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THERMAL COMPRESSION EFFECT ON SEVERAL PROPERTIES OF 8-YEAR-OLD WARU GUNUNG (*HIBISCUS MACROPHYLLLUS*) WOOD

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The study aimed to investigate the effect of thermal compression on the properties of 8-year-old waru gunung wood. Wood specimens with the dimension of 25 mm × 100 mm × 150 mm in thickness, width and length, respectively were heated at 180 ˚C and 200 ˚C for 4 h. During the heating process, the specimens were subsequently compressed at 2.452 N mm^{-2} for 40 mins at each temperature. Changes in the wood chemical structure were examined using the pyrolysis-gas chromatography-mass spectrometry, the wood physical properties were tested according to ASTM D143-94, the wood surface hardness was tested using a Universal Testing Machine and the wood discoloration was examined using the CIELab method. The changes in crystallinity degree was examined using x-ray diffraction and cell structure were collected as supporting data. Results showed that the thermal compression at 180 ˚C and 200 ˚C caused severe degradation on the specimen carbohydrate but less for lignin. The sum effect of thermal compression on the specimens also consecutively increased the density by 34.24% and 41.87% and surface hardness by 52.78% and 63.09% and decreased the swelling thickness by 38.54% and 47.03% and the equilibrium moisture content by 36.53% and 38.59% at 180 ˚C and 200 ˚C, respectively. Thermally compressed specimens also had darker colours, glossier surfaces and smoother texture compared to non-thermally compressed specimens.

Keywords: Eight-year-old wood, thermal compression, properties

INTRODUCTION

Waru gunung *(Hibiscus macrophyllus)* is one of the fast-growing tree species widely planted in Java island, Indonesia. The trunk of the 9-year-old tree can reached a height of 28 m and a diameter at breast height of 25–30 cm (Susanto & Mashudi 2017). The wood appearance is slightly brown in colour, slick and shiny and has a straight fibre direction. Due to these characteristics, waru gunung wood has been used as raw materials for furniture by small to medium enterprises in Java island.

Presently, the use of young waru gunung is still limited to extremely light furniture. The wood needs to be modified in order to expand its usage spectrum as heavy furniture and/or construction material. Densification technology is an effective method to improve the quality of inferior wood. The technology not only improves the wood strength properties but also enhances its surface quality. Therefore, the densified wood can be further converted into products with high commercial value such as flooring and furniture (Belt et al. 2013, Song et al. 2018).

The densification of wood can be performed physically with or without heating process. Thermal compression which combines the use of high-temperature heating and mechanical pressing to produce densified wood is currently gaining attention (Candan et al. 2013, Rautkari et al. 2013, Choowang & Hiziroglu 2015, Dogu et al. 2016). A single heat treatment above 150 ˚C will affect the wood hemicellulose generally present in a range of 18%–35% within the trees (Moliński et al. 2016). The degradation of wood hemicellulose may cause some amorphous regions to change into crystalline regions. These regions are void of effective free OH groups, thus decreasing the wood moisture content (Esteves et al. 2011). As a result, the equilibrium moisture content of the wood will decrease due to the reduced hygroscopic properties of wood and the dimensional stability of the wood will further

increase (Pelit et al. 2015, Shukla 2019, Priadi et al. 2019). Furthermore, the hemicellulose degradation (Gasparik et al. 2019) is similar to lignin degradation (Shmulsky & Jones 2019) as both contribute to the discoloration of wood.

Heating treatment also soften the wood structure and makes it easier to be compressed. Wood structure often changes because of combined action between heat and compression (Harsono 2016, Tu et al. 2014). Depending on tree species, exposure to heat treatment and thermal compression cause morphological changes of wood. The changes include reduced wood cell lumen dimension, collapsed and flattened vessels, cracked fibre cell walls, ruptured bordered pits and minor damage in ray cells (Darwis et al. 2017, Tu et al. 2014, Dogu et al. 2016).

The use of thermal compression on waru gunung wood had not yet been attempted. The study was necessary to provide information on possible efforts to improve the properties of the wood as they had become more popular among local communities for their source of income. The main aim of this study was to investigate the effect of using thermal compression technology on several properties such as chemical degradations, density, thickness swelling, equilibrium moisture content, surface hardness, colour changes, crystallinity degree and cell structure of 8-year-old waru gunung wood.

