Influence of Durian Rind Cellulose Microfiber as the Fillers on the Mechanical Properties of Polylactic Acid Bio-composites

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Received: 31 March 2024, Revised: 13 May 2024, Accepted: 13 May 2024

Abstract
This work aims to study the influence of durian rind cellulose (DRC) microfiber as a reinforcing filler on the mechanical properties of polylactic acid (PLA) bio-composites. Cellulose from durian rind will be extracted using sodium hydroxide solution and bleached treatment with hydrogen peroxide. X-Ray fluorescence technique was used to examine the chemical composition of durian rind cellulose, and functional groups were analyzed using FT-IR technique. PLA was mixed with DRC microfibers particle size of 300 μm by using a mixing apparatus, and then melt-blending by extruder in varying ratios of 100/0, 95/5, 93/7, 90/10, and 80/2 0 wt%. The results revealed that the mechanical properties of PLA/DRC composites in the tensile modulus, flexural modulus, and shore-D hardness of the composites at DRC microfibers of 20 wt% was increased a maximum of 64%, 21%, and 16%, respectively, as compared to the neat PLA. On the other hand, the tensile strength, and flexural strength of the composites at DRC microfibers of 20 wt% was depressed by 9%, and 23%, respectively, as compared to the neat PLA. Polymer composites reinforced with DRC microfiber had increased water absorption when increasing DRC microfiber particles. PLA biocomposites as a new eco-friendly alternative material, and can be used to as compostable chopsticks.
Keywords: Durian rind cellulose, Polylactic acid, Biocomposite, Mechanical properties

1. Introduction

Polymer biocomposites are green alternative materials that can be fabricated to replace the rare and expensive woods, and can also serve as environmentally friendly alternatives to petroleum-based plastics such as high-density polyethylene (HDPE), polypropylene (PP), etc. These plastics are durable and have a long lifespan, but they cannot be decomposed naturally. Consequently, the amount of plastic waste has increased worldwide along with the increasing population, leading to the greenhouse effect and global warming. Polymer composite materials have garnered attention from researchers due to their advantages in good mechanical properties and suitability for a wide range of applications in the automotive industry, agricultural industry, interior architecture, and various creative works that require durability and beauty.

Durian is another important economic fruit crop of Thailand, and it has important cultivation areas in the eastern and southern regions of the country. Durian production throughout the year in Thailand amounts to 1,537,000 tons. [1]. After consuming a durian fruit, the durian rind is considered waste. Durian rind fibers have good mechanical properties, environmentally friendly, and biodegradable [2-4]. Xing et al. [5] studied preparation and extraction of cellulose nanofibers from durian peel using sodium carbonate, and found that cellulose nanofiber aerogel had the porosity high to 99%, and compressive strength of cellulose aerogel composite was highest when increased cellulose nanofibers of 5 wt%. Nadondu et al. [6] studied the mechanical of polylactic acid/glass-carbon/durian skin fiber hybrid composites, and found that tensile and impact strength, and hardness of PLA composites increased when a mixture content of E-glass fiber/carbon fiber/durian skin fiber was 5.05/10/10 wt%, respectively. Polypropylene hybrid-composites reinforced with cellulose nanofibers (CNFs)/Talc, and mix with a maleic-anhydride were investigated by Kinoshita et al. [7]. They found that the mechanical properties of the composites (elastic constant, and tensile strength) increased when increasing the cellulose nanofibers and maleic-anhydride. Zhao et al. [8] used an epoxy solution-modified pine-wood fibers as a reinforced on PLA composites. It was found that the PLA composites reinforced with

