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Impregnation of *Sesenduk* (*Endospermum diadenum*) Wood with Phenol Formaldehyde and Nanoclay Admixture: Effect on Fungal Decay and Termites Attack

(Rawatan Kayu Sesenduk (*Endospermum diadenum*) dengan Menggunakan Campuran Fenol Formaldehid dan Lempung Nano: Kesan ke atas Kulat Reput dan Serangan Anai-Anai)

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ABSTRACT

The aimed of this study was to evaluate the resistance of sesenduk *(*Endospermum diadenum*) wood, treated using admixture of low molecular weight phenol formaldehyde (LmwPF) resin and nanoclay, against white rot fungus (*Pycnoporus sanguineus*) and subterranean termites (*Coptotermes curvignathus *Holmgren). Seven sample groups including untreated* sesenduk *wood and treated* sesenduk *wood using 10, 15 and 20% LmwPF resin and the admixture of the 1.5% nanoclay with every level of resin concentrations. Air-dried samples were impregnated using vacuum-pressure process. After impregnation, the treated samples were heated in an oven at 150°C for 30 min. Five test blocks from each treatment group were tested separately against* P. sanguineus *and* C. curvignathus *in accordance with AWPA E10-12 and AWPA E1-13 standards, respectively. The results showed that both treatments had significant effects on the percentage weight loss and decay rate of the samples. The weight loss due to termite attack was found reduce with the increasing PF concentration. Generally, the addition of 1.5% nanoclay in PF resin slightly increased the resistance against both deteriorating agents compared to the wood treated using PF alone. It was found that the PF resin can be used as an effective method to improve the durability of* sesenduk *wood.*

Keywords: Coptotermes curvignathus; *nanoclay; phenolic resin;* Pycnoporus sanguineus*;* sesenduk

ABSTRAK

*Tujuan penyelidikan ini adalah untuk menilai rintangan kayu sesenduk (*Endospermum diadenum*) yang dirawat dengan menggunakan campuran resin fenol formaldehid berkeberatan molekul rendah (LmwPF) dan lempung nano, terhadap kulat reput putih (*Pycnoporus sanguineus*) dan anai-anai bawah tanah (*Coptotermes curvignathus *Holmgren). Tujuh kumpulan sampel termasuk kayu sesenduk yang tidak dirawat dan kayu sesenduk yang dirawat dengan menggunakan 10, 15 dan 20% LmwPF serta campuran 1.5% lempung nano bagi setiap kepekatan resin. Sampel kering telah dirawat dengan menggunakan proses tekanan vakum. Selepas rawatan, sampel yang telah dirawat dipanaskan dalam ketuhar pada 150°C selama 30 min. Lima blok ujian daripada setiap kumpulan rawatan telah diuji secara berasingan terhadap* P. sanguineus *dan* C. curvignathus *masing-masing mengikut standard AWPA E10-12 dan AWPA E1-13. Hasil kajian menunjukkan bahawa kedua-dua rawatan mempunyai kesan yang ketara ke atas peratusan kehilangan berat dan kadar kemerosotan sampel. Kehilangan berat sampel akibat serangan anai-anai berkurangan dengan peningkatan kepekatan PF. Secara amnya, penambahan sebanyak 1.5% lempung nano dalam resin PF meningkatkan rintangan kayu terhadap kedua-dua ejen kemerosotan berbanding dengan kayu yang dirawat dengan menggunakan PF sahaja. Kesimpulannya, resin PF boleh digunakan sebagai kaedah yang berkesan untuk meningkatkan ketahanan kayu sesenduk.*

Kata kunci: Coptotermes curvignathus*; lempung nano;* Pycnoporus sanguineus*; resin fenolik; sesenduk*

INTRODUCTION

Recently, a series of work has been conducted at the Faculty of Forestry, Universiti Putra Malaysia in enhancing the properties of low density tropical hardwood through impregnation treatment with phenol formaldehyde resin. Impregnation with 30% low molecular weight phenol formaldehyde (LmwPF, mw 600) followed by curing under heat has increased the mechanical strength, dimensional stability and resistance of *sesenduk* (*Endospermum diadenum*), jelutong *(Dyera costulata*) and mahang *(Macaranga* sp.) against decay and termite (Ang et al. 2014; Lee & Zaidon 2015; Nur Izreen et al. 2011; Purba et al. 2014). This product is known as *impreg* (Rowell 2005). Impregnation followed by compressing at high temperature (*compreg)* has enhanced the bending strength, dimensional stability and durability against fungal attack (Rabi'atol Adawiah et al. 2012; Zaidon et al. 2010). This product had superior mechanical properties than untreated wood due to increase in density. In another study, Bakar et al. (2013)

found that the durability of oil palm wood increased once it was converted into *compreg* product.

