Evaluation of Selected Properties of Modified *Sapium Baccatum* and *Neolamarckia Cadamba*

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Abstract

This study was to evaluate the selected performances of modified Ludai (*Sapium Baccatum*) and Kelempayan (*Neolamarckia Cadamba*) wood species. Ludai and Kelempayan were impregnated with acetic anhydride at 80°C, 100°C, and 120°C for 15, 30, 60, 180, 300, and 420 min without catalyst. The highest WPG of 16% and 14% for Ludai and Kelempayan were obtained. VC% ranging from 0.5% to 10% were obtained. The ultimate OH-groups substituted of acetylated Ludai and Kelempayan at 120°C were 3.77 mmoles/g and 3.98 mmoles/g, respectively. Unlike the conventional preservative treatment in the industry today, wood-acetyl bonds pose a positive impact on the ecosystem.

Keywords: Chemical modification, Weight per cent gain, Volume Change. Hydroxyl (OH) substitutions.

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1.0 Introduction

Cellulosic-containing materials, including wood, are prone to shrinkage, swelling and bio-deterioration. These two main problems occur due to the ability of wood-OH to attract water from the surrounding. Changing the molecular structure of wood-OH into wood-acetyl could lessen the problems. Changing the molecular structure of wood-OH into wood-acetyl can be done by reacting the wood with acetic anhydride or acetylation. Therefore the aim of wood modification is to bring about an improvement in decay resistance or dimensional stability, reduce water sorption and improve weathering performance (Siti Rafedah Abdul Karim, et al., 2020, Hill C.A.S et al., 2008; Kwon et al, 2007, K, Siti Rafidah et al, 2006, Hill, C.A.S, 2008, Rowell, R.M 2005,). Inferior wood properties can be overcome by changing the molecular structure of wood-OH into hydrophobic structure, hence reducing the shrinkage and swelling of the modified wood (Hill, 2008 and Rowell, 2005). Chemical modification of wood with acetic anhydride or acetylation results in changes to the physical and biological properties of the material (Azizul I, et al., 2013). The majority of these properties appear to be determined by the weight per cent gain (WPG) of the material rather than the extent of hydroxyl substitution. The moisture content of wood influences other properties, such as dimensional stability, mechanical properties, decay resistance (Epmier, H. et al., 2007; Chang, H.T and Chang, S.T., 2002)., and the swelling behaviour of modified wood (Hill, C.A.S, 2008). The hydroxyl groups contained in wood affect the water sorption of wood, and the number of hydroxyl groups of lignocellulosic materials decreases after the chemical modification. The hydrophilic nature of the cell wall is reduced by the bonding of the hydrophobic groups (Rowell, R.M. and Ellis, W.D., 1978). In the case of acetylation, wood-OH, which is hydrophilic or water-liking in nature, is changed into wood-acetyl, which is hydrophobic or water-hating in nature.

Various studies of the relationship between the swelling of the material due to modification have been published. It has long been established that the modification of a wood substrate with acetic anhydride causes an increase in the volume of the wood. The cell wall of wood swells as the OH groups of the cell wall polymers are substituted. This swelling occurs because the covalently bonded groups occupy space within the cell wall and cause a localised deformation of the cell wall polymeric network. The volume degree due to modification is equal to the volume of the acetyl groups in the wood and the associated void volume. Acetylation is a single-site reaction, which suggests that one hydroxyl group will be substituted by one acetyl group with no polymerisation. Because small hydroxyl groups are substituted with larger acetyl groups, the wood will remain in a permanently swollen state and become heavier. In essence, acetylation depicts a chemical reaction that introduces the acetyl functional group into natural fibres. In other words, the hydroxyl (OH groups) are replaced by a hydrophobic acetyl group (CH_3CO), inducing the change of the hydrophilic nature to the hydrophobic nature of the fibre. Due to the potential of acetylation to wood materials, studies on acetylated bamboo have also grown. A study on wood acetylation using vinyl acetate (VA) and acetic anhydride (AA) showed that VA could acetylate cellulose and lignin, but AA reacted more readily with lignin than VA (Saisai Huang, et al., 2019).

Ludai and kelempayan are abundantly found pioneer wood species in the tropics, which have inferior properties to other unmodified wood. Unmodified wood contained wood-OH in the molecular structure. OH is capable of attracting water from the surrounding. This phenomenon is the main reason for dimensional instability and bio-deterioration of wood products. No literature was found on the chemical modification of these species.