pine-wood, and add an epoxy solution an amount of 0.5-10 wt% increased in tensile strengths and elastic modulus of the composites up to 20% and 82%, as compared to pure PLA. In addition, also found that the PLA composites/pine-wood with an epoxy solution (1.0 wt%), had occurred fewer voids on the fracture surface of the composites, as comparing with PLA/pine-wood composites without surface-modified of pine-wood fibers by epoxy solution. Sitticharoen et al. [9] studied the mechanical and thermal properties of polylactic acid (PLA) hybrid biocomposites reinforced with hemp microfibers (Hf) and microtalc (Tc) were investigated. Young's modulus and hardness properties of biocomposites for chemical surface-modified Hf-Tc particles were higher, as comparing with non-modified particles, also found that the impact strength of biocomposites for modified Hf-Tc content at a ratio of 14:6 wt%, and 6:14 wt% were significantly increased. The crystallinity of PLA-Hf-Tc biocomposites for chemical surface modified Hf-Tc and non-modified Hf-Tc a little increased, but the melting temperatures of biocomposites were didn't different to pure PLA. Thermal stability of pure PLA was higher, as compared to PLA-Hf-Tc biocomposites. Rafiqah et al. [10] studied the miswak fibers treatment with a sodium hydroxide (NaOH) on the mechanical- thermal properties of polylactic acid composites, and found that the tensile strength of the composites significantly increased with increasing concentration of a chemical solution treatment the miswak fibers, but modulus of the composites tends to decline. The thermal stability of the composites slightly increased as comparing with the un-treated fiber composites. Jesus et al. [11] studied the mechanical and thermal properties of polylactic acid (PLA) reinforced with curaus microfibrillated cellulose, and found that the tensile and flexural strength, and flexural modulus of composites increased with 0.5 wt% of microfibrillated cellulose as compared to the neat PLA, and thermal stability of the composites lower than that the neat PLA.

This research aims to study cellulose microfibers from waste durian rind as reinforcements in polylactic acid (PLA) composites, and to use them in the production of biodegradable prototype chopsticks.

2. Materials and Methods

2.1 Materials

Polylactic acid (PLA) injection grade 3251D, with a melt flow rate (MFR) of 35 g/10 min (190 °C, 2.16 kg), and obtained in pellets form by Nature Works LLC, USA. Durian rind (DR) as waste, and obtained from Muang Mai Market in Chiang mai, Thailand, as displayed in Fig. 1(a). Durian rind cellulose microfibers (DRC) were an

Figure 1 (a) DR and (b) DRC microfibers.

average of particles size of 300 μm, as shown in Fig. 1(b). The chemical used in this work as sodium hydroxide (NaOH) solution, and hydrogen peroxide (H_2O_2) solution were purchased from Northern Chemicals and Glassware Ltd., Partnership (Chiang Mai, Thailand). The chemical constituent of DRC microfibers were investigated with X-ray fluorescence spectroscopy (XRF) technique, (Fischerscope X-RAY XUV 773), as displayed in Table 1.

2.2 Preparation of DRC Microfibers

DR was washed and cut into small, thin pieces. The small DR pieces were boiled in a sodium hydroxide solution with a concentration of 15 wt% at 90°C for 2 hours. Then, the DR was stirred to separate it into fibers. Finally, the DR fibers were rinsed with running tap water. A 35 wt% hydrogen peroxide solution of was bleached treatment the DR fibers, and rinsed the DR fibers

with running water from tap water until the pH value achieved 7.0. DRC microfibers were dried in an oven at 80 °C for 48 h [9, 24], and then grinded and sieved with ASTM E11 No.50 mesh (particles size: 300 μm).

Table 1 Examination of chemical constituent of DRC microfibers with XRF technique.

Chemical constituent of DRC microfibers	Content (%)
Iron (III) oxide (Fe_2O_3)	47.8
Calcium oxide (CaO)	18.3
Silicon dioxide (SiO_2)	16.9
Sulfur trioxide (SO_3)	6.28
Potassium oxide (K_2O)	3.39
Phosphorus pentoxide (P_2O_5)	3.05
Zinc oxide (ZnO)	1.67
Titanium (IV) oxide (TiO_2)	1.35
Manganese (IV) oxide (MnO_2)	1.26

2.3 Fourier Transform Infrared Spectroscopy

The functional groups of DR and DRC microfibers was examined with a Fourier transform infrared (FT-IR) instruments (Thermo Fisher Scientific, Nicolet 6700). The samples were scanned in the range of 4000 cm^{-1} to 500 cm^{-1}, and infrared spectra was used at a resolution of 4 cm^{-1}.

