DECAY RESISTANCE OF ACETIC, PROPIONIC AND BUTYRIC ANHYDRIDES MODIFIED RUBBERWOOD AGAINST WHITE ROT (TRAMETES VERSICOLOR)

Hisham HN^{1,2,*}, Nuraishah H¹, Fitri-Yazid Z¹, Paridah MT² & Salmiah U³

¹Faculty of Forestry, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia
 ²Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia
 ³Mycology Unit, Product Development Division, Forest Research Institute Malaysia, 52109 Kepong, Kuala Lumpur, Malaysia

*h_noroul@upm.edu.my

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The effectiveness of rubberwood modified with acetic, propionic and butyric anhydrides against white rot *Trametes versicolor* was investigated. Specimens measuring 5 mm × 20 mm × 20 mm ($l \times w \times t$) underwent a Soxhlet extraction with toluene/methanol/acetone (4:1:1 by volume) for 8 h, and then oven-dried at 103 °C for 24 h. The specimens were vacuum-impregnated with anhydrides for durations ranging from 0.25 to 24 h at 120 °C. The weight percent gain (WPG) was calculated and the chemical bonding was analysed with Fourier transform infra-red (FTIR). The specimens were leached in deionised water according to EN 84 (1997) and exposed to white rot in an incubation room at 22 °C for 16 weeks. The study found that the acetic, propionic and butyric anhydrides reduced the rate of decay but did not totally protect rubberwood from *T. versicolor*. All the modified rubberwoods were classified as durability class 1 comparable to Scots pine and European beech. The final moisture content had a positive correlation with weight loss following decay. Scanning electron microscopy showed that hyphae penetrated cells in both untreated and modified rubberwood.

Keyword: Rubber tree, modification, deterioration, basidiomycete.

INTRODUCTION

Rubberwood (Hevea brasiliensis) is the most popular wood for manufacturing both local and export oriented furniture as it is inexpensive, readily availabile and adaptable to processing technology. Rubberwood sawn timber is the top raw material used in the furniture industry, which made up 85% of total wood furniture exports for Malaysia in 2013 (Hazmira et al. 2015). Rubberwood emerged as an alternative source of timber for the wood industry when restrictions on logging activities were implemented by the Malaysian government. Rubberwood is recognised as the most ecologically-friendly lumber used in today's furniture industry. Rubber trees for latex production are cut down and replanted after 26-30 years. The wood is then used as sawn timber and to produce mouldings, furniture, medium density fibreboard, chipboard, builders, joinery and carpentry and wooden frames (Shigematsu et al. 2011).

Despite the success story of rubberwood products, issues with low dimensional stability and biodegradation make rubberwood less attractive as a building material. Rubberwood products have been found to be more susceptible to biodeterioration agents than solid wood, (Zaidon et al. 2003). Rubberwood was more susceptible to soft rot decay than other nondurable timbers such as punggai (*Coelostegia* griffi) and jelutong (*Dyera costulata*). Other studies reported rubberwood as more susceptible than the non-durable jelutong and ramin (*Gonystylus* bancanus), to white rot (*Coriolus versicolor*) (Hong & Yamamoto 1989, Teoh et al. 2011). The high susceptibility of rubberwood to decay may be due to high carbohydrate (sugar and starch) reserves deposited in the parenchyma, and the absence of phenolic compounds (Ujang 1997).

Increasing emphasis has been placed on using less or non-toxic chemicals instead of copper chromium and arsenic (CCA) in wood modification to extend the wood service life. The CCA in wood leaches out into soils, and CCAtreated wood with potential for direct skin-contact has been banned from use in playgrounds (Hill 2006). As a viable alternative to CCA, chemical modification can improve wood dimensional stability and decay resistance without altering important mechanical properties particularly load-bearing capacity (Hisham & Hale 2012, Rowell 2014).

One of the best chemical modification treatments is acetylation (Rowell 2014). The increased resistance of acetylated wood against fungi is caused by the bulking effect of the chemical in wood cell walls and the reduction of hydroxyl (OH) groups, which reduces cell wall moisture content (Hill 2006, Rowell 2014). In acetylated rattan (*Calamus manan*), which had low rates of fungal attack compared with untreated rattan, modification was thought to reduce moisture content by decreasing the cell wall void volume (Hisham & Hale 2012, 2013). The moisture content of acetylated rattan was lower than that of untreated rattan in those studies.

