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# WOOD FIBER REINFORCED POLY(LACTIC ACID) COMPOSITES

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## Abstract

Natural fiber-reinforced composites are increasingly being used in applications in the automotive, furniture, or building industry. The processing and physical properties of these composite materials are the very important parameters in respect to the design layout and product guaranty. This paper presents the results of the study of processing and physical properties of environmentally friendly wood fiber reinforced poly(lactic acid) (PLA) composites that were produced by a micro-compounding molding system. Wood fiber-reinforced polypropylene (PP) composites were also processed and compared to PLA/wood fiber composites. The mechanical, thermal-mechanical and morphological properties of these composites have been studied. PLA/wood fiber composites have mechanical properties of sufficient magnitude to compare with conventional thermoplastic composites. The tensile and flexural properties of the PLA/wood fiber composites were significantly higher when compared with the virgin resin. The addition of 20 wt % of wood fibers in PLA/wood fiber composite improved the flexural strength of PLA by 19 %, the flexural modulus by 115 %, and the tensile strength and tensile modulus by 5 wt % and 77 % respectively. The flexural modulus (8.9 GPa) of the PLA/wood fiber composite (30 wt % fiber content) was comparable to that of traditional (i.e. polypropylene/wood fiber) composites (3.4 GPa). Incorporation of the wood fibers in PLA resulted in a considerable increase of the storage modulus (stiffness) and a decrease in the tan delta values. The addition of the maleated polypropylene coupling agent (MAPP) improved the flexural and Izod impact properties of the wood fiber reinforced composites. The morphology as indicated by scanning electron microscopy (SEM), showed good dispersion of wood fiber in the PLA matrix. Microstructure studies also indicated a significant interfacial bonding between the matrix and the wood fibers.

The specific performance evidenced by the wood fiber reinforced PLA composites may hint their potential application, for example, in automotive industry and in geotextiles sectors.

## 1. Introduction

Wood fiber reinforced plastic composites are a group of new materials made from a combination of wood particles and thermoplastic resins [1]. Recently, wood fiber-reinforced polymers are increasingly being used in applications in the furniture, automotive, or building industry [1-3]. However, thermoplastic users are continually seeking new ways to improve

product performance. In addition to processing and physical properties, the long-time behavior of these materials is a very important parameter in respect to the design layout and the product liability [3-5]. Moreover, recent expansion of wood fiber reinforced plastic composites brings requirements for a more uniform and accurate evaluation of product properties across the industry. Though when combined with thermoplastic polymers, wood fibers produce composite materials that are lightweight, recyclable, and offer high strength to weight ratios; the combination of wood fibers and thermoplastic polymers presents a number of problems [1,5-7]. In many instances incompatibility between the fiber and matrix results in an inferior interface that does not adequately transfer stress to the load bearing fiber. Several reinforcing natural fibers and biodegradable polymers have been analyzed considering the structure-property relationship [6,7]. The performance of these biomaterials was greatly influenced by properties of the fiber and fiber-polymeric matrix adhesion. In order to improve the poor compatibility between hydrophobic thermoplastics and hydrophilic cellulosic fibers, many researches have been done to achieve high-performance natural fiber reinforced thermoplastics [8-11]. Further development of the applications of renewable bio-fibers (e.g., wood fibers) in the plastic industry can provide attractive new value-added markets for agro-products while simultaneously displacing petrochemical-based plastic resins.

One of the most promising biodegradable polymers is polylactic acid (PLA), and PLA can be manufactured from renewable resources, most commonly from corn [12]. Most research on PLA composite ultimately seeks to improve the mechanical properties to a level that satisfies a particular application [13]. Some researchers consider the enhanced toughness the main advantage of biofibers in composites [7,13]. Biocomposites consist of biopolymers as the matrix material and biodegradable fibers or fillers, usually biofibers. Since both components are biodegradable, the composite as the integral part is also expected to be biodegradable [8,12,13]. Since PLA can open many new opportunities in industrial bioplastic applications, there is a need to better understand and describe its properties as the matrix material for biocomposite materials.

A review of the literature shows that a comparative study of thermal and mechanical properties of PLA based composites containing different amount of wood fibers is very limited. Therefore, the purposes of this study are to evaluate the mechanical and thermo-mechanical properties of wood fiber reinforced PLA composite materials that were processed by a micro-compounding molding system. Wood fiber-reinforced polypropylene (PP) composites were also processed using the micro-compounding molding system and compared to PLA/wood fiber composites. Since coupling agents help overcome the polarity disparity to increase the composite strength, the maleated polypropylene coupling agent (MAPP) was also used in this study. The mechanical and thermal-mechanical properties of these composites have been studied.

## 2. Experimental

### 2.1. Materials

Poly (lactic acid) (PLA; molecular weight, $M_w$ 20 kDa; $M_n$ 10.1 kDa) was obtained from Biomer, Krailling, Germany (product name - Biomer L 9000). Wood Fibers (product name: 2010 MAPLE wood flour) was supplied by American Wood Fibers, Schofield, WI. Polypropylene (ProFax 6523) was obtained from Basell Polyolefins, Elkton, MD. Coupling agent (maleated polypropylene: EPOLENE G-3015 Polymer) (MAPP) was supplied by Eastman Chemical Company, Kingsport, TN.

### 2.2. Composites Processing

Prior to processing, the wood fibers and PLA were dried under vacuum at 80°C for 24 hrs. The PP matrix, however, was not dried. The polymer and the fibers were extruded at 100 rpm with a Micro 15cc compounding system, (DSM Research, Geleen, The Netherlands) at 183°C for 10 minutes. The extruder has a screw length of 150 mm, a L/D of 18, and a net capacity of 15 $cm^3$. In order to obtain the desired specimen samples for various measurements and analysis, the molten composite samples were transferred after extrusion, through a preheated cylinder to a mini-injection molder, which was pre-set with the desired temperature and cooling system. Injection-molded samples were placed in sealed polyethylene bags in order to prevent moisture absorption.

### 2.3. Measurements

*2.3.1. Mechanical testing*

A mechanical testing machine, United Calibration Corp SFM 20, was used to measure the tensile properties, according to ASTM D 638 standard, and the flexural properties according to ASTM D 790. System control and data analysis were preformed using Datum software. The Notched Izod impact strength was measured with a Testing Machines Inc. (TMI) 43-02-01 Monitor/Impact machine according to ASTM D256. All results presented are the average values of five measurements.

*2.3.3. Dynamic mechanical analysis (DMA)*

The storage modulus, loss modulus, and loss factor (Tan Delta) of the composite specimen were measured as a function of temperature (20°C to 100°C for PLA based composites and -50°C to 150°C for PP based composites) using a TA 2980 DMA equipped with a dual-cantilever bending fixture at a frequency of 1 Hz and a heating constant rate of 5°C /min.

*2.3.4. Scanning electron microscopy (SEM)*

The morphology of impact fracture surfaces of the composites was observed by scanning electron microscope (SEM) at room temperature. A JEOL (model JSM-6300F) SEM with field emission gun and accelerating voltage of 10 kV was used to collect SEM images for the composite specimen. A gold coating of a few nanometers in thickness was coated on impact fracture surfaces. The samples were viewed perpendicular to the fractured surface.

## 3. Results and discussions

### *3.1. