Research Article

Characterization of Polypropylene Green Composites Reinforced by Cellulose Fibers Extracted from Rice Straw

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Polypropylene (PP) based green composites containing 10, 20, 30, 40 and 50 wt% of cellulose fibers (CFs) which were extracted from rice straw were successfully prepared by melt blend method. The CFs washed with H_2O_2 after alkaline extraction showed lower water absorption than that not washed with H_2O_2 . The thermal, mechanical, and biodegradation properties of composites were also investigated. The 10% weight loss temperature of the composites was decreased with the increasing CFs content, but all the composites showed over 300°C. Young's modulus and flexural properties of PP were improved by blending PP with CFs. The pure PP showed no degradability, but the PP/CFs composites degraded from about 3 to 23 wt%, depending on CFs content after being buried in soil for 50 days. These PP/CFs composites with high thermal, mechanical properties and biodegradability may be useful as green composite materials for various environmental fields.

1. Introduction

Natural fibers as reinforced materials in polymer composites have attracted much attention for being applicable in many fields such as automotive, aerospace, packaging, construction, and transportation industries [1–6]. Natural fibers which are used as fillers or reinforcement materials in polymer composites including palm sheath [7], palm leaf [8], guayule biomass [9], bagasse [10], sunflower stalk flour [11], banana [12], sugarcane, pineapple, ramie, and cotton [13] show many advantages such as renewability, biodegradability, CO_2 neutrality, nontoxicity, wide availability, low cost, low density, low energy consumption during fabrication, and high specific strength compared to synthetic fibers [14–16]. Therefore, the polymer composites reinforced by natural fibers are becoming significantly important for the production of a large variety of composites due to being relatively cheap, lightweight, and eco-friendly materials [17].

The choice of polymer as a matrix material is important for the kinds of natural fiber reinforcement. Polyethylene, polystyrene, polyvinyl chloride, polylactides (PLAs), polypropylene (PP), and so on are thermoplastic materials and can be used as matrix materials for composites. PLAs

are biodegradable polymers, are available from renewable sources, and degrade completely to water and carbon dioxide. However, the cost of PLAs is significantly higher than other polymers such as PP. Among the types of plastics, PP is widely used in the industrial products and household goods and especially as a matrix material in composites, due to low production cost, design flexibility, and recyclability, compared with other polymers. Several potential properties of PP include heat distortion temperature, flame retardant, transparency, and dimensional stability. Besides, PP also suits filling, reinforcing, and blending [12]. Therefore, many researches have reported composite materials with PP as a matrix and CFs of various plants as reinforcement materials [12, 18–22]. CFs have a number of -OH groups and hence lead to a poor interaction between PP and CFs in composite material and even would decrease mechanical and thermal properties such as tensile strength, flexural strength, and thermal stability [23]. The problem can be solved by the modification of CFs surface using physical or chemical treatment methods. Chemical treatment methods are known as silane treatment, alkaline treatment, maleated coupling, acetylation, and enzyme treatment [24]. Akhtar et al. studied the mechanical properties of alkaline treated and untreated

