Some physical properties of delignified and compressed *Melia dubia* wood

Melia dubia tree.
Photo A. Kavitha, N. Deepthi, R. Ganesan, S. C. Gladwin Joseph. [online] India Biodiversity Portal, Species Page: {name of species field}, Available at: https://indiabiodiversity.org/species/show/31551?pos= [Accessed date 29 june 2019].

Shailendra Kumar[1]
Nautiyal Ismita[1]
Shikhar Shukla[1]

[1] Forest Research Institute
Forest Products Division
Dehradun
India

Auteur correspondant / Corresponding author:
Shailendra Kumar –
Shailendra354@gmail.com

Doi : 10.19182/bft2019.341.a31758 – Droit d'auteur © 2019, Bois et Forêts des Tropiques – © Cirad – Date de soumission : 21 septembre 2018 ; date d'acceptation : 26 février 2019 ; date de publication : 1er juillet 2019.

Citer l'article / To cite the article
Kumar S., Nautiyal I., Shukla S., 2019. Some physical properties of delignified and compressed *Melia dubia* wood. Bois et Forêts des Tropiques, 341 : 71-77. Doi : https://doi.org/10.19182/bft2019.341.a31758

S. Kumar, N. Ismita, S. Shukla

RESUMÉ

Quelques propriétés physiques du bois de *Melia dubia* délignifié et comprimé

Cette étude explore les effets de la délignification dans l'amélioration de quelques propriétés physiques du bois comprimé. Les billes de bois d'une plantation de six ans d'âge de *Melia dubia* (densité = 0,39 g/cm^3) ont été délignifiées dans une solution alcaline de NaOH et Na_2SO_3 pendant 4 heures. Les billes de bois délignifiées ont été séchées jusqu'à contenir 12 % d'humidité, et comprimées à deux températures (120 °C et 150 °C) pendant 2 heures et 4 heures. Les améliorations dans la densité, la recouvrance de la compression (%) et la perte d'épaisseur (%) après la compression ont été observées et comparées avec des échantillons non délignifiés. La densité moyenne du bois délignifié et comprimé (DCW) était de 0,825 g/cm^3 (SD = 0,109) ; pour le bois comprimé sans délignification (CW), la densité moyenne était de 0,889 g/cm^3 (SD = 0,049). Ces densités diffèrent significativement : les échantillons de DCW ont montré une basse recouvrance de la compression (2,97 % à 5,22 %), tandis que, comparativement, les échantillons de bois CW ont montré une haute recouvrance de la compression (48,47 % à 38,05 %). La perte moyenne d'épaisseur (%) dans les échantillons de DCW a varié de 47,22 % à 52,26 %, et elle était approximativement de 10 % plus haute (58,77 % à 61,91 %) dans les échantillons de CW, sans délignification.

Mots-clés : bois comprimé, délignification, recouvrance de la compression, perte d'épaisseur, Inde.

ABSTRACT

Some physical properties of delignified and compressed *Melia dubia* wood

This study explores the effects of delignification in improving some physical properties of compressed wood. Blocks of six year-old plantation-grown *Melia dubia* wood (density = 0.39 g/cm^3) were delignified in an alkaline solution of NaOH and Na_2SO_3 for 4 h. The delignified wood blocks were dried to 12% moisture content and compressed at two temperatures (120 °C and 150 °C) for 2 hours and 4 hours. Improvements in density, set recovery (%) and thickness loss (%) after compression were observed and compared with the non-delignified samples. The mean density of delignified and compressed (DCW) wood was 0.825 g/cm^3 (SD = 0.109); for compressed wood without delignification (CW), the mean density was 0.889 g/cm^3 (SD = 0.049). These densities differ significantly, with the DCW samples showing very low set recovery (2.97% to 5.22%), while comparatively, the CW wood samples showed very high set recovery (48.47% to 38.05%). Average loss of thickness (%) in the DCW samples varied from 47.22% to 52.26%, and was approximately 10% higher (58.77% to 61.91%) in the CW samples with no delignification.

Keywords: compressed wood, delignification, set recovery, thickness loss, India.

RESUMEN

Algunas propiedades físicas de la madera comprimida y deslignificada de *Melia dubia*

Este estudio explora los efectos de la deslignificación en la mejora de algunas propiedades físicas de la madera comprimida. Varias trozas de madera de plantaciones de seis años de edad de *Melia dubia* (densidad = 0,39 g/cm^3) se deslignificaron en una solución alcalina de NaOH y Na_2SO_3 durante 4 h. Las trozas de madera deslignificada se secaron hasta alcanzar un contenido en humedad del 12 % y se comprimieron a dos temperaturas (120 °C y 150 °C) durante 2 horas y 4 horas. Se observaron mejoras en la densidad, recuperación de la compresión (%) y pérdida de espesor (%) después de la compresión. Estas propiedades se compararon con las de las muestras no deslignificadas. La densidad media de la madera deslignificada y comprimida (DCW) fue de 0,825 g/cm^3 (SD = 0,109); para la madera comprimida sin deslignificación (CW) la densidad media fue de 0,889 g/cm^3 (SD = 0,049). Estas densidades difieren significativamente: las muestras DCW presentaron una recuperación de la compresión muy baja (2,97 % a 5,22 %), mientras que, comparativamente, las muestras de madera CW presentaron una recuperación de la compresión muy elevada (48,47 % a 38,05 %). La pérdida de espesor promedio (%) en las muestras DCW varió de 47,22 % a 52,26 %, y fue aproximadamente el 10 % más elevada (58,77 % a 61,91 %) en las muestras CW, sin deslignificación.

Palabras clave: madera comprimida, deslignificación, recuperación de conjunto, pérdida de grosor, India.

Introduction

Over-exploitation of natural grown long rotation timber along with public awareness has forced the industries to use plantation based woods for furniture, joinery, flooring and decking etc. However, certain demerits like lower density, lower strength properties etc. are associated with the fast growing plantation wood. Compressing wood at higher temperature, pressure in absence or presence of resins and other additives have been attempted by various researchers to improve the wood properties.

It has been reported that the wood compression can improve the trade value of low density plantation grown wood manifold (Senol and Budakci, 2016). Set recovery or spring back is the excessive swelling in compressed wood when subjected to high humidity or liquid water conditions. Set recovery is a major challenge in utilization of compressed wood (Morsing and Hoffmeyer, 1998).

Many researchers have adopted different approaches to lower the set recovery of compressed wood. Inoue (1993a) and Ito *et al.* (1998) have used pre-steaming and temperature increase to reduce spring back. Partial hydrolysation of para-crystalline region of cellulose was identified as the main reason behind effectiveness of pre-steaming. Dwianto *et al.* (1999) proposed chain scission of hemicelluloses accompanied by a slight cleavage of lignin during pre-steaming. Hemicelluloses leach out during steaming, followed by cellulose degradation has been found to be responsible for fixation of compressed wood (Higashihara *et al.*, 2004). Other workers have used heat treatment (190 °C) after delignification for effective reduction in compression set recovery (Esteves *et al.*, 2017). Senol and Budakci (2016) reported the use of hydrothermal post treatments at 200 °C for fixation of compressive deformation. Inoue *et al.* (1993b) reported the use of melamine formaldehyde resins to decrease the set recovery. Use of wood delignification and compression to achieve improvement in wood properties is reported recently by Song *et al.* (2018).

Melia dubia is a fast grown plantation species which has potential to replace long rotation, traditional furniture and joinery timber species like mango in India. However, low density (0.39 g/cm^3) is a major deterrent in efficient utilisation of this species (Kumar *et al.*, 2018). This work is centred around exploring the effect of delignification and compression on some physical properties of *M. dubia* wood like density, set recovery and thickness loss.

Materials and methods

Wood samples preparation

Freshly felled wood logs were selected from six years old plantation of *M. dubia* (Dehradun, India, 30.3165° N, 78.0322° E). The logs were sawn into planks of thickness 5 cm. The planks were dried to bring moisture content up-to 12%. The dried planks were planed and reconverted to 64 blocks of size 6 cm x 4.5 cm x 6 cm (length x thickness x width). The wood blocks were randomly divided in two equal sets. First set of 32 blocks was used for delignification and compression (DCW) and the second set was used for compressed without any delignification treatment (CW).