The objectives of this study were to determine WPG%, volume change%, and OH substituted of acetylated Ludai and Kelempayan.

The results of this study provide preliminary information on the performances of Ludai and Kelempayan after modification with acetic anhydride, without using the catalyst and vacuum-impregnation.

The initial part of the article presents the literature review of acetylation on wood species. Most literature found on wood modification was on acetylation. Samples were then modified in the reactor flask at different times and temperatures. Performances of modified wood were evaluated by determining the WPG%, volume change%, and OH substituted. Results were then analysed and discussed.

2.0 Literature Review

For the past 20 years, many studies on acetylation have been carried out on wood (Lisbeth, G.T., et al., 2021; Hill, C.A.S. et al., 2008, 2005, 2004, 1999, 1996). Wood modification aims are to bring about an improvement in decay resistance or dimensional stability, to reduce water sorption, and to improve weathering performance (Saisai Huang, et al., 2019, Hill C.A.S et al., 2008; Kwon et al, 2007, K, Siti Rafidah et al, 2006, Hill, C.A.S, 2008, Rowell, R.M 2005,). Furthermore, inferior wood properties can be overcome by changing the molecular structure of OH into a hydrophobic structure, hence reducing the shrinkage and swelling of the modified wood (Rowell, 2005). In essence, acetylation depicts a chemical reaction that introduces the acetyl functional group into natural fibres. In other words, the hydroxyl OH- groups are replaced by a hydrophobic acetyl group (CH_3CO), inducing the change of the hydrophilic nature to the hydrophobic nature of the fibres. Due to the potential of acetylation to wood materials, studies on acetylated bamboo have also grown.

Decay properties have been studied, and various hypotheses have been proposed to explain how acetylated wood is protected from decay. These include the blocking of enzyme recognition, reduction in cell wall moisture content (MC), and physical blocking of the cell wall micropores (Hill et al., 2008; K. Siti Rafidah, et al., 2006). The mechanism of resistance to fungal attack by acetylation is said to be due to the suppression of fungal growth (Rowell, R.M., 2005). The fibre Saturation Point (FSP) of the acetylated wood was reduced, thus imparting decay resistance. OH-group substitution with acetyl groups was reported to suppress decay (Greeley, B., et al., 2018). Hill (2004 & 2008) reported that FSP of acetylated Corsican pine was reduced with increasing Weight Percent Gain (WPG). In the said report, FSP was determined by solute exclusion technique, and the reduction of FSP was simply due to bulking effect of the wood cell wall by bonded acyl groups. Solute

exclusion is a very comprehensive study that needs modelling to interpret the result. The latest study on furfurylated wood hypothesised that furfurylation enhances moisture exclusion within the cell wall through impregnation polymerisation and offers long-term protection compared to acetylation that depends on the replacement of hydroxyl groups with ether-bound adducts that can be removed by fungi (Lisbeth, G.T., et al., 2021). No other studies report on furfurylation.

For comparisons, acetylation of bamboo with potassium acetate as a catalyst obtained WPG% of 19.6% (Saisai Huang, et al., 2019), acetylation of bamboo with vinyl acetate and methyl methacrylate reaches 18.95% (Saisai Huang, et al., 2019). With acetylation of rubberwood without catalyst, a 27-hour reaction period resulted in an average WPG of only 13%, and two further reactions were performed at 94 and 144 hours to give average WPGs of 15 and 16.6%, respectively (K. Siti Rafidah, et al.,2006).

Acetylation resulted in bulking of the wood cell wall. This bulking effect is permanent where the chemical does not leach out. The change from a single bond to a double bond of the treated wood is proven by FTIR or ATR. Chemical modification is non-toxic because the chemical is grafted to the wood matrix, and the permanence of the bonding makes it superior as compared to conventional chemical impregnation techniques. Chemical modification represents a process that is used to improve material properties and can be disposed of at the end of a product life cycle without presenting an environmental hazard. Improvement of all these properties is determined by WPG%, and FSP% is a function of WPG% (Gauss, C., et al.,2021, Lisbeth, G.T., et al., 2021 and Hill, C.A.S 2008). Previous studies have proven that reduction in FSP as determined by solute exclusion correlates with the degree of bulking of the cell wall due to the presence of the covalently bonded acyl substituents (acetyl in this case). As the decay resistance was confirmed to be related to WPG% (Hill et al., 2008, 1999, 1996), therefore it is anticipated that reduction in mass loss% (decay indication) has some correlation to WPG%. Since acetyl groups occupy a certain space within the wood cell wall, it can be hypothesised that only a limited space is available for bound water attraction to the wood cell wall. A theoretical FSP at a range of WPGs can be calculated from the bonded volume of the bonded acetyl as determined by a helium pycnometer, and these data correlate well with FSP values from solute exclusion. Mass Loss% data is correlated to data of FSP and WPG, and a previous study had shown that zero mass loss occurs at an FSP value of 20%.