2.4 Scanning Electron Microscopy Analysis

Fracture surface of PLA/DRC biocomposite samples were performed in liquid nitrogen for 2 min, and then the top of the samples were coated with thin layer of gold by sputter coating apparatus. The fracture surface of biocomposites in this work were analyzed on scanning electron microscopy (SEM) apparatus (JEOL, JSM-5410LV), and conducted with an accelerating voltage of 15 kV.

2.5 Preparation of biocomposites

PLA were mixed with DRC microfibers using a mixer machine in varying ratios of 100/0, 95/5, 93/7, 90/10, and 80/20 wt%, and each compound ratio was mixed for 3 min. Then, melt-blend was done on a single screw extruder (SMC500) machine. The temperature profiles of melt-blend by extruder from hopper to melt-area zones (a capillary die) were 150 °C to180 °C, and a screw rotation speed was set low at 40 rpm, and then cut into the bio-composite pellets with a pelletizer apparatus. PLA/DRC biocomposites were placed in an oven at 80 °C for 24 h [9, 24]. All the test specimens, and compostable chopsticks workpiece were performed using an injection molding (HYF - 1000) machine, with used an injection temperature of 185 °C. The specimen dumbbell-shape of PLA/DRC biocomposites for different DRC ratio were done by mold, as shown in Fig. 2.

(a) (b) (c) (d) (e)

Figure 2 PLA/DRC biocomposites specimen images for different DRC ratio: (a) 100/0 wt%, (b) 95/5 wt%, (c) 93/7 wt%, (d) 90/10 wt%, and (e) 80/20 wt%.

2.6 Mechanical Properties

The Hounsfield50-KS tester machine was used to perform the tensile and flexural test of the PLA/DRC biocomposites. Tensile tests were conducted at running the speed of 50 mm/min, according to ASTM D638-2014, and flexural test was done at running the speed of 1 mm/min, according to ASTM D790-2007. Hardness (Shore-D) test was evaluated using a Durometer (HH-337-11) apparatus. All values of the mechanical properties of PLA/DRC biocomposites were evaluated using independent measurements to ensure data reliability. The averages were calculated from 5 replicate test samples in each mixing ratio, as illustrated in Fig. 2, and then the results were reported.

2.7 Water Absorption Measurement

Water absorption measurements of *PLA/DRC* biocomposites were conducted in accordance with ISO-62-2008, as a modified-standard. All the specimens were performed by immersion in water for 14 days, and then weighed. The water absorption of biocomposites was evaluated, and the results were reported based on the average of measurements from 5 independent testing replications in an experiment. The water absorption (W_{abs}) values

are reported as the percentage of water absorption ($\%W_{abs}$), as follows [9,12]:

$$\%W_{abs}=\frac{W_a-W_b}{W_b} \quad (1)$$

where W_a and W_b are the sample weight after immersion, and the sample weight prior immersion in water, respectively.

vibration of the hydroxyl groups, because of the hydrogen bonded between the molecules. In this work was used a sodium hydroxide on eliminate the lignin and hemicellulose from the DR microfibers, as shown in the curve of DR. The absorption band of DR microfibers at the wavenumber of 2919 cm^{-1} was assigned to the C-H stretching vibration in molecules from

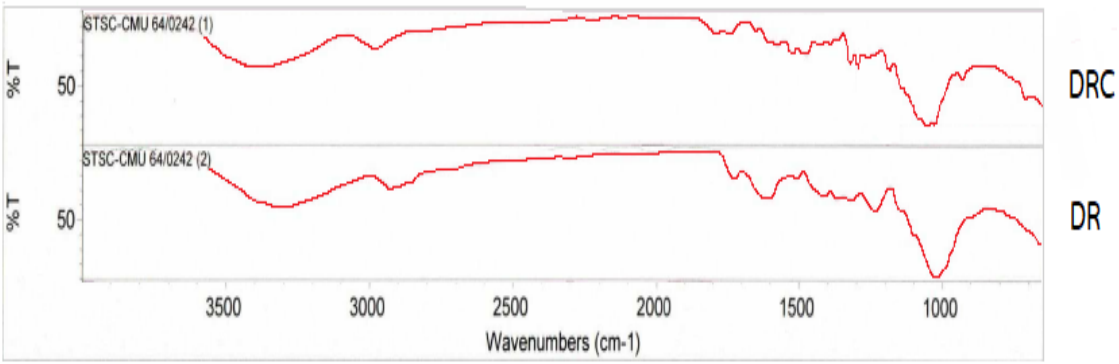

Figure 3 FT-IR spectroscopy of DR, and DRC.