Delignification and drying

A glass container filled with 2 L water was used for preparation of delignifying solution. 240 grams of sodium hydroxide (NaOH) was mixed in the water by stirring with a glass rod. After NaOH got dissolved, 120 g of the sodium sulphite (Na_2SO_3) was added and stirred. The solution was put on hot plate at a constant temperature 100 °C. Eight wood blocks from the first set were dipped in the solution for 4 h. As water from the solution evaporated, additional water was added to maintain a constant level. After completion of 4 h, a new solution was prepared for another set of wooden blocks. Thus, in 4 cycles of delignification all the 32 wood samples in the first set were treated. The delignified samples were again kiln dried to bring moisture content to 12%.

Compression

The 64 wooden samples (32 DCW and 32 CW) were pressed at 96.5 bar in a multi day-light press. The temperature and time durations used for pressing are given in table I:

After the compression, the specimens were kept in room conditions for 48 h.

Table I.
Combinations of temperature and time durations in treatments.

Time (h)	Temperature (°C)
2	150
	120
4	150
	120

Determination of density

Small blocks of size of 3 cm x 3 cm on tangential face were cut from each of the 4 compressed wood specimens. The dimensions of all the specimens were measured with the help of digital micrometer to calculate volume. These specimens were then oven dried at 103 ± 2 °C until a constant weight was achieved. The oven dry weight was measured. The density was calculated using formula:

$$\text{Density} = \frac{W_o}{V_g}$$

Where, W_o = oven dry weight of test sample (gm)
V_g = Initial volume at test (cm^3)

Determination of compression set recovery (C_r) (%)

To examine the effect of water absorption on compressed wood a set recovery test was conducted on each sample. This test consisted of soaking of specimens in water at 60 °C for 4 h and drying in room temperature for 24 h.

The compression set recovery was calculated using following expression:

$$C_r(\%) = \frac{L_o - L_i}{L_i} \times 100$$

L_i = initial thickness before immersion
L_o = thickness after immersing at 60 °C for 4 h

Determination of thickness loss (T_l) (%)

Thicknesses of the specimens were measured before and after pressing. To obtain thickness loss (%), following formulae was used:

$$T_l(\%) = \left(\frac{T_i - T_f}{T_i}\right) \times 100$$

T_l = thickness loss
T_i = initial thickness
T_f = thickness after pressing

Figure 1.
Compressed wood specimens:
A. Compressed without delignification (CW).
B. Delignified and compressed.

Results and discussion

Appearance of compressed wood specimens

Figure 1 presents the cross section of compressed wood blocks. Upper two layers of blocks (A) denotes compressed wood without delignification (CW), whereas, lower two layers (B) denotes compression after delignification (DCW).

It can be seen from the figure 1 that the color of wood specimens changed from pale white to brown due to delignification. By seeing layer B color profile, it is observed that the delignification could not be achieved evenly along the entire cross section of the wood. It is likely that delignification followed by compression could have resulted in vertical density profile due to differential extent of delignification across thicknesses.

Density

Table II presents mean densities of delignified and undelignified wood specimens are 0.825 g/cm³ and 0.889 g/cm³ respectively. The densities varied from 0.73 g/cm³ to 0.91 g/cm³. Thermo-hygro-mechanic (THM) densification of wood carried out by Navi and Girardet (2000) on Fagus (*Fagus silvatica*), Norway spruce (*Picea abies)* and maritime pine (*Pinus pinnaster)* at temperature and pressure 150 °C and 130 bar, resulted in mean densities of 1.27 g/cm³, 1.30 g/cm³, 1.32 g/cm³ respectively. In the current study, the densities are lower as compared with Navi and Girardet (2000). There may be two reasons behind this: the initial wood densities of the woods taken by Navi and Girardet (2000) were already higher i.e. 0.67 g/cm³ (89.5% increase in density), 0.42 g/cm³ (209% increase in density), 0.50 g/cm³ (164% increase in density) respectively, and the pressure applied was also approximately 35% higher.

Higher density in DCW compressed for longer duration (4 h) as compared with short duration (2 h) may be attributed to reduced spring-back due to re-arrangement of wood fibrils was helped by extended heating duration as well removal of lignin (Song *et al.*, 2018).

Figure 2 presents mean densities of compressed samples with and without delignification irrespective of pressing time and temperature. Mean density of DCW was 0.825 g/cm³ (SD = 0.109) and that of CW, 0.889 g/cm³ (SD = 0.049). Thus, the DCW and CW samples showed overall improvement of 112.8% and 125% over the raw wood density.

