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Soil Burial Degradation of Oil Palm Shell (OPS) Nanofiller and Phenol Formaldehyde (PF) Resin-Impregnated Oil Palm Trunk Lumber (OPTL): Dimensional Stability and Mechanical Properties

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In the present study, oil palm trunk lumber (OPTL) was chemically modified by impregnation with oil palm shell (OPS) nanofiller and phenol formaldehyde (PF) resin as a bulking agent (polymer matrix). A vacuum-pressure technique was used to impregnate OPTL stakes with phenol formaldehyde (PF) and OPS nanoparticles at 1, 3, 5, and 10% w/w. The resulting composite materials were buried in soil according to the BS EN ISO 846 standard for 6 and 12 months. After the designated burial period, structural and morphological changes in OPTL stakes were analyzed by Fourier Transform Infrared Spectroscopy (FT-IR) and scanning electron microscopy (SEM), respectively. Furthermore, dimensional stability and mechanical properties of the samples were assessed according to the standards. Water absorption of OPTL stakes was greatly reduced with PF-OPS nanofiller, resulting in less decay. Weight loss of the OPTL samples decreased with increasing OPS nanofiller concentration. Moreover, there was no significant decrease in physical and mechanical properties after 12 months of soil burial when compared with those at 6 months. The results of the study indicated that OPS nanofiller impregnation protected OPTL against microbial degradation during soil burial and 5% addition of OPS nanofiller in PF resin were sufficient to limit the decay by microbes. Therefore it can be concluded that PF-OPS nanofiller impregnation into OPTL might be a good alternative for microbial resistant of wood products.

Keywords:

RESEARCH ARTICLE

1. INTRODUCTION

Since oil palm trunk lumber (OPTL) waste was first developed for use as building materials, lightweight construction materials, and furniture,[1–3] scientists have made intense efforts to increase the quality of OPT with regard to their diverse environmental influences. A significant number of scientific reports reveal that chemical modification of OPTL may improve properties such as dimensional stability[2–4] and biological resistance.[5,6] One such modification technique that may improve the quality of OPT is impregnation of resin. In prior studies, where synthetic resins were used, OPT degraded upon its exposure to soil and water.[7] With the increased use of OPT to satisfy the demands of wood-based industries, decay resistance is an important area of focus.

Decay is arguably the most important and widespread type of degradation caused by microbes, specifically fungi.[8] Water in combination with microbial activity greatly impacts decay and surface degradation.[8] In circumstances where there is little or no oxygen, such as in deep soil, the wood is primarily degraded by by microbes.[9] Degradation mechanisms are very complex and are influenced by many factors such as water, temperature, and oxygen. Therefore, it is difficult to improve the decay resistance properties of lignocellulosic materials such as OPTL. However, wood modification through reducing the oxidation reactions might prove to be useful in improving the decay resistance profile.

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Various chemical modification techniques have been used to improve the decay and weather resistance of wood by blocking the hydroxyl groups of cell wall polymers.[10–12] Mohebby[8] demonstrated that substitution of the hydroxyl groups from the cell wall polymers by the acetyl groups might be effective, as the acetyl groups are not accessible to the microorganisms. Thus far, phenol formaldehyde (PF) impregnation into OPTL has been widely reported in many studies as a means of protecting against degradation.[13–15] However, addition of a filler in PF could be used as reinforcement to further improve OPTL degradation properties.[16] Both organic and inorganic nanofillers in PF can be used to improve the degradation properties of OPTL. AS far as can be seen in the current literature, no prior work has done to assess the decay resistance properties of OPTL impregnated with organic nanofiller. Thus, this study investigated how oil palm shell (OPS) nanofiller impregnation in OPTL influenced the microbial degradation resistance properties of OPTL when tested by the soil burial method. Dimensional stability, mechanical properties, and structural and morphological changes during soil burial were the main focus of the study.