The efficacy of this impregnation treatment depends on molecular weight of PF resin, concentration of PF resin and thickness of the material (Zaidon et al. 2010). PF resin with molecular weight 290-480 is able to penetrate cell wall and impart dimensional stability (Rowell 2005), but PF resin with molecular weight of 820 remains in the cell lumen without resulting in any significant stability (Furuno et al. 2004). It has also been found that PF resin with molecular weight of 600 can successfully improve dimensional stability and properties of low density tropical hardwood, oil palm wood and bamboo (Amarullah et al. 2010; Anwar et al. 2011; Paridah & Loh 2009; Zaidon et al. 2010). It has been reported that for an optimum bulking of the resin, the wood are treated to 25 to 35% resin-forming materials on the basis of the weight of dry untreated wood (Rowell & Youngs 1981). One of the drawbacks associated with the using of LmwPF is its high formaldehyde emission during curing (Furuno et al. 2004). This scenario is worse if high concentration resin is used (Nur Izreen et al. 2011). Incorporating formaldehyde scavenger in the PF resin to reduce formaldehyde emission has been widely explored. Nevertheless, the performance of the treated product was not as good as those treated without formaldehyde scavenger (Purba et al. 2014; Rabi'atol Adawiah et al. 2012).

Mixing nano particle in phenol formaldehyde could possibly reduce the use of high concentration of resin in the treatment system and as a result would lower the formaldehyde emission and further increased the properties of the treated wood. Lu and Zhao (2008) reported that incorporating nanoclay in phenolic resin resulted in improvement on strength properties of low density wood. Cai et al. (2008) also found that addition of nanoclay in phenolic matrix significantly improved the properties of the modified aspen wood. In earlier study, it was reported that *impreg sesenduk* had significantly higher elasticity, compression stress, hardness and dimensional stability when treated with 10-20% admixture of LmwPF and nanoclay compared to *impreg* treated with LmwPF *per se*. The admixture-impregnated wood had 2-5% lower formaldehyde emission than PF-treated wood (Nabil et al. 2015). Thus, to conclude the overall performance of this so-called *impreg* product, the effect on biological properties is worth investigation. This paper reports the effects of *impreg sesenduk* (*Endospermum diadenum)* treated with admixture of LmwPF and modified montmorillonite nanoclay on fungal decay and termites attack. The dispersion method of nanoclay in the resin system is also discussed.

Materials and Methods

Air dry *sesenduk* (*E. diadenum*) wood with average density of 360 kg/m^3 was used in this study. Resol type low molecular weight phenol formaldehyde (LmwPF) resin (Mw, 600) with 45% solid content which was obtained from Malaysian Adhesive Chemical Sdn. Bhd. Shah Alam was used as the matrix. The initial pH value of the LmwPF resin was 8.64 and having a viscosity of 0.213 poise. Nanoclay modified with Octadecyl ammonium/ silane was obtained from Nanocor Inc. and used in this study. Specifications of the nanoclay are shown in Table 1.

SYNTHESIZATION OF LMWPF/NANOCLAY ADMIXTURE

Earlier study showed that nanoclay of concentration ranged from 0.5-1.5% (based on solid PF resin) can be well dispersed in 45% LmwPF through ultrasonication technique (Nabil et al. 2015). In the present study, this dispersion technique was adopted. Nanoclay (1.5%, based on solid PF) were dispersed in different concentrations of LmwPF resin (10, 15 and 20%), respectively. The following sonication profile was employed: 50 kHz amplitude for 60 min and for every 60 s of sonication, the process was paused for 5 s to avoid heat generation in the solution.

