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Effect of wood flour on structural and thermal properties and antibacterial activity of PLA filled with triclosan

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ABSTRACT

Poly(lactic acid) (PLA) were compounded with wood flour and triclosan. The wood/PLA composites were then characterized using differential scanning calorimeter (DSC) and the antibacterial activity was also assessed via plate count agar (PCA) under a wide range of contact times using *Escherichia coli (E.coli*) as the testing bacteria. The characterization results suggested that the addition of wood flour produced two melting peaks behaviour which resulted in an enlargement of degree of crystallinity (X_c) in the wood/PLA composites. These two metling peaks were probably related with re-crystallization of the PLA by the presence of wood. The antibacterial efficiency of triclosan/PLA was improved considerably by the presence of wood flour, suggesting that the wood particles could act as "anti-bacterial promoter" for the PLA.

Introduction

A biodegradable aliphatic polyester Poly(lactic acid) (PLA) can be obtained from rural plants, such as, sugarcane and corn starch. In food packaging applications, PLA products must be safe from foodbrone pathogen which is usually carried out by incorporating or blending with antimicrobial agents with PLA. Wood flour (WF) is one of the most widespread natural fibers for making wood polymer composites (WPC) because of cost saving, good mechanical properties, environmental concern and their suitabilities for both indoor and outdoor applications. However, recent literature surveys has indicated that the information and experimental data, regarding mechanical and thermal properties of wood/PLA composites are still rare, likely due to difficulties in processing which are related to fiber-polymer incompatibilities and low melt strength of PLA [1-2]. Based on available published research, the thermal behaviour and antibacterial evaluations for wood/PLA composites are required in order to understand and determine the processing windows, and possibilities in prediction of the final properties for wood/PLA composite products. The main advantages for the wood/PLA composites include not only the environment friendly materials, but also abilities to biodegrade after their service life. It was presumed that addition of hydrophilic wood particles should accelerate the hydrolysis reaction of PLA after service use. Therefore, the aim of this work was to study the effect of wood on the structural and thermal properties, and antibacterial performance of PLA. The wood/PLA composites were thermally characterized using differential scanning calorimeter (DSC) and the results were used to relate with their antibacterial performances.

Experimental

Materials and chemicals

Poly(lactic acid) (PLA, 2002D, by NatureWorks, USA) was used. Triclosan (2,4,4'-trichloro-2'-hydroxydiphenylether) was used as an antibacterial agent (Koventure Co. Ltd., Thailand). Wood flour with an average particle size of 100–300 µm was supplied by V.P. Wood Co., Ltd. (Bangkok, Thailand) and the surface treatment of the wood flour was carriedout using N-2(aminoethyl)-3-

aminopropyltrimethoxysilane (KBM 603, Shin-Etsu Chemical Co. Ltd., Japan), the experimental procedure being given elsewhere [3]. Finally, Gram-negative *Escherichia coli (E. coli,* ATCC 25922) was used as the testing bacteria.

Specimen preparation

PLA was dried 70 ^{o}C overnight in an oven before blending with triclosan and KBM603-treated wood flour by high-speed mixer. Then, melt-bending using a twin screw extruder (Polylab-Rheomex CTW 100P, Haake, Germany) to produce pellets of triclosan/PLA blends and triclosan/wood/PLA composites was performed. The pellets were compression-molded afterwards to produce flat sheets with 1mm and 3 mm thick for antibacterial and thermal property testing, respectively

Antibacterial activity evaluations

Antibacterial activity of PLA was performed using Plate count agar (PCA) method following ASTM E2149 (2001). Overnight inoculums of *E. coli* in5 ml liquid nutrient broth (NB) was diluted to Optical density values (OD) of 0.1with growing media peptone solution. Flasks filled with OD of 0.1 peptone solution were put in with the two pieces of 2.5 x 5 cm^2 film specimens. The flasks were then shaken using a reciprocal shaker at the shaking rate of 100 rpm at 37 ^{o}C ± 0.5 ^{o}C for 60, 120, 180 and 240 min contact times, respectively. 100 μL of the bacteria solution was dropped on agar in sterilized Petri dishes for every contact time. The percent reduction in term of of bacteria colony-forming-units (CFU) was calculated using Eq. 1:

$$\%R = \frac{A - B}{A} \times 100 \quad (1)$$

where: *R* is the decreasing of bacteria (%), *A* is the average amount of bacterial colonies from composites without triclosan for a given contact time (CFU/ml), and *B* is the average amount of bacterial colonies from composites with triclosan for a given contact time (CFU/ml)

Characterizations

Differential scanning calorimetry (DSC**;** DSC822, Mettler-Toledo, USA) was used to observe the physical and thermal properties of PLA, triclosan, wood flour and PLA composites with triclosan and wood. The interested properties include the glass transition temperature (T_g), melting temperature (T_m) and percentage of crystallinity (X_c).

