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ICWEP2022

https://icwep.uitm.edu.my/index.php
International Conference on Wood and Eco-Products 2022
Best Western Hotel iCity, Shah Alam, Selangor, Malaysia 15-16 Nov 2022
Organiser: Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM), Shah Alam Malaysia & Research Nexus UiTM,, Office of Deputy Vice-Chancellor (Research & Innovation)

E-B Environment - Behaviour Proceedings Journal

e-IPH e-International Publishing House Ltd., United Kingdom

Performances of *Ludai* (*Sapium Baccatum*) and *Kelempayan* (*Neolamarckia Cadamba*) after Acetylation

Siti Rafedah Abdul Karim[1*], Yanti Abdul Kadir[2], Azrena Abdul Karim[1]
**Corresponding Author*

[1] Faculty of Applied Science, Universiti Teknologi MARA 40450 Shah Alam Selangor, Malaysia
[2] Research Officer, Forest Research Institute Malaysia, Kepong, 52109 Selangor Malaysia

srafidah@uitm.edu.my; yanti@frim.gov.my; rena184@uitm.edu.my
Tel *: 019 346 6030

Abstract
The study changed the chemicals that make up Ludai (Sapium Baccatum) and Kelempayan (Neolamarckia Cadamba) molecules that have been modified with acetic anhydride. The wood species was treated at 80°C, 100°C, and 120°C. The changes were assessed by replacing the WPG and OH groups. The WPG range is 3%–16%, and therefore, at 16%, Ludai had the highest WPG, while Kelempayan had 14%. At 120°C, a 15% weight per gallon (WPG) solution changed Kelempayan volume by 9%. The Ludai volume changed 5.5% under the same conditions. The Ludai and Kelempayan had the highest OH-substitution rate at 120°C, 3.5–4 mmoles/g.

Keywords: Acetylation, Weight percent gain, Hydroxyl substitution, Volume change

DOI: https://doi.org/10.21834/e-bpj.v9iSI17.5977

1.0 Introduction

Moisture content in wood causes dimensional instability, reduces the mechanical properties and decreases the decay resistance (Chang 2002; Hill, 2008). Therefore, wood needs to be treated, especially for outdoor applications. Conventional impregnation treatments with preservatives such as boron and CCA are toxic and cause pollution to the environment because the preservatives leach out. Wood modification with chemicals is a possible approach. Chemical modification with acetic anhydride, or acetylation, causes a permanent swelling of wood because the hydrophobic acetyl molecules are grafted to the wood matrix, thus conferring protection. The hydroxyl groups contained in wood affect the water sorption of wood and the number of hydroxyl groups (OH groups) decreases after chemical modification (Hill et al., 2008; Rowell & Ellis, 1978).

Currently, only acetylation and furfurylation modification methods are commercialised for production due to the lower cost, simple manufacturing process, and excellent modification effect (Wenzhai et al., 2021; Antonios, 2019). Acetylation was successfully done for many wood species to improve dimensional stability and durability. Ludai and Kelempayan are lesser-known wood species found in many parts of the tropics. No literature was found on chemical modification on these wood species. In this study, Ludai and Kelempayan were chosen for acetylation. The effect of acetylation treatment on these wood species was evaluated by determining the weight gain after modification. The weight gain results determined volume change and OH-group substitution values. Due to the potential of acetylation to wood materials, studies on acetylated bamboo have also grown and reported. Acetylation of bamboo with potassium acetate as a catalyst obtained a WPG% of 19.6% (Saisai et al., 2019), acetylation of bamboo with vinyl acetate and methyl methacrylate reaches 18.95% (Saisai 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 (Karim et al., 2006).

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DOI: https://doi.org/10.21834/e-bpj.v8iSI17.5977