MATERIALS AND METHODS

Wood specimen

Approximately 10 tangential boards of 8-year-old waru gunung wood were prepared. Each board dimension was 25 mm (radial (r)/thickness)×100 mm (tangential (t)/width)×1000 mm (longitudinal (l)/length). The boards were dried at the temperature range of 40 °C–65 °C until the moisture content reached 12%. Each board was further sawn into specimens with a dimension of 25 mm (thick)×100 mm (width)×150 mm (length). These specimens were divided into groups of control and thermal densification specimens at random.

Thermal compression

All wood specimens were dried in an oven at 180 °C for 3 h. The next stage was followed by thermal compression at 180 °C and 200 °C, respectively. A pressure of 2.452 N mm^{-2} for 40 min was applied on both temperature treatments.

Changes in the chemical structure of the specimens primarily lignin and carbohydrate due to thermal densification were analysed using pyrolysis-gas chromatography mass-spectrometry. Changes in the physical properties of thermally compressed control specimens were observed after 1 month of storage at room temperature between 23 °C and 30 °C and relative humidity between 76% and 90%. In each treatment, 10 control specimens were examined for density, thickness swelling and equilibrium moisture content according to the ASTM D143 standard (ASTM 2014). The surface hardness of the specimens was tested using a universal testing machine and dropping a half-circle steel ball with a diameter of 11.25 mm on to the specimen surface.

Colour changes or discoloration of the specimens due to thermal compression were observed quantitatively using the CIELAB method. This method could measure the values for L, a and b (Hunter Lab 2012). L indicated lightness with colour values ranging from 0 (black) to 100 (white), a indicated colour values from green to red with −a from −80 to 0 corresponding to green and +a from 0 to 80 referring to red and b denoted colour values from blue to yellow with −b from −70 to 0 corresponding to blue and +b from 0 to 70 referring to yellow. In each specimen, five location points were used for the determination of L, a and b values. The colour change (ΔE) were calculated as a mathematical function of ΔL, Δa and Δb based on the formula below. The colour change values were classified into several criteria (Table 1).

$$\Delta E = f(\Delta L, \Delta a, \Delta b)$$
$$= ([\Delta L]^2 + [\Delta a]^2 + [\Delta b]^2)^{0.5}$$

where ΔE = the colour change value; ΔL = the difference in L value between non-densified

Table 1 Colour change class and criteria

Class	Colour change (ΔE) values	Criteria
I	ΔE < 0.2	Invisible change
II	0.2 < ΔE < 2.0	Very small change
III	2.0 < ΔE < 3.0	Small changes (colour changes is visible by high-quality filter)
IV	3.0 < ΔE < 6.0	Medium (colour changes is visible by high-quality filter)
V	6.0 < ΔE < 12.0	Large (distinct colour changes)
VI	ΔE > 12.0	Different colour

Source of reference Hunter Lab (2012)

specimen (control) and densified specimen; Δa = the difference in a value between non-densified specimen (control) and densified specimen and Δb = the difference in b value between non-densified specimen (control) and densified specimen.

The wood crystallinity degree was observed using x-ray diffraction. The visual changes in wood cell structures were examined using a stereo-capable microscope.

RESULTS AND DISCUSSION

Chemical component changes

The changes in lignin and carbohydrate components of the specimen due to thermal compression were shown in Table 2. The pyrolysis-gas chromatography mass-spectrometry results showed hot pressing severely degraded the carbohydrates in the specimens which consisted of hemicellulose and cellulose. However, the effect of heat on lignin was minimal.

The depolymerisation percentage of lignin compounds (Total A + B) tended to be much lower than that of carbohydrate compounds (Total C). The observation showed lignin was least degraded and had a greater impact on the low-thickness swelling performance of the densified specimens at 180 °C and 200 °C than of carbohydrates. Lignin which was inherently thermoplastic in nature became plastic after exposure to heat. Lignin melted and migrated to the specimen surface, filling all cell cavities, voids and spaces between fibres and microfibrils (Sjostrom 2013). After the cooling process, lignin became hardened and turned into strong adhesive between microfibrils and between fibrils (Choowang & Hiziroghu 2015).

The degraded lignin compound consisted of phenylpropane and benzene/phenol derivatives were shown in Table 2. The concentration of eugenol, cis-propenylsyringol, trans-propenylsyringol, and coniferyl alcohol which were derivatives of phenylpropane had decreased in the thermally compressed specimens at 180 °C. Eugenol and coniferyl alcohol continued to decrease in specimens being thermally compressed at 200 °C. The decrease was due to the fragmentation of two phenylpropane monomers into simpler molecular weight compounds such as the benzene derivatives. The concentration of propenylsyringol initially decreased in specimens thermally compressed at 180 °C but had increased in specimens exposed to thermal compression at 200 °C. The results suspected that lignin polymer in the specimens which was thermally compressed at 200 °C underwent more intensive depolymerisation to monomer units of cis-propenylsyringol and trans-propenylsyringol.

