Mechanical Properties and Chemical Changes of Mahoni Wood (*Swietenia mahagoni*) by Close System Compression Hot Press Machine

W. Dwianto[1,], T. Darmawan[1], D.S. Adi[1], Y. Amin[1], I. Wahyuni[1], M. Karina[2]*

[1]Research and Development Unit for Biomaterial, Indonesian Institute of Sciences, Bogor, Indonesia; wahyudwianto@yahoo.com
[2]Research Center for Physics, Indonesian Institute of Sciences, Bandung, Indonesia

Abstract. This paper deals with mechanical properties (modulus of elasticity and modulus of rupture) of compressed wood by Close System Compression (CSC) and its chemical component changes. A conventional hot press is equipped with an air-tight seal frame and placed between the two hot plates machine. The high moisture content of wood is laid in the CSC and pressed. The trapped wood moistures will produce steam and act as self steam treatment. Previous study showed that permanent fixation by a conventional hot press could be achieved at a heating temperature of 180°C for 20 h and accompanied by the great reduction of the mechanical properties of compressed wood. On the other hand, that for steam treatment in an autoclave was achieved at 180°C for 10 min and no marked decrease in the modulus of rupture were observed. The wood species used in this research was Mahoni (*Swietenia mahagoni*), with 0.39 ~ 0.42 g/cm^3 density and 61% moisture content. The wood specimens were cut into 30 cm (L) x 2 cm (T) x 2 cm (R) and compressed into 50% of their initial thickness in radial direction inside the CSC at 160, 180, and 200°C for 10 and 20 min. Chemical components of 40~60 mesh wood powder were also analyzed to observe the changes of their holocellulose, α celulose, lignin and extractives contents. The result showed that density of compressed woods increased between 0.58 ~ 0.81 g/cm^3; fixation was achieved at 180°C for 20 min with RS 0.36% and mechanical properties increased by 50% compression level. However, mechanical properties decreased with the increasing of temperature and time. It also revealed that holocellulose and α-cellulose decreased around 13~33% and 1 ~ 10%, respectively. Therefore, we considered that the fixation of compressive deformation by CSC resulted from the release of internal stresses stored in the cell wall by a structural change in the cellulose and partial hydrolysis of hemicelluloses due to their degradations.

Keywords: CSC, fixation, mechanical properties, chemical changes

Introduction

Surface hardness and strength of low density wood could be increased by compression process. When a compressed wet wood specimen is dried under restraint, the stress gradually decreases until it disappears and the wood is fixed in the deformed state.

However, the fixation of deformation is impermanent [1], because it can be almost completely reversed by boiling. Therefore, permanent fixation of deformation is required to utilize compressed wood.

Many attempts have been made to fix the compressive deformation of wood permanently. Heat treatment, i.e. heating of wood under dry conditions at high temperatures, is effective method of fixing. Inoue and Norimoto [2] investigated the permanent fixation of compressive deformation of Sugi (*Cryptomeria japonica* D. Don) wood by heat treatment under dry conditions. They reported that permanent fixation could be achieved at heating temperature of 180°C for 20 h, 200°C for 5 h or 220°C for 3 h. Heat treatment can be performed easily using a conventional hot press and is of practical use for small-scale production. Unfortunately, not only it takes a long time to achieve complete fixation, it also causes a great reduction of mechanical properties of the compressed wood.

Steam treatment, i.e. heating of wet wood or having high moisture content at high temperatures, is also an effective method of fixing the compressive deformation of wood. Steaming is performed in an autoclave [3] or using a hot press equipped with an airtight seal [4]. Inoue *et al*. [3] compressed wood under restraint at vapor pressures of 9 to 20 kgf/cm^2 in an autoclave. They reported that permanent fixation was achieved at 180°C for 10 min or at 200°C for 1 min. They also observed no effect of steam treatment on fixation for dry specimens by using a hot press equipped with an airtight seal [4], and concluded that the moisture content of the wood affected fixation. No marked decrease in the modulus of rupture or drastic color changes, which were seen following heat treatment of wood, were observed with steam treatment.

Although complete fixation can be achieved by steaming in a very short time, the apparatus is expensive and the operation is difficult. This problem can be solved by Close System Compression (CSC) method. A conventional hot press is equipped with an air-tight seal frame placed between the two hot plates of the machine. The wood with high moisture content is laid in the CSC frame and pressed. The trapped wood moisture will produce steam and act as self steam treatment [4].

Amin and Dwianto [5] reported that wood compression using CSC method at 180°C temperature for 30 min with vapor pressure of 9.5 kg/cm^2 was still recovered to a 8.92% from its initial thickness and lost 12.79% of its weight. Moisture content of the wood, pressing temperature and time, and vapor pressure has an effect on the decreasing of recovery of set (RS). It needed 180°C temperature and 10 kg/cm^2 vapor pressure to get the permanent fixation. Therefore, it was necessary to add water to produce the steam pressure

besides the one that was produced from the evaporation of wood moisture content.