2.0 Literature Review

For the past 40 years, most acetylation were carried out on wood (Yue et al', 2022; Lisbeth et al., 2021; Wenzhai et al., 2021; Karim et al., 2020; Mingming et al., 2020; Papadopoulos et al., 2019; Hill et al., 2009; 2006; 2005; 2004; 1996). Acetylation is a single-site reaction, which suggests that one hydroxyl group will be substituted by one acetyl group. Since smaller hydroxyl groups are substituted with larger acetyl groups, the wood will remain permanently swollen or bulking and become heavier. Acetyl groups occupying space in the wood cell wall aim to improve the physical properties, hygroscopicity and resistance against mold, decay fungi, marine borers, and subterranean termites of the modified wood (Karim et al., 2020; Papadopoulos et al., 2019; Wenzhai et al., 2021; Mingming et al., 2020; Loh et al., 2011; Hill et al., 2009; Kwon et al., 2007; Karim et al., 2006, Hill, 2006; Rowell, 2005). Acetylation depicts a chemical reaction that introduces the acetyl functional group into the wood cell wall, reducing the shrinkage and swelling. In essence, the OH- groups are replaced with an acetyl group (CH_3CO), inducing a change from a hydrophilic nature to a hydrophobic nature. 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 (Mohamed et al., 2011). The modification methods include filling the wood cell lumen by physical or chemical means, such as physical filling with poly (glycidyl methacrylate) or phenolic resin, physical or chemical filling with polyacrylic resin, or physical or chemical filling with inorganic silica compounds. However, this strategy implies the use of large amounts of filler, resulting in high costs. Another method is to fill the dynamic nanopores of the hydrophilic hydroxyl group with a novel hydrophobic group. Thus, eradicating the affinity between wood and moisture. Polyethylene glycol, maleic anhydride, and silicon compounds have been explored for diffusion into the wood cell wall to fill the dynamic nanopores or bond to the hydroxyl group, preventing water from penetrating the cell wall, thus improving the dimensional stability and the decay resistance of wood (Mingming et al., 2020).

Acetylation resulted in the bulking of the wood cell wall. This bulking effect is permanent where the chemical did 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 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 (Hill, 2006). Improvement is determined by weight per cent gain, WPG. Fibre saturation point, FSP is a function of WPG (Hill, 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 covalently bonded acyl substituents (acetyl in this case). As the decay resistance was confirmed to be related to WPG (Hill et a., 2009), it is anticipated that reduction in mass loss (decay indication) has some correlation to WPG. Since acetyl groups occupy a specific space within the wood cell wall, it can be hypothesized that only a limited space is available for bound water attraction to the wood cell wall. A theoretical FSP at a range of WPG 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. Previous studies have shown that zero mass loss occurs at an FSP value of 20%.

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., 2009). The mechanism of resistance to fungal attack by acetylation is said to be due to suppression of the fungal growth. 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 et al., 2018). Hill (2004 & 2009) reported that FSP of acetylated Corsican pine was reduced with increasing Weight Percent Gain (WPG). In the report, FSP was determined by solute exclusion technique and the reduction of FSP was simply due to the 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 hypothesized that furfurylation enhances moisture exclusion within the cell wall through impregnation polymerization and offers long-term protection compared to acetylation depending on the replacement of hydroxyl groups with ether-bound adducts that can be removed by fungi (Lisbeth et al., 2021). Very few studies were found on the effects of acetylation on mechanical properties. Papadopoulos and Pougioula (2010) found out 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. In another study, the acetylation process increased the density by about 5.3% and decreased the equilibrium moisture content (EMC) by about 50% of the Acetylated Birch Plywood specimens, compared with the unmodified ones. Both increased density and decreased moisture content are associated with changes in mechanical properties (Yue et al., 2022).

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, 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. 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.

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

$$WPG\ (\%) = [(W_{mod} - W_{unmod}) / W_{unmod}] \times 100 \quad (1)$$

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:

$$VC\ (\%) = [(V_{mod} - V_{unmod}) / V_{unmod}] \times 100 \quad (2)$$

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:

$$OH\ groups\ (mmoles/g) = [(W_{mod} - W_{unmod}) / W_{unmod}] / (MW - 1) \quad (3)$$

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 1 mass unit is subtracted to account for the hydrogen atom lost during reaction. [acetyl acyl = 43].

4.0 Findings

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 had also been observed in acetylated and hexanoylated Rubberwood. 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.