Result and Discussion

Thermal properties

DSC curves of PLA, 5 and 10 %wood /PLA composites with and without 1.5% triclosan are given in Fig. 1. The T_g and T_m of PLA were around 60–62 ^{o}C and 152–153 ^{o}C, respectively. The T_g values of wood/PLA and /triclosan/wood/PLA composite specimens had lower T_g than those of neat PLA. This suggests that the triclosan and wood fibers softened the PLA in molecular scale, which may relate to more hydrophilicities of wood fibers and triclosan in comparison with PLA. The T_m peak characteristics were also affected by the presence of wood fibers. The Tm value for neat PLA was around 153^{o}C whereas the T_m values for the wood/PLA composite displayed a double melting peaks, the first melting peak being around 151-152 ^{o}C and the second melting peak exhibiting around 156–159 ^{o}C. The first peak represented the PLA whereas the second was thought to be associated with the crystallization behavior of PLA and wood fibers. In other words, these two melting peaks probably involved with the recrystallization of PLA occurring due to the presence of wood fibers. Shi et al. [4] found the recrystallization behaviour of PLA due to highly hydrophilic fiber. This recrystallization behaviour has occurred due to PLA imperfect crystals as a result of wood fibers during first processing (before being tested in DSC). These double melting peaks were also responsible for the increases in crystallinity levels of PLA from 7.9 to 36.4% as also shown in Fig. 1. However, it was found that the crystallinity level of PLA decreased slightly with triclosan, suggesting a molecular interaction between PLA and triclosan which will be discussed later. Another interesting point was that, on DSC heating step, the exothermic peaks have appeared at 90–135 ^{o}C, only for the PLA samples with wood fibers. This confirmed the recrystallization process of the PLA with the presence of wood particles.

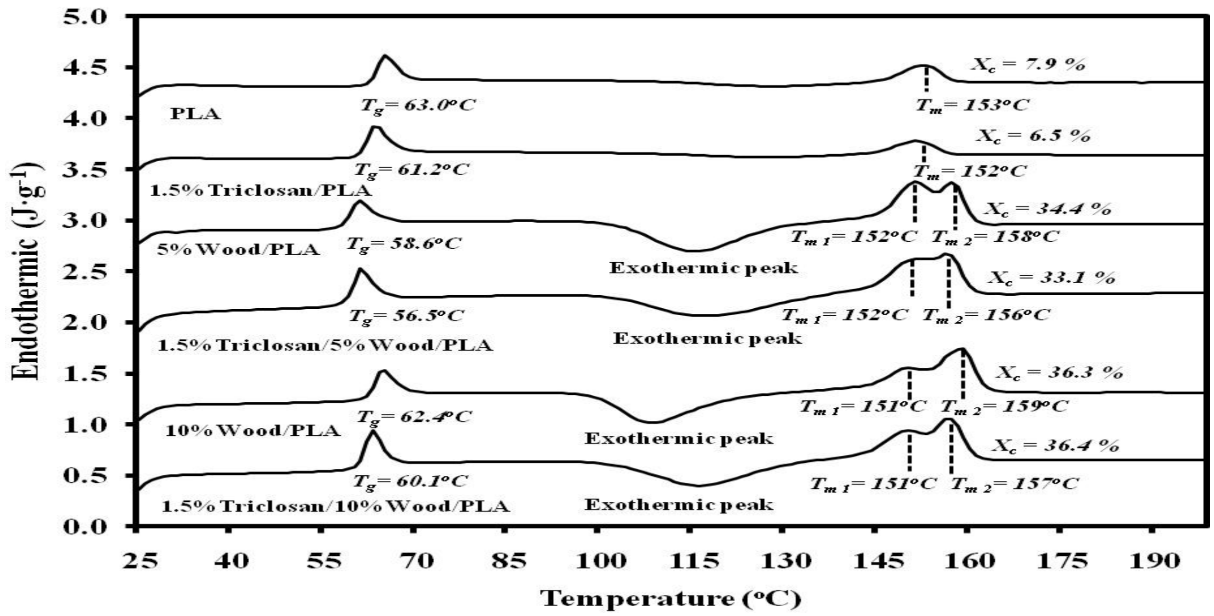

Figure 1. DSC curves for PLA, wood flour/PLA, triclosan/PLA and triclosan/wood flour/PLA