kenaf reinforced PP composite with fibers volume fractions of 10 to 50% [18]. The results were that PP composites with alkaline treated kenaf showed greater mechanical properties than alkaline untreated kenaf and composite with 40% of treated kenaf exhibited highest mechanical properties. The natural fibers are treated using alkaline, the lignin, hemicellulose, and oils and other substances on the fiber surface are removed, leading to the improvement of the bonding between the fiber and matrix [20]. In addition, moving them on the fiber surface increases the number reaction sites on the fiber surface and improves surface roughness of fibers [18, 25, 26]. The CFs treatment using alkaline not only increases mechanical properties of PP matrix composites but also contributes to other matrix composites. Ray et al. reported that the jute fibers which were treated using 5% NaOH for 4, 6, and 8 h at 30°C increased their flexural modulus by 12, 68, and 79%, respectively [27]. To date, some studies have reported to use wheat straw in polymer composites as a reinforcement material, and the matrix material in composites was commonly used as PP [28–31]. Zou et al. demonstrated that, for PP matrix composites, split wheat straw fibers showed better reinforcement material than whole wheat straw fibers, due to the increase in the surface area and aspect ratio of split configuration [31]. However, to the best of our knowledge, relatively little research is available on cellulose natural fibers from rice straw reinforced composite materials, especially no report in CFs from rice straw. In Vietnam, about 45 million tonnes of grains equivalent to 54 million tonnes of rice straw was produced in 2015, and it tends to increase in the coming years. However, most of the postharvest rice straw is burnt on open fields, causing ecological environment pollution and wasting potential resources. According to a survey by the Food and Agriculture Organization of the United Nations (FAO) in 2016, global paddy production reached 761.9 million tonnes, especially in Asia, which is set to lead the global recovery, with 680.1 million tonnes of grains being produced [32]. The agricultural wastes including rice straw are one of the most important problems that must be resolved for the conservation of global environment [33]. Therefore, research to convert them to useful materials is essential.

This study focuses on thermal and mechanical properties of composites consisting of PP and CFs which were extracted from rice straw using alkaline treatment method. Cellulose from rice straw is abundant in nature and contributes to environmental improvement due to its excellent biodegradable properties [34]. In this paper, the biodegradability of PP/CFs composites is also reported. This research is especially important in Asia in general and in Vietnam in particular. The results are expected to contribute to environmental protection and solve the problem of wasting resources.

2. Materials and Methods

2.1. Materials. Commercial PP used as a matrix material was purchased from the Polyolefin Co., Private Limited, Singapore, with melting temperature of 170°C, density of 0.90 g·cm⁻³, and melt-flow index of 10 g/(10 min) at 230°C. Rice straw was collected from a rural area of Vietnam. The chemical composition of rice straw depends on the rice straw

varieties, producing area, and so forth. It contains on average 32–47% cellulose, 19–32% hemicellulose, 5–24% lignin, and 13–20% other components [35–38]. The rice straw used in this study contained 39.20% cellulose, 19.02% lignin, 18.52% hemicellulose, 14.26% ash, and 9.18% other components. NaOH and H₂O₂ used in this work were supplied by Sigma-Aldrich Corporation.

2.2. CFs Extraction from Rice Straw. Alkaline treatment or mercerization is in the greatest popularity regarding chemical treatments of natural fibers to reinforce thermoplastics. In this study, CFs of rice straw were obtained using alkaline treatment method as follows. Rice straw was cut with dimension of about 2 mm using screen and then immersed in the NaOH 2 M solution with the rate of solid/liquid of 1/10 g/ml for 2 h at 90°C below stirring speed of 200 rpm. After reaction time, the resulting mixture was filtered and collected. The solid residue was washed with acetic acid to neutralize (pH 7–8) the remaining NaOH [39]. Many researches have reported that the alkaline treated fibers resulted in high physical and mechanical properties of composites, due to the removal of lignin, hemicellulose, and other compounds on the cellulose surface [20, 21]. However, obtained CFs showed yellow color (Figure 1(a)), which means that a part of the lignin and other compounds remained on the fiber surface. Therefore, the CFs in this study were also washed with H₂O₂ (Figure 1(b)) to remove them from the external surface of CFs, since they could limit the adhesion of CFs with the PP matrix [40, 41]. The CFs were finally dried in an oven for 24 h for any further use.

2.3. Composite Preparation. CFs with various concentrations (0, 10, 20, 30, 40, and 50 wt%) were blended with PP matrix in a plastic mixer (Haake Rheocord 9000, Germany) using a rotor speed of 60 rpm at 185°C for 8 min. Then, the obtained mixture was compression molded at 185°C for 15 min under 10 MPa. The samples were left at room temperature for 5 days before use.