An independent samples t-test for equality of means of densities of DCW and CW samples indicated that the means were significantly different ($t(56) = -2.757$, $p = 0.008$). This significant difference can be attributed to the loss of lignin material during delignification, which ultimately could have resulted in loss of wood mass. The ray cells on outer wood surfaces were apparently diluted during delignification. Some thermal degradation of lignin can also occur at lower temperature resulting in breakdown of various phenolic compounds (Sandermann and Augustin, 1964).

Table II.
Mean densities of different treatments of delignification, temperature and time duration.

Lignin status	Pressing time (h)	Pressing temperature (°C)	Mean density (g/cm³)
Delignified and compressed	2	150	0.79
		120	0.73
	4	150	0.89
		120	0.90
Compressed without delignification	2	150	0.87
		120	0.91
	4	150	0.88
		120	0.90

Figure 3 presents the mean densities of compressed wood with respect to two compressions time durations irrespective of the temperatures.

The difference in densities of DCW and CW was significant ($F(58) = 10.955$, $p = 0.002$). Moreover, the densities of DCW pressed at 2 h and 4 h were also significantly different ($t(30) = -4.331$, $p \leq 0.001$). 2 h pressing resulted in mean density of 0.758 g/cm³ (SD = 0.08) where as 4 h pressing resulted in 0.892 g/cm³ (SD = 0.92) density (figure 3). Thus, it can be concluded that pressing time affects the density of delignified wood during compression. Difference in the densities due to the pressing temperature used in the study (120 °C and 150 °C) was not significant ($F = 0.013$, $p = 0.911$).

Set recovery (%)

Set recovery or spring back is a form of excessive swelling when the compressed wood is exposed to moisture content and heat. Table III presents mean set recovery (%) of both sets compressed wood samples (DCW and CW). The DCW wood samples showed very small set recovery around 2.97% (2 h) and 5.22% (4 h). On the other hand, the CW wood samples showed comparatively very high set recovery of 48.47% (2 h) and 38.05% (4 h). In the case of beech wood without delignification, the set recovery was 44.72% with simple pressing while it was from 15 to 23% for compression through thermo-hygro-mechanical method (Hwang and Lee, 2011). It can be seen from the present study that delignification of wood helps in reducing set recovery during preparation of compressed wood.

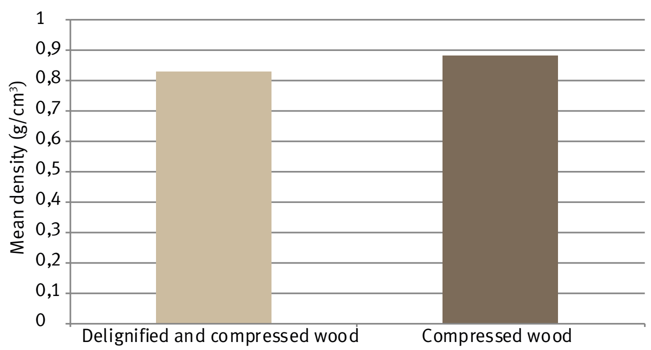

Figure 2.
Mean densities of DCW and CW wood specimens.

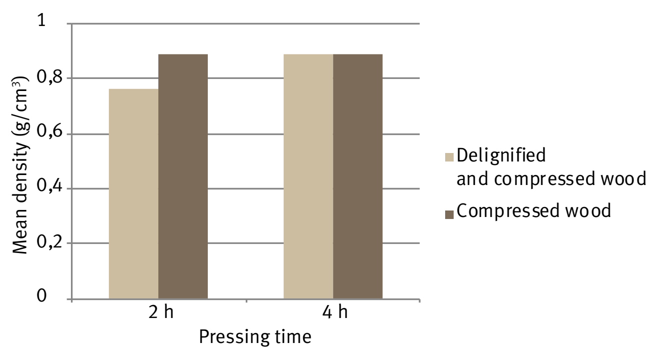

Figure 3.
Effect of pressing time of wood density.

Table III.
Mean set recovery of the wood specimens.