Wood is lignocellulosic material which is susceptible against thermal degradation. Steam treatment inside the CSC frame is needed to fix the deformation, though it causes degradation of its chemical components. Amin dan Dwianto [5] stated that weight loss of compressed wood by CSC method was higher than that by heat treatment [6], because heated wood in wet condition had more degraded wood chemical components. However, how much the degradation of its chemical components has been not yet known.

This paper deals with mechanical properties of compressed wood by CSC method and its chemical component changes. The aim of this research was to observe the changes of its holocellulose, α-celulose, lignin and extractive contents and the decreasing of mechanical properties at the fixation state.

Experimental

The wood species used in this research was Mahoni (*Swietenia mahagoni*), with 0.39 ~ 0.42 g/cm^3 density (average = 0.41 g/cm^3) and 61% moisture content. The wood specimens were cut into 30 cm in longitudinal (L) x 2 cm in tangential (T) x 2 cm in radial (R) and compressed into 50% of their initial thickness in R direction inside the CSC frame at 160, 180 and 200^{o}C for 10 and 20 min. The compressed wood specimens were then dried at 60^{o}C under restraint for 24 h.

Density and fixation measurements were conducted based on their oven dried weight. The wood specimen size was 2 cm (L) x 2 cm (T) x 1 cm (R). Fixation level was calculated by a formulation as follows [7]:

$$RS\ (\%) = [(Tr - Tc) / (To - Tc)] \times 100, \qquad (1)$$

where To = thickness of wood specimen before compression (mm), Tc = thickness after compression (mm), dan Tr = thickness after boiling for 30 min (mm).

Static bending tests were done by using Universal Testing Machine (UTM) according to ASTM D 143-94 [8]. The wood specimen size after compression was 30 cm (L) x 2 cm (T) x 1 cm (R). Chemical components of 40 ~ 60 mesh wood powder were also analyzed based on Mokushitsu Kagaku Jiken Manual [9] to observe the changes of their holocellulose, α-celulose, lignin and extractives contents. Chemical components of initial wood and after treatments were then compared to obtain the percentage of loss.

Results and Discussion

Physical Properties

The results show that density of compressed woods were between 0.58 ~ 0.81 g/cm^3 (average = 0.69 g/cm^3), increased almost 2-fold compared to the average density of uncompressed wood. Narrowing of wood pores by the compression caused the increase of wood density [10]. These results fitted the experiments done by Murhofiq [11] that 50% compression increased the density of agathis wood from 0.41 g/cm^3 to 0.79 g/cm^3 and sengon wood from 0.23 g/cm^3 to 0.48 g/cm^3. Sulistyono [12] also observed that the density of agathis wood increased between 0.43 ~ 0.46 g/cm^3 to 0.70 ~ 0.85 g/cm^3 by 50% compression.

RS decreased with the increasing pressing temperature and time. Fixation state has been achieved at 180^{o}C temperature for 20 min, resulting in RS = 0.36%. This result was better than the previous research done by Amin and Dwianto [5]. This was due to the vapour pressure inside CSC frame was maintained at 10 kg/cm^2. However, the compressed woods were damaged at 200^{o}C because pressing time to reach fixation at this temperature was shorter [3, 13].

Mechanical Properties

The results of static bending tests were shown by Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) values (Fig. 1).

