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INFLUENCE OF WATER ABSORPTION ON MECHANICAL PROPERTIES OF COCONUT COIR FIBER/POLY-LACTIC ACID BIOCOMPOSITES

N.P.G. Suardana[1*], I.P. Lokantara[1], Jae Kyoo Lim[2]

[1]Mechanical Engineering Department, Udayana University, Kampus Bukit Jimbaran 80361, Bali Indonesia

[2]Automobile Hi-Technology Research Institute, Chonbuk National University,

Deokjin 1-664-14, Jeonju 561-756, Republic of Korea

*email: npgsuardana@me.unud.ac.id

Abstract. Biocomposites were prepared using coconut coir fiber that has been subjected to various treatments and polylactic-acid (PLA) as a matix. The volume fraction of the fibers in the PLA composites was fixed at 40 %. The composite with NaOH-treated fiber absorbed the most water. In general, biocomposites made with coconut coir fiber that had been treated with NaOH followed by acrylic acid (AA), had a greater tensile and flexural strength than did untreated or fibers treated only with NaOH. Tensile and flexural strengths of the biocomposites decrease after 6 h and decrease drastically for 192 h following soaking in the water. Tensile fracture and surface biodegradation were observed using the SEM. We also studied the comparison of biocomposites with coir fibers that had been treated with alkali followed by 0.5 % AA for 0.5 h at room temperature and at 70 ^{o}C.

1. Introduction

Coconut coir is a natural fiber. Long ago, this material was used in traditional building construction, in clothing, and even frequently as a source of energy [1]. Coconut coir fiber is one of the most lignocellulosic fibers. It is not brittle, is amenable to chemical modification, is non-toxic [2, 3] and biodegradable, has a low density, and is less abrasive to plastic processing equipment [3]. In addition, coconut coir poses no waste disposal problems. However, it is highly hydrophilic because of the predominance of –OH groups on its surface [4]. Coconut coir also contains a central hollow portion that runs along the fiber axis (lacuna) [5], so it can be used in acoustic and thermal insulating applications where its reduced bulk density and lightweight properties are advantageous [6].

In the last decade, coconut coir has been investigated as reinforcement for polymer composites, such as polyethylene, epoxy, polyester and phenolic resins [5, 7, 8, 9, 10]. The aim is to create a low-cost, high strength-to-weight ratio material to replace glass fiber composites.

The natural fibers-matrix bonding can be improved by physical and chemical modifications of fiber surfaces. Some chemical solutions have been used for treating the natural fiber. Rout et al. [8] studied the influence of fiber treatments (alkali, blenching, vinyl grafting) on the performance of coconut coir polyester composites. Rahman and Khan [2] subjected coconut coir fibers to alkali treatment 5-50 % for 0.5 h at temperatures ranging from 0-100 ^{o}C. Alkali treatment of coconut coir fiber was followed by grafting with 50 % ethylene dimethylacrylate (EMA) and curing under UV radiation, as studied by Kumar et al. [10].

Combined treatment of bagasse can also be achieved by treatment of the fibers with NaOH solution, followed by acrylic acid (Vilay et al. [11]). Our previous study showed that single coconut coir fiber with 6% alkali followed by 0.5 % acrylic acid treated had higher tensile properties than that untreated and alkali treated [12].

Poly-lactic acid (PLA) is a natural biodegradable polymer, but does not possess the necessary thermal and mechanical properties desirable for engineering plastics. The best engineering plastics are synthetic, but they do not biodegrade and they pollute the environment [13]. The advantages of using natural fibers as reinforcements in natural polymers are ease of disposal, in addition to reduction of weight, resulting in reasonable stiffness and strength.

The interest in natural fiber-reinforced biodegradable polymers has grown recently because of increasing environmental concerns [14]. These polymers are environmentally sound, i.e. renewable and recyclable, and have a very low raw material cost [2, 8].

Environmental conditions affect such properties of the composite systems as dimensional stability and other physical, mechanical, and electrical properties of polymer composites [15]. When a composite is exposed to water, there are three possible mechanisms that affect the composite systems. These can include matrix plasticization, degradation of the fiber, and degradation of the fiber–matrix interface [15, 16]. Many researchers have studied the water absorption of natural fiber reinforced polymer composites, such as phenol formaldehyde, recycled plastic (HDPE and PP), and polyester [17-19]. In this paper, coir fiber was treated with acrylic acid (AA) to improve mechanical properties and reduce water absorption of coconut coir fiber reinforced PLA biocomposites. AA treatment of fiber is one of the chemical treatments that are more efficient than other treatments [20]. Besides, the acrylic acid treatment of fiber at room temperature, including the treatment at 70 ^{o}C and biodegradation of biocomposites system were also evaluated. It is expected that the results can guide in developing of mechanical properties and reducing water absorption of coconut coir/PLA biocomposites.

2. Experimental procedure

Materials. Polylactic-acid (PLA) is produced by Cargill Dow LLC (USA). Coconut husk/coconut coir is obtained from Vietnam, and the fiber is separated from the outer shell of the coconut fruit. Chemical composition of the coconut coir (coconut husk = CH) is given in Table 1 [21]. Sodium Hydroxide (NaOH) was purchased from Showa Chemical Co. Ltd Japan and Acrylic acid (CH2:CHCOOH) was produced by Junsei Chemical Co. Ltd Japan.

