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Preparation and Characterization of Composites from Coconut Fiber and Biodegradable Polymers

Pensiri SRIBURI** and Toshio OGAWA*

Abstract

The composites were prepared by molding coconut fiber with either polylactic acid (PLA) or polybutylene succinate (PBS) in the cyclohexanone and characterized. Formulations of the composites (types of biodegradable polymer and ratios of fiber to polymer) were optimized. Physical and mechanical properties, including water content, microscopic examinations by CCD and SEM, apparent density, and mechanical properties of coconut fiber, polymers and composites were investigated. The incorporation of the PLA and PBS polymers to coconut fiber significantly improved the physical and mechanical properties of the composites. Both PLA and PBS polymers produced composites with similar appearances. Increasing polymer contents essentially increased the apparent density, flexural strength and modulus of elasticity, and all samples exhibited enhanced adhesive properties. However, increasing more polymer contents could adversely affect the composite preparation, particularly the difficulty of mixing and homogeneity. Generally, composites made from PLA had almost the same flexural strength at break and modulus of elasticity when compared to those of PBS composites at certain polymer concentrations. Nevertheless, thin film layers of some PBS composites may clearly be appeared if higher polymer contents were used, as examined by CCD. The composite with approximately 50% PLA and the coconut fiber of 50% has been satisfied our requirements of physical characteristics and mechanical properties.

1. INTRODUCTION

Due to environmental impact of persistent non-degradable materials is growing global concern, biodegradable composites are one of the key factors to become an acceptable alternative. However, the suitability of material properties for composites preparation, the technical feasibility of processing options; the versatility of applications; and ultimately, commercial viability of production and processing are also considered to be essential. More generally, it seems accepted that ap-

plications of different materials developed will be suitable for certain areas, but that general applications remain limited.^{1,2)}

Composites represent an essential class of engineering materials and have used in a wide variety of applications. Many materials are effectively composites. This is particularly true of natural biological materials, which are often made up of at least two constituents. In many cases, a strong and stiff component is present, often in elongated form, embedded in a softer constituent forming the matrix. Composite materials typically consist of two basic elements: reinforcing fiber and binder or matrix.^{3~6)}

Coconut fiber has widely been known as a biomaterially agricultural resource, especially in tropical countries. It is primarily lignocellulose-based. The use of coconut fiber in the preparation of agro-material filled biodegradable polymer composites is

* Laboratory for Ecological Polymer Chemistry
Kanazawa Institute of Technology
7-1 Ohgigaoka, Nonoichi, Ishikawa 921-8501, Japan

** Department of Chemistry
Faculty of Science
Chiang Mai University, Chiang Mai 50200, Thailand
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attracting much attention because of its potential supply as a biomass material.^{7,8)} The use of natural fibers such as coconut fiber in plastic composites is increasing. It has been reported since 2001 that the luxury car manufacturer is using coconut fibers combined with natural latex rubber (caoutchouc) from the Amazon rain forest and molded into headrests, sun visors, back cushions and seat parts for small Mercedes-Benz sedans, such as the A-class as well as for trucks sold in Brazil.^{9,10)} It was also mentioned that the coconut fiber products meet the same quality standards that DaimlerChrysler has set for all of its automotive products. More recently, Dan Palil, a scientist from Malaysia presented a cycling crash helmet made with coconut fibers which adhere to government safety standards at the 33rd International Exhibition of Inventions in Geneva, Switzerland in 2005.¹¹⁾

Fully biodegradable synthetic polymers such as polylactic acid (PLA), polybutylene succinate (PBS) are commercially available since 1990 with many interesting properties, such as biodegradability, melt processing and chemical resistance. However, both are considerably more expensive than conventional plastics.^{10~13)} It is therefore necessary to find out the possible way of its application, for example, in combination with other agricultural less-valued materials or by-products to produce composites. This should encourage society particularly in terms of environmental protection. Recent reports from Kyodo News International, Tokyo on 23 February 2005 and The Japan Times on 3 March 2005 have addressed that an increasing number of Japanese corporations have begun producing biodegradable materials less harmful to the environment than conventional ones, reflecting rising environmental consciousness.^{16~17)}

The specific objective of this research is to produce PLA-based and PBS-based composites that are truly reinforced from coconut fiber by-products as well as biodegradable. They may also be used in the same application as existing polymer composites, thereby enhancing mechanical properties through the composites. The question of how best to prepare the coconut fiber-PLA and coconut fiber-PBS composites is of our particular interest. Therefore, we prepared these composites and characterized the physical and mechanical properties. These results obtained were also compared for the feasibility of further applications and addressed the

limitations.