2. MATERIALS AND METHODS

2.1. Sample Preparation

Small stakes of $300 \times 50 \times 20$ mm in size were prepared from the commercially available OPT (Banten, Indonesia). All stakes were initially dried at 60 °C for 24 hours and later dried at 103 ± 2 °C for another 24 hours, to get a moisture content of 10–12% before impregnation. The dried stakes were stored in a conditioning room until impregnation. Five different batches of stakes containing at least 25 stakes in each batch were prepared from the same OPT.

OPS was collected from a palm-oil processing mill in Kertajaya, Banten, Indonesia, in the form of chips. The OPS chips were ground using a grinder/refiner to produce powder, and the powder was dried to a total evaporable moisture content of 1.5%. The dried OPS powder samples were further ground to produce OPS nanoparticles using a high-energy ball milling (Pulverisette, Fritsch, Germany) for 30 hours at 170 rev min^{-1}. The ball mill was loaded with a ball-to-powder weight ratio of 10:1 in a stainless steel chamber using stainless steel balls having the diameter of 19, 12.7 and 9.5 mm. The OPS nanoparticles were kept at 250 °C in drying oven for 24 hours to prevent the agglomeration and finally kept in a dry place to avoid contact with moisture. The particle size was analyzed by Transmission Electron Microscope (TEM) (Phillips CM 12, Germany).

2.2. Impregnation of OPTL

The dried OPT stakes were placed in a chamber for impregnation with high molecular weight (4000 Mn) PF resin obtained from Palmolite Adhesive Ltd., Indonesia for a concentration of 15% w/w. OPS nanoparticles of 1, 3, 5, and 10% (w/w) having the size of 50 to 100 nm were mixed with PF resin for getting different concentrations. The mixtures were compounded using a mechanical stirrer at 3000 rpm for 15 minutes. Resin impregnation was carried out in a closed cylinder by the vacuum-pressure method using the initial vacuum at 3 bar for 15 minutes, pressure at 5 bar for 60 minutes and the final vacuum at 3 bar for 10 minutes. Finally, the PF resin-impregnated stakes were cured at 150 °C for 3 h. A total of five different impregnations (PF and PF with four different OPS nanoparticles) were carried out. The weight gain of the stakes were measured and calculated as percentage according to Eq. (1).

$$\mathrm{WG}(\%) = \frac{\mathrm{Wai} - \mathrm{Wod}}{\mathrm{Wod}} \times 100 \tag{1}$$

Where—Wai = Weight after impregnation (g); Wod = Oven dried weight (g); WG (%) = Weight gain percentage.

2.3. Testing of Materials

2.3.1. Soil Burial Test

A soil burial test in a glass box ($100 \times 60 \times 55$ cm) was performed according to the BS EN ISO 846: 1997 standard (Plastic-evaluation of the action of micro-organism). Six different glass boxes were used for six different types of samples including the control samples (dried OPTL). Containers were filled with natural soil having 90% water holding capacity where the moisture content was 50%. The water-holding capacity of the soil was measured and calculated by the similar method applied for moisture content determination of wood by oven dry method in Soil Science Laboratory under Faculty of Civil and Environment Engineering, Institut Teknologi Bandung, Indonesia. The samples were in permanent contact with the soil and exposed to a temperature of 29 ± 1 °C. Shirley cotton strips were used to determine the biological activity of the soil (cotton material to monitor the attack by soil microorganism clearly). The cotton strip retained less than 25% of the original tensile strength at the end of 7 days after soil burial. At least twenty stakes from each batch of sample were planted vertically into the soil with sorted distance of about 3 cm from each other. The samples assemblies

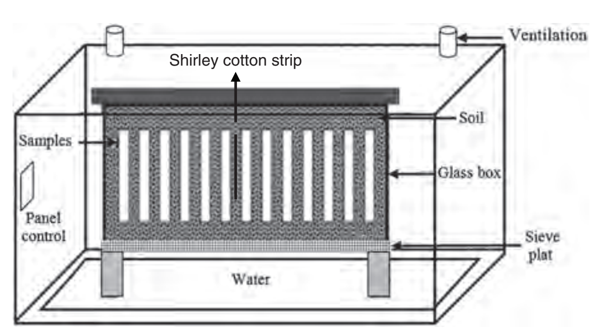

Fig. 1. Test assemblies of soil burial test.

are shown in Figure 1. Half of the samples were tested at 6 months and the rest were tested at 12 months. Mass losses were calculated after the desired period of time. The weight loss prevention ratio (WLPR) (%), defined as the ability to limit the weight loss from the sample, was calculated and compared with the untreated samples.