2.4. Characterization

2.4.1. Scanning Electron Microscopy (SEM) Observation. The morphology evaluation of rice straw was analyzed using a JEOL 6490 (JEOL, Japan). The morphology evaluation of CFs which were extracted from rice straw using alkaline treatment method and the interfacial bond between CFs and PP matrix in prepared composites and the morphological evaluation of PP/CFs composites before and after biodegradation were performed using a Hitachi S-4800 scanning electron microscope (Hitachi, Japan).

2.4.2. Water Absorption Test. The water absorption tests of pure PP and various PP/CFs composites were carried out following ASTM D 570-99. Rectangular samples were cut with the dimension of 39 × 10 × 3 mm, dried at 105°C until the weight remained unchanged, cooled to room temperature in a desiccator using silica gel, and immediately weighed with an accuracy of 0.001 g. To investigate the water absorption of PP/CFs composites, the samples were immersed in the

FIGURE 1: CFs images of before (a) and after (b) being washed with H_2O_2 .

distilled water for 24 h at room temperature. Then, the samples were taken, with the excess water on their surface removed using a soft cloth, and weighed. The percentage of water absorption (W) of the samples was calculated using the following:

$$W \text{ (wt\%)} = \frac{W_2 - W_1}{W_1} \times 100. \quad (1)$$

W_1 is weight of the specimen before immersion.

W_2 is weight of the specimen after immersion.

2.4.3. Thermogravimetric Analysis (TGA). The thermal degradation behavior of the pure PP and various PP/CFs composites (CFs was washed with H_2O_2) was analyzed by TGA (SSC/5200 SII Seiko Instruments Inc.). TGA patterns were carried out from room temperature to 650°C at a heating rate of $10^\circ\text{C}\cdot\text{min}^{-1}$ in a nitrogen atmosphere with a flow rate about $250 \text{ ml}\cdot\text{min}^{-1}$.

2.4.4. Mechanical Test. The tensile and flexural tests of pure PP and PP/CFs composites (CFs was washed with H_2O_2) were carried out following ASTM D 638 and ASTM D 790 standards, respectively. For the tensile strength test, the specimens were cut with dimension of $165 \times 19 \times 3 \text{ mm}$, and crosshead speed was $2 \text{ mm}\cdot\text{min}^{-1}$. For the 4-point bending flexural strength test, the specimens were cut with dimension of $76 \times 25 \times 3 \text{ mm}$; crosshead motion rate was $2.8 \text{ mm}\cdot\text{min}^{-1}$.

2.4.5. Biodegradation Test. Throwing daily waste in the landfills is the most widely used method of waste disposal today. Landfills are commonly found in developing countries. Therefore, in this study, the rectangular specimens of pure PP and various PP/CFs composites (CFs was washed with H_2O_2) were cut with dimension of $50 \times 50 \times 3 \text{ mm}$ and then were buried in the soil for 50 days. The degraded samples were washed thoroughly with distilled water at room temperature and then dried at 105°C for 24 h. The change in shape of

specimens before and after being buried in soil was observed by SEM and weighed.

The percentage weight remaining of biodegraded specimens ($W_{\text{remaining}}$) was calculated using the specimen weights before (W_{before}) and after (W_{after}) biodegradation as the following:

$$W_{\text{remaining}} \text{ (wt\%)} = \frac{W_{\text{after}}}{W_{\text{before}}} \times 100. \quad (2)$$

3. Results and Discussion

3.1. The Morphology and Composition of CFs and PP/CFs Composites. The CFs which were treated by alkaline and then neutralized by acetic acid showed yellow color. However, after being washed with H_2O_2 , the CFs appeared white, indicating that lignin, hemicellulose, and other compounds which remained on the fiber surface were also removed. The SEM micrographs of alkaline untreated rice straw and H_2O_2 washed CFs and PP/CFs 80/20 wt% composite are shown in Figure 2. Rice straw possessed a block and impurities (Figures 2(a) and 2(b)) but obtained CFs after alkaline treatment and showed clean and rough cylindrical shape with average about $5 \mu\text{m}$ diameter (Figure 2(c)). The removal of lignin, hemicellulose, and wax from the outer cellulose is necessary to strengthen the interfacial bonding between CFs reinforcement and PP matrix [18, 20]. Figure 2(d) is SEM image of representative fractured surface of the PP/CFs 80/20 wt% composite and shows the presence of CFs in the composite.