3. Results and Discussion

3.1 Characterization of DRC Microfibers

An image of DRC microfibers, as shown in Fig. 1. It is clearly that DRC microfibers was fine white powder particles, as a result of bleached treatment with hydrogen peroxide used. The chemical constituent of DRC microfibers was examined by XRF instrument. It was obviously visible that the main composition of DRC microfibers, approximately 47.8% as ferric oxide, 18.3% of calcium oxide, 16.9% of silicon dioxide, and the rest composed of the sulfur trioxide, potassium oxide, phosphorus pentoxide, zinc oxide, etc., as depited in Table 1.

3.2 Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

The principle functional groups of DR and DRC microfibers were analyzed with FT-IR technique in the scanning range of 4000 cm^{-1} to 500 cm^{-1}, and the FT-IR spectra results of the samples at each range were recorded, and then reported, as displayed in Fig. 3. The FT-IR spectrum of the DR and DRC microfibers were exhibited severe peaks, which was indicated the presence of O-H stretching vibration in the DR and DRC microfibers at the wavenumber of 3283 cm^{-1} and 3329 cm^{-1}, respectively. This stretching peak in the DR and DRC microfibers indicated the hydrophilic of fibers surface. This view also supported by the works of Xing et al. [5], Zhao et al. [8], Jumaidin et al. [13], Edhirej et al. [14], and Kunthadong et al. [15] who stated that the peak in these range were attributed to the stretching of CH_2 and/or CH_3 groups [13,14,16,17]. At the wavenumber of 1730 cm^{-1} of DR was indicated the presence of the stretching vibration of C=O, which was an ester group that presence in the hemicellulose, and had been widely found by the works [5,13,15,18]. The stretching vibration C=C bond of DR at the wavenumber of 1510 cm^{-1}, which was indicated the existence of the benzene ring of lignin [5,18,19]. It was found that at the wavenumber of 1420 cm^{-1} of DR was assigned to the aromatic-skeletal vibration of lignin. The C-O stretching vibration peak of lignin in the natural fibers was represented at 1235 cm^{-1} [5,18-21]. After used a sodium hydroxide treatment, those characteristic of the peaks invisible in the curve DRC. The results clearly shown that the lignin and hemicellulose in the DR microfibers had been successfully eliminated. The FT-IR spactra results of DRC microfiber appeared in curve DRC, it was obvious that the durian rind cellulose composed of the characteristic absorption peaks of O-H stretching vibration, C-H stretching vibration, C-H bending vibration, and C-O stretching vibration at a wavenumber of 3329 cm^{-1}, 2891 cm^{-1}, 1366 cm^{-1}, and 1157 cm^{-1}, respectively. It was also found that at a wavenumber of 894 cm^{-1} were defined to the β-glycosidic linkage [5,15-17,22].

3.3 Mechanical Properties

Elastic modulus and tensile strength results of PLA/DRC biocomposites for different DRC

ratio are presented in Table 2, and Fig. 4. It was found that when added the reinforcing filler into PLA biocomposites, the elastic modulus values of PLA/DRC biocomposites significantly increased. This was because of the effect of the rigidities of the silica and calcium-based DRC microfibers that can be inserted and dispersion within the PLA-matrix biocomposites [9,23,24].The elastic modulus value of the biocomposites at DRC microfibers of 20 wt% was increased a maximum of 64% as comparing with the neat PLA. On the other hand, the tensile strength values of the biocomposites slightly decreased with the increasing in the DRC microfibers content loading. At the DRC microfibers of 20 wt%, the tensile strength value of the biocomposites was reduced by 9% as compared to the neat PLA. The decrease in tensile strength of the biocomposites also has been reported by other researchers [6,9,23-25].