Table 1. Chemical compositions (wt %).

Sample	Coconut coir (CH)
Cellulose	21.22± 1.44
Hemicellulose	12.69± 2.34
Lignin	46.50± 1.73
Partial amount, PA	80.41± 5.51
Extractives	8.77 ± 0.39
Moisture	13.68± 0.05
Ashes	1.05 ± 0.90
Sum	103.91 ±6.85

Fiber preparation. Coconut coir fibers were cut to a length of 5-15 mm. The fibers were rinsed with water to remove dust and impurities and then boiled in 40 ml water per 1 g fiber at 100 ^{o}C for 1h. The purpose is to remove the impurities which cannot be removed by washing at room temperature and also for killing the bacteria. This procedure also makes

the same condition of the untreated fibers. After these processes, the fibers were rinsed in tap water and then dried in the oven at 70 ^{o}C for 12 h.

Chemical treatment. The fibers were treated using 6 % NaOH immersion in distilled water at 95 ^{o}C for 3 h (using a ratio of 1 g fiber to 40 ml NaOH solution) and then washed with tap water to eliminate any residual NaOH solution. Next, they were dried in the oven for 24 h at 70 ^{o}C. Subsequently, they were soaked in 0.5 % acrylic acid (AA) (using a ratio of 1 g fiber to 40 ml AA solution) for 0.5, 1 and 3 h and then rinsed in tap water, respectively. Finally, the fibers were dried in the oven at 70 ^{o}C for 72 h.

Composite fabrication. Thin sheets of PLA were cut to 180 x 155 mm. The volume fraction of the fibers in the PLA composites was fixed at 40 %. The mold frame (180 x 155 mm) was aluminum. The surfaces of the mold were sprayed with Blue Coat-R release agent to reduce adhesion between the composites and the mold. The compounded materials were molded from a stack of nine PLA films interleaved with fibers. The mold and specimen were placed into a hot press machine (Carver #3912) that was used for compression molding. The specimens were heated at 175 ^{o}C and 1.3 MPa for 15 min and then removed from the hot press machine and pressed under 40 kg until the mold reached room temperature. The nominal thickness of the specimens was 3-4 mm.

Water absorption of composites. The specimens were prepared with dimension 30 mm x 20 mm (length x wide). All the specimens were dried in an oven at 70 ^{o}C for 3 h. The specimens were immersed in water for 3, 6, 9, 12, 24, 48, 96, 192 and 384 h, respectively. Moisture absorption measurements were performed under 52 % relative humidity at 27 ^{o}C. The experiments were performed according to ASTM D570. After immersion, the surfaces of the specimens were dried with tissue paper and weighed immediately to measure their wet weight.

Tensile and flexural tests on the composites. The tensile and three-point bending (flexural) tests were performed using an Instron Universal Testing Instrument (Model 4206). The specimens were tested at crosshead 5 mm/min, and tests were performed in accordance with ASTM D3039 and ASTM D790, respectively. The span length-thickness ratio determined in the flexural test is 16:1. A minimum of four tensile tests were performed for each experimental composite group. The treatment assigned to each group of composites is identified in Table 2.

Table 2. Samples identification.

Samples identification	Chemical treatment of coconut coir fiber reinforced PLA composites
C1	Untreated-fiber composite
C2	6% NaOH-treated fiber composite.
C3	6% NaOH followed by soaking in 0.5% AA solution for 0.5 h - treated fiber composite.
C4	6% NaOH followed by soaking in 0.5% AA solution for 1 h - treated fiber composite.
C5	6% NaOH followed by soaking in 0.5% AA solution for 3 h - treated fiber composite.
C6	6% NaOH followed by soaking in 0.5% AA solution for 0.5 h at 70 ^{o}C - treated fiber composite.

Scanning electron microscopy (SEM). The surfaces of the fibers and the tensile fracture surface of composite samples were coated with gold and then examined using a scanning electron microscope (SEM) (JEOL JSM-6400 Japan).

3. Results and discussion

Water absorption. Fig. 1 illustrates water absorption as a function of square root of time for coconut coir/PLA biocomposites and various chemical treatments of coconut coir fiber reinforced PLA immersed in distilled water at room temperature. The water uptake process for all specimens sharply increased and approached saturation after 169 h. The composite with NaOH-treated fiber (C2) absorbed the most water. This may be attributed to the NaOH treatment removed all the lignin from the fibers surface, and PLA is the biodegradable polymer also, hence composite easy to absorb the water. According to Rozman et al [4], lignin on the coconut fiber surface reduces the water absorption of the composites. SEM micrograph of the surface of untreated and treated coconut coir fiber (Fig. 2) also supports this statement. Fig. 2a exhibits the SEM micrograph of the untreated single fiber surface, which indicates that it is full of randomly distributed organic materials, whereas, after alkali treatment, the hemicellulose and lignin are removed, and pits are revealed on the surface of the fiber (Fig. 2b) [8, 22]. This statement was also supported by FTIR that shows the peak at 1736 cm^{-1} (C=O stretching of the acetyl groups of hemicelluloses) (Fig. 3a) and disappears after treatment with NaOH (Fig. 3b) [23-25]. Therefore, capillary flow carries water along the fiber-matrix interface if bonding is incomplete [26].