Table 2 showed as the pressing temperature increased, the concentrations of benzene derivatives guaiacol, syringol and cresol had increased while the vanillin and syringaldehyde concentration decreased. At the temperature of 200 °C, the concentration of syringaldehyde was undetected. The increase of guaiacol, syringol and cresol could be attributed to the more intensive fragmentation of their phenylprophane monomer derivatives. The decrease in the concentration of vanillin and syringaldehyde could be due to their volatilisation or further fragmentation into a

Table 2 Changes of lignin and carbohydrate polymers in densified and undensified specimens

No	Identified fragment compounds from the degradation of wood constituents	Chemical aspects[1]			Concentrations (%)		
		Chemical formula	BP (°C)	MW (g/mole)	Treatment		
					Control	180 (°C)	200 (°C)
A	Phenylpropane derivatives*						
	1 Eugenol	$C_{10}H_{12}O_2$	254	164.20	1.36	1.12	0
	2 Propenylsyringol (cis & trans)	$C_{11}H_{14}O_3$	306	194.23	3.64	1.99	3.01
	3 Coniferyl alcohol	$C_{10}H_{12}O_3$	163	180.08	3.72	2.47	1.92
B	Benzene/phenol derivatives**						
	1 Guaiacol	$C_7H_8O_2$	205	124.14	1.88	2.00	2.20
	2 Syringol	$C_8H_{10}O_3$	261	154.16	3.73	3.78	3.80
	3 Cresol[2]	C_7H_8O	202	108.13	1.58	1.60	1.70
	4 Vanillin	$C_8H_8O_3$	258	152.15	0.83	0.80	0.75
	5 Syringaldehyde	$C_9H_{10}O_4$	192	182.17	0.88	0.31	0
	Total: A + B				17.62	14.07	13.38
C	Degraded wood Carbohydrate						
	1 Acetic acid	CH_3COOH	117.9	60.05	8.70	10.71	8.53
	2 Glucosan[3]	$(C_6H_{10}O_5)$			5.42	5.29	7.56
	3 Furans/furfurals		< 150		1.80	7.35	6.40
	Total: C				15.92	23.35	22.49
D	Carbondioxide[5]	CO_2	<< 0	44.01	17.30	13.83	23.71
	Total: A + B + C + D				51.14	51.25	59.58
E	Other carbon-containing fragments[4,5]				48.86	48.75	40.42

MW = molecular weight, BP = boiling point;
[1] = source of reference (Rumble 2020), [2] = from phenol-propane-type monomers,
[3] = degree of polymerization (x) less than that (n) of cellulose, [4] = low molecular weight or low-boiling fragments, [5] = could be partially from the cell wall degraded chemical components and partially from the degraded/volatilised extractives;
* = monomer units from the depolymerized lignin,** = from the further degradation or fragmentation of phenyl-propane derivatives

much simpler inorganic chemical structure such as H_2O and CO_2. Lignin fragmentation at high temperatures experienced volatilisation or was converted into simpler chemical structures such as CO_2 (Rumble 2020). When the temperature was increased from 180 °C to 200 °C, the concentration of CO_2 in the pressed specimens had increased from 13.83% to 23.71%, respectively.

The depolymerisation of the wood carbohydrate yielded compounds consisted of acetic acid, glucose and furans/furfurals (Table 2). Acetic acid and furans were assumed to be the product of hemicellulose degradation, whereas glucosans were from cellulose depolymerisation (Shmulsky & Jones 2019). Furans played an important role in creating lignin–furfural bonds between

fibres in thermally compressed specimens (Widyorini et al. 2005). Therefore, the polymer also contributed to the dimensional stability of thermally compressed specimens. When the pressing temperature increased from 180 °C to 200 °C, the concentration of acetic acid and furans in the specimens had decreased. This was assumed to be due to the continued fragmentation of hemicellulose, which eventually converted into a simpler structure such as CO_2. Acetic acid, as one of the product of carbohydrate degradation, could also accelerate the fragmentation of carbohydrates to a much simpler structure (Kucerova et al. 2016).