2.3.2. Changes of Dimensional Stability and Mechanical Properties

Changes in dimensional stability were measured according to BS EN 325: 1993 standard and determined by estimating the volumetric swelling coefficient (SC) and antiswelling efficiency (ASE) using water immersion test. Tensile and flexural strength were measured according to ASTM D 3039: 2000 and ASTM D 790: 2003 standards, respectively. There were at least five replications for the test.

2.3.3. IR Spectroscopy

Fourier transform infrared spectroscopy (FT-IR) (Nicolet Avatar 360, USA) was used to characterize the surface features of the treated stakes. The samples with three replications were pounded in a mortar cooled with liquid nitrogen and 1–2 mg of the obtained powder was dispersed in 100 mg of potassium bromide. Spectra were recorded using a Bruker Vectra 22 FTIR Spectrometer equipped with a DuraSampl IR II™ detector.

2.3.4. Scanning Electron Microscopy (SEM)

Biological decay of stakes was studied using SEM (ZEISS type EVO 50, Germany). After taking the samples from the soil, they were kept in a refrigerator at 4 °C temperature to cease further microbial activity. Two specimens (control and impregnated) having the size of $5 \times 5 \times 5$ mm were prepared for the test. Samples were sputter coated with 20 nm thick layer of gold using a Polaron Equipment Ltd. (Model E500) set at a voltage of 1.2 kV (10 mA) and a vacuum of 20 Pa for 10 min prior to their morphological observation. The SEM micrographs were obtained under conventional secondary electron imaging conditions with an acceleration voltage of 5 kVimens.

2.4. Statistical Analysis

Univariate Analyses of variance (ANOVA) were done with linear models in a completely randomized design (CRD) by using SPSS version 16.0 having the 95% confidence limit.

3. RESULTS AND DISCUSSION

The weight gain percentage increased with the increase of OPS nanofiller percentage up to 5 and then decreased for 10 (Table I), though this negative change was not significant. This might be related to the increase of concentration of higher OPS nanofiller in PF resin which inhibits the penetration of PF resin into OPTL. In addition, decrease of weight gain percentage at higher OPS nanofiller content is probably due to the formation of OPS nanoparticle agglomeration and the presence of unexfoliated aggregates.[17]

Table I. Variations in weight gain (%) of impregnated samples with different OPS nanofiller concentration.

OPS nanofiller (%)	Weight gain (%)
0	30.1
1	31.8
3	33.5
5	36.5
10	35.8

3.1. Degradation Properties of Impregnated OPTL

Table II summarizes the average weight loss (%), statistical test result and weight loss prevention ratio (%) of the impregnated stakes (PF and PF-OPS nanofiller) after 6 and 12 months of soil burial. In general, the PF-OPS nanofiller impregnated stakes showed a higher resistance to weight loss compared to the PF impregnated stakes. Untreated samples showed the lowest resistance to weight loss during the test.

The trend of weight loss after soil burial was opposite to the weight gain trend observed in the study, i.e., the average weight loss decreased with the increase of PF-OPS nanofiller impregnation percentage up to 5%, and then increased slightly the same for 10%. The WLPR was higher in the PF-OPS nanofiller impregnated group compared to the PF impregnated group. Additionally, impregnation of 5% PF-OPS nanofiller concentration showed the best performance against microbial decay as the weight loss was minimal. Statistical analysis revealed that there were no significant differences between 3, 5 and 10%, and 0 and 1% PF-OPS nanofiller impregnation for 6 and 12 months of burial. Control samples were situated completely in a different category.

Table II. Effects of PF resin impregnation on weight loss and weight loss prevention ratio after 6 and 12 months of soil burial.