IMPREGNATION PROCESS

Air-dry, defect-free *sesenduk* wood was cut into tangent samples with nominal dimension of 150 mm long, 50 mm width and 10 mm depth. All samples were conditioned in a conditioning room at temperature of 25±2°C and a relative humidity of 65±2%. The weight and measurement of each sample were determined. They were placed in a vacuum-pressure apparatus and evacuated under 85 kPa pressure for 15 min to remove air from the samples. Then, the treating solutions were introduced separately into the apparatus until the samples were completely submerged in the solution. Additional pressure of 689 kPa was then applied to the system for 60 min. After the impregnation process was completed, the samples were removed and excess impregnant on the surface were wiped and blotted followed by curing at 150°C in an oven. The *impregs* were stacked in a conditioning room until constant weight was obtained. The density and weight percent gain (WPG) for each impregnated wood were determined.

DECAY AND TERMITE TEST

The test was carried out according to American Wood Preserves' Association (AWPA) Standard E10-12 (AWPA

TABLE 1. Specification of nanoclay

Commercial grade	Organic Modifier	Modifier Concentration	Specific gravity	X-ray diffraction (d_{001})
Nanomer® I.31PS	Octadecyl ammonium and aminopropyltriethoxysilane	28-32%	1.9 g/cm^3	18-22A

2012). The untreated and treated wood was randomly selected and cut into nominal dimension of 20×20×10 mm with five replicates for each treatment group and left in a conditioning room (25±2°C, 65±2%) until constant weight. White rot fungus, *Pycnoporus sanguineus* was used in this study. Test block which were previously sterilized in an autoclave at 121°C for 1 min were placed on mycelium covered strips in the bottles and left incubated at temperature of 25±3°C for 16 weeks. At the end of the incubation period, the test blocks were removed and the mycelium was brushed off. The specimens were then conditioned until they reached constant weight. The percentage weight loss for each block was calculated.

Termite tests were carried out according to American Wood Preserves' Association (AWPA) Standard E1-13 (AWPA 2013). Test blocks of 20×20×10 mm were cut from the treated and untreated *impreg*. Subterranean termites, *Coptotermus curvignathus* Holmgren were used in this test. Each of the test blocks was placed in a culture bottle filled with approximately 200 g of sterilized sand mixed with 40 mL of distilled water. Approximately 350 termites comprising 10% soldiers and 90% workers were introduced into each bottle. The cultured bottles were then sterilised, covered with black paper and left at room temperature for 4 weeks. At the end of the test period, the test blocks were removed and conditioned until they reached constant weight. The percentage weight loss for each block was then calculated. Each of the test blocks was examined and rating was given in accordance with AWPA rating system (Table 2).

TABLE 2. Rating system of termite damage

Rating	Resistance class
10	Sound, surface nibbles permitted
9	Light attack
7	Moderate attack, penetration
4	Heavy attack
0	Failure

STATISTICAL ANALYSIS

Analysis of variance (ANOVA) was employed to determine any difference in weight loss values among the treatment groups. Mean values were separated using Tukey's test. Correlation between WPG and weight loss was conducted using Pearson's correlation analysis.

RESULTS AND DISCUSSION

DENSITY AND WEIGHT PERCENT GAIN

Figures 1 and 2 illustrate the density and weight percent gain (WPG) of *impreg* from different treatment combinations. The results showed that the *impreg* products had higher density compared to that of the untreated *sesenduk* wood. The density of the *impregs* was ranging from 467-565 kg/m^3 and the density of the untreated wood was 360 kg/m^3. The results also showed that the density of *impregs* with the presence of nanoclay had density higher than those without nanoclay. With regards to polymer loading as described by WPG, it was also found that the polymer loading of the former was relatively higher than the latter and the difference was prominent for the *impreg* treated at lower admixture concentration. The WPG values for 20% PF resin *impreg* and 20% admixture *impregs* were 68% versus 69%, for 15% formulated resin *impregs* were 57% versus 65% and for 10% formulated resin *impregs* were 34% versus 50%. The increment in density is presented in Figure 3. One can see that the density increment increased along with increasing PF concentration and addition of nanoclay.