While the decay resistance of acetylated wood is excellent, acetylation has been found to create a by-product-acetic acid, which needs further separation to recycle the acetic anhydride (Hill 2006, Rowell 2014). Studies using longer chain carboxylic acid anhydrides such as propionic and butyric anhydrides have reported good decay protection that varies based on whether the material to be modified is from dicotyledon or monocotyledon plants, which have different microstructures and amounts of structural organic chemicals. This included the decay resistance of modified rubberwood against brown rot, *Coniophora puteana* (Nuraishah et al. 2017)

The aim of the present study was to investigate the effectiveness of acetic, propionic and butyric anhydrides in protecting rubberwood against *Trametes versicolor*.

MATERIALS AND METHODS

Source of material

Rubberwood was obtained from 25 to 30 yearold rubber trees in Kampung Relau, Batu Kurau, Perak (4° 53' N, 100° 22' E). The wood was processed at the Forest Research Institute Malaysia (FRIM), Kuala Lumpur. The heartwood at diameter breast height was cut into boards measuring 25 mm × 140 mm × 1000 cm (r × t × l) and kiln-dried to a 10–12% moisture content. Specimens sized 20 mm × 20 mm × 5 mm (r × t × l) were then prepared using small band saw.

Preparation of the specimens

The rubberwood specimens were sanded to remove loosely adhering fibres, then placed in a Soxhlet extractor for solvent extraction using toluene/methanol/acetone (4:1:1 by volume) for 8 h. The specimens were then oven-dried for 24 h at 103 °C, weighed to three decimal places, and specimen volume was measured using Mitutoyo digimatic height gauge. The specimens were transferred to a vacuum desiccator and allowed to cool to ambient temperature over silica gel. The rubberwood specimens were arranged in containers by their reaction time (0.25, 4, 10)and 24 h), ballasted under metal wrapped with aluminium foil, and submerged in absolute acetic, butyric and propionic anhydrides (Table 1). The containers were vacuum-impregnated at 0.7 kPa and the specimens were kept submerged in anhydride for three days to achieve a wet and swollen state before the reaction.

Reaction procedures

The reaction procedures were conducted according to Hisham et al. (2014). The impregnated specimens were heated with oil in a bath for 0.25, 4, 10 and 24 h at 120 $^{\circ}$ C to obtain weight percent gains (WPG) representing lowest (4.7-8.3%), low (7.2-12.4%), medium (10.2–14.0%), and highest (13.3–15.5%) WPGs, respectively (Figure 1). At the end of the reaction period, the reaction was quenched in ice until the liquid temperature reached 20 °C and underwent an hour-long cold acetone wash three times. The modified rubberwood was finally Soxhletextracted with a toluene/methanol/acetone mixture (4:1:1) for 8 h and oven-dried at 103 °C for 24 h. The dry specimens were cooled, weighed and measured, and then air-dried to a constant weight in a conditioned room at 20 °C and 65% relative humidity (RH). The WPG was calculated using the following formula:

WPG (%) =
$$[(W_m - W_{um})/W_{um}] \times 100$$

where $W_m = mass$ of modified wood and $W_{um} = mass$ of unmodified wood.

Fourier transform infrared (FTIR) analysis

The Fourier transform infrared (FTIR) analysis was performed using a spectrometer to confirm

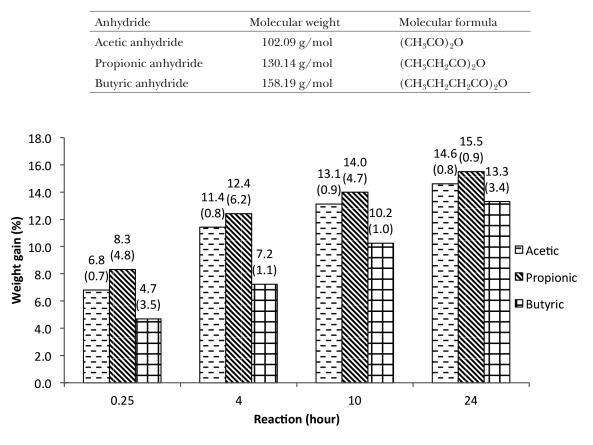


 Table 1
 The molecular weight of anhydrides used in the study

Figure 1 The averaged WPG of acetylated, propionylated and butyrylated rubberwood; values in parentheses are standard deviations

the bonding of the wood and the anhydrides. The specimens were shaved prior to the FTIR test. The infrared spectra of the specimens, which represented unmodified and modified with anhydrides, were obtained and evaluated via a FTIR analysis.