	Weight loss (%) and Weight loss prevention ratio (%)					
	6 months			12 months		
Nanofiller (%)	WL[a]	HS[b]	WLPR	WL[a]	HS[b]	WLPR
Controlled	21.8 (1.11)	A	–	22.5 (1.10)	a	–
0	11.7 (1.20)	B	+46.1	12.2 (1.04)	b	+45.9
1	10.3 (1.20)	B	+50.0	11.4 (1.23)	b	+49.4
3	7.9 (1.20)	C	+63.6	8.6 (1.19)	c	+61.9
5	6.6 (1.34)	C	+69.8	7.2 (1.50)	c	+67.9
10	7.4 (1.02)	c	+66.1	7.8 (0.96)	c	+65.4

Notes: [a]Values in parentheses are standard deviations; [b]HS (Homogeneous subsets): Different letters indicate significant difference by Duncan's Homogeneity test ($p < 0.05$).

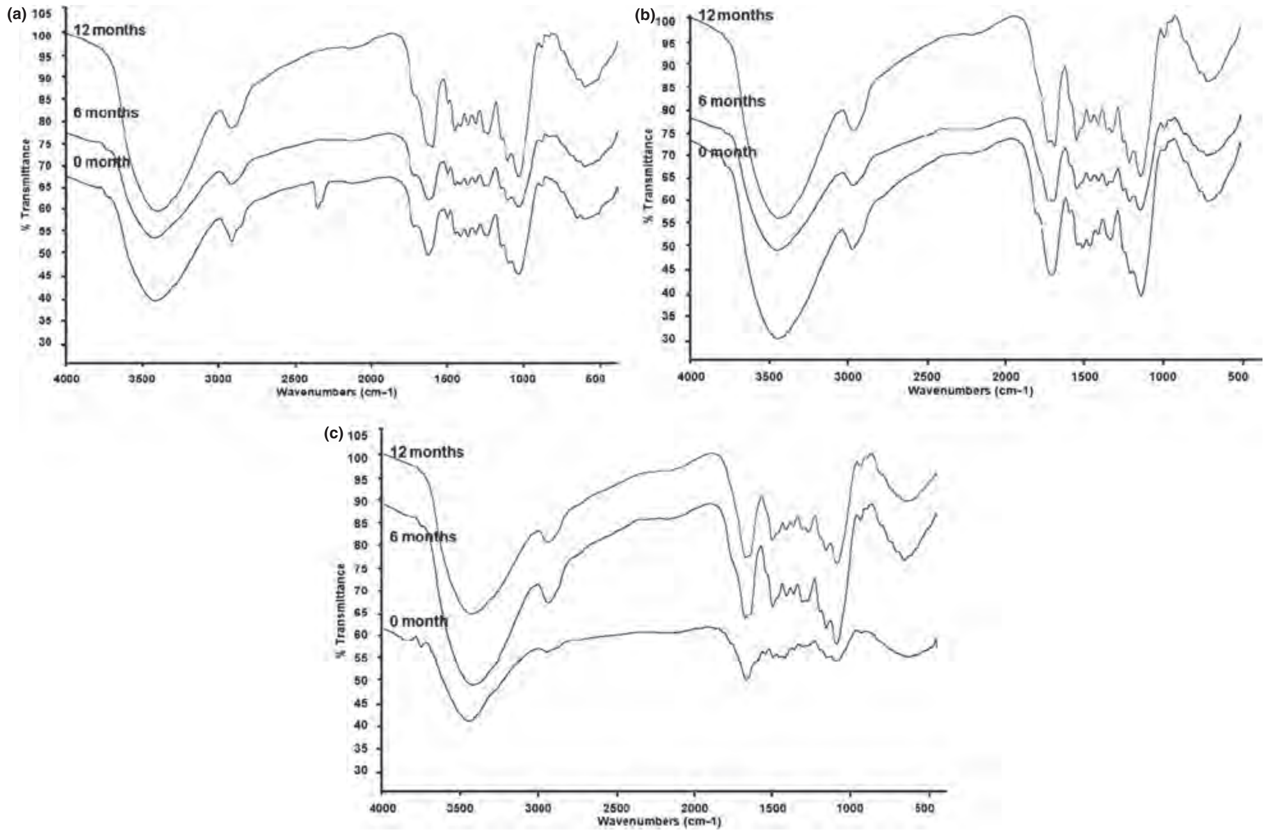

Fig. 2. IR spectra of OPTL (a) controlled OPTL, (b) PF-impregnated OPTL, and (c) PF-nanofiller impregnated OPTL.