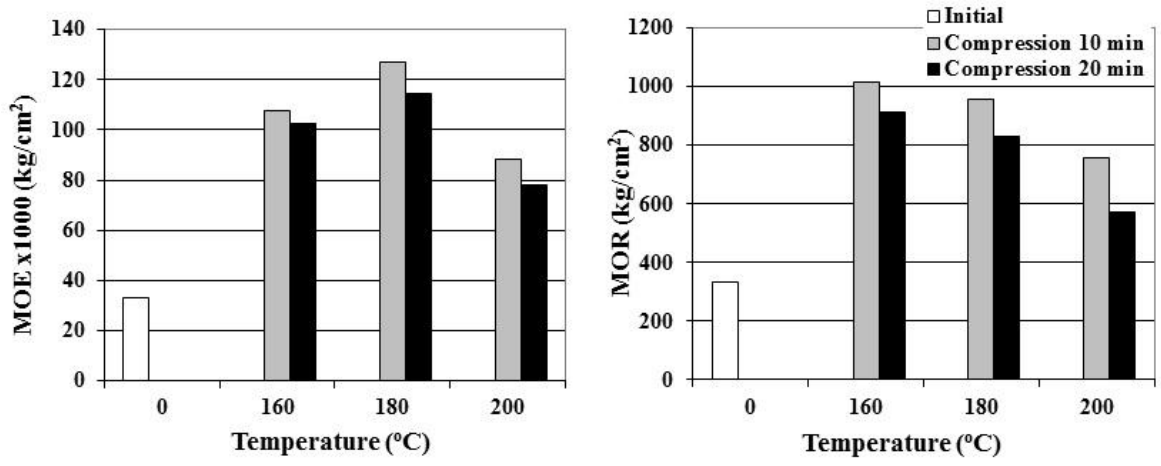

Figure 1. MOE and MOR values of compressed wood by CSC method with various pressing temperatures and times.

By 50% compression, MOE and MOR values increased more than 2-fold compared to the uncompressed wood. Besides as the effect of the increase of wood density, these values were probably due to the partly formed crystalline cellulose [14]. The highest MOE value

was achieved at pressing temperature of 180°C for 10 min, while the highest MOR value was reached at pressing temperature of 160°C for 10 min. However, the values declined with the increase of pressing temperature and time, especially at 200°C temperature. The decline of MOE and MOR values indicated the starting degradations of chemical components of wood occurred. MOE dan MOR values at the fixation state were 114.3 x 10^3 kg/cm^2 and 830.9 kg/cm^2, respectively.

Chemical Component Analysis

Wood chemical component analysis of holocellulose, α-cellulose, lignin and extractive content are given in Table 1. It can be seen that the higher the temperature and the longer the pressing time, the lower holocellulose content. According to Fengel and Wegener [15], when lignin is removed from wood, the holocellulose represents the amount of cellulose and hemicellulose. The low level of the holocellulose was suspected as the effect of thermal degradation of hemicellulose since the content of α-cellulose did not significantly change.

Table 1. Wood chemical component analysis.

Chemical components	Control (%)	160ºC		180ºC		200ºC	
		10'	20'	10'	20'	10'	20'
Holocellulose	77.60	66.80	66.43	58.57	51.86	55.72	53.84
α-cellulose	47.89	44.12	45.76	45.34	42.85	47.11	47.84
Lignin	26.10	35.31	34.28	35.88	35.36	33.95	33.99
Extractive	4.98	4.61	4.53	5.96	7.64	8.69	10.61

Hsu *et al.* [16] stated that the steam treatment can cause wood fixation due to the hydrolysis of hemicellulose. Hemicellulose has a lower degree of polymerization compared to cellulose [17], and has lower and unstable molecular chains that are easily degraded by heat. Besides, hemicellulose is a polymer consisted of six carbon sugars such as mannose, galactose, glucose and 4-O-methyl-D-glucuronic acid, and five carbon sugars such as xylose and arabinose [18-19]. Therefore, as the pressure and pressing time increase, the sugar polymer chains of hemicellulose will be broken and turn into simple sugars. These simple sugars will become more unstable against temperature which leads to the excessive degradation.

The decrease of α-cellulose at fixation state, at the temperature of 180°C for 20 min, was 10.52% of the initial, where the degraded part was suspected to be its amorphous region.

At the same time, the holocellulose was degraded for 33.17% of the initial, in which most of this was its hemicellulose fraction.

The reason of the fixation can be explained as the effect of the removal of some of OH-functional group that decreased the ability of wood cellulose to bind water (hygroscopicity). This removal of some hemicellulose fraction would lead the contiguous cellulose components to link to each other or to form new chemical bond, i.e. ether linkage [17]. Actually, the α-cellulose fraction did not degrade significantly since it has long molecular chain with high degree of polymerization [17] that makes it resistant to heat degradation under 200^{o}C.

To clarify the mechanism of the permanent fixation of compressive deformation of wood by high temperature steaming, the stress relaxation and stress-strain relationships in the radial compression for Sugi wood has been measured under steam at temperatures up to 200^{o}C by Dwianto [20-21]. The relationship between the residual stress at the end of relaxation measurements could be expressed by a single curve regardless of time and temperature. This fact proved that the permanent fixation of compressive deformation was resulted from the release of stresses stored in the cell wall polymers by their degradation.

Table 1 also shows that the lignin content increases with increasing pressing temperature and time. This trend of increasing lignin content is relatively similar to the research conducted by Akyildiz *et al.* [22]. The same result was also uncovered by Fengel and Wegener [15] that the lignin content remains constant in a wide temperature range and will increase at the temperature above 140 ~ 150^{o}C. At temperature above 200^{o}C, lignin will be degraded while at the temperature below 200^{o}C, it will only soften. Glassy transition of lignin occurs at temperature of 130 ~ 150^{o}C [18]. Lignin is thermoplastic, which will soften at its glass transition temperature and will harden below that temperature.