Decay test

All rubberwood specimens were leached in water following EN 84 (1997), and then dried and weighed. Specimens were then sterilised with propylene oxide for 5 days and exposed to decay over 60 ml of 4% malt extract agar in vented 500 ml squat jars following the procedures in EN 113 (1996). The 4% malt agar was made up of 40 g L21 Oxoid powdered malt extract, 20 g L21 Oxoid No. 3 agar and deionised water. The jars were sealed with lids vented with non-absorbent cotton wool plugs. The jars were autoclave sterilised then *T. versicolor* (CTB863A) was allowed to grow on the agar medium at 22 °C, 65% RH for 2 weeks before exposure of

the specimens. Two specimens, one modified and one untreated, were placed on a sterilised polypropylene mesh within each jar, over the fungus culture. Twelve replicates were used for each reaction period and anhydride. Six replicates of similarly sized Scots pine (Pinus sylvestris) sapwood and European beech (Fagus sylvatica) blocks were also exposed as reference specimens. All specimens were incubated for 16 weeks. At the end of the test, excessive mycelium was removed and moisture content and mass loss due to decay were determined. The durability classification was then determined following the guidance given in EN 350-1 (1994) where a ratio, expressed as the 'x value' is determined in comparison to the reference species (Scots pine and beech), i.e., x is the ratio of the average mass loss of test specimens to the average mass loss of reference species. In the study, x values using both reference species were calculated. Durability classes were assigned according to EN 350-1, for the following x values: $x \le 0.15$, class 1 (very durable); $0.15 < x \le 0.30$, class 2 (durable); $0.30 < x \le 0.60$, class, 3 (moderately durable); $0.60 < x \le 0.90$, class, 4 (slightly durable) and; x > 0.90, class 5 (not durable).

Scanning electron microscopy (SEM)

A thin surface layer from the decayed specimens was cut using a sharp razor-blade to obtain a smooth surface. The specimens were separated into vials and fixed in 4% glutaraldehyde for 2 days at 4 °C, then washed with 0.1 m sodium cacodylate buffer three times for 30 minutes each time, and post fixed in 1% osimium tetroxide for 2 h at 4°C. The specimens were washed again with 0.1 m sodium cacodylate buffer three times for 30 minutes each time. The specimens were dehydrated three times with acetone at concentration of 35, 50, 75 and 95% for 30 minutes, and lastly at 100% concentration for 1 h. The specimens were critically dried with critical point drying for 30 minutes and coated with gold using a sputter coater. Then scanning was performed using an electron microscope.

RESULTS AND DISCUSSION

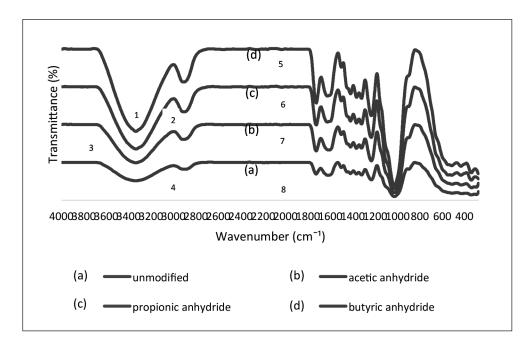
Fourier transform infra-red (FTIR) analysis

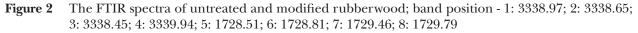
The FTIR spectra were differentiated by larger and smaller peaks of the line graph in the spectra. A larger peak indicates high intensity while a smaller peak indicates a lower intensity of intermolecular bonded hydroxyl groups. The FTIR spectra of untreated and modified rubberwood is shown in Figure 2. The unmodified rubberwood spectrum had a lower intensity of intermolecular bonded hydroxyl groups at 3339.94 cm⁻¹. The peak intensity of bonded hydroxyl groups was higher in acetylated rubberwood at 3338.45 cm⁻¹, 3338.65 cm⁻¹ for propionylated rubberwood and 3338.97 cm⁻¹ for butyrylated rubberwood.

