, visualization. S.S.O.A.: Writing-review and editing, visualization.

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Declaration of interest

The authors declare no conflict of interest.

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