The process of impregnation of PF resin causes cell wall bulking and cavity filling, resulting in the blockage of water movement pathways resulting the reduction of shrinkage and swelling properties of wood. By blocking the movement of water, wood decay is reduced. In a study by Ohmae,[18] lower molecular weight PF resin treatment of hinoki wood by the vacuum-pressure method decreased the anti swelling efficiency (ASE) by 74% when the weight gain was 30%. The ASE increase was attributed to cell wall bulking and cross-linking of the resin components with the cell wall. Cell wall bulking and cross-linking help to reduce the growth and penetration of fungal hyphae. The results of this study indicated that the weight loss of a buried sample was the function of time and decay. Weight loss increased with an increase in burial duration. During the burial period, water gradually penetrates into sample and the impregnated chemicals migrate into the soil, causing degradation by microbial attack.[19]

3.2. FT-IR Spectra of Impregnated OPTL

The soil burial test process induces chemical changes to the sample surface. Therefore, determination of infrared spectra of the decayed sample surface contributes to the understanding of the chemical changes caused by soil burial. As shown in Figure 2(a), the band at 1641 cm^{-1} for dried OPTL has markedly reduced, and the band at 2358 cm^{-1} for dried OPTL has observed to disappear rapidly with soil burial. C═O stretching vibration diminishes greatly, possibly due to the breaking of the C═O bond.[20] These bands seem to vary continuously with soil exposure and not found after 6 and 12 months of exposure. Such spectral changes indicate that soil burial decomposition caused by microbes occurs readily. The reduction of these bands is nearly completed by 12 months.

Figure 2(b) shows the IR spectra of PF impregnated OPTL. The band at 1246 cm^{-1} disappears rapidly after soil

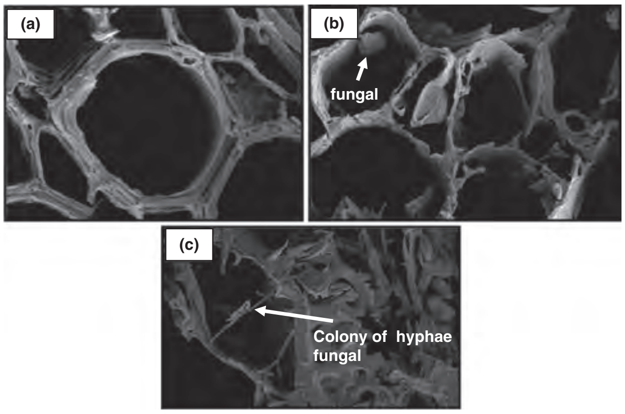

Fig. 3. Effects of 0, 6 and 12 months of soil burial test on morphologic changes of dried OPTL: (a) before test (0 month); (b) after 6 months; (c) after 12 months (500× magnification).