Very few studies were found on the effects of acetylation on mechanical properties. Papadopoulos, A.N., and Pougioula, G., (2010) found that the improvement of compression strength imparted by chemical modification is independent of the degree of bulking of the cell wall but correlates well with the degree of substitution of the cell wall hydroxyl groups.

3.0 Methodology

Freshly-felled kiln-dried Ludai (*Sapium spp*) and Kelempayan (*Neolamarckia Cadamba*) were processed into 20 x 20 x 5 mm (radial x tangential x longitudinal) sample dimensions. Samples were smoothed with sandpaper to remove loosely adhering fibres, labelled, and

then solvent extracted using a mixture of toluene/ethanol/acetone (4:1:1 by volume) for 6 hours in the Soxhlet apparatus. Samples were then air-dried for 3 hours and then oven-dried overnight at 105°C. Samples were removed from the oven, transferred to a vacuum desiccator, and allowed to cool to ambient temperature over silica gel, and weight and dimensions were determined. Extractive-free samples (10 replicates) were then added periodically to a reaction time of 15, 30, 60, 180,300, and 420 min in heated acetic anhydride at 80°C, 100°C, and 120°C respectively. Impregnation was done without a vacuum to reduce treatment costs. At the end of the reaction period, the hot reagent was decanted off, and the samples were added to ice-cold acetone for two hours and subjected to Soxhlet extraction toluene/ethanol/acetone (4:1:1 by volume) for 8 hours to remove by-product, and then air dried and oven dried at 105°C for 12 hours. Weight gain (g) and dimensions (mm) were recorded.

Volume change % was determined by vernier calliper for calculation of external volumetric in mm^3. A helium pycnometer was not available at the time of measurement.

Weight per cent gain (WPG) due to the reaction was calculated according to the well-known formula:

$$WPG\ (\%) = [(\ Wmod - W\ unmod\ )\ /\ Wunmod] \times 100 \quad (1)$$

Where,

Wmod is the oven-dried weight of the modified wood, and W unmod is the oven–dried weight of the same unmodified wood sample.

Percentage volume change (VC) as a result of modification was calculated based on the original volume of the same sample by measurement of external dimensions:

$$VC\ (\%) = [(\ Vmod - V\ unmod\ )\ /\ Vunmod] \times 100 \quad (2)$$

Where,

Vmod is the oven-dried volume of the sample after modification, and V unmod is the oven–dried weight of the same unmodified wood sample.

For a given WPG, acetic anhydride will have reacted with a different number of hydroxyl (OH) groups. The amount of substituted OH of wood after modification was calculated as follows:

$$OH\ groups\ (mmoles\ g\text{-}1) = [(\ Wmod - W\ unmod\ )\ /\ Wunmod] / (MW\ -1) \quad (3)$$

Where,

Wmod is the oven-dried weight of the modified wood, and W unmod is the oven – dried weight of the same unmodified wood sample. MW is the molecular weight of the respective acyl, and one mass unit is subtracted to account for the hydrogen atom lost during the reaction. [acetyl acyl = 43].

4.0 Results

4.1 Weight gain due to modification

A range of weight gain was obtained with varying reaction times and different temperatures, as shown in Fig 1a. The reaction curves show an asymptotic profile, and this type of profile has also been observed in acetylated and hexanoylated rubberwood (K, Siti Rafidah,et al., 2006). The appearance of rapid reaction at the initial stage of modification is primarily due to the high concentration of reagent of anhydride molecules and the wood hydroxyl groups. As the reaction proceeds, the reagent will penetrate the interior of the sample. During the reaction of the chemical modification with acetic anhydride, the reagent must travel via the cell wall microporous network to reach the reactive sites. The majority of the reactive OH sites of the cell wall are located within the interior. The mechanism for such transport is diffusion, driven by differences in concentration gradient between reagent molecules distributed at the surface cell wall and within the cell wall. A linear relationship in Fig. 1b showed that the reaction was driven by diffusion reaction.