EVALUATION OF DECAY RESISTANCE

Table 3 shows the percentage of weight loss of the untreated and the *impregs* after 16-weeks of exposure to *P. sanguineus*. The increment in resistance against the white rot fungus is also listed. The results showed that the untreated wood was severely attacked by the white rot with an average weight loss of 31.86%. The weight

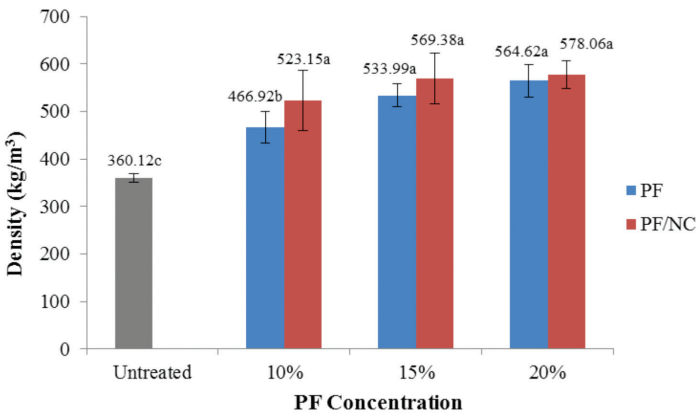

FIGURE 1. Density of *sesenduk* wood treated using phenol formaldehyde resin alone (PF) and combination with nanoclay (PF/NC)

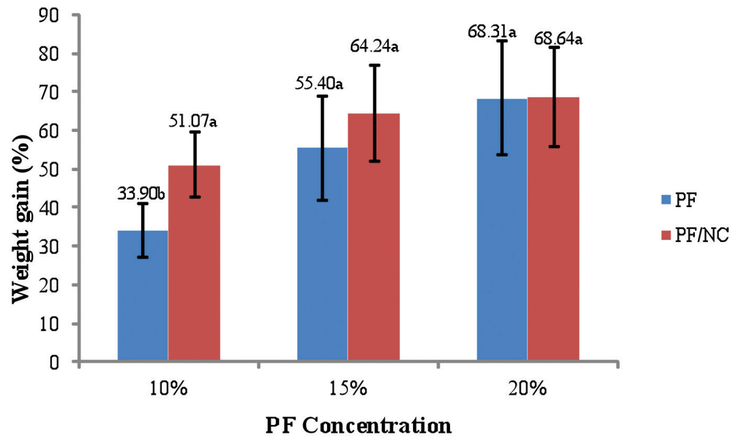

FIGURE 2. Weight percent gain (WPG) of *sesenduk* wood treated using phenol formaldehyde resin alone (PF) and combination with nanoclay (PF/NC)

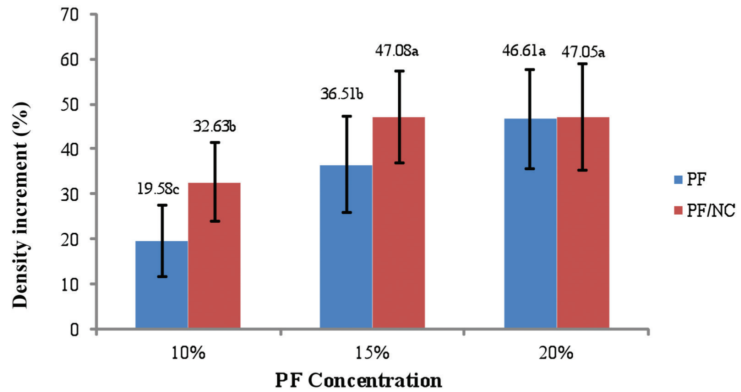

FIGURE 3. Increment in density of *sesenduk* wood treated using phenol formaldehyde resin alone (PF) and combination with nanoclay (PF/NC)

TABLE 3. Mean weight loss of treated and untreated *sesenduk* wood after 16-weeks exposure to white-rot fungus (*P. sanguines*)

Treatment	Weight loss (%)	Increment in resistance (%)
20%PF	2.24±1.17[a]	92.96±3.66
20%PF/NC	2.11±0.58[a]	93.38±1.82
15%PF	4.37±1.19[a]	86.29±3.73
15%PF/NC	3.08±1.14[a]	90.32±3.59
10%PF	4.85±2.00[a]	84.78±6.23
10%PF/NC	4.74±1.22[a]	85.11±3.85
Untreated	31.86±4.39[b]	-

Means followed by the same letter in the same column are not significantly different at $p \leq 0.05$; ± is standard deviation

loss values for *impregs* containing nanoclay ranged from 2.11 to 4.74%, whilst for *impregs* without nanoclay had weight loss values ranged from 2.24 to 4.85%. Obviously, phenolic resin treatment successfully imparted decay resistance ability to the wood. Although the improvement is insignificance, decay resistance of the wood is more pronounced when 1.5% nanoclay was added in the PF solution. These findings were compatible with Bari et al. (2015) who showed that nanoclay considerably inhibited the growth of wood-deteriorating fungi, although the reduction in weight losses were very low. Higher decay resistance might be due to higher WPG and density gain was attained in the wood treated using nanoclay. WPG is a very important factor in improving the durability of the wood. A significant negative correlation (r=-0.804) was found between WPG and weight loss of the wood block (Table 4) suggesting that higher WPG resulted in lower mass loss. Previous study conducted by Ang et al. (2014) found that WPG of 30% was enough to protect the wood from decay fungus.