A weak C-H stretching signal appeared at 2900 cm⁻¹ for all spectra. A sharp intensity ester carbonyl vibration appeared in the 1720 cm⁻¹ to 1730 cm⁻¹ region, which confirmed the formation of ester bonds, or esterification. The unmodified rubberwood spectrum had a lower intensity of 1729.70 cm⁻¹ in this region. An increase in the intensity of the C=O bonds occurred for all of the modified woods, with 1729.46 cm⁻¹ for butyrylated rubberwood and 1728.81 cm⁻¹ for propionylated rubberwood.

Decay resistance

The percent weight loss of untreated and modified rubberwood decayed by *T. versicolor* are shown in Figures 3(a-c). The weight loss of untreated rubberwood was almost identical when exposed together with each set of acetylated





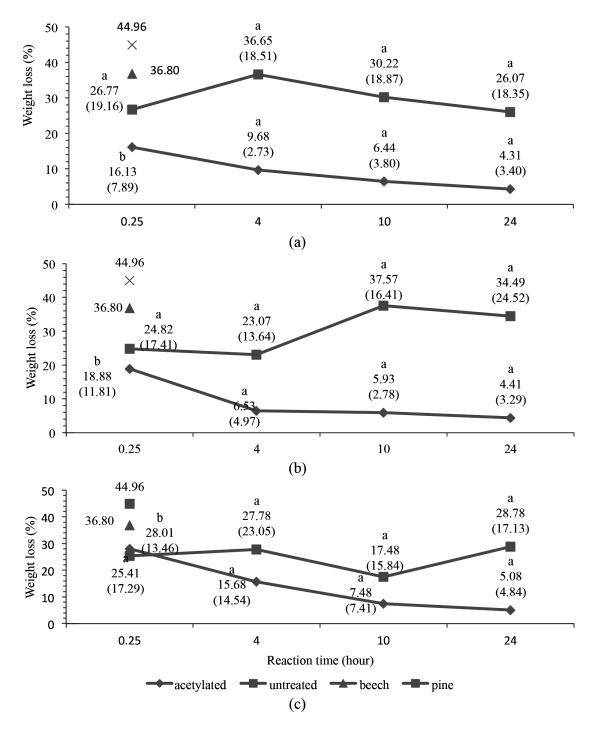


Figure 3 Mean weight loss following decay by *Trametes versicolor*, of untreated rubberwood and rubberwood modified by a) acetylation, b) propionylation and c) butyrylation; values in parentheses are standard deviations; mean values followed by the same letter in the same bar are not significantly different at p < 0.05 (Duncan's test)

rubberwood (26.07–36.65%), propionylated rubberwood (23.07–37.57%) and butyrylated rubberwood (17.48–28.78%). Weight loss gradually declined with increasing reaction time. Weight loss gradually declined from 16.13–4.31%, 18.88–4.41% and 25.41–5.08% for acetylated, propionylated and butyrylated rubberwood respectively. These lowest percent weight losses were realised by the highest WPGs of 14.6, 15.5 and 13.3% for acetylated, propionylated and butyrylated rubberwood reacted for 24 h, respectively.

The percent weight losses of all the modified rubberwood specimens in this study were above 3% and the test was valid according to EN113 (1996). Modifications with anhydrides were not effective in protecting rubberwood against T. versicolor. With chemical modification, the decay protection of wood is referred to as the decay protection threshold or the percent weight gain above which no microbial degradation occurs (Hill 2006). The decay protection threshold of 10% WPG in acetylated Corsican pine (Pinus nigra) gave 0% weight loss after 16 weeks of exposure to T. versicolor (Hill et al. 2003). In acetylated beech (Fagus sylvatica), a decay protection threshold of 20% WPG gave 0% weight loss. In acetylated rattan (C. manan) aged 10 and 13 years, thresholds of 13.4% and 9% WPG gave 0% weight loss after exposure for 16 weeks (Hisham & Hale 2012). A low effectiveness of anhydrides in protecting rubberwood was due not only to its low WPG but also because rubberwood is not very reactive with anhydrides. Acetylated rubberwood achieved a 13% WPG after a reaction time of 27 h, while extending reaction times for an additional 94 and 144 hours only gave 15% and 16.6% WPGs respectively (Siti Rafidah et al. 2006). This is probably caused by a lower kinetic reaction in rubberwood. The kinetic reaction is influenced by the accessibility of a reagent to the reaction site, and the actual chemical reaction (Minato & Ogura 2003). In the present study, anhydride treatments were evidently not sufficient in protecting rubberwood from T. versicolor.