2. EXPERIMENTAL

2.1 Materials

Fibers from coconut (*Cocos nucifera* Linn.) were originally obtained from Thai Coconut Fibre Industries, Thailand. Pellets of polylactic acid (PLA, Lacea H-100) and polybutylene succinate (PBS, Grade 1001) were respectively supplied by Mitsui Chemicals and Showa Highpolymer, Japan, and both pellets had approximately the same density of 1.26 g/cm³.^{18,19)} Data for water content, melting temperature and some mechanical properties of materials were shown in Table 1. Cyclohexanone (C₆H₁₀O) was of analytical grade and purchased from Wako Pure Chemical Industries, Japan. Teflon sheet with the thickness of 0.05 mm was obtained from Nitto Denko Corporation, Japan.

Table 1 Water content, melting temperature (T_m), tensile strength, modulus of elasticity and elongation of coconut fiber and polymers.

sample	water content (% w/w)	T_m (°C)	tensile strength (MPa)*	modulus of elasticity (MPa)*	elongation (%)*
coconut fiber	7.73	-	53	132	1439
PLA pellet	0.30	164	59	1701	285
PBS pellet	0.39	111	38	388	1875

* Measurements taken at the average diameter of coconut fiber and average thicknesses of PLA and PBS sheets at 0.47 ± 0.10 , 0.080 ± 0.010 and 0.096 ± 0.011 mm, respectively

2.2 Preparation of composites

The composites from the fiber and polymers were prepared by hot-dipping in the solvent with various ratios. For coconut fiber-PLA composite, in a 600 mL beaker, 100 mL of cyclohexanone was added and heated at over 100°C under stirring for about 15 min and PLA pellets were added. Composites were prepared by six different PLA contents of 22.2, 26.3, 30.0, 34.9, 37.8 and 48.8%. Similarly, composites from coconut fiber and PBS were prepared in the same way as those described for PLA-composites and the PBS contents were expressed as following: 22.2, 26.3, 36.4, 41.7, 44.7 and 48.8%. The mixture was continuously heated and stirred until complete dissolution of the polymer was obtained. Coconut fibers were hot-dipped and deeply soaked, depending on the ratio of fiber to polymer. The samples were then left in the draft chamber to remove the remained solvent and until eventually dried.

The dried samples were finally molded by hot-pressing (Table Type Test Press, Tester Sangyo, Japan). The conditions for molding were as followed: temperature of 190°C and pressure of approximately 13.3-18.4 MPa and for 30 min using the 3-mm thickness tailor-made steel plate and under Teflon sheets. Composites were prepared at least two replicates. The test specimens were stored in sealed zipped polyethylene bags to avoid moisture uptake. Coping saw frame (No. 01105, Mitsutomo Manufacturing Co., Ltd, Japan) was manually used to cut the test pieces for mechanical testing.

2.3 Tests for physical and mechanical properties

We investigated physical properties in terms of water content, thermal properties by differential scanning calorimetry (DSC), microscopic appearances both by camera with charge-coupled device (CCD) and by scanning electron microscope (SEM) and mechanical properties such as tensile and flexural strengths, and modulus of elasticity in order to predict which composite would essentially exhibit the most effective mechanical properties and other qualities. Water content was analyzed by drying the samples in drying oven (Yamato DX 41, Japan) at 103°C until constant weight was obtained and at least three replicates were done. The thermal properties were measured by Rigaku TAS 100 in nitrogen atmosphere. A sample of 7 ± 0.1 mg was heated from room temperature to 200°C. Heating rate was 10°C/min. At least five replicates were run under certain DSC condition. DSC was mainly performed at raw materials included coconut fiber, PLA and PBS pellets. Microscopic characteristics were investigated either by CCD using Shimadzu CCD-f2 Micro CCD Scope and Digital Image File DF-20, Japan or by SEM using Topcon SM-200, Japan and at least two replicates were carried out. XPS (ESCA-850, Shimadzu, Japan) was used to characterize the surface of coconut fiber. Acceleration voltage was 8kV and current was 30 mA, and X-ray of 1253.6 eV was generated using magnesium as an anticathode, and spectra were measured under the vacuum of over 10^{-5} Pa. Determination of apparent density of the composites was basically performed at 24°C and 65% RH by weighing the sample in air and divided by the volume of it. For mechanical properties, we used the universal testing machine (Shimadzu, Autograph AGS-1000B, Japan). The tensile properties were conducted according to JIS K7113