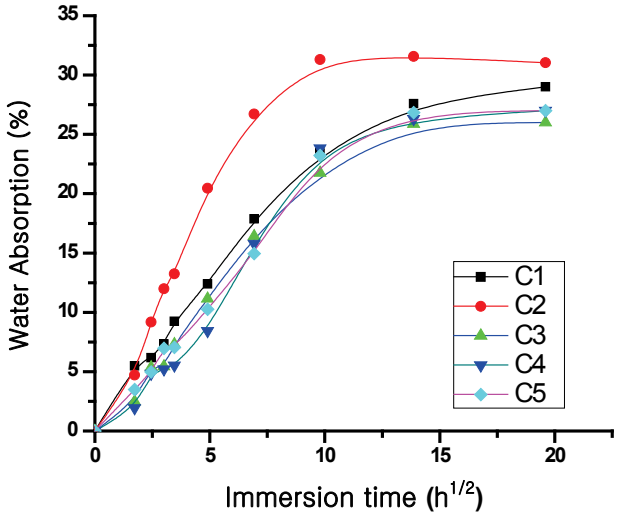

Fig. 1. Water absorption curves for composites of PLA/coconut coir fiber with different chemical treatments.

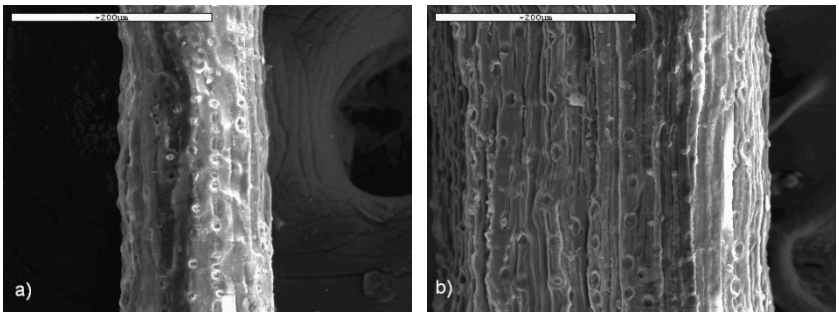

Fig. 2. SEM of (a) untreated and (b) alkali-treated single fibers.

Lignocellulosic materials absorb water molecules by hydrogen bonding between water molecules and the hydroxyl groups of cellulose and hemicellulose in the cell wall [4]. Nevertheless, the percentage of cellulose and hemicellulose in coconut coir is less than the percentage of lignin (Table 1); hence, untreated fiber composite materials (C1) absorb less water than composites with a NaOH-treated fiber (C2).

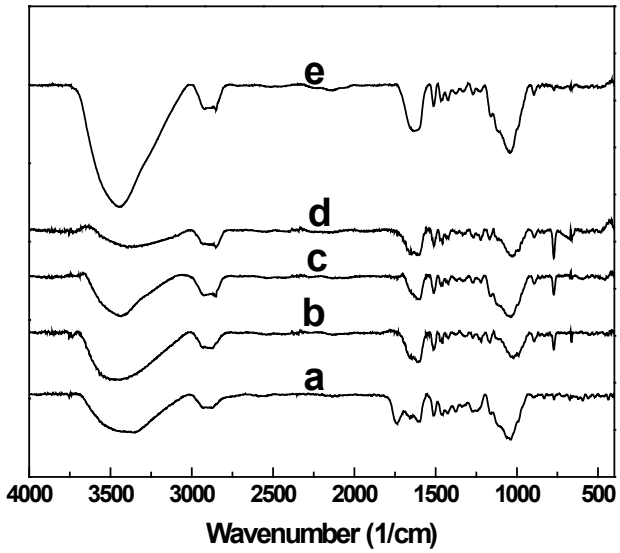

Fig. 3. FTIR spectra of coconut coir fibers: (a) untreated, (b) NaOH-treated, (c) AA 0.5h, (d) AA 1h, (e) AA 3h.

AA-treated fibers reinforced PLA composites (C3, C4, and C5) are better than C1 and C2 to reduce water absorption. This is due to the AA treatment coating the fiber surface, hence decreasing the water absorption of the composites themselves [11, 27]. As expected, the acrylic acid treatment leads to the decrease in the water sorption in the composites. Nevertheless, the difference in water absorption was not clearly shown with different AA treatment. The order of decreasing water absorption is as follows: NaOH < Untreated < AA. In addition, the reaction scheme of acrylic acid with coconut coir fibers is shown in Fig. 4. The reaction result can be seen from FTIR, which is shown at peak at 2850 cm^{-1}. This peak -CH2- and -CH-group formation more marked compared to untreated spectrum [27].

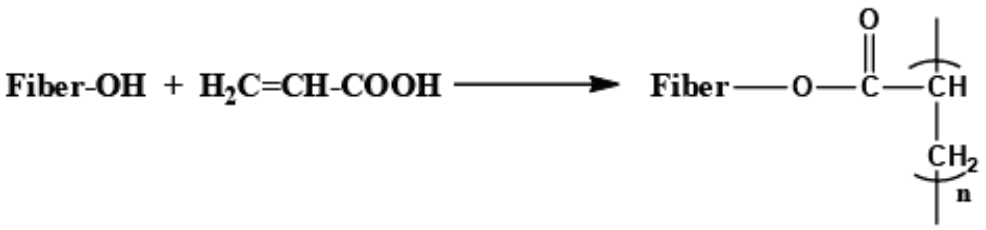

Fig. 4. Chemical reaction between fiber and AA.