Moisture content

The final moisture content of untreated and modified rubberwood is shown in Figures 4(a-c). With the exception of untreated rubberwood exposed alongside butyrylated rubberwood, untreated rubberwood took up more moisture than rubberwood modified with anhydrides. Moisture uptake ranged from 132.8–159.4%, 107.6–161.3% and 84.9–143.4% for acetylated, propionylated and butyrylated rubberwood respectively.

In all cases, the moisture content of modified rubberwood was greatly reduced with increasing reaction time. This was consistent with the percent weight loss after decay by *T. versicolor*. The moisture content ranged from 17.8–84.3%, 15.8–70.9% and 23.54–111.5% for acetylated, propionylated and butyrylated rubberwood respectively. The final moisture content at the decay protection threshold against *T. versicolor* was 12.5% and 14.4% for acetylated rattan aged 10 and 13 years respectively (Hisham & Hale 2012), which was slightly lower than that for rubberwood in the present study. A higher final moisture in modified rubberwood as compared to acetylated rattan may facilitate the access of fungal degradative agents in the former.

Correlation between the basic properties and percent weight loss of decayed untreated and modified rubberwood

Referring to Table 2, the final moisture content following decay was the only property positively correlated with percent weight loss in the untreated and modified rubber specimens (Table 2). Overall, the strongest correlation was obtained by butyrylated rubberwood (p = 0.84) followed by acetylated rubberwood (p = 0.69) and propionylated rubberwood (p = 0.65). WPG was negatively correlated with percent weight loss, a relationship also reported for acetylated rattan decayed by T. versicolor (Hisham & Hale 2012). Hill et al. (2005) concluded that acetylation reduced decay by reducing cell wall moisture content. Decay protection is thought to be due to the blocking of cell wall microcapillaries, thereby preventing access of low molecular weight degradative agents produced by the fungus (Papodopoulus & Hill 2003, Papodopoulus et al. 2010). Both factors are valid as the role of water is to transport food, degradative agents and enzymes as catalysts to the breakdown process (Bowyer et al. 2003).

The durability classes of untreated and modified rubberwood

When Scots pine was used as the reference specimen, almost all the untreated rubberwood specimens were classified as moderately or slightly durable (Table 3). The acetylated, propionylated and butyrylated rubberwood at minimum WPGs of 13.1, 14.0 and 10.2% respectively, were classified as very durable (class 1).

When beech wood was used as the reference specimen, all the untreated rubberwood

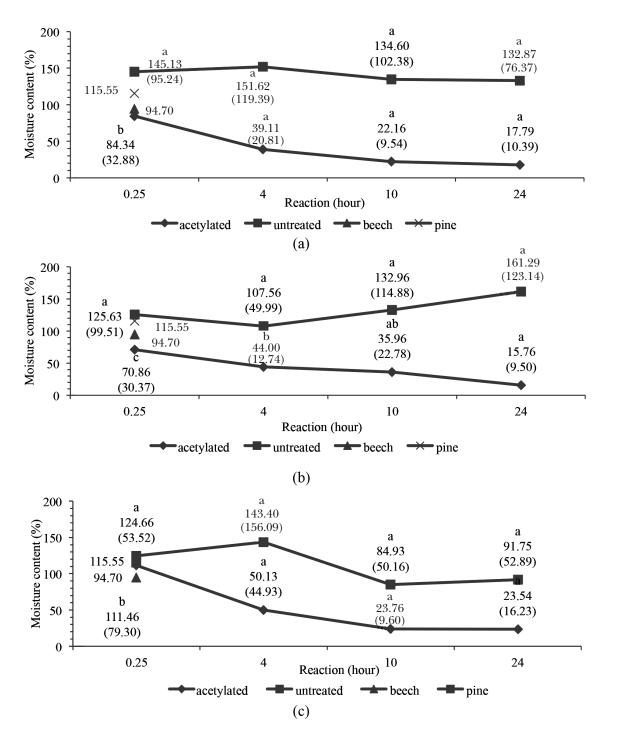


Figure 4 Mean final moisture content following decay by Trametes versicolor, of untreated rubberwood and rubberwood modified by a) acetylation, b) propionylation and c) butyrylation; values in parentheses are standard deviations; mean values followed by the same letter in the same bar are not significantly different at p < 0.05 (Duncan's test)

specimens were classified as moderately or slightly durable. Akin to Scots pine as the reference specimen, the acetylated, propionylated and butyrylated rubberwood at minimum WPGs of 13.1, 14.0 and 10.2% respectively, were classified as very durable (class 1).