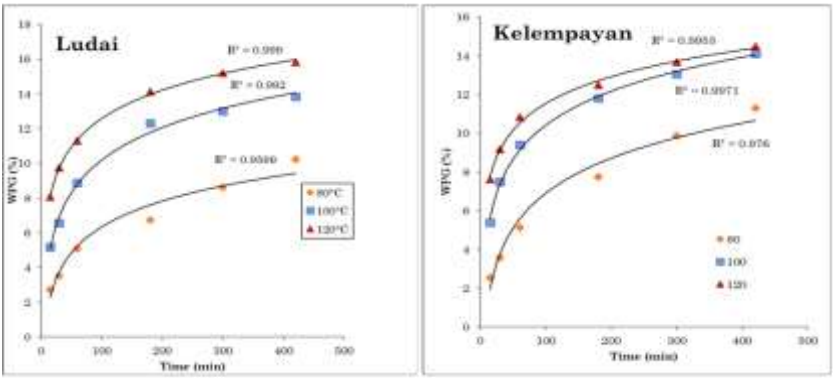


Fig.1a: Reaction Profile of acetylated Ludai and Kelempayan at three different temperature

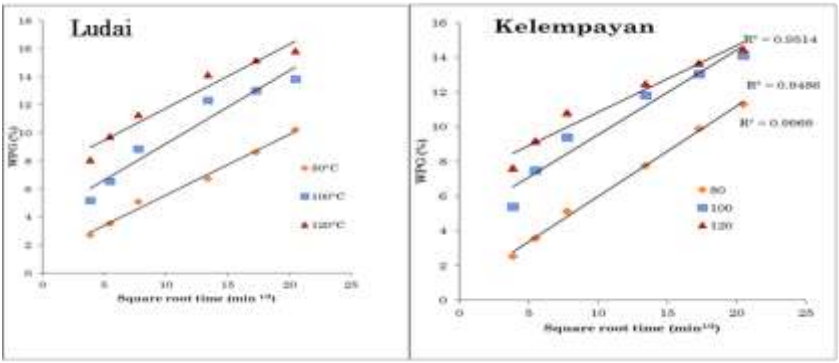


Fig 1b: Relationship between Weight Percent Gain (WPG%) and Square Root Time (Min 1/2) for Ludai and Kelempayan

For diffusion to take place during the modification process, the diameter of the reagent should be smaller than wood pores. The microvoids of sound wood have a diameter of 20 – 40 Å, and the diameter of the acetic anhydride used in this study is 6.76 Å. Theoretically, diffusion of acetic anhydride via cell wall microvoids is possible. Furthermore, the form of this profile is influenced by temperature. At 80°C, the very slow reaction and a satisfactory WPG were not achieved, as explained by the insufficient penetration of acetic anhydride into the wood cell wall because both the wood swelling and concentration in the reagent solution were limited at that temperature. The WPG increased greatly at 120°C. WPG increased with increasing temperature. Acetylation is effective at high temperatures as it expedites the kinetic reaction of reagents without time consumption. An increase in the concentration gradient of the reagent is assumed to lead to the increasing diffusion mechanism. The rate of chemical reaction at the reactive site is rapid compared with the rate of diffusion.

4.2 Volume change due to modification

A constant relationship between volumetric increase and WPG of acetylated Ludai and kelempayan is shown in Fig. 2. This swelling is determined by measuring the external dimensions of the oven-dry wood samples before and after modification. An increase in volume indicates more stable dimensional stability as the hydrophilic OH groups have been substituted by a hydrophobic acetyl group and thus occupy space in the cell wall or known as the bulking effect. Volume changes due to modification result from the volume occupied by the reagent and associated void volume (K, Siti Rafidah et al., 2006).

As shown in Fig 2, a linear relationship was obtained for both acetylated Ludai and Kelempayan. An increase of 5% WPG resulted in 1.5% VC for Ludai, while an increase of 5% WPG resulted in 3% VC for Kelempayan. Lower VC% was found for Ludai due to the fact that Ludai wood possesses a higher density than Kelempayan. Fewer acetyl groups were inserted into the dense microvoid, resulting in less VC%. According to K, Siti Rafidah et al., (2006), 5% WPG resulted in 8% VC for acetylated rubberwood, which is higher than the current study. It is to be noted that in the referred study, rubberwood was modified by vacuum impregnated in a reaction flask containing a hot reaction solution at 110^0C. In this study, 144 hours of reaction time resulted in 16.6% WPG. The current study resulted in a WPG of 14% and 16% at 7 hours for Ludai and Kelempayan, respectively. Both studies showed an asymptotic profile which levels up at 7 hours (420 minutes) to 10 hours (600 minutes) reaction times. Fig 1a shows an asymptotic reaction profile which starts to level up at 420 minutes of reaction times. Seven hours of reaction time is optimum for modification because if the reaction time is extended to 144 hours, the maximum WPG obtainable is only 16.6%. For commercial purposes, extended reaction time is unnecessary because it will incur a cost. This justifies why in the current study, 7 hours is the maximum reaction time conducted.