Phenolic resin treatment of the wood specimens was able to provide protection as much as 84.78 to 92.96% to the *sesenduk* wood treated using PF alone against *P. sanguineus* compared to the untreated wood. Meanwhile, the increment in resistance ranged from 85.11 to 93.38% for the wood treated with addition of nanoclay (Table 3). Overall, addition of nanocaly in the phenolic resin showed better results, however, there was no significant difference at $p \leq 0.05$ levels found between treatments of PF with and without the addition of nanoclay. Although no significant difference was observed, the addition of nanoclay was found to further decreased the weight loss of the test block. This finding is in agreement with Mamatha et al. (2013) who found that the addition of nanoclay in the treatments improved the resistibility of the wood against white rot fungus, *Polyporous versicolour*. The addition of nanoclay inhibited moisture absorption in the treated wood thus might have had some impact on additional decay resistance properties. Cai et al. (2008) reported that incorporation of nanoclay increased the tortuous path for moisture transport and hence reduced the water diffusivity in the wood. Lower moisture content resulted in insufficient moisture for decay survival and prevented the wood from being attacked readily by wood-deteriorating fungi.

Figure 4 exhibits the visual analyses of the *sesenduk* wood after 16-weeks exposure to the white rot fungi. The results showed that the untreated *sesenduk* wood (Figure 4(a)) was colonized by the fungus and the entire test block was covered by mycelium. Nevertheless, no mycelium was observed on the surface of the treated test block (Figure 4(b)-4(d)). All of the treated wood showed almost no visible fungal infection. Generally, the results showed that the application of PF alone directly inhibited the growth rate of fungi and performed well in wood resistance. This is due to the poisonous nature of the PF resin which inhibited the fungal growth (Okino et al. 2005). This finding was in an agreement with Furuno et al. (2004), where the cell walls and cell lumina were able to be filled by LmwPF and resulted in better biological properties. The resin will prevent fungus from incubating the wood by penetrating deep into the cell wall and lumen. Some studies were also reported that LmwPF resin with 600 Mw had successfully prevented the weight loss by *P. sanguineus* on *Dyera costulata* (Nur Izreen et al. 2011) and *Macaranga* sp. (Ang et al. 2014). The addition of nanoclay in PF resin reduced the water absorption of wood samples and hence lead to reduction in mass losses, since a high correlation were found between water absorption

TABLE 4. Pearson's correlation analysis between WPG and weight loss of the *impreg* wood

	WPG	Density	WL (F)	WL (T)
WPG	-	0.990 (0.000)	-0.855 (0.030)	-0.883 (0.020)

Notes: WPG: Weight percent gain; WL(F): Weight loss caused by fungus; WL(T): Weight loss caused by termite