Microstructure of decayed untreated and modified rubberwood

The microstructures of untreated rubberwood, acetylated, propionylated and butyrylated rubberwood decayed by fungi are shown in Figures

Specimen	Basic properties	Trametes versicolor		
Acetylated rubberwood	Percent weight gain	-0.68**		
	Final moisture content	0.69**		
ropionated rubberwood	Percent weight gain	-0.62**		
	Final moisture content	0.84**		
Butyrylated rubberwood	Percent weight gain	-0.26		
	Final moisture content	0.65*		

Table 2 Correlation between basic properties and percent weight loss for treated specimens

*significant at p < 0.01, ** is significant at p < 0.001

Table 3X values and durability classes of untreated rubberwood and rubberwood modified with acetic,
propionic and butyric anhydrides using Scots pine and European beech as reference

	Scots pine						European beech			
	Untreated		Modified		Untreated	Modified				
Anhydride	Reaction time (h)	x	class	х	class	x	class	x	class	
Acetic	0.25	0.51	3	0.35	3	0.62	4	0.41	3	
	4	0.74	4	0.21	2	0.90	5	0.26	2	
	10	0.60	3	0.15	1	0.73	4	0.14	1	
	24	0.50	3	0.11	1	0.61	4	0.13	1	
	Average	0.59	3	0.20	2	0.72	4	0.24	2	
Propionic	0.25	0.48	3	0.37	3	0.58	3	0.45	3	
	4	0.46	3	0.13	1	0.56	3	0.17	2	
	10	0.77	4	0.12	1	0.93	5	0.14	1	
	24	0.73	4	0.10	1	0.89	4	0.12	1	
	Average	0.61	4	0.18	2	0.74	4	0.22	2	
Butyric	0.25	0.49	3	0.58	3	0.6	4	0.71	4	
	4	0.53	3	0.36	3	0.65	4	0.44	4	
	10	0.38	3	0.13	1	0.57	3	0.15	1	
	24	0.62	4	0.12	1	0.75	4	0.14	1	
	Average	0.51	3	0.32	3	0.64	4	0.39	3	

x = average mass loss of test specimen: reference species

5 to 7. In almost all cases hyphae penetration was more prominent in the longitudinal direction than in the transverse direction. All untreated and modified rubberwood specimens were penetrated by hyphae, even at the highest WPG. This indicated that the fungi hyphae were able to penetrate the wood cells, but access was limited due to the slightly lower moisture content.

CONCLUSIONS

Modification of rubberwood with anhydrides to a degree, only improved decay resistance to *T. versicolor* but did not totally protect against deterioration. All the modified rubberwood specimens at maximum WPG were classified as durability class 1 when compared to Scots pine

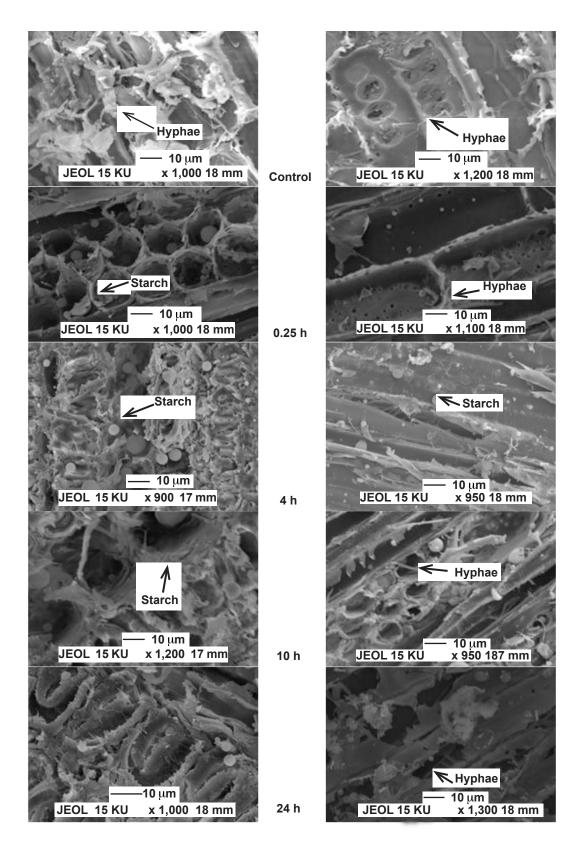


 Figure 5
 The microstructure of untreated rubberwood and acetylated rubberwood decayed by Trametes versicolor (left: transverse, right: longitudinal)