3.2. Water Absorption. Increasing natural CFs content in the composite materials is desirable because it assists in reducing the cost, protecting the environment, and increasing the modulus of composite materials. The natural CFs are abundant in nature and stiffer than polymer matrix. However, they may not be suitable for several application fields because of their moisture absorption. Therefore, the water absorption is one of the important factors to evaluate properties of material application. To limit the ability of moisture absorption,

FIGURE 2: SEM images of (a) exterior surface of rice straw, (b) interior surface of rice straw, (c) CFs after being washed with H_2O_2 , and (d) representative fractured surface of PP/CFs 80/20 wt% composite.

the surface of natural CFs is modified by various methods including alkaline treatment method. When CFs are treated with alkaline, the hydrophilic $-\text{OH}$ groups in the cellulose structure are converted to hydrophobic $-\text{ONa}$ groups [42] as the following reaction:

The water absorption of PP/CFs composites with various ratios of matrix and reinforcement material after immersion in the distilled water for 24 h at room temperature is evaluated according to (1) and shown in Figure 3. The water absorption was increased with increasing fiber content in the composites. The number of $-\text{OH}$ groups in the cellulose structure, amount of lignin and other compounds, and NaOH remaining on fibers decide the amount of water absorption. The water absorption of the composites reinforced by 10 wt% CFs showed 0.69 wt% when CFs were not washed with H_2O_2 but decreased to 0.29 wt% when CFs were washed with H_2O_2 . The CFs content increased 20, 30, 40, and 50 wt%, the water absorption of the corresponding composites increased 2.16, 4.10, 5.63, and 6.98 wt% when not washed with H_2O_2 and 0.90, 1.72, 2.39, and 2.92 wt% when washed with H_2O_2 , respectively. These results demonstrated that the composites reinforced by CFs washed with H_2O_2 indicated higher water absorption ability than those not washed with H_2O_2 . The CFs content increased in the composite materials, which means the number of OH groups increased, leading to increasing the amount of water absorption [42]. Haque et al. reported that the amount of the water absorption of

FIGURE 3: The water absorption properties of pure PP and various PP/CFs composites with respect to CFs content and washed or not washed with H_2O_2 (average value of three tests).

PP/coir composites was dependent on the type of chemical treatment [42]. The composite materials with untreated coir reinforcement showed the highest water absorption ability, followed by neutral (pH 7), acidic (pH 3), and alkaline (pH 10.5) treated coir fiber reinforced composites, respectively. In

FIGURE 4: Representative TG and DTG curves of pure PP (a), PP/CFs 80/20 wt% (b), and PP/CFs 70/30 wt% (c) composites at heating rate of $10^{\circ}\text{C}/\text{min}$ under atmosphere at a flow rate of ca. $250\text{ ml}\cdot\text{min}^{-1}$.

this study, the CFs after alkaline treatment were washed with acetic acid to be neutralized and also washed with H_2O_2 ; therefore the remaining NaOH, lignin, hemicellulose, and other compounds were removed. In other words, the water absorption of PP/CFs composites can be controlled by the treatment methods or CFs content.

3.3. Thermal Properties. The thermal property investigation of the polymer composites is necessary to determine the influence of reinforcement materials into polymer matrixes on thermal stability of composites and to confirm any thermal pyrolysis process during composites production. The thermal stability behavior of PP/CFs composites was investigated using thermogravimetric analyzer under nitrogen atmosphere. The representative TGA curves of the pure PP and PP/CFs 80/20 and 70/30 wt% are shown in Figure 4. The pure PP showed a one-step process of decomposition, while PP/CFs composites clearly showed a two-step process. Decomposition of the CFs reinforcement and PP matrix occurred in the first and second stages, respectively. PP/CFs

composites showed intermediary thermal stability between PP matrix and CFs reinforcement [43].