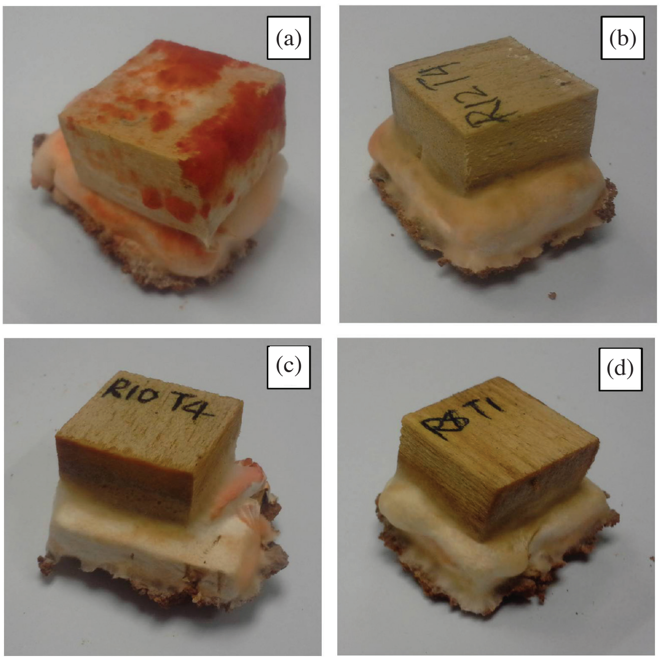

FIGURE 4. *Sesenduk* wood after 16 weeks exposure to *Pycnoporus sanguineus* (a) untreated *sesenduk*, (b) treated using 20% PF concentration, (c) treated using 15% PF concentration and (d) treated using 10% PF concentration

and mass loss ($R^2 > 70$) in a study conducted by Bari et al. (2015).

EVALUATION OF TERMITE RESISTANCE

The percentage of weight loss of the untreated and treated *sesenduk* wood after 4-weeks exposure to *C. curvignathus* Holmgren are shown in Table 5. The increment in resistance, termite mortality and block evaluation of the treated wood were also listed in the same table. The degree of the weight loss decreased as the PF concentration increased. Average weight loss of less than 0.50% was recorded in the wood samples treated using 20% PF concentration. Apparently, all of the treated wood showed lower weight loss compared to that of the untreated wood (17.95%). Although treatment using PF alone significantly enhanced the durability of the wood against termites attacks, the addition of nanoclay was found further reduced the weight loss of the wood. This phenomenon can be explained by higher WPG which was attained in the wood treated using nanoclay. Based on Table 5, a significant negative correlation was found between WPG and weight loss due to termite attack (r=-0.85) indicated that higher WPG lead to better resistance against termite attack.

However, there is no significantly difference at $p \leq 0.05$ was found in the treatment with the addition of 1.5% (w/w based on solid PF) nanoclay and without the addition of nanoclay. It was found that the PF concentration was in favour of the weight loss reduction. The results suggested that the higher PF concentration resulted in milder attack by termites due to the higher amount of polymer penetrate into wood intercellular cell wall. A study by Nur Izreen et al. (2011) and Rabi'atol Adawiah et al. (2012) reported that higher concentration would result on higher polymer attained inside wood intercellular cell thus forming polymer wall to the wood. Similar study by Deka et al. (2000) reported that the bulking of PF resin in the wood could inhibit the termite attack. Nevertheless, since wood moisture is one of the important factors that affected the termite feeding activity (Delaplane & La Fage 1989; Kulis et al. 2008), nanoclay particles formed a bond with the hydroxyl group of the wood and reduce the hydroxyl groups that available to absorb water molecules and eventually resulted in better resistance against termites compared to PF treatment alone (Bari et al. 2015).

EVALUATION OF TERMITE MORTALITY

The termite mortality was observed at the end of the testing period (Table 5). All of the treated wood showed 100% termite mortality at the end of testing period. Meanwhile, untreated wood showed low mean mortality rate (24%). The complete mortality attained on treated wood was due to the PF acted as a slow poison to the termites. This study was in an agreement with Bakar et al. (2013) and Loh et al. (2011) who suggested that the mortality of termites was due to the toxicity of PF resin. Zaidon et al. (2003) also reported that the toxicant of resin could influence the termite mortality and the inability of the termites to digest the materials.

EVALUATION OF WOOD BLOCK

The test blocks were examined and visually rated using the rating system shown in Table 2. The results of the block evaluation are shown in Table 5. Untreated wood gave a mean rate of 4 (heavy termite attack) as heavy nibbles were observed on the surface of the wood blocks due to termite attack (Figure 5(a)). The treated wood had mean rate of 7 to 10, respectively, as the rate value increased along with the increasing PF concentration. Sound or surface attack (10) was found in the wood treated using 20% PF concentration (Figure 5(b)), light attack (9) was observed in the wood treated using 15% PF concentration (Figure 5(c)) and wood treated using 10% PF concentration (Figure 5(d)) showed moderately attack by termite (7). From Figure 5, it was confirmed that LmwPF can be consider as repellent to *C. curvignathus* and the addition of nanoclay could further improved the repellent system.