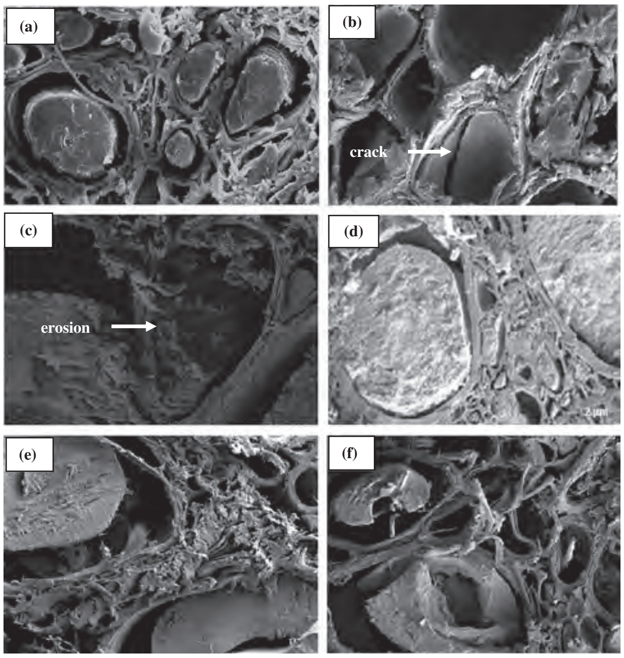

Fig. 4. Effects of 0, 6 and 12 months of soil burial test on morphology of PF and PF-nanofiller impregnated OPTL: (a) PF impregnated OPTL before test (0 month); (b) PF impregnated OPTL after 6 months; (c) PF impregnated OPTL after 12 months; (d) PF-nanofiller impregnated OPTL before test; (e) PF-nanofiller impregnated OPTL after 6 months; (f) PF-nanofiller impregnated OPTL after 12 months (500× magnification).

burial. The other band at 1620 cm^{-1} shows minimal spectral changes after 6 and 12 months of soil burial. This band is produced due to the skeletal stretching of the hydrogen bond. The reduction of the band intensity at 1246 cm^{-1} indicates the removal or decomposition of a guaiacyl unit. The reduction of the band at about 608 cm^{-1} indicates the removal or decomposition of an aromatic ring of ether.[21]

Structural units of PF-OPS nanofiller impregnated OPTL undergo various changes in the functional groups compared to the untreated and PF impregnated OPTL samples. Figure 2(c) shows the IR spectra of PF-OPS nanofiller impregnated OPTL before and after soil burial at 6 and 12 months. The band at 3740 cm^{-1} and 598 cm^{-1} disappear rapidly after soil burial. Meanwhile, the bands at 3435, 2925, 1637 and 1045 cm^{-1} show few spectral changes after 6 and 12 months of soil burial.

3.3. Morphologic Changes of Impregnated OPTL

Figure 3(a) shows the cross section of an OPTL fiber clearly showing the cell lumen and cell wall. The cross section of treated OPTL after 6 and 12 months of soil burial, are shown in Figures 3(b and c) respectively. It is important to note that fungal hyphae have colonized the cell lumen and ray cells after 6 and 12 months of soil burial. One may theorize that the fungi use open pathways to penetrate into OPTL, preferably through vessel lumina and rays at the earlier stage of soil burial, and then later penetrate into fiber lumina through inter-fiber pits or cross-fields between rays and fibers.

Cells of untreated OPTL have enough nutrients for hyphal growth, which permits the rapid penetration of hyphae into the adjacent cells. Bacteria may also present in the cell lumina with the fungi as there is a close association between bacteria and fungi for wood decay.[22] A synergism between the two microbes may ease decay of the dried OPTL. However, the decaying process starts after several months by the wood-degrading bacteria and fungi[23] which is consistent with the present study.

The PF and PF-OPS nanofiller impregnated OPTL do not show any hyphal growth even after 12 months of soil burial (Figs. 4(b, c, e and f)). However, Figure 4(c) shows the erosion of PF resin after 12 months of soil burial. PF resin penetrated and deposited in the cell lumen with a surface coating which contributed less against either erosion or decay.[5] With time, erosion will increase which might tempt microbes to grow in OPTL resulting the decay. Addition of nanofiller in PF resin helps to reduce the rate of erosion as is seen in Figure 4(f). The PF resin and OPS nanofiller matrix caused cross-linking between the matrix and cell wall which contributed to erosion resistance. The impregnation process might help to remove or modify the nutrients present in the OPTL cell used by the fungal hyphae initially.[12] Lack of nutrients and protection against microbes by the OPTL cell wall-PF matrix after impregnation could be the reasons for little or no decay in the OPTL after 6 and 12 months of soil burial. The weight