Table 1 shows the weight loss corresponding to the decomposition temperature and decomposition temperature peaks of the pure PP and various PP/CFs composites. The pure PP showed one decomposition peak; the PP/CFs composites showed two decomposition peaks corresponding to the peaks of CFs and PP. The thermal stability of composites tends to decrease with increasing CFs content. However, 10 wt% weight loss temperature of all composites showed high temperature at over 300°C . Pure PP practically did not lose weight at 400°C ; however its weight loss occurred rapidly from 462°C , resulting in minimum residue. The saturated and unsaturated carbon atoms in PP were degraded at about 460°C , which was higher than that for CFs.

3.4. Mechanical Properties. Mechanical properties of the pure PP and various PP/CFs composites were investigated for tensile strength, Young's modulus, flexural properties, and elongation at break. Figures 4 and 5 and Table 2 showed

TABLE 1: Thermal properties of pure PP and various PP/CFs composites.

Sample	Weight loss temperature (°C)			Decomposition temperature peak (°C)	
	10 wt%	20 wt%	30 wt%		
Pure PP	420	437	445	-	462
PP/CFs 90/10 wt%	364	439	455	350	465
PP/CFs 80/20 wt%	348	438	453	348	467
PP/CFs 70/30 wt%	331	355	431	348	470
PP/CFs 60/40 wt%	320	332	418	347	471
PP/CFs 50/50 wt%	306	316	398	346	468

FIGURE 5: Effect of CFs content on the tensile properties of PP/CFs composites (average value of six tests).

FIGURE 6: Effect of CFs content on the flexural properties of PP/CFs composites (average value of six tests).

mechanical properties of pure PP and various PP/CFs composites which were averaging results of 6 specimens for each green composite. The tensile strength of PP/CFs composites was decreased, whereas their Young's modulus was increased with increasing CFs content, as expected (Figure 5). A similar behavior was reported for jute strands [44], wood floor and olive stone flour [45], *Thespesia lampas* fibers [46], and rice husk [47] for PP matrix composites. The tensile strength depends on the weakest part of the composite materials and further interfacial interaction between PP matrix and CFs is weak, leading to the decrease in the tensile strength of PP/CFs composites with increasing CFs content. However, the flexural properties of PP were improved by blending with CFs up to 50 wt% content (Figure 6). Improvement of the Young's modulus and flexural properties is expected to be due to the high stiffness of the CFs compared to the PP matrix. In addition, partially separated microspaces which were created during tensile loading, obstructed stress propagation between CFs and PP matrix, hence the obstruction degree increased with increasing CFs content, leading to the increase in the stiffness [38]. The elongation at break of pure PP showed 57.5%, but it was significantly decreased with the loading

level of CFs (Table 2). The poor elongation of the PP/CFs composites is probably due to the weak interactions between PP matrix and CFs, which generates stress concentration points and agglomeration. On the other hand, this result can prove that CFs improved the stiffness of composite materials.

3.5. Biodegradation. Plastics including PP which are synthesized from petroleum products are often burnt or buried after use. The burning of plastic wastes releases gases and chemicals into the air, leading to smog, acid rain, and toxic air pollution. Most of these plastic wastes show no degradability and accumulate in the environment when being buried, considerably increasing environmental pollution. In recent years, to reduce the burden on the environment, the composites between synthetic plastics composite with natural fibers have attracted much attention. There are many reports on these composite materials. However, most of them have reported their thermal and mechanical properties, without mentioning their biodegradability. In this study, we evaluate the biodegradability of composite of PP and CFs which were extracted from rice straw using alkaline treatment method and washed with H_2O_2 . The samples after being buried in the soil for a certain period of time were washed, dried,

TABLE 2: Mechanical properties of pure PP and various PP/CFs composites.