CONCLUSION

Durability of the *sesenduk* wood could be enhanced by using the impregnation treatment of LmwPF resin with the combination of nanoclay. The durability of treated *sesenduk* wood against white rot fungus and termite attack suggested that treatment of phenolic resin, even

TABLE 5. Mean weight loss of test block of the untreated and treated *sesenduk* wood after 4-weeks exposure to *Coptotermes curvignathus* Holmgren

Treatment	Weight loss (%)	Increment in resistance (%)	Termite mortality	Block evaluation
20%PF	0.49±0.21[a]	97.26±1.18	100	10
20%PF/NC	0.37±0.01[a]	97.94±0.08	100	10
15%PF	2.56±1.58[ab]	85.76±8.80	100	9
15%PF/NC	2.19±0.89[ab]	87.83±4.93	100	9
10%PF	3.40±0.78[b]	81.07±4.34	100	7
10%PF/NC	3.20±0.13[b]	82.18±0.70	100	7
Untreated	17.95±0.86[c]	-	24	4

Means followed by the same letter in the same column are not significantly different at $p \leq 0.05$; ± is standard deviation

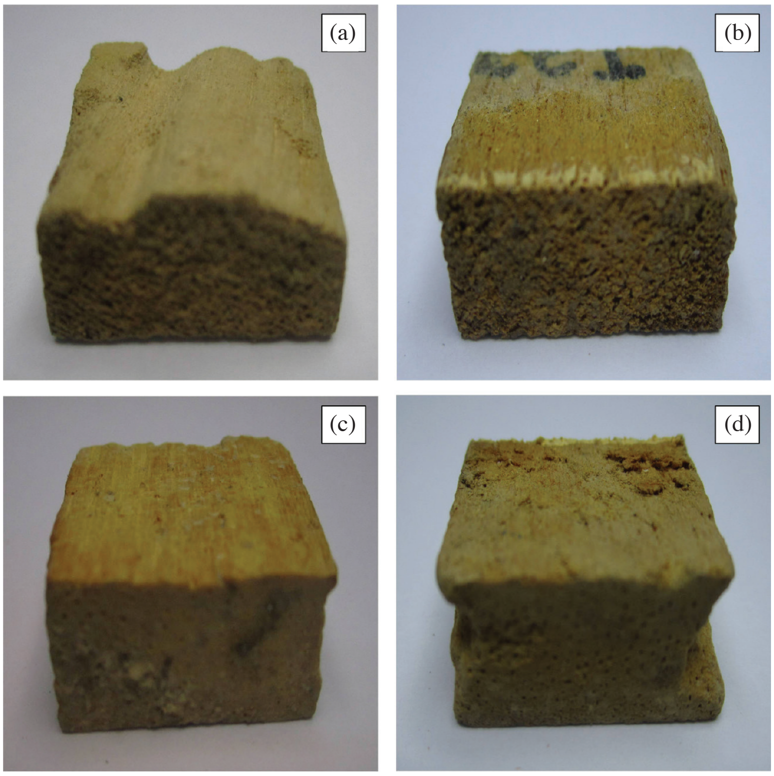

FIGURE 5. *Sesenduk* wood after 4 weeks exposure to *Coptotermes curvignathus* Holmgren (a) untreated *sesenduk*, (b) treated using 20% PF concentration, (c) treated using 15% PF concentration and (d) treated using 10% PF concentration

with the minimum concentration (10%), could obstruct the degradation of the wood by both bio-deterioration agents. Incorporation of nanoclay into the PF resin further improved the durability of the treated *sesenduk*. However, this improvement was not significance compared to the wood treated using solely PF resin. Nevertheless, it should be noted that the increment in resistance against both white rot fungus and termite attack of the wood treated using admixture of 10% PF and 1.5% nanoclay are more or less equivalent to the wood treated using 15% PF solely (10% PF/NC : 15% PF = 85.11% : 86.39% and 82.18% : 85.76%, respectively). This finding suggesting that, with the addition of nanoclay, lower PF concentration can be used to attain desired level of resistance and therefore lower formaldehyde emission can be anticipated. Besides that, previous study conducted by Nabil et al. (2015) also reported that better dimensional stability has been acquired in the wood treated using admixture of PF and nanoclay compared to that of the wood treated using PF solely with higher concentration. Furthermore, the presence of the nanoclay in PF resin also worked effectively in reducing formaldehyde emission. Therefore, despite the less significance improvement (with the addition of nanoclay) on both the decay and termite tests, lower PF concentration can be used along with nanoclay to produce wood with good durability while maintaining its properties.

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