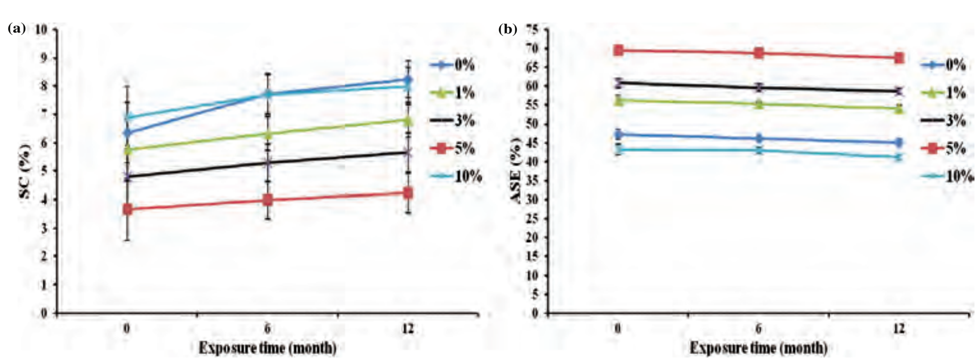

Fig. 5. Changes of dimensional stability after 6 and 12 months of soil burial for different percentage of PF-nanofiller impregnated OPTL.

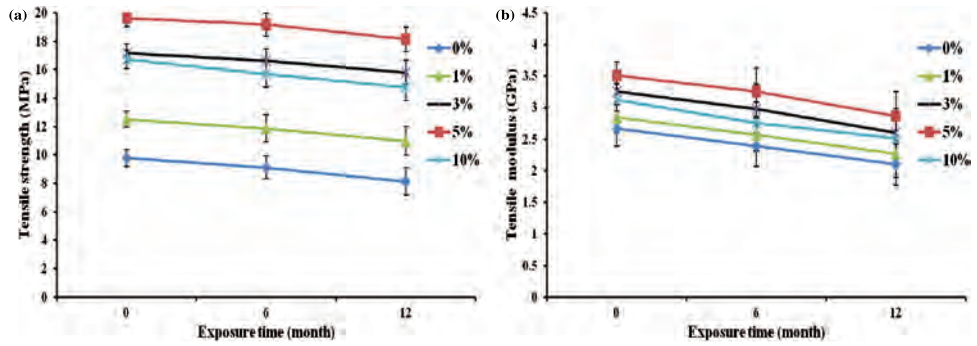

Fig. 6. Changes of tensile properties after 6 and 12 months of soil burial for different percentage of PF-nanofiller impregnated OPTL.

loss due to fungal attack decreased with the increase of OPS nanofiller concentrations.

3.4. Dimensional Stability and Mechanical Properties of Impregnated OPTL

Figures 5(a and b) show the effects of soil burial on the SC and ASE of impregnated OPTL after 6 and 12 months duration, respectively. The SC of PF impregnated OPTL increased by approximately 20.3% at final exposure (12 months) by soil burial when compared to the before impregnation. The 5% PF-OPS nanofiller impregnated OPTL showed the lowest SC followed by 3, 1, 10 and 0% PF-OPS nanofiller impregnation.

The ASE decreased with an increase in soil burial duration. The ASE increase was linear with the percentage increase of OPS nanofiller concentrations at each burial duration except 10% (Fig. 5(b)). As was seen with the SC, 5% PF-OPS nanofiller impregnation showed the highest ASE. The ASE of treated OPTL decreased with the soil burial duration, though this decrease was not statistically significant.

In Figures 6(a and b), tensile strength and tensile modulus of 5% PF-OPS nanofiller impregnation decreased by about 7.6 and 9.6%, respectively after 12 months of burial duration. Meanwhile, PF impregnation reduced by about 16.9%, and 21.35%, respectively for tensile strength and tensile modulus.

Figure 7 shows the flexural properties of impregnated OPTL stakes after soil burial. PF-OPS nanofiller impregnation showed higher flexural properties compared to the PF impregnation for any duration of burial. The 1, 3 and 10% PF-nanofiller impregnation showed the flexural strength reduction of about 8.0, 6.8 and 6.0%, respectively after 12 months of soil burial. Meanwhile, 5% PF-OPS nanofiller impregnation retained the flexural strength up to 95%. Flexural strength decreased by about 43.2% after 12 months of soil burial, though the decrease was not substantial for the flexural modulus.