At high temperatures, hemicellulose which was inherently amorphous in nature could fragment significantly. As a result,

the amorphous region shrunk, whereas the proportion of crystalline cellulose increased (Kucerova et al. 2016). The reduction in the free OH group at high temperatures brought the fibrils and microfibrils closer and increased the crystallinity proportion of the heated wood (Shmulsky & Jones 2019, Esteves et al. 2011). Other than hemicellulose, hardened melted lignin also contributed to the increase in surface hardness of the wood (Akgul & Korkut 2012, Choowang & Hiziroghu 2015).

Glucose concentration decreased in the specimens when thermally compressed at 180 °C but increased in specimens when thermally compressed at 200 °C. This happened as glucose underwent further fragmentation to a much simpler structure like H_2O and CO_2 at 180 °C. However, at 200 °C cellulose underwent further intensive depolymerisation, thereby increasing glucose concentration. The glucose concentration in the thermally compressed specimen was lower than that of acetic acid. This indicated that the semi-crystalline structure of glucose was more resistant to degradation by heat than the amorphous hemicellulose structure (Srinivas & Pandey 2012).

Physical and mechanical properties

Table 3 showed the densification temperatures at 180 °C and 200 °C significantly increased the specimen density at 34.24% and 41.87% and surface hardness at 52.78% and 63.09% but decreased the thickness swelling at 38.54% and 47.03% and equilibrium moisture content at 36.53% and 38.59% when compared to the undensified or control specimens. The density increase was caused by the decrease in specimen volume and thickness due to the hot press process. The volume and thickness decrease was attributed to decreasing volume of the wood cell cavities in the specimen structures (Budakci et al. 2016, Darwis et al. 2017).

Figure 1(b)–(c) showed the effect of high temperature and pressing movement of hot metal plates on wood cells which consisted of pores, rays and fibres to become more elastic and thereby narrowing their shapes in the densified specimens. Rays structures in the densified specimens appeared crushed and buckled when compared to those in the control specimens (Figure 1(a)). The wood fibres, especially the ones near to the surface

Table 3 Average values of physical properties in densified and undensified (control) specimens

Treatment	Density (kg m⁻³)	Thickness swelling (%)	Equilibrium moisture content (%)	Hardness (N mm⁻²)
Control	406 ± 45	5.89 ± 0.28	12.10 ± 0.43	9.32 ± 0.83
Densified 180 °C	545 ± 18	3.62 ± 0.09	7.68 ± 0.18	14.24 ± 2.76
Densified 200 °C	576 ± 10	3.12 ± 0.05	7.43 ± 0.07	15.20 ± 2.70

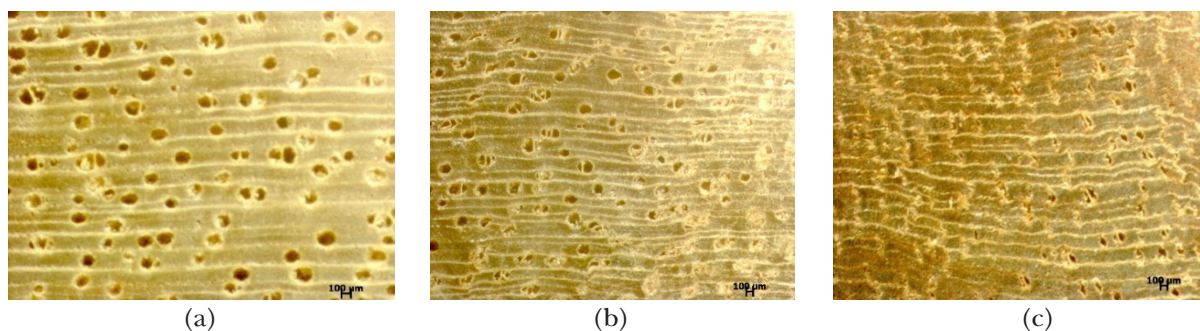Average of 10 replications

| (a) | (b) | (c) |

Figure 1 Cross sectional views of wood cell shape in densified and undensified (control) specimens
a = control, b = densified at 180 °C, c = densified at 200 °C

became closer to each other, denser and compacted, thereby increasing the hardness of the densified specimens as compared to control specimens (Table 3) (Budakci et al. 2016, Fang et al. 2019, Augustina et al. 2020). Increased hardness in wood was essential as they were beneficial for flooring (Chowang & Hiziroghu 2015).