Pressing time	Treatment	Mean set recovery (%)
2 h	DCW	2.97
	CW	48.47
4 h	DCW	5.22
	CW	38.05

Moreover, permanent fixation is not reported to be associated with decomposition of cell wall polymers, the cross linking of cell wall and constituents, or crystallisation but is dominated by formation of cohesive structures (Higashihara *et al.*, 2004). The delignification of wood helps in re-arranging of the cellulose nanofibers due to weakening of the cell wall. Delignification in NaOH and Na_2SO_3 followed by hot pressing leads to total collapse of cell walls and complete delignification of wood (Song *et al.*, 2018). Thus, removal of lignin helps in realignment of cells to form compact cellulose nanofibers.

Some other reports suggest that the set recovery is also affected by compression ratio. Higher the compression ratio, higher is the time required for uptake of water (Abe *et al.*, 2001). Oxidation during heat treatment is reported to affect positively on fixation of compression (JieYing *et al.*, 2000).

Thickness loss (%)

After compression of the wood specimens to equal thickness, the wood specimens immediately showed spring back after removing them from pressing machine. However, the dimensions of the specimens attained stability after putting them in room condition for two days. Thereafter, thickness loss was calculated as per methodology described earlier. Table IV presents total thickness loss in compressed wood specimens with and without delignifications.

It can be seen from the table IV that mean thickness loss for delignified samples varied from 47.22% to 52.26%, whereas, compressed samples without any delignification showed approximately 10% higher thickness loss (58.77% to 61.91%). Figure 4 presents mean thickness loss in DCW and CW samples.

Mean of thickness loss of delignified samples (49.11%, SD = 3.06) was significantly lower to that of without delignification (60.45%, SD = 2.61) ($F = 284.26$, $p \leq 0.001$). Higher thickness loss in the samples without delignifications can be attributed to the breaking of cell walls during the compression, whereas, in deliginified samples, cell re-alignment occurs.

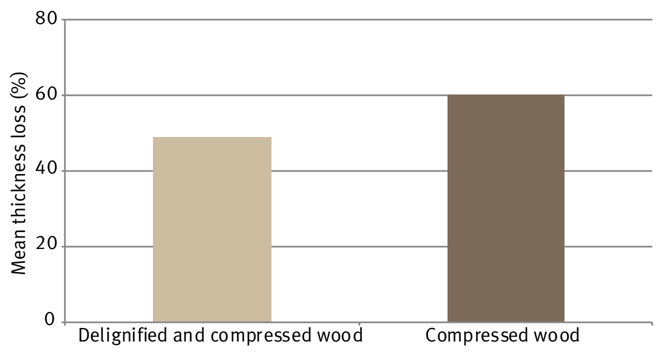

Figure 4.
Thickness loss in sample with delignification and without delignification.

Conclusion

Mean density of delignified and compressed wood was 0.825 g/cm^3 (SD = 0.109) and that of only compressed wood was 0.889 g/cm^3 (SD = 0.049) and these densities were found to be significantly different. The delignified wood samples showed very small set recovery in the range of 2.97% to 5.22%. Whereas, the wood samples without delignifications showed comparatively very high set recovery in the range of 48.47% to 38.05%. Mean thickness loss for delignified samples varied from 47.22% to 52.26%, whereas, compressed samples without any delignification showed approximately 10% higher thickness loss (58.77% to 61.91%). The study shows that partial delignification can be an important tool to reduce set recovery in compressed wood.

Table IV.
Mean thickness loss (%) at different temperatures and times.

Treatments	Pressing time (h)	Pressing temperature (°C)	Mean thickness loss (%)
Delignified and compressed (DCW)	2 h	120 °C	47.22
		150 °C	48.22
	4 h	120 °C	48.75
		150 °C	52.26
Compressed without delignification (CW)	2 h	120 °C	58.77
		150 °C	59.64
	4 h	120 °C	61.91
		150 °C	61.51

References

Abe H., Funada R., Kuroda N., Furusawa O., 2001. Confocal laser scanning microscopy of water uptake during the recovery of compressed and drying-set wood. IAWA Journal, 22 (1): 63-72. https://doi.org/10.1163/22941932-90000264

Dwianto W., Morooka T., Norimoto M., Kitajima T., 1999. Stress relaxation of sugi (*Cryptomeria japonica* D. Don) wood in radial compression under high temperature steam. Holzforschung, 53 (5): 541-546. https://doi.org/10.1515/HF.1999.089