Effect of water absorption on tensile properties. Figure 5 depicts the effects of fiber chemical treatments and of water absorption on the tensile properties of coconut coir fiber/PLA biocomposites. The tensile strength and tensile modulus of biocomposites increased after immersing in water for 3h, except composite with NaOH-treated of coir fiber (C2). This

could be due to the swelling of the fibers caused by water uptake, which fills the gaps and enhances the friction between the fiber and PLA matrix interface, eventually increasing the tensile strength and tensile modulus of the composites [28, 29]. As apparent from the tensile stress–strain behavior of the C3 composite without and with immersion, composites became more elastic upon immersion time. This is evident is shown in Fig. 6. The strain at break changed after water absorption. Without immersion, the composite is brittle and strains at break lower than do composites that were immersed. The C3 composite, as shown in Fig. 7a, was observed to have better interfacial bonding between the coconut coir fiber and the PLA, since the fibers and matrix break together. After soaking in water for 6 h and even 192 h, excess swelling occurred, which caused damage to the fiber structure, providing additional channels for water to diffuse into the composite, as well as debonding and further weakening the fiber/matrix interface. At these stages, the tensile strength of the composites decreases with increasing duration of water immersion [29]. The fractured surface of C3 composite after soaking for 6 h showed debonding between the fiber and the PLA matrix, leading to pullout (see Fig. 7b). In Fig. 7c, C2 composite surface after soaking for 192 hours showed some cracks and regardless of the PLA from the fiber that causes porous surfaces, while its fracture surface (Fig. 7d) indicated some debris and matrix separation without a residual on the fiber surface.

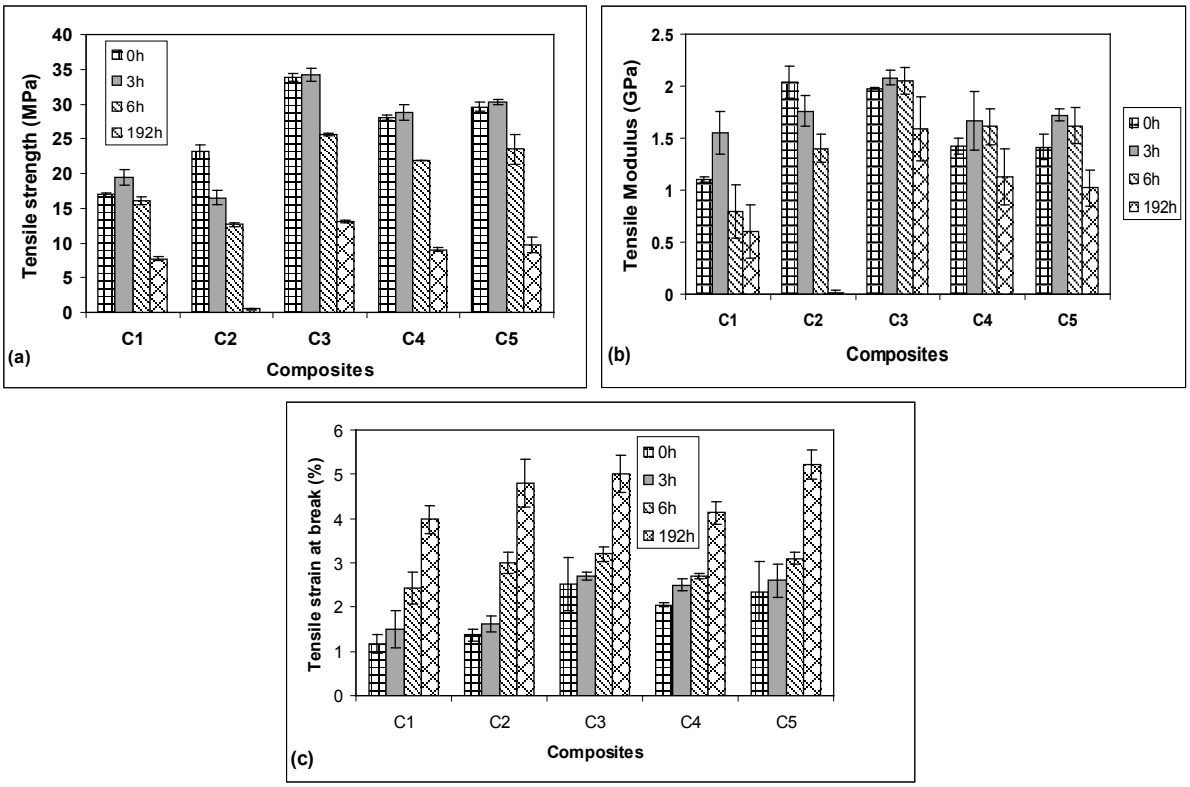

Fig. 5. Tensile properties of the PLA/coconut coir composites (a) tensile strength, (b) tensile modulus, (c) tensile strain at break.

From Fig. 5c, it can be seen that tensile strain at break for all the composites increased after soaking in water. The increasing strain at break may be due to the plasticization effect in the fibers and fiber/PLA interfaces [29]. Chow et al. [29] have reported for sisal fiber reinforced polypropylene composites in which the elongation at break sharply increased after water immersion for 3 h.