Table 2 Experimental elastic modulus (E.M.) and tensile strength (T.S.) results.

PLA/ DRC (wt%)	100/0	95/5	93/7	90/10	80/20
E.M. (MPa)	1,273.28	1,594.68	1,767.51	1,889.14	2085.60
T.S. (MPa)	68.92	61.80	59.84	56.75	62.52

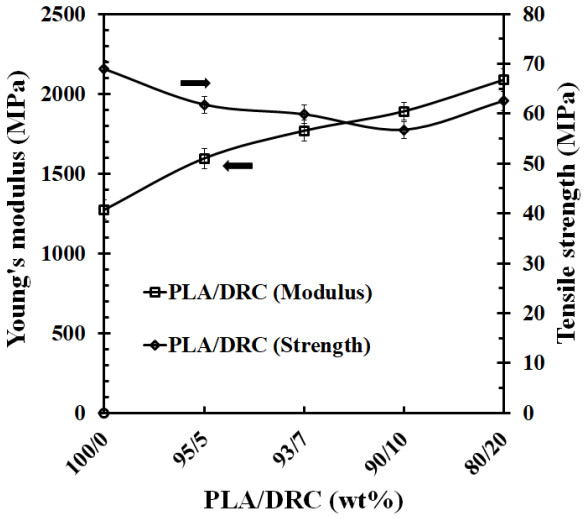

Figure 4 Elastic modulus and tensile strength of PLA/DRC biocomposites for different DRC ratio.

This may be attributed to an agglomeration of fibers to fibers, leading to the decline interfacial area between PLA-matrix surface and DRC microfibers when the mixture of fiber increases up to 20wt%, resulted in a decrease in the tensile strength. As displayed in the SEM images of the biocomposites in Fig. 6(d), and Fig. 6(e), which was obviously appeared the voids, pore, crack-texture, and also the pull-out of the DRC fibers from the PLA matrix surface that indicate a weak of adhesion in the PLA biocomposites. The flexural modulus and flexural strength results of the PLA/DRC biocomposites for different ratio are shown in Table 3, and Fig. 5. It was observed that the flexural modulus gradually increased with increasing the DRC microfibers filler content. The flexural modulus value of the biocomposites at DRC microfibers of 20 wt% increased a maximum of 21% as compared to the neat PLA.

Table 3 Experimental flexural modulus (F.M.) and flexural strength (F.S.) results.

PLA/ DRC (wt%)	100/0	95/5	93/7	90/10	80/20
F.M (MPa)	1,896.88	1,988.45	2,148.23	2,237.14	2,288.60
F.S. (MPa)	104.12	98.75	94.70	86.46	80.37

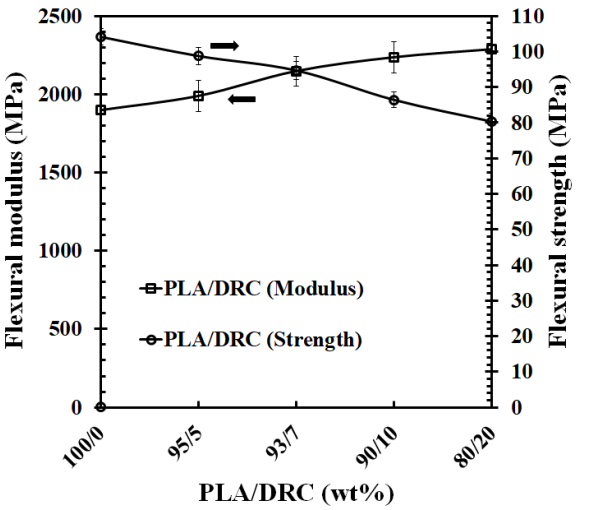

Figure 5 Flexural modulus and flexural strength of PLA/DRC biocomposites for different DRC ratio.