Esteves B., Ribeiro F., Cruz-Lopes L., Domingos J. F. I., 2017. Densification and heat treatment of maritime pine wood. Wood Research, 62 (3): 373-388. http://ce3c.ciencias.ulisboa.pt/research/publications/ver.php?id=833

Higashihara T., Morooka T., Hirosawa S., Norimoto M., 2004. Relationship between changes in chemical components and permanent fixation of compressed wood by steaming or heating. Mokuzai Gakkaishi, 50: 159-167. https://jglobal.jst.go.jp/en/detail?JGLOBAL_ID=200902214551284397&rel=0

Hwang S., Lee W., 2011. Hardness and Dimensional Stability of Radiata Pine (*Pinus radiata* D. Don) Heat-Compressed Wood - Effect of Press Temperature & Time. Journal of the Korean Wood Science and Technology, 39 (3): 206-212. http://www.jwst.or.kr/past/list_sub.asp?srcCate=&i_key=6021&p_key=3020&v_key=39&n_key=3&n_key1=3&i_kname=&p_name=

Inoue M., Ogata S., Kawai S., Rowell R. M., Norimoto M., 1993a. Fixation of Compressed Wood Using Melamine-Formaldehyde Resin. Wood and Fiber Science, 25 (4): 404-410. https://wfs.swst.org/index.php/wfs/article/view/623/623

Inoue M., Norimoto M., Tanahashi M., Rowell R. M., 1993b. Steam or heat fixation of compressed wood. Wood and Fiber Science, 25 (3): 224-235. https://wfs.swst.org/index.php/wfs/article/view/288

Ito Y., Tanahashi M., Shigematsu M., Shinoda Y., 1998. Compressive-molding of wood by high-pressure steam-treatment. Part 2: Mechanism of permanent fixation. Holzforschung - International Journal of the Biology, Chemistry, Physics and Technology of Wood, 52 (2): 217-221. https://doi.org/10.1515/hfsg.1998.52.2.217

JieYing W., GuangJie Z., Iida I., 2000. Effect of oxidation on heat fixation of compressed wood of China fir. Forestry Studies in China, 2 (1): 73-79.

Kumar S., Bhushan U. K., Mishra A. K., Jena S. K., 2018. Variability in physical properties of plantation-grown progenies of *Melia composita* and determination of a kiln drying schedule. Journal of Forestry Research, 29: 1435-1442. https://doi.org/10.1007/s11676-017-0527-z

Morsing N., Hoffmeyer P., 1998. Densification of Wood. The influence of hygrothermal treatment on compression of beech perpendicular to gain. Kongens Lyngby, Denmark, Technical University of Denmark (DTU). http://orbit.dtu.dk/en/publications/densification-of-wood(5e00be8d-891d-4a4d-b5e8-102e7bbaeadd).html

Navi P., Girardet F., 2000. Effects of thermo-hydro-mechanical treatment on the structure and properties of wood. Holzforschung - International Journal of the Biology, Chemistry, Physics and Technology of Wood, 54 (3): 287-293. https://doi.org/10.1515/HF.2000.048

Sandermann W., Augustin H., 1964. Chemical investigations on the thermal decomposition of wood. Part III: Chemical investigation on the course of decomposition. Holz als Roh- und Werkstoff, 22: 377-386. https://doi.org/10.1007/BF02628346

Senol S., Budakci M., 2016. Mechanical wood modification method. Mugla Journal of Science and Technology, 2 (2): 53-59. http://dergipark.ulakbim.gov.tr/muglajsci/article/view/5000205925

Song J., Chen C., Zhu S., Zhu M., Dai J., Ray U., 2018. Processing bulk natural wood into a high-performance structural material. Nature, 554 (8): 224-228. https://doi.org/10.1038/nature25476

Kumar *et al.* – Author's contributions

Contributor role	Contributor names
Conceptualization	S. Kumar
Data Curation	S. Kumar
Formal Analysis	S. Kumar
Project Administration	S. Kumar
Resources	I. Nautiyal, S. Shukla
Supervision	S. Kumar
Writing – Original Draft Preparation	S. Kumar
Writing – Review & Editing	S. Kumar

Bois et Forêts des Tropiques - Revue scientifique du Cirad

Cirad - Campus international de Baillarguet, 34398 Montpellier Cedex 5, France - Contact : bft@cirad.fr - ISSN : L-0006-579X