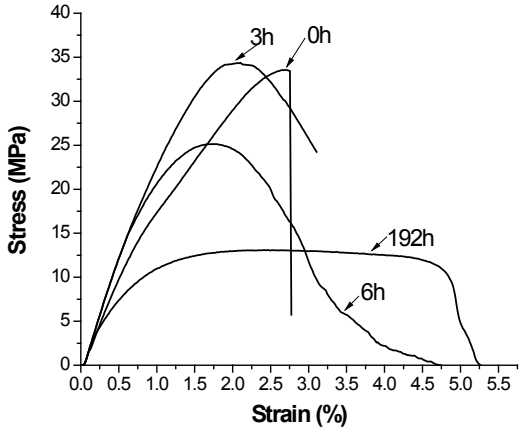

Fig. 6. The typical tensile stress-strain curves of 0.5-h AA-treated fiber composites (C3) with varying duration of water immersion.

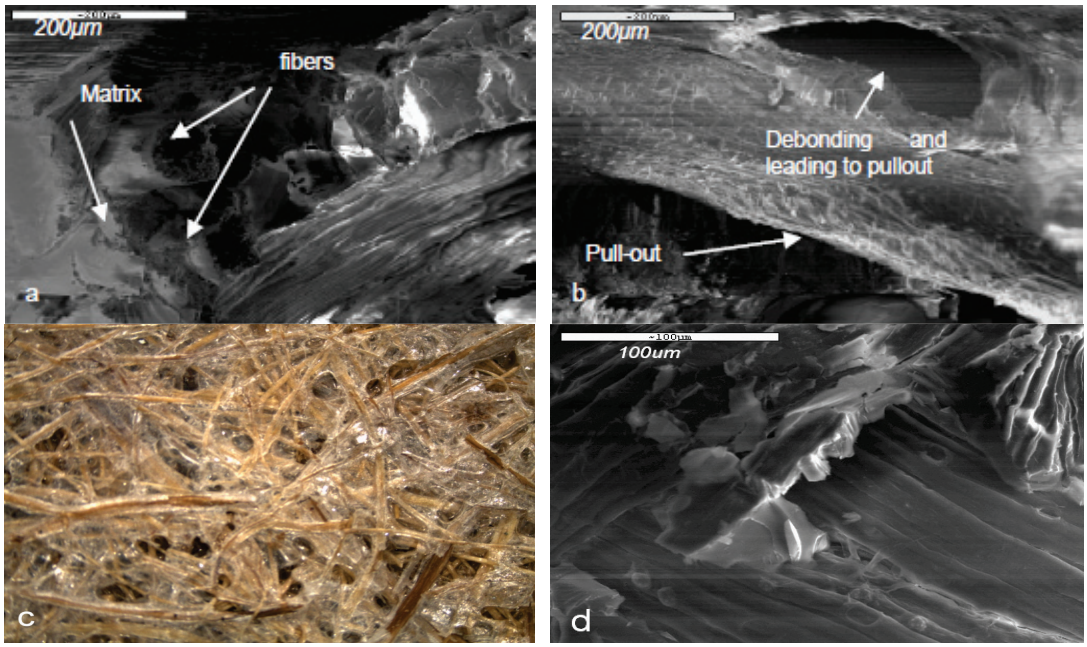

Fig. 7. SEM morphology of C3 composite after water immersion (a) 3h and (b) 6h, while (c) and (d) micro-photo and SEM of C2 composite with water immersion 192h, respectively.

In our previous study, the tensile strength of coconut coir single fiber increased slightly after alkali treatment. NaOH-treated single fibers have higher tensile strength, modulus of elasticity, and strain at break than do untreated single fibers [12]. Similar tendency occur in their composites, because treatment with NaOH removed some lignin and hemicelluloses from the fiber surface [22]. Nevertheless, C2 is not surprised to observe its tensile strength drastically decreased after soaking in the water.

The tensile strength, modulus, and strain at break of the NaOH followed by AA-treated fiber composites (C3, C4, and C5) improved in comparison to untreated (C1) and even their tensile strength (C3, C4, and C5) are higher than those of alkali-treated fiber composites (C2). AA treatment was introduced to ensure chemical bonding at the fiber matrix interface, because the waxy layer was also removed from the fiber surface [27]. The same results have been reported in the previous study with AA treated bagasse fiber composites [11]. In that

study, the AA-treated fiber composites also improved in tensile strength, modulus of elasticity, and elongation at break when compared to untreated fiber composite.

For all composites, the maximum tensile strengths occurred when the AA-treated was 0.5 h (C3). Among the AA-treated composites, tensile strength decreases when 1 h AA-treated and then increased slightly with 3 h AA-treated.

The AA-treated fiber composites reduced the water absorption and exhibited better compatibility with PLA than did untreated and alkali fiber composites [30].

Effect of water absorption on bending properties. Flexural strength and modulus of biocomposites were found with varying the immersion duration. These values decreased after water immersion [31] for 6 h and more drastically decreased after immersion for 192 h for all composites. Fig. 8a and 8b show the changing in the flexural strength and modulus of different specimens immersed in water. The water uptake reached a maximum value for the fiber of the alkali-treated composite (C2). Therefore, its flexural strength was the lowest. This behaviour can be explained by water uptake weakening the fiber matrix interfacial bond which also explained in sub chapter 3.1.