It was found that the flexural strength value of the biocomposites decreased as the increases the filler loading. At the DRC microfibers of 20wt%, the flexural strength value of the biocomposites was 23% reduced as compared to the neat PLA. When increased in filler concentrations the flexural modulus of the composites progressively increased but slightly decreased its flexural strength (intersection point at

mixing ratio: 93/7wt%) as displayed in Fig. 5. The decrease in flexural strength of the composites can be revealed by the SEM image in Fig. 6(c), due to

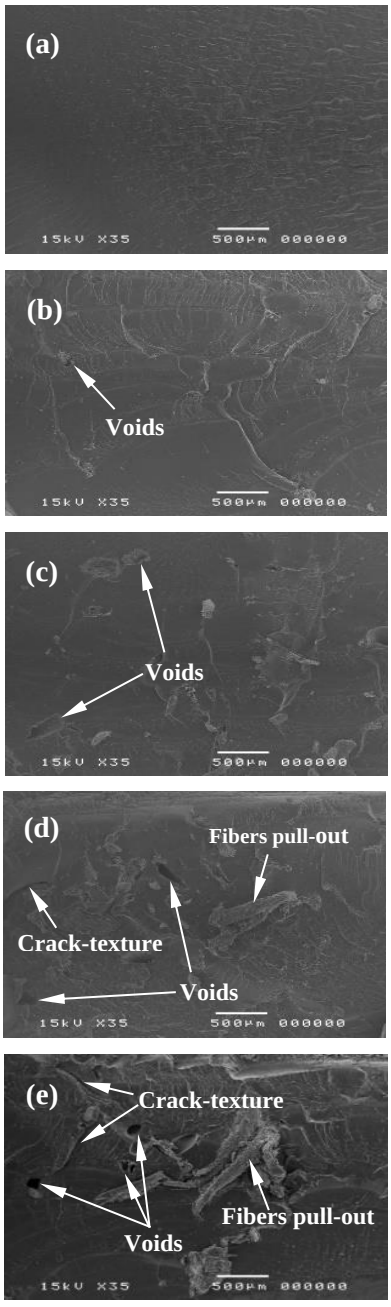

Figure 6 SEM images of PLA/DRC bio-Composites at different DRC ratio: (a) 100/0 wt%, (b) 95/5 wt%, (c) 93/7 wt%, (d) 90/10 wt%, and (e) 80/20 wt% (x35).

poor fiber dispersion. This may lead to the formation of voids and pores on the PLA matrix surface [3,9]. Fig. 6 demonstrated the SEM microstructure of the PLA/DRC biocomposite samples. Obviously, when increased the filler loading in the biocomposites, the microstructure gradually changes. This was led to incompatibility between PLA-matrix and the DRC microfibers, and then the generate more the voids, pore, crack-texture and the fibers pull-out within the bio-composite samples, which was a reduced in the tensile and flexural strength. The results of shore-D hardness of PLA/DRC biocomposites are depicted in Table 4, and Fig. 7. It was obviously that when the DRC filler concentration was increased, the hardness values of the biocomposites progressively increased, because of these the DRC microfibers composed of the strong filler particles. The hardness values of the biocomposites with DRC microfibers of 20 wt% was 83.64 as compared to the neat PLA was 72.3.

Table 4 Experimental hardness results.

PLA/ DRC (wt%)	**100/0**	**95/5**	**93/7**	**90/10**	**80/20**
Shore-D	72.30	77.47	80.53	81.76	83.64

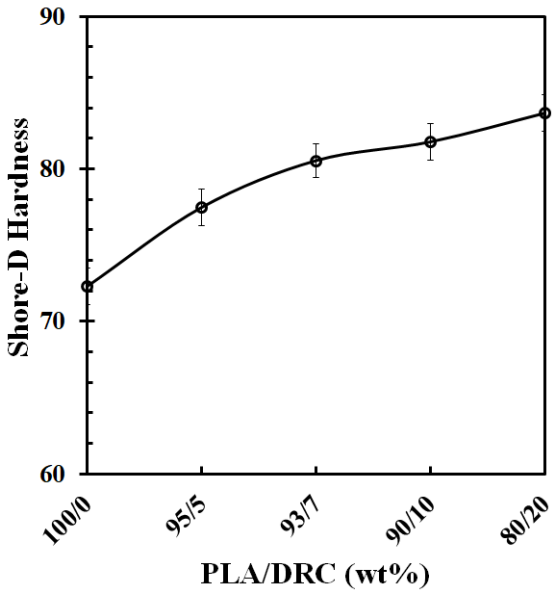

Figure 7 Hardness of PLA/DRC biocomposites for different DRC ratio.