The reduction of equilibrium moisture content in the thermally compressed specimens (Table 3) could be attributed to the reduced free OH group in the specimen cells which resulted from the water condensation during the desorption process. As a result, the hygroscopicity of thermally compressed specimens decreased and the situation was known as the hysteresis phenomenon (Frederikson & Thybring 2019). The decrease in thickness swelling of the densified specimens (Table 3) could be due to the role of the most heat-stable wood component such as lignin. The observation was because under further hot press densification at 180 °C and 200 °C, hemicellulose and cellulose would be degraded or depolymerised first, whereas lignin might still be able to withstand heat or remained intact. The heat-treated densification caused the evaporation of free water, bound water molecules as well as low-boiling temperature extractives. The water evaporation could draw the fibrils and microfibrils closer together, thus increasing specimen crystallinity. Figure 2 showed the hot-pressed specimens had an increased degree of crystallinity compared to the control specimens. Higher press temperature could cause higher degree of crystallinity in specimen. Furthermore, the densified specimens thickness swelling at the hot-pressing temperature of 200 °C was smaller than at 180 °C. The results implied that the recovery–prevention effort at the 200 °C densified specimens was greater than the 180 °C densified specimens. The recovery–prevention was related to the phenomena called spring back, whereby the more intense the recovery–prevention effort, the greater would be the capability of the specimen to prevent spring back action (Kocaefe et al. 2015, Darwis et al. 2017). The decreasing capability of wood in adsorbing or desorbing water vapour from and to the air was an essential factor to maintain the quality of wood products, especially those that would be placed in an air-conditioned room or exported to four-season countries with extreme changes in air humidity.

Table 4 showed the ΔE values of the control specimens and the thermally compressed specimens at 180 °C and 200 °C were 26.18 and
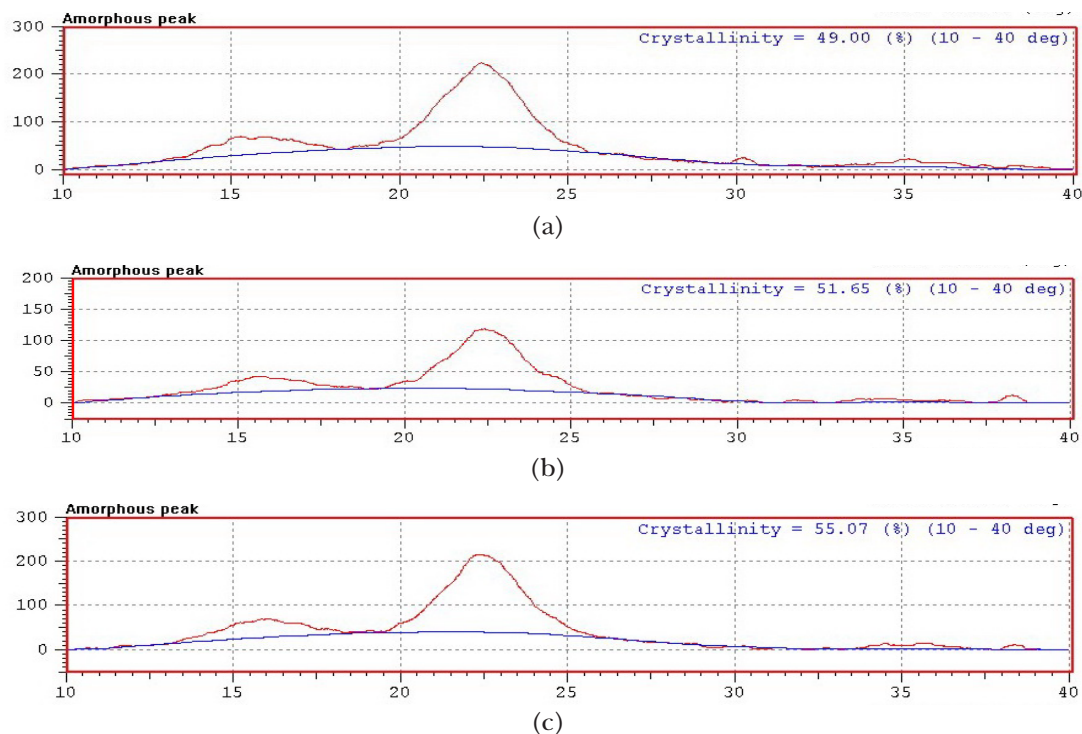

Figure 2 Crystallinity degree in densified and undensified (control) specimens
          a = control, b = densified at 180 °C, c = densified at 200 °C