The changes in dimensional stability and mechanical properties were significantly higher for untreated OPTL samples compared to the treated ones. The PF resin and PF-OPS nanofiller samples occupied the cell lumen to form a rigid cross-linked polymer which improved strength and stiffness of the OPTL resulting in better dimensional stability and mechanical properties.[24] Thus, treated samples had better physical and mechanical properties compared to the untreated OPTL even after decay.[4] The tensile properties of all samples decreased after 12 months of soil burial. Given that the resin and nanofiller matrix have hydrophobic properties causing less moisture absorption than what is expected for untreated OPTL during soil burial, the swelling and shrinking properties of the treated samples were minimized.

The decrease in the mechanical properties due to the presence of moisture content during soil burial was reported by several researchers.[20,24,25] The ability of OPS nanofiller to absorb moisture increased the degradation of PF-OPS nanofiller impregnated stakes. The degradation

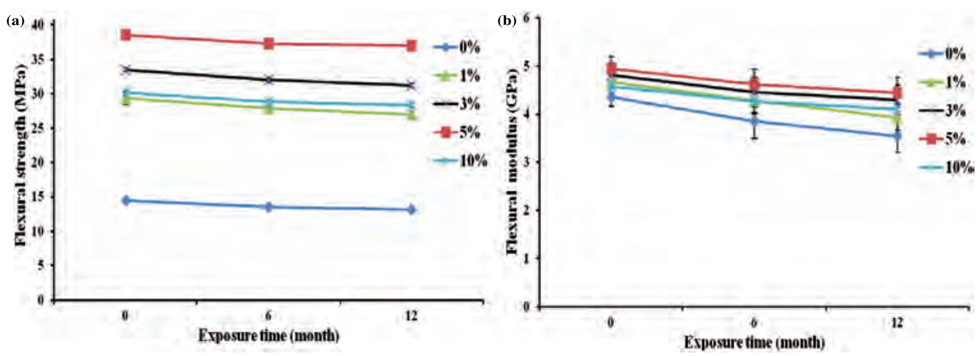

Fig. 7. Changes of flexural properties after 6 and 12 months of soil burial for different percentage of PF-nanofiller impregnated OPTL.

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would also further increase as the PF resin erodes over time. The OPS nanofiller contains a hydroxyl group which is able to absorb moisture from the environment. Due to this moisture absorbing property, the decay process would be higher for the PF-OPS nanofiller impregnated OPTL when compared with untreated OPTL. Nevertheless, PF-OPS impregnated OPTL resin forms a matrix with the cell wall. The resulting higher bonding strength reduces water absorption. This caused decreased swelling and shrinking properties of the impregnated OPTL.

4. CONCLUSIONS

PF resin impregnation provides extended service life to OPTL and protects against microbial invasion. These benefits were further enhanced by OPS nanofiller addition up to 5%. PF resin and PF-OPS nanofiller impregnation limits the penetration and colonization of microbes in the cell lumina, which otherwise colonize in the cell lumina and start the decay of untreated OPTL.

The 12-month soil burial test proved that increased OPS nanofiller up to 5% in PF matrix and long duration exposure caused the lowest percentage of degradation. The 5% PF-OPS nanofiller impregnation showed the lowest SC and the highest ASE for any duration of soil burial, which contributed to the lowest degradation profile of these stakes. Of all groups tested, the mechanical properties were optimal for 5% PF-OPS nanofiller stakes after 12 months of soil burial. This trend decreased accordingly, corresponding with 3, 10, and 1% OPS nanofiller concentrations. The dried OPTL showed the poorest mechanical properties after soil burial. On the other hand, elongation at break decreased with an increase in nanofiller concentration. Thus, it is concluded that addition of OPS nanofiller up to 5% in the PF resin improves the mechanical and other properties of OPTL, providing an alternative green composite for use in building materials.

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