3.4 Water Absorption Measurement

Water absorption measurement was one necessary properties of the biocomposites that must be performed, as presented in Table 5, and Fig. 8. It was found that the water absorption of PLA/DRC biocomposites properties gradually increased with increasing the DRC microfibers. It can be explained in two possible manners, as

follows: 1) Natural fibers loaded into biocomposites generally had hydrophilic properties, and it was affirmed from FT-IR results at 3329 cm^{-1}, indicated the O-H groups of the nature fibers as hydrophilic [8,13,14], and also confirmed this view from the literatures [9,25], who stated that the composites with high natural fibers content, the water absorption will also be high, 2) When natural fibers added to polymer-matrix composites, they encourage the formation

Table 5 Experimental water absorption (W_{abs}) results.

PLA/DRC (wt%)	100/0	95/5	93/7	90/10	80/20
W_{abs} (%)	0.0	0.29	1.15	4.14	5.50

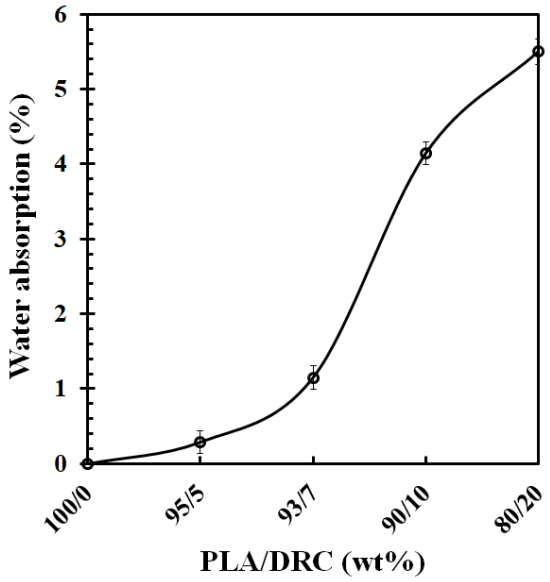

Figure 8 Water absorption of PLA/DRC biocomposites for different DRC ratio.

of voids, pores, and fiber debonding within the composite samples. Thus, the water can be easily inserted into the composites [9,25].

3.5 Application of the PLA/DRC Bio-composites

Fig. 9 shows the photograph of the compostable chopsticks sample made from PLA/DRC bio-composites with the ratio of PLA/DRC microfibers at 93/7 wt%. These compostable chopsticks were done by an injection molding method at the melting temperature of 185 °C, and had the dimension of a chopstick was 5 mm in diameter, and 206 mm in length.

Figure 9 Compostable chopsticks made from PLA/DRC biocomposites.

4. Conclusions

Durian rind cellulose microfiber is an alternative reinforcing filler used in polylactic acid bio-composite materials. The results showed that durian rind cellulose constitute approximately 18.3% of calcium oxide, and 16.9% of silicon dioxide which was potential as a reinforcing filler. It was found that the mechanical properties of the polylactic acid/durian rind cellulose biocomposite had the value of the tensile modulus, flexural modulus and hardness (shore-D) increased with increasing durian rind cellulose microfiber particles, but the value of the tensile strength and flexural strength depressed when the amount of durian rind cellulose particles increased. Polymer composites reinforced with durian rind cellulose microfiber particles was found to be increased water absorption when increased the durian rind cellulose. PLA can be mixed with 7 wt% DRC fibers, results in the tensile and flexural modulus, and hardness of composites clearly increased but slightly decreased its tensile and flexural strength. PLA/DRC biocomposites are eco-friendly, lightweight, non-toxic, and naturally biodegradable materials, its suitable for use in consumer products such as biodegradable spoons and chopsticks.

5. Acknowledgments

The authors thank the Division of Tools and Die Engineering, Faculty of Engineering, Rajamangala University of Technology Lanna for kindly providing a place for the experiment, and support this research.

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