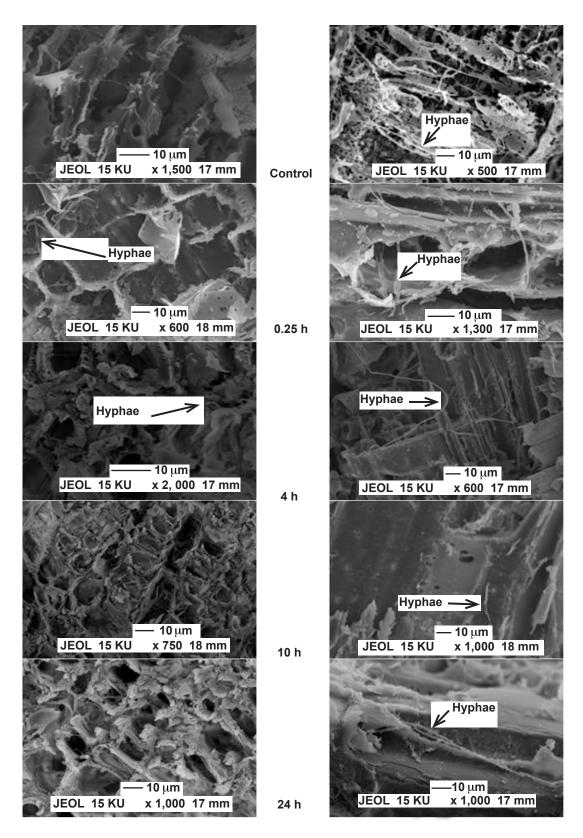


 Figure 6
 The microstructure of untreated rubberwood and propionylated rubberwood decayed by *Trametes versicolor* (left: transverse, right: longitudinal)

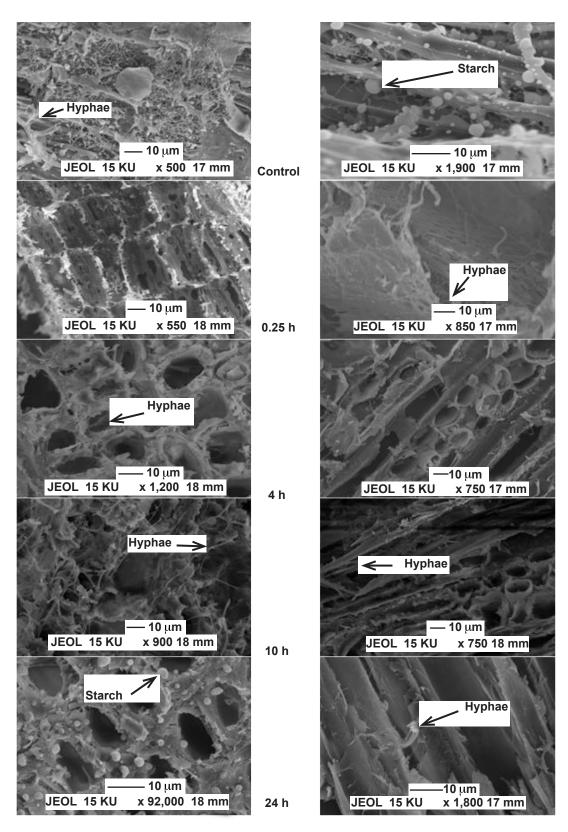


 Figure 7
 The microstructure of untreated rubberwood and butyrylated rubberwood decayed by *Trametes versicolor* (left: transverse, right: longitudinal)

and European beech. The final moisture content had a positive correlation with the percent weight loss following decay. The SEM observation confirmed that the hyphae penetrated the cells of both untreated and modified rubberwood.

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