(1981) and the flexural properties were carried out using three point bending according to JIS K 7203 (1982) to characterize the composite materials and to make comparisons. Test results were obtained with at least 7 test pieces and reported.

3. RESULTS AND DISCUSSION

3.1 Characteristics of coconut fiber and polymers

An ever-increasing variety of materials is now available, each having its own characteristics, applications, advantages, and limitations.^{2,3)} Fundamental properties of materials are therefore important in design and manufacture of composites. Material cost will also be important. Coconut fiber was an alternative good choice for this study. It is a low cost material with low density, renewable resource attributes, non-abrasive properties, excellent specific mechanical properties, and is potentially outstanding reinforcing fillers in thermoplastic composites.¹⁾ However, one of the main disadvantages of using lignocellulosic materials as fillers is their high water absorption, which originates from their inherent numerous hydroxyl groups. Moisture penetration into biocomposites occurs via the direct diffusion of water molecules into the filler and matrix polymer interface.⁷⁾

Factors that must be considered in selecting a material for a given part/product are properties required, previous experience, availability, cost, environment and processing method. Since material characteristics influence the design and manufacture of products, a material will be selected for its properties.^{4,5,20)} However, when selecting materials for products, generally we first consider their mechanical properties. The mechanical properties specified for a product, and its components, should be appropriate to the conditions under which the product is expected to function. Data from Table 1 has clearly shown that coconut fiber with water content of 7.73% exhibited tensile strength almost the same as of PLA polymer but rather higher than the PBS one. Modulus of elasticity and elongation percentage of coconut fiber were also determined and the results were of between 103-166 MPa (average of 132) and 730-2500% (average of 1439), depending on the diameter of coconut fiber, respectively. The diameters of coconut fibers characterized by CCD were found in a range of 0.3-0.7 mm (average of 0.5 mm) (Fig. 1 (a)), which appeared to be almost invariably high. The

Fig. 1 Micrographs of coconut fiber taken from (a) CCD at magnification of 50, a diameter of the fiber (distance between x and x) was 0.513 mm and (b) SEM of cross section of coconut fiber; scale bar represents 50 μ m.

diameter value of fibers might lie in collective properties which develop when they are in composites with a variety of polymers. Scanning electron micrographic examination of coconut fiber (Fig. 1 (b)) obviously demonstrated that it consisted of numerous microfibrils which would be beneficial for reinforcing the composites. Measurement of thermal properties such as melting temperature of raw materials was shown also in Table 1. They can be used to point out the performance of the polymer under different environmental conditions.^{12, 13, 21)} For the biodegradable polymers (Table 1), it was found that not only the tensile strength of PLA was higher than the PBS but also melting temperature was.

Results from XPS have demonstrated the surface analysis of coconut fiber to consist atomic concentration of C : N : O of 73.07 : 2.71 : 24.22 atom %, respectively. This was corresponded to a report from a group of scientists who have developed lignocellulosic-based composites from kenaf core. According to the elemental analysis, it was found that this core consisted 0.30% of nitrogen and 1.90% of protein (as anhydrous basis).²²⁾

3.2 Preparation of composites

Initially, composites were prepared by lamination PLA sheet with the fiber (without any solvent) with various means (ratio of fiber ratio to PLA, number of layered laminate and pressing conditions). Also, dipping coconut fiber in PLA mixture of available solvents was carried out. However, the composites were unfavorably poor. Therefore more attempts have been put for other possible methods, particularly by trying to dissolve PLA in suitable solvents such

Fig. 2 Water content of composites; coconut fiber-PLA (■) and coconut fiber-PBS (□), as a function of polymer content.

as chloroform, cyclohexanone and so on, and later molding as the same method of laminates. Hot-dipping of fiber into polymer-cyclohexanone mixture was finally decided to use for composite preparation throughout this study. Two factors for formulating the composites were considered; two types of biodegradable polymer (PLA and PBS) and six different polymer contents.