Table 4 Colour and colour change of densified and undensified specimens

Wood specimens	Quantitative colour system[1]			Colour change (ΔE)	Implication[2]
	L	a	b		
Control/undensified	79.86	4.06	20.86		
Densified (at 180 °C)	56.40	11.20	30.04		
Densified (at 200 °C)	46.43	13.83	30.73		
NDS vs DS180				26.18	Different colour [3]
NDS vs DS200				36.20	Different colour [3]
DS180 vs DS200				10.33	Distinct colour changes [4]

[1] = averaged from 5 location points, [2] = reference from Table 1, [3] = colour of the densified specimens at 180 °C became darker or stronger (blackening and yellowish/reddish red) and intensively stronger at 200 °C, [4] = wood darkening colour at 200 °C was more distinct than that at 180 °C heat press; NDS = undensified (control) specimens, DS180 = densified specimens at 180 °C, DS200 = densified specimens at 200 °C

36.20, respectively. The ΔE values in both cases were greater than 12.00, indicating colour differences between the thermally compressed specimens and the control specimens. The ΔE value between the specimens thermally compressed at 180 °C and 200 °C was 10.33 (Table 4). The results indicated the use of hot pressing at 200 °C tended to produce a darker colour on the specimen surface than at the temperature of 180 °C. The discoloration of the smoked-wood specimens, where they appeared darker and stronger compared to the non-smoked specimens occurred due to heat exposure during the wood-smoking process (Hadi et al. 2020). Such discoloration of the thermally compressed specimens was due to the presence of the heat-stable or the least degraded lignin. Wood lignin was characteristically light in colour but its chemical structure easily changed and caused the colour to darken (Shmulsky & Jones 2019). The colour change was also caused by the

combustion of the specimen surface in contact with a hot-pressing metal. The combustion was due to the release of hydrogen and oxygen elements from the carbon element, which was naturally black in colour (Kucerova et al. 2016, Gasparik et al. 2019).

Although surface colours of the thermally compressed specimens were darker, their texture was smoother than that of the control specimens (Figure 3(b)–(c)). The surface of the thermally compressed specimens appeared glossy, which was possibly due to the flow of melting, glass-like shiny lignin (Budakci et al. 2015). The lignin flow transition process had rearranged, filled the space and levelled the uneven specimen surface more intensively at a high pressing temperature of 200 °C (Figure 3(c)). Therefore, the surface of the thermally compressed specimens would tend to be more attractive and had aesthetic characteristics than that of the non-thermally pressed or control specimens.
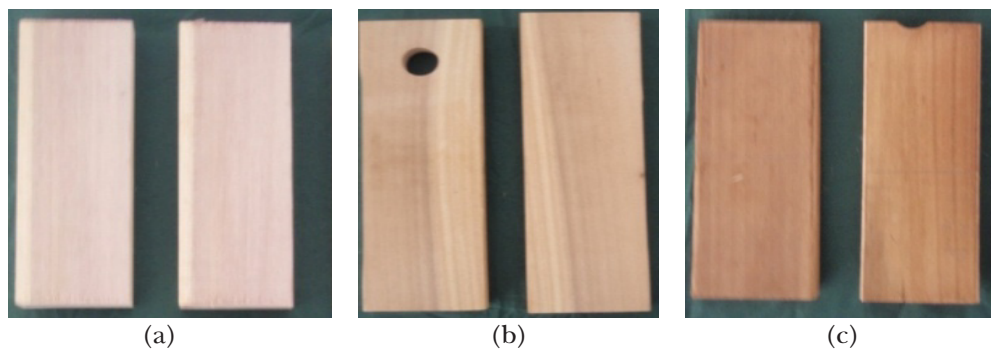

(a)   (b)   (c)

Figure 3 Visual appearance of densified and undensified (control) specimens
a = control, b = densified at 180 °C, c = densified at 200 °C

CONCLUSION

The thermal compression treatment caused extensive depolymerisation or fragmentation of the chemical components such as hemicellulose, cellulose and lignin of 8-year-old waru gunung specimens. Hemicellulose was the most severely degraded, whereas cellulose experienced less severe degradation and lignin was shown to be the least to degrade. When compared to control specimens, the thermally compressed specimens at the temperature of 180 °C–200 °C and at the pressure of 2.452 N mm^{-2} had increased density and surface hardness but decreased thickness swelling and equilibrium moisture content. The thickness swelling and equilibrium moisture content of the thermally compressed specimens were lower at 200 °C than at 180 °C, whereas their density and surface hardness were higher at 200 °C than at 180 °C. Nevertheless, the thermally compressed specimens had darker colours, glossier surfaces and more aesthetic appearance at 200 °C than at 180 °C.

ACKNOWLEDGEMENTS

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