Another reason for the lower VC% was that VC% calculation was based on the external dimension of wood samples which is inaccurate. Cell wall volume cannot be determined using vernier caliper. The use of a helium pycnometer allows for extremely accurate

determination of the cell wall volume of wood samples. The amount of helium gas in the helium pycnometer chamber determines the accurate volume of the internal cell wall volume since the molecular weight of helium gas is much smaller (4Å) than the size of the wood microvoids of 20 – 40 Å. Obviously, the initial volume of the external dimension is much greater than the cell wall volume, therefore lower VC% was obtained.

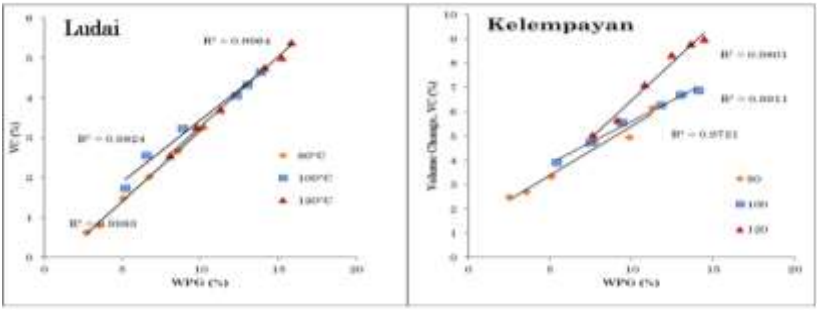


Fig 2: Relationship between Volume Change(%)and Weight Percent Gain(%) of acetylated Ludai and acetylated Kelempayan

4.3 Hydroxyl (OH-groups) substitution

The calculation of the OH-groups substitution in this study assumes that one mole of wood–OH is reacted with one mole modifying chemical without any cross-linking or polymerisation. The substitution of OH-groups and the volume occupied caused the irreversible volume change of modified wood. The relationship between OH-groups substituted and reaction time is as in Fig 3a. Asymptotic profiles are exhibited in all cases, showing that almost all of the OH-groups substitution had taken place in the early stage of the reaction. As an example, the ultimate OH-groups substituted of acetylated Ludai at 120°C with 3.77 mmoles/g is higher as compared to acetylated Ludai at 80°C with 2.43 mmoles/g. This can be explained by the desorption behaviour of bound water by substitution of the acetyl group, which results in the absorption of energy for breaking hydrogen bonds. Higher energy which comes from the higher temperature of the reaction medium, contributes to this acetylation process. The diffusion process results in a moisture flux, which is equal to both phases and is linked by sorption isotherm to satisfy the mass balance. The desorption of the bound water will be accompanied by the adsorption process and, in this case, the substitution of the acetyl group to the wood cell wall.

The reaction of wood with acetic anhydride or acetylation is a single-site reaction, where one acetyl substitutes one OH-groups of wood with no polymerisation. Meaning all weight gain in acetyl can be converted directly into a unit of OH-groups blocked. The reaction of anhydride with wood results in the esterification of the accessible OH-groups in the cell wall with the formation of carboxylic acid as a by-product. Fig. 3b demonstrated the ATR of both species before and after modification. The stretching band of OH-groups can be seen for unmodified samples, and the ester stretching band can be seen for modified samples.