3.3 Characteristics of composites

3.3.1 Water content of composites

The amounts of water (Fig. 2) in either PLA or PBS composites were surprisingly very little (less than 2% wet weight) and they did not vary differently from each other. No changes were found for all of them as a function of polymer concentrations. This may exhibit that the polymer contents seemed unlikely to affect the water content of final products. However, some of the water could be trapped in the porous structure. Further research with more sensitive instrumentation is required.

Fig. 3 CCD micrographs of coconut fibers-PLA composites at magnification of 50; (I) top-view; (a)-(f) were of 22.2, 26.3, 30.0, 34.9, 37.8 and 48.8%, and (II) cross-sectional view; (g)-(l) were of 22.2, 26.3, 30.0, 34.9, 37.8 and 48.8% of polymer content in composites.

3.3.2 CCD and SEM of composites

Figs. 3 and 4 showed CCD micrographs both top- and cross-sectional views of all composites at different polymer concentrations. It can be seen that coconut fiber and polymer could be adhered considerably well. From this result, it could be expected that no significant changes were found for all of them as a function of polymer concentrations. However, looking at the top-view of the PBS composites at higher concentration

(>40%), it can be markedly seen the thin film covered at the top layer of the composites (Fig. 4(I) (d)-(f)). This probably resulted from the accumulation of excessive PBS content while preparation due to the hydrophobic nature of PBS.^{7,8)} Additionally, this distinct appearance might stem from the molding temperature because both PLA- and PBS-composites were molded at the same temperature of 190°C. This temperature appeared to be rather higher than the temperature of

Fig. 4 CCD micrographs of coconut fibers-PBS composites at magnification of 50: (I) top-view; (a)-(f) were of 22.2, 26.3, 36.4, 41.7, 44.7 and 48.8%, and (II) cross-sectional view; (g)-(l) were of 22.2, 26.3, 36.4, 41.7, 44.7 and 48.8% of polymer content in composites.

melting of PBS (111°C). The film would also increase more thickness of the composites and may lead to the difficulties for further application such as coating or bonding with other additives. This may exhibit significant difference of PBS composite when compared to the PLA ones which did not exhibit this appearance.

SEM micrographs of PLA and PBS composites taken from 26.3 and 48.8% of polymer content were presented in Figs. 5 and 6, respectively. The results

clearly showed that the polymers have been better distributed into the microfibrils of coconut fibers. They also indicated well adhesion between the fiber and polymer. However, clear distribution was better seen mostly in the coconut fiber-PLA composites.

Micrographs either from CCD and SEM could substantially confirm the presence of void or air pockets structure of these composites which is likely similar to the cellular-type plastics. It is widely known that voids

Fig. 5 SEM micrographs of coconut fiber-PLA composites, bar represents 100 μm ;
(I) top-view; (a) 26.3% and (b) 48.8%, and (II) cross-sectional view;
(c) 26.3% and (d) 48.8% of polymer content in composites.

are in general harmful to the resulting composite.⁴⁾ This is because portions of a fiber passing through the void are not supported by surrounding PLA or PBS which may influence lower strength or modulus of elasticity. The fiber-polymer matrix adhesive strength in specific region, i.e. void region, is broken, the fiber would separate from the matrix and leaves discontinuities that may cause failure.

3.3.3 Apparent density of composites

One of significant considerations of composite applications is the density.³⁾ This value would involve in the voids present in the composite materials which can variably be found in all forms of composites. These voids can commonly form during the composite manufacturing.

The density of tested specimen in this study was practically evaluated by weighing procedure. All PLA and PBS composites at 22.2-48.8% of polymer contents

tended to have low apparent density with the average value between 0.38-0.62 g/cm³ at 24°C and 65% RH. This was illustrated in Fig. 7. The highest densities were found at 20% polymer concentration at both composites. Alternatively, the lowest values appeared at the lowest amount of used polymers. This could be implied that void contents were much exhibited in composites with the lower polymer to fiber ratio. It was, additionally, shown that increasing polymer contents clearly increased the apparent density of composites.