Table 1. Percent bacteria reductions for PLA and wood flour/PLA composites with triclosan loadings of 0.5–1.5%wt for different contact times

Materials	Contact time (min.)	Percent bacteria reduction (%)		
		0.5% Tricolsan	1.0% Tricolsan	1.5% Tricolsan
PLA	60	14.86	9.46	17.57
	120	53.16	63.16	65.26
	180	62.95	63.86	74.11
	240	74.92	78.81	83.40
5%WF/PLA	60	10.77	24.62	32.31
	120	49.58	77.73	87.39
	180	45.74	81.91	83.69
	240	54.95	88.16	91.65
10%WF/PLA	60	21.67	48.33	40.00
	120	63.27	74.83	86.39
	180	64.20	82.72	96.71
	240	63.68	88.05	96.78

Antibacterial performances

The percent bacteria reductions of PLA and wood/PLA composites are given in Table 1. It was observed that, for all tested specimens and tested conditions, when triclosan contents and contact time were increased the percent bacteria reductions also increased. This suggested that triclosan had the capacity to kill or retard the bacteria growth. Previous work [5] has shown that triclosan became molten at normal processing temperatures (above 150^{o}C) and could diffuse through polymeric materials, like polyethylene, polypropylene, polystyrene and poly(vinyl chloride), to kill the *E.coli*.

The results in Table 1 also show that the antibacterial properties of the PLA enhanced when the wood content was increased to 5 and 10%wt. This suggested that the wood fibers promoted the bacteria killing efficiencies for the PLA. It was thought that when adding the wood fibers into the triclosan/PLA the highly hydrophilic wood particles [6-7] would interact with PLA and leave the triclosan to be free, and also absorb more water molecules into the PLA composites. These two concurrent phenomena have promoted the migration of the triclosan molecules to the PLA sample surfaces and thus, increased the bacteria killing efficiency. Therefore, it was concluded that the wood fibers could act as antibacterial promoter for the triclosan/PLA blend. The interactive model for promoting the antibacterial character for triclosan/PLA by the wood fibers is given in Fig. 2.

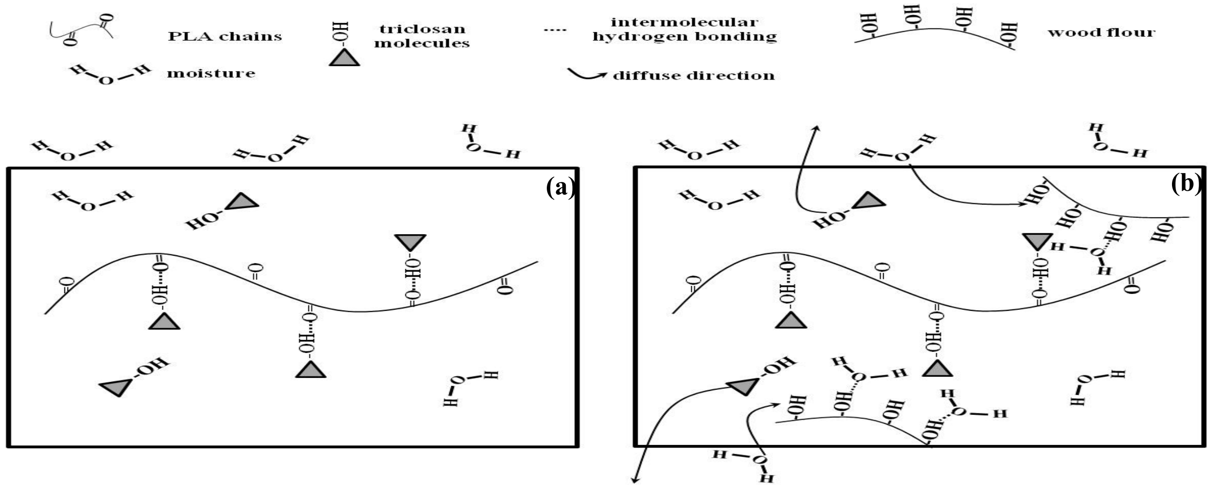

Figure 2. A possible model of moisture regain by wood in triclosan/PLAs:(a) no wood; (b) with wood.

Conclusion

In this work, triclosan and wood were incorporated with PLA, and the thermal and antibacterial performances were examined and discussed. Triclosan was found to slightly lower the T_g of PLA and the wood fibers resulted in recrystallization of the PLA, exhibiting two melting peak characteristics. The wood fibers could enhance the bacteria killing efficiencies for the PLA by interacting with PLA and absorbing more water molecules into the PLA composites to promote the migration of the triclosan molecules to the PLA sample surfaces.

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