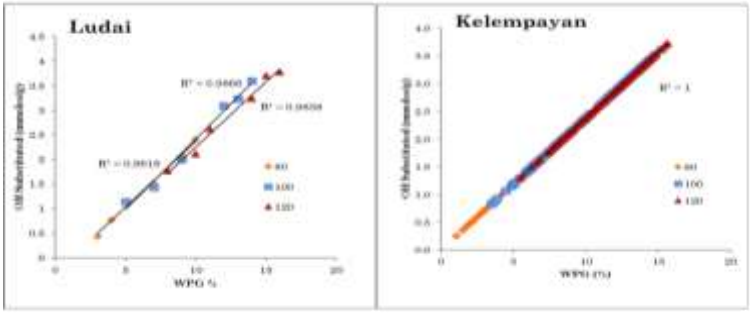
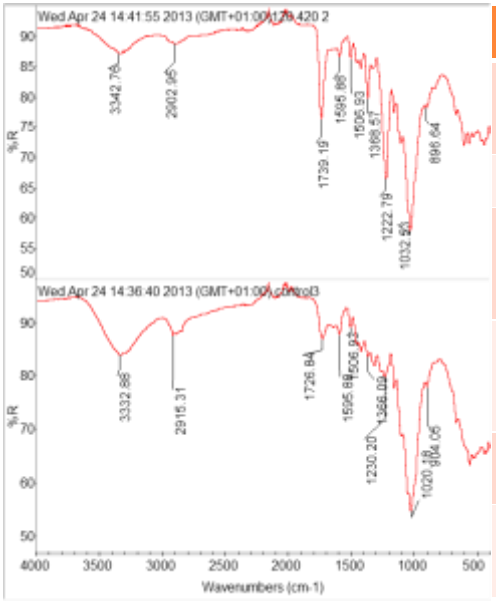


Fig 3a: Relationship between OH- groups substituted and reaction time of acetylated Ludai and Kelempayan



Frequency [cm^{-1}]	Assignment	Remark
3200-3550	OH	Stretching frequency of hydroxyl
2850-2950	C-H	Alkane C-H bonds
1720-1740	C=O	Stretching frequency of carbonyl goup
1500-1700	C=C	Stretching frequency of aromatic goup
1222,1230	C-O-C	Ester stretching bands
896, 904	C-H	Stretching of ß-glucosidic linkage

Fig 3b: Attenuated Total Reflectance, ATR of Ludai and Kelempayan

5.0 Discussion

Acetylation resulted in bulking of the wood cell wall. This bulking effect is permanent where the chemical does not leach out. The change from a single to a double bond of the treated wood is proven by the ATR result. Acetylation is non-toxic because the chemical is grafted to the wood matrix, and the permanence of the bonding makes it superior to the conventional chemical impregnation techniques. Chemical modification represents a process that is used to improve material properties and can be disposed of at the end of a

product life cycle without presenting an environmental hazard. Improvement of all these properties is determined by WPG%, and FSP% is a function of WPG%. Previous studies have proven that reduction in FSP as determined by solute exclusion correlates with the degree of bulking of the cell wall due to the presence of the covalently bonded acyl substituents (acetyl in this case). As the decay resistance was confirmed to be related to WPG%, therefore it is anticipated that reduction in mass loss% (decay indication) has some correlation to WPG%. Since acetyl groups occupy a certain space within the wood cell wall, it can be hypothesised that only a limited space is available for bound water attraction to the wood cell wall. A theoretical FSP at a range of WPGs can be calculated from the bonded volume of the bonded acetyl as determined by a helium pycnometer, and these data correlate well with FSP values from solute exclusion. Mass Loss% data is correlated to data of FSP and WPG, and a previous study had shown that zero mass loss occurs at an FSP value of 20%.

6.0 Conclusion

Ludai and Kelempayan were successfully modified using acetic anhydride. It has been shown that the increase in WPG, volume change, and percentage of OH-groups substitution is temperature dependence. Replacing the hydroxyl group with the acyl group, which has greater molecular volume, results in irreversible volume change and thus reduces the moisture to the sorption site. It should be noted that the samples in this study did not undergo vacuum-impregnated and without using the catalyst. The presence of existing air inside the void spaces will begin to compress when the reagent starts to enter the wood. Penetration will be slowed down because of the increasing back pressure. It is predicted that with vacuum impregnation, WPG% would be higher. Acetylation can reduce the problem of shrinkage and swelling of inferior wood species, including Ludai and Kelempayan. The problem of joints in furniture products can be solved with acetylated wood. The decay problem can be reduced where the FSP% of treated wood is reduced below 30%. With lower FSP%, absorbed water into wood is reduced. Moisture content, MC% of less than 20%, can stagnate the fungi growth that causes decay in wood. In short, modification can solve many problems of wood products in utilisation.

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Article Contribution to Related Field of Study

The article contributes to a greener approach to treating wood, bamboo, and other biomass from bio-deterioration and dimensional instability. On the same note, acetylated wood is dimensionally stable, and therefore longer utilisation of products is anticipated. Acetylated

wood is suitable for construction purposes. Permanent bulking of the acetylation means no leachate is released into the environment.

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