It is necessary to consider the relationship between apparent density and mechanical properties. The apparent densities of the composites were then plotted against the flexural strength at break and modulus of elasticity in Figs. 8(a) and 8(b), respectively. It was obviously shown that the linear relationships were seen for all parameters. Therefore, it can be concluded that the more apparent density of the composites was, the

Fig. 6 SEM micrographs of coconut fiber-PBS composites, bar represents $100\ \mu\text{m}$; (I) top-view; (a) 26.3% and (b) 48.8%, and (II) cross-sectional view; (c) 26.3% and (d) 48.8% of polymer content in composites.

Fig. 7 Apparent density of composites; coconut fiber-PLA (■) and coconut fiber-PBS (□), as a function of polymer content.

higher the mechanical properties of the composites were.

3.3.4 Mechanical properties of composites

More importantly, to consider for selecting a composite, mechanical properties are one of significant

factors.³⁾ Therefore, flexural strength at break and modulus of elasticity of all composites as a function of polymer contents were investigated and the results were found to range between 1.38-2.86 and 76-162 MPa, respectively (Figs. 9(a) and 9(b)). Mostly, the values did not markedly high when compared to other non-biodegradable plastic based composites.⁶⁾ These results were also reflected in slightly varied in flexural strength at break and elasticity of samples having polymer contents less than 48.8%. This usually seemed to happen when biological materials such as coconut fiber are produced.³⁾ Nevertheless, these fiber composites offer the attractive combination of low cost and easy processing.

In general, when considered at specified polymer concentrations, it was found that flexural strength at break (Fig. 9(a)) and modulus of elasticity (Fig. 9(b)) of PLA and PBS composite were not much different. A

(a): flexural strength at break

(b): modulus of elasticity

Fig. 8 Relationship between mechanical properties and apparent density of composites; coconut fiber-PLA (■) and coconut fiber-PBS (□) and apparent density of the composites.

decrease in mechanical properties was markedly found as a decreased polymer contents for both composite types. Alternatively, it can be seen that increasing polymer contents essentially increased both mechanical properties. However, incorporating more polymer contents (more than 20%) may cause difficulties in composite preparation, especially mixing and fiber dipping.

By basic material combination, the composites in this study can be classified as fiber composite which mainly composed of coconut fiber and either PLA or PBS biodegradable polymer. Therefore, the success of the interaction between the composite constituents practically depends to a significant degree on the polymer. It is obvious that the intrinsic properties of the materials of which the constituents are composed are of critical importance. A new set of combined properties, particularly the mechanical properties, derives from those of the individual materials.

The adhesion properties of the added polymers and its ability to transfer stresses are of course of paramount importance. In many fiber composites, uniformity in orientation of the fibers, control of matrix

(a): flexural strength at break

(b): modulus of elasticity

Fig. 9 Relationship between mechanical properties of composites; coconut fiber-PLA (■) and coconut fiber-PBS (□) and polymer content in the composites.

content, quality of the interfacial bonds and many other processing variables influence the composite's performance directly.⁴⁾

Some representative mechanical properties were shown in Table 2 for coconut fiber and a few representative composites (48.8% of polymer content) for comparison. Inspection of these data shows that some modulus of elasticity was attracting. Consequently, we conclude that the composite prepared from formulation containing 51.2% (approximately of 50%) coconut fiber and 48.8% (approximately of 50%) PLA has met our requirements, especially in terms of physical characteristics and mechanical properties.