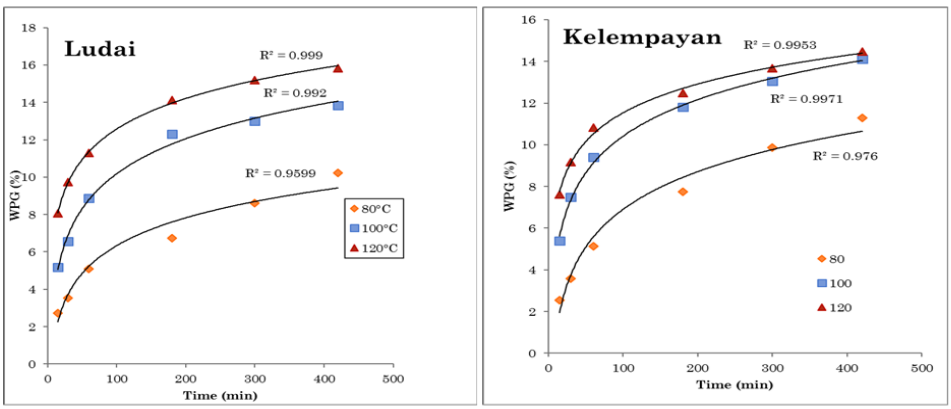


Fig.1a: Reaction Profile of acetylated Ludai and Kelempayan at three different temperature.

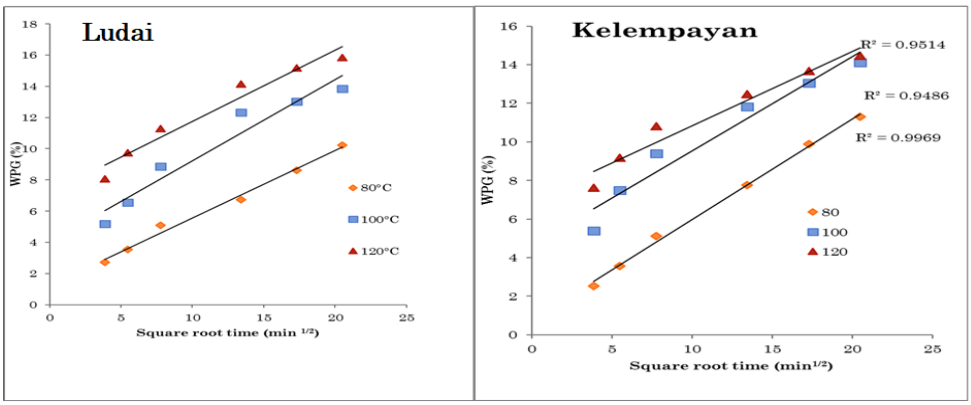


Fig 1b: Relationship between Weight Percent Gain (WPG%) and Square Root Time (Min $^{1/2}$) for Ludai and Kelempayan.

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 known as the bulking effect. Volume changes due to modification result from the volume occupied by the reagent and associated void volume (Rafidah, K.S, et al., 2006). Volume change in Kelempayan is higher as compared to Ludai, meaning more bulking effect takes place in Kelempayan. For example, at 15% WPG and 120^{0}C, volume change in Kelempayan was 9%, while the volume change in Ludai was only 5.5%.

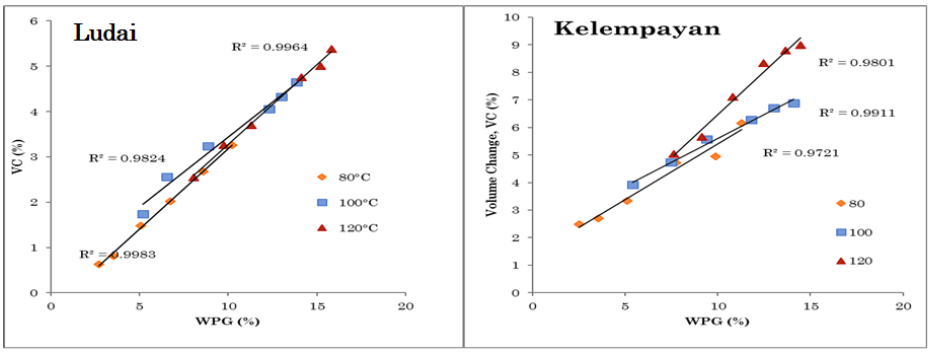


Fig 2: Relationship between volume change (%) and weight percent Gain (%) of acetylated Ludai and Kelempayan.

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 polymerization. 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 substitutions have 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 behavior 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.