Composites with 0.5-h AA-treated fiber (C3) show maximum flexural strength and modulus. Flexural properties decrease after one hour of AA immersion (C4) and increased again after three hours of AA immersion for fiber composite (C5) but remained lower than that seen with a 0.5 h immersion time (C3) [30]. This is due to the effectiveness of the 0.5h immersion in AA solution, which improves the fiber-matrix interfacial bonding.

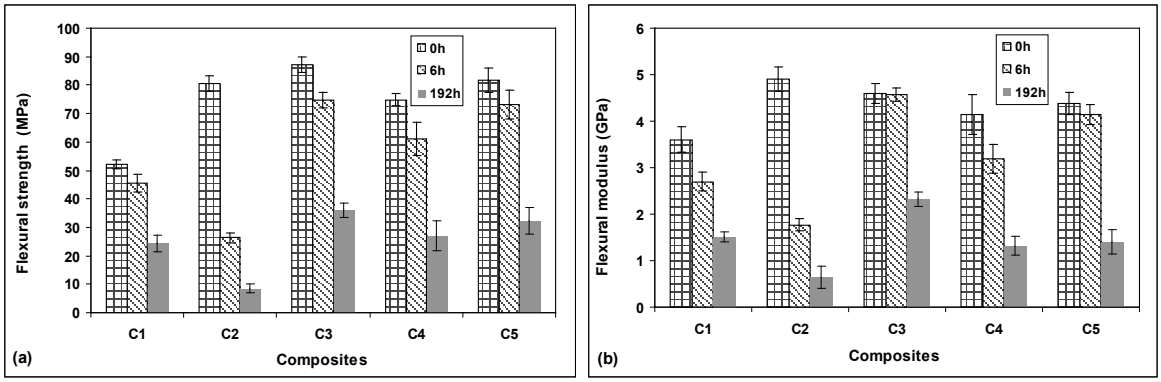

Fig. 8. Flexural properties of the PLA/coconut coir composites:
(a) flexural strength, (b) flexural modulus.

Comparison of C3 and C6 Composites. This study is to compare the best result on tensile and flexural strengths of biocomposites as described above, when the fiber is treated by AA at room temperature (C3) with treated at 70^{o}C (C6). The objective of this study is to know the effect of reaction temperature on the fibers and subsequently on its composite properties.

a. Water absorption. The coconut coir fibers from two treatment groups (those immersed in 6 % alkali followed by 0.5 % AA for 0.5 h at room temperature and those immersed in 6 % alkali followed by 0.5 % AA for 0.5 h at a temperature of 70 °C) were used to fabricate composites C3 and C6, respectively. Fig. 9 shows the water absorption of C3 and C6 composites after soaking them in water. C6 absorbed less water than C3 may be attributed to the effect of heating on AA reaction with the fibers. According to Gurdag et al. [20], the temperature is one of the most important factors affecting the extent of grafting AA on the fibers. Therefore, we suspect that the AA is more readily reactive with heating than without, and that the improved wettability of the fiber and of the matrix at the interface region [11] subsequently decreases water absorption.

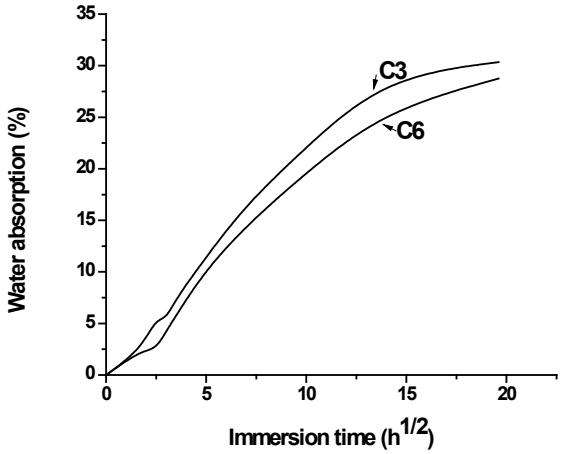

Fig. 9. Water absorption curves for C3 and C6 composites.

b. Mechanical properties. Fig. 10 reveals the mechanical properties of both composites C3 and C6 that were immersed in water for 6h. The strain at break of the composite increases when immersion time increases. On the other hand, the elastic modulus and tensile strength increases slightly when water soaking time is 3h and decreases as the immersion time in water for 6 h. Fig. 10 indicates that the strain of C3 is higher than that of C6, but the tensile strength and elastic modulus of C3 was lower than those of C6 for all immersion times. The composite prepared from coconut coir fiber with 6 % alkali solution followed by 0.5 % AA for 0.5 h at temperature 70 ^{o}C (C6) showed an improvement in tensile strength and elastic modulus by about 5.11 % and 18.3 % over the C3 composite, respectively. Again, with further increasing in immersion time (3 h and 6 h), the tensile strength and elastic modulus of C6 decreased in value. If we compare the tensile strength values of C3 and C6 composites, C6 exhibits comparatively better results than C3. In our research, the enhanced tensile strength of the C6 composite with AA-treated fiber by heating may be attributed to an improved compatibility between PLA and the coconut coir, resulting in better wettability and adhesion between the fiber and the PLA [11].