It has been widely accepted that composite materials have numerous characteristics which differ from the conventional engineering materials and some characteristics are totally new and require new analytical and experimental procedures. Also, most composite materials are often heterogeneous and more complex.⁴⁾

The mechanical properties measured with the composites may not always directly reflect the strength of the composites; however they provide information and

Table 2 Summary of mechanical properties of coconut fibers and composites taken from 48.8% polymer content in composites.

sample	tensile properties			
	tensile strength (MPa)		modulus of elasticity (MPa)	
	range	average	range	average
coconut fiber	44-61	53.4	103-166	132
coconut fiber-PLA	6.5-12.1	9.5	93-128	112
coconut fiber-PBS	6.9-15.2	11.0	67-97	81
	flexural properties			
	flexural strength at break (MPa)		modulus of elasticity (MPa)	
coconut fiber-PLA	1.5-3.1	2.3	123-202	162
coconut fiber-PBS	2.2-3.5	2.9	88-166	127

guidelines for preliminary part design and material selection. A study of these mechanical properties may also help one to understand the effects of various material variables in the composite formulation.⁶⁾

Tensile and flexural strengths are important for the materials that are going to be stretched and bend, respectively. Typically, fibers need good tensile strength and they have been added to polymer to improve strength, but no one knew exactly how they worked. In general, flexural properties follow the same trend as the tensile properties. They are affected by the fiber content, length, type, and orientation in the same way as tensile properties are affected by these parameters. Tensile strength of composites increases significantly with increasing fiber content; however, the tensile modulus is affected only moderately. In general, flexural strengths are higher than the tensile strengths, but the flexural modulus values are similar to the tensile modulus values.⁶⁾

Flexural properties of composites in this study were determined by three-point flexural tests of rectangular specimens. According to the linear relationship between the mechanical properties and the polymer content as illustrated in Fig. 9, it could therefore be predicted that the higher flexural strength and modulus in the composites should also indicate the better mechanical properties and physical characteristics of the composites.

4. CONCLUSIONS

The addition of the PLA and PBS polymers to coconut fiber significantly improved the performance of the composites and offered a renewable resource and biodegradable alternative for composite applications

(Table 2). More importantly, strong relationship among types of biodegradable polymer, ratios of fiber to polymer, and mechanical properties which influenced the adhesive characteristics was found. Although both composites seemed likely to have almost the same values of flexural strength at break and modulus of elasticity at specified amounts of polymer, the advantage of good appearance of PLA composite had drawn conclusions that composites prepared by coconut fiber and PLA polymer appeared to be better than those from PBS.

The composite with approximately 50% PLA and the amount of coconut fiber of 50% has preferentially been targeted for commercial use. These also confirmed by micrographs from CCD and SEM. The flexural strength and modulus of elasticity of this composite were approximately 2.3 and 162 MPa, respectively. Limitations regarding the preparation of hot-dipping may be of critical importance.

The results obtained from this research will additionally enhance the utilization of coconut fiber in industrial materials. Research is being undertaken to use other agricultural natural by-products and on other biodegradable polymers in manufacturing biodegradable composite materials as well as the biodegradability of both composites of coconut fiber-PLA and coconut fiber-PBS.

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<論文>

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ヤシの繊維と生分解性樹脂から成る複合材料 の調製とキャラクターゼーション

Pensiri SRIBURI*・小川 俊夫**

要 旨

ヤシの繊維とポリ乳酸 (PLA) あるいはポリブチレンサクシネート (PBS) からシクロヘキサンを溶媒として複合材料を作成し、キャラクターゼーションを行った。ヤシの繊維量と樹脂の種類の比を変えて複合材料としての最適化を検討した。具体的には、水分量、顕微鏡観察、見掛け密度、樹脂および成形物の物理的および機械的性質について調べた。その結果、PLA および PBS のヤシの繊維への含浸状態が複合材料の性質に著しく影響を与えた。両樹脂を含浸した複合材料はほぼ似たような性質を示した。すなわち、樹脂含量の増加とともに、見かけ密度、曲げ強度、および弾性率は増加し、内部の接着力も向上した。しかし、あまり樹脂量を増やすと、複合材料の均一性が損なわれてくる結果となった。一般に、PLA からの複合材料と PBS からのそれと曲げ強度や弾性率は同じような値を示した。ただし、PBS が高濃度の場合、あまり良い性質が得られなかった。結局、ヤシの繊維約 50% と PLA 約 50% から成形した場合、複合材料として最も好ましい物理的、機械的性質を示した。

* チェンマイ大学, 科学部, 化学科

タイ国, チェンマイ市 50200

** 金沢工業大学, 環境高分子化学教室

石川県石川郡野々市町扇が丘 7-1 〒921-8501

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