Conclusion

The density of woods compressed into 50% of their initial thickness almost increased by 2-fold, i.e. from the average of 0.41 g/cm^3 to 0.69 g/cm^3.The increase of pressing temperature and time decreased the value of RS. Fixation was achieved for the specimen pressed at temperature of 180^{o}C for 20 min, with RS value of 0.36%. With 50% compression level, the values of MOE and MOR increased more than 2-fold compared to the initial values. The MOE and MOR values at fixation state were 114.3 x 10^3 kg/cm^2

and 830.9 kg/cm^2, respectively.The decrease of α-cellulose content at fixation state was 10.52% of the initial which was assumed to be its amorphous region. At the same time, the degraded holocellulose was 33.17% of the initial with most of it was hemicellulose. It also revealed that holocellulose and α-cellulose decreased around 13 ~ 33% and 1 ~ 10%, respectively. Therefore, we considered that the fixation of compressive deformation by CSC was resulted from the release of internal stresses stored in the cell wall by a structural change in the cellulose and partial hydrolysis of hemicelluloses due to their degradations.

[1] M. Norimoto, J. Gril. *J. Microwave Power. Electromagn. Energy*, **1989**, *24* (4), 203.
[2] M. Inoue, M. Norimoto. *Wood Res. Tech. Notes*, **1991**, *27*, 31.
[3] M. Inoue, M. Norimoto, M. Tanahashi, R. M. Rowell. *Wood Fiber Sci.* **1993**, *25* (3), 224.
[4] M. Inoue, M. Norimoto. Proc. the International Symposium on the Utilization of Fast-Growing Trees. China Forestry Publishing House, Beijing 1994, 56–64.
[5] Y. Amin, W. Dwianto. *J. Trop. Wood Sci. Technol.* **2006**, *4* (2), 55.
[6] W. Dwianto, M. Inoue, M. Norimoto. *Mokuzai Gakkaishi*. **1997**, *43* (4), 303.
[7] M. Inoue, K. Minato, M. Norimoto. *Mokuzai Gakkaishi.* **1994**, *40* (9), 931.
[8] American Standard Testing Method (ASTM). D143-94 : Standard Test Methods for Small Clear Specimens of Timber, 2005.
[9] Mokushitsu Kagaku Jiken Manual, Japan Wood Research Society Publisher, 2000.
[10] A. Darwis, W. Dwianto, I. Wahyudi. Proc. the First International Symposium of Indonesian Wood Research Society. Bogor. 2009, 71–78.
[11] S. Murhofiq. Jurusan Teknologi Hasil Hutan, Fakultas Kehutanan, Institut Pertanian Bogor. Skripsi. Unpublished, 2000 (In Indonesian).
[12] Sulistyono, N. Nugroho, S. Surjokusumo. *Buletin Teknik Pertanian* **2003**, *17* (1) (In Indonesian).
[13] Y. Ito, M. Tanahashi, M. Shigematsu, Y. Shinoda, C. Ohta. *Holzforchung*. **1998**, *52* (2), 211.
[14] R. Hartono. Program Studi Ilmu Pengetahuan dan Kehutanan. Program Pascasarjana IPB. Thesis Doktor. Unpublished, 2011 (In Indonesian).
[15] D. Fengel, G. Wegener, "*Wood, Chemistry, Ultrastructure, Reactions*", De Gruyter, New York, 1984.
[16] W. E. Hsu, W. Schwald, J. Schwald, J. A. Shields. *Wood Sci. Technol*. **1988**, *22*, 281.
[17] S. Prawirohatmodjo, S, "*Kimia Kayu*". Diktat Kuliah Kimia Kayu Mahasiswa Jurusan Teknologi Hasil Hutan Fakultas Kehutanan. Universitas Gadjah Mada, Yogyakarta, 1996 (In Indonesian).
[18] C. J. Biermann, in: "*Hand Book of Pulping and Papermaking*", 2nd ed., Academic Press, California USA 1996.
[19] S. N. Marsoem. "*Pulp dan Kertas*". Bahan Kuliah Mahasiswa Jurusan Teknologi Hasil Hutan Fakultas Kehutanan Universitas Gadjah Mada, Yogyakarta, 2005 (In Indonesian).
[20] W. Dwianto, T. Morooka, M. Norimoto. *Mokuzai Gakkaishi*. **1998**, *44* (2), 77.
[21] W. Dwianto, T. Morooka, M. Norimoto, T. Kitajima. *Holzforschung*. **1999**, *53* (5), 541.
[22] H. M. Akyildiz, S. Ates, H. Osdemir. *African J. Biotechnology*. **2009**, *8* (11), 2565.