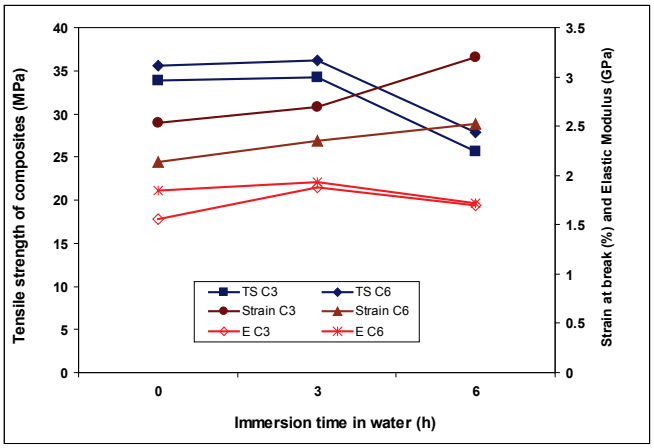

Fig. 10. Mechanical properties of the C3 and C6 composites.

c. Biodegradation. The compost was made by mixing fertilized soil and dry sawdust in a 1:1 ratio to a weight of 500 g. Fertilized soil with an initial moisture content of 50-60 %, pH

5.8-6.8, and density 0.18-0.2 g/cc was produced by Sanglim Co., Ltd., Korea. Water was supplied constantly everyday, and the weight of the compost was maintained at a minimum of 1000 g. The composites (30 mm x 25 mm) were weighed and placed on a 2 cm deep layer of the compost medium. Five replicates were prepared for each time interval, and then the samples were incubated for 27 ^{o}C for 7, 14 and 25 days, respectively. Each specimen was dug out from the compost, washed with tap water, and finally dried in an oven at 50 ^{o}C for 12 h. Biodegradability was determined by measuring weight loss of the specimens after burial in the compost. This method is the same as that used in a previous study by Naozumi Teramoto et al. [32] and Lifang Liu et al. [33]. The weight loss W_{loss} (%) was calculated using the formula [33]:

$$W_{loss}(\%) = \frac{W_o - W_t}{W_t} \times 100, \quad (1)$$

where W_o and W_t are the weights of specimens before and after burial in compost, respectively. The weight loss of the corresponding composites is illustrated in Fig. 11. The C6 composite is less biodegradable than C3 for all burial times. Weight loss is accompanied by water absorption. As can be seen from Fig. 9, water absorption in the C6 composite is less than that of C3 for all immersion times. This demonstrated that there is better interfacial compatibility of fiber and PLA in C6 than in C3. Therefore, the weight loss in C6 becomes lower than C3 for all burial times.

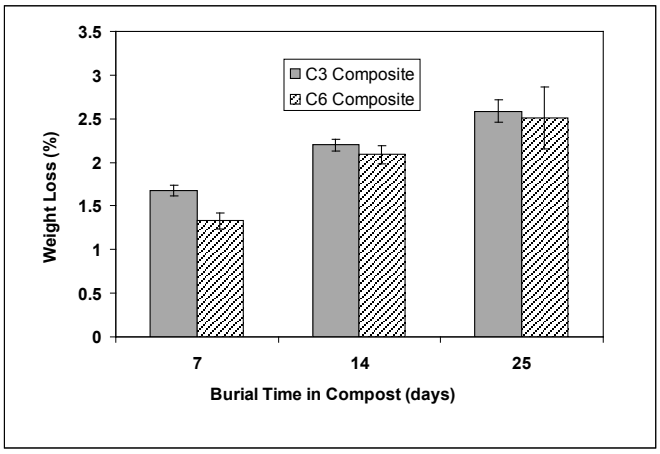

Fig. 11. Weight loss of C3 and C6 composites during burial in compost.

About 1.33, 2.09, and 2.51 % of the weight loss was observed for C6 composites, while C3 composites decreased their weight by 1.68, 2.19, and 2.58 % after 7, 14 and 25 days, respectively (Fig. 11). The percentage weight loss of C6 in the compost soil burial test was significantly lower than C3 at any given time point. A low degradation rate of the C6 sample may indicate that coconut coir fibers treated with alkali followed by 0.5 % AA for 0.5 h at 70 ^{o}C, were afforded significant decay protection. These results were supported by the observation in micro-photo of degraded surfaces. Significant fragmentation was observed randomly dispersed all along the surface of the C3 composite (Fig. 12a). Fewer areas of fragmentation were noted on the C6 composite (Fig. 12b), indicating that the fibers in the C3 were less protected by AA than the C6 composites, so the fibers decay more easily, hence biodegradation of PLA proceeds from both external and interface surfaces. The surface of composites near the fiber broke. SEM (Fig. 13a-c) images support the C3 result, as the

composite surfaces appeared increasingly cracked and broken with increasing burial time in the compost. The length of the burial time changes the surface morphology of the C3 composites. After burial for 7 days, fragmentation growth on the surface was observed (Fig. 13a). After 14 and 25 days, fragmentation and some destruction became apparent (Figs. 13b and c), which may have been due to biodegradation by bacterial.