Sample	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)	Flexural strength (MPa)	Flexural modulus (GPa)
Pure PP	34.3 ± 1.2	638.5 ± 60.1	57.5 ± 5.6	42.4 ± 12.6	1.29 ± 0.22
PP/CFs 90/10 wt%	30.1 ± 3.0	686.4 ± 28.5	15.2 ± 1.2	44 ± 2.2	1.61 ± 0.11
PP/CFs 80/20 wt%	28.1 ± 2.1	731.3 ± 34.6	10.9 ± 0.9	46.6 ± 4.6	1.83 ± 0.16
PP/CFs 70/30 wt%	25.5 ± 5.2	767.6 ± 53.2	8.0 ± 2.1	48.5 ± 5.2	2.39 ± 0.21
PP/CFs 60/40 wt%	22.8 ± 1.7	803.2 ± 67.2	6.2 ± 0.8	49.8 ± 2.6	2.58 ± 0.26
PP/CFs 50/50 wt%	21.4 ± 2.6	824.2 ± 48.8	4.5 ± 1.1	49.6 ± 0.9	2.64 ± 0.19

FIGURE 7: Biodegradation behavior of pure PP and various PP/CFs composites with respect to CFs content (average value of three tests).

weighted, and evaluated for the biodegradability according to (2). Figure 7 shows the weight remaining percentage of the pure PP and PP/CFs composites with 90/10, 80/20, 70/30, 60/40, and 50/50 wt%. As expected, the weight of the pure PP was not changed after being buried in the soil for 50 days; in other words, pure PP was not degraded. However, the remaining weight of PP/CFs composites decreased with the burying time in the soil and their degradation rate increased with increasing CFs content. After 50 days being buried in the soil, the remaining weight percentage of PP/CFs composites with 90/10, 80/20, 70/30, 60/40, and 50/50 wt% was 96.98, 92.28, 88.82, 83.00, and 76.89 wt%, respectively.

The SEM observation provided further information on the morphology of the representative PP/CFs 80/20 wt% composite and the pure PP during biodegradation (Figure 8). Before being buried in the soil, both pure PP and composite displayed smooth surfaces (Figure 8(b)). After being buried in the soil for 50 days, the surface of the pure PP did not show deformation and remained as a flat surface without holes (Figure 8(a)), whereas that of the PP/CFs 80/20 wt% composite showed the existence of many holes (Figure 8(c)). Moreover, the fractured surface of the pure PP was not changed after being buried in soil for 50 days (Figures 8(d) and 8(e)), which means PP was not degraded. However, the various CFs in PP/CFs 80/20 wt% composite were observed

on the fractured surface before being buried in the soil (Figure 2(d)), but they were biodegraded with many holes formed after 50-day burial in soil (Figure 8(f)). According to this research, environmental degradation of the PP/CFs composites was affected by natural factors, including not only rainwater and underground water but also microbial activities.

4. Conclusions

Green composite materials from PP and various content of CFs extracted from rice straw were successfully prepared by a simple melt blending method. The water absorption of PP/CFs composites could be controlled by CFs content and treatment methods. The thermal stability of PP was decreased with the loading level of CFs, due to the low thermal properties of CFs, but their 10% weight loss temperature showed to be over 300°C. Young's modulus flexural properties of PP were improved by being blended with CFs; in other words, CFs improved the stiffness of composites. Particularly, PP showed no biodegradability, whereas PP/CFs composites showed that the opposite and their weight loss from 3.02 to 23.11 wt% depended on the CFs content after being buried in soil for 50 days. These results proved that the composites materials may be applied to various environmental fields. This

FIGURE 8: Representative biodegradation behavior in SEM images of the surface of pure PP for 50 days (a), PP/CFs 80/20 wt% composite for 0 days (b) and 50 days (c), the fractured surface of pure PP for 0 days (d) and 50 days (e), and PP/CFs 80/20 wt% composite for 50 days (f).

study is expected to contribute to environmental protection and solving the problem of wasting resources.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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