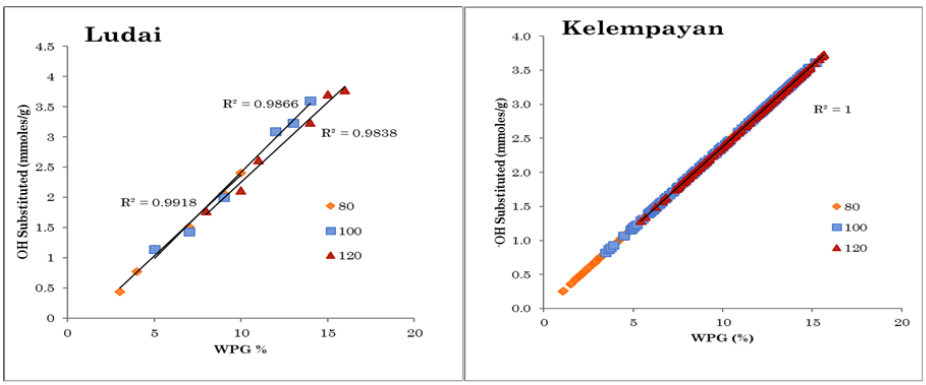
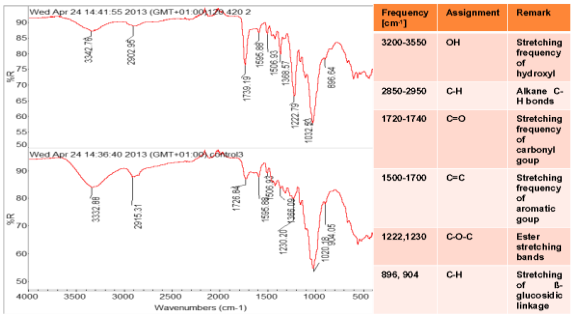


Fig 3a: Relationship between OH- groups substituted and reaction time of acetylated Ludai and Kelempayan.

The reaction of wood with acetic anhydride is a single-site reaction, where one acetyl substitutes one OH-group of wood with no polymerization. Meaning, that all weight gain in acetyl can be converted directly into units of OH-group 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.



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 B-glucosidic linkage

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

5.0 Discussion

Acetylation resulted in the 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 in 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 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's 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 a reduction in FSP as determined by solute exclusion correlates with the degree of bulking of the cell wall due to the presence of covalently bonded acyl substituents (acetyl in this case). As the decay resistance was confirmed to be related to WPG% (Hill et al., 2009), it is anticipated that a 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. We can figure out a theoretical FSP for a range of WPGs by measuring the bonded volume of the bonded acetyl with a helium pycnometer. These results agree well with the FSP values found by excluding solutes. Mass loss data is correlated to data from FSP and WPG, and a previous study had shown that zero mass loss occurs at an FSP value of 20%.

6.0 Conclusion and Recommendations

Ludai and Kelempayan were successfully modified using acetic anhydride. It has been shown that the increase in WPG, volume change, and percentage of OH substitution is temperature dependent. Changing the hydroxyl group to the acyl group, which has a bigger molecular volume, causes a volume change that cannot be undone. This makes the sorption site less wet. It should be noted that the samples in this study did not undergo vacuum impregnation 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 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 fibre saturation point (FSP%) of treated wood is reduced below 30%, and with a lower FSP%, water absorbed into the wood is reduced. Moisture content, MC% of less than 20%, can stagnate the fungi growth, causing decay in wood. Further decay and mechanical tests should be done to leverage the potential of local Ludai and Kelempayan as structural resources. For the past 40 years, wood chemical modification has focused on acetylation and furfurylation, which have been scaled up and exploited. Solutions in the science of nanotechnology can offer wood protection as an alternative, an innovative green approach. It is well known that the wood cell wall shows a porosity of molecular scale dimensions due to the partial filling of spaces between the cellulose microfibrils, mainly by polyoses and lignin. Small nanoparticles can profoundly and effectively penetrate the wood, alter its surface chemistry, and improve its properties, resulting in improved performance. The difficulty that acetylation modification needs to overcome in the future is to reduce the influence of byproducts such as acetic acid on the strength of wood and the recovery of chemical reagents, which can be achieved through optimising catalysts and modification processes. Due to environmental and legislative issues surrounding the use of biocides, new avenues for chemical modification have been created. Acetylated wood is suitable for construction purposes, for which longer service is expected. Longer service life means the carbon is sequestered in acetylated wood with no carbon emissions. Furthermore, permanent bulking of the acetylation means no leachate is released into the environment.

Acknowledgements

The author would like to thank UiTM for financing the research and the University of Wales, Bangor, for providing the facilities.

Paper Contribution to Related Field of Study

Contributes to a greener approach to treating wood, bamboo, and another biomass.

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