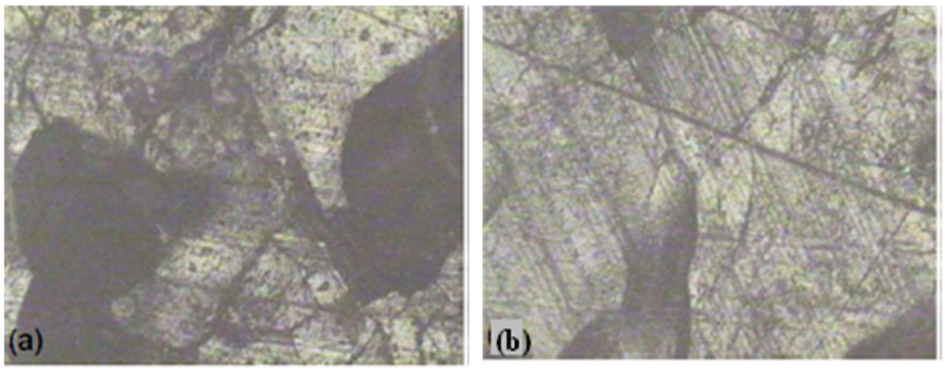

Fig.12. Biodegradation of (a) C3 and (b) C6 composites.

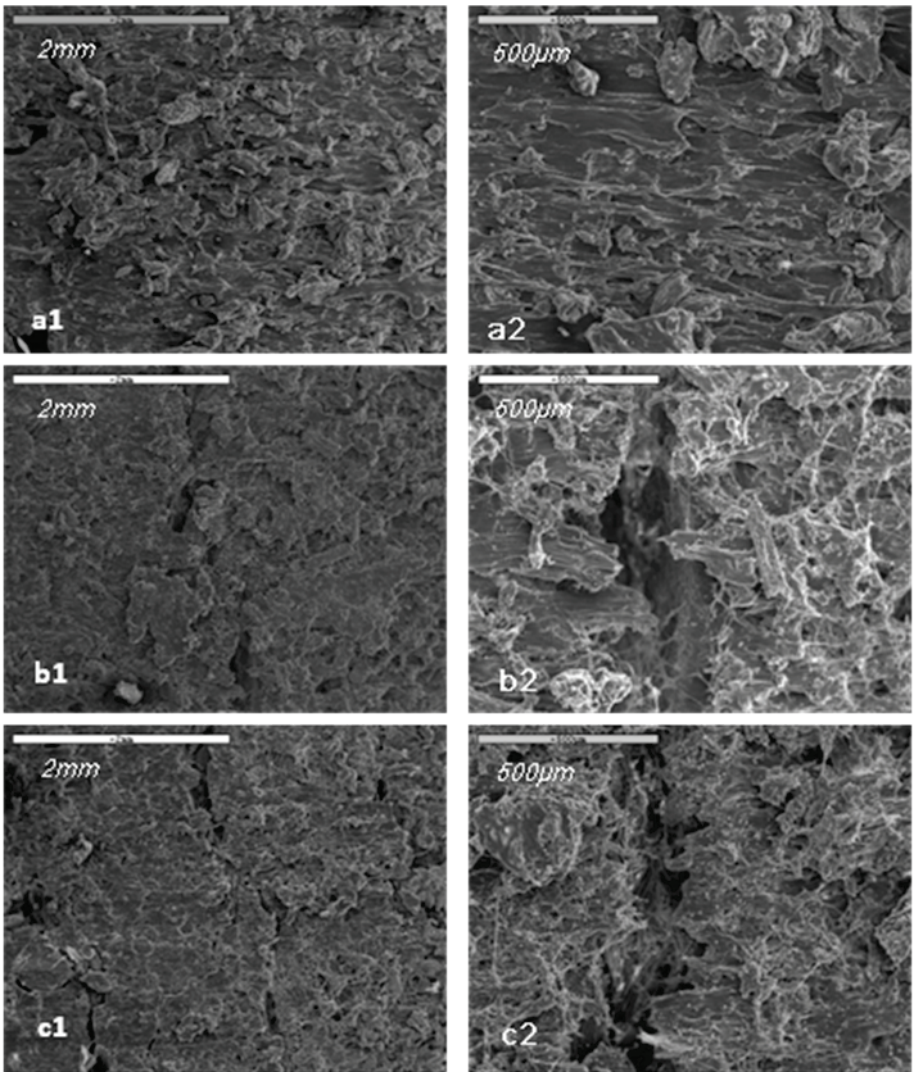

Fig. 13. Biodegradation of C3 composite with burial time: (a) 7, (b) 14, and (c) 25 days.

4. Conclusion

The water uptake process for coconut coir reinforced PLA composites sharply increased and approached saturation after 169h. The composite with NaOH-treated fiber (C2) absorbed the most water and caused its tensile and flexural strength dropped drastically. In General, composites with coconut coir fiber treated with NaOH followed by AA (C3, C4 and C5) had greater tensile and flexural strengths and less water sorption than those of untreated fibers (C1) or fibers treated only with NaOH (C2). Based on the AA-treated of coconut coir at room temperature, C3 composite has the maximum tensile and flexural strength for all water immersion time of composites. The flexural properties of biocomposites decreased after 6h of water immersion and drastically decreased after 192h. In comparison with C3 and C6 composites, the effect of reaction temperature on the graft copolymerization of AA on the fibers is effectively used. It can be seen that the tensile strength and elastic modulus of C6 were higher than those of C3 for all immersion durations. In addition, C6 composite absorbed less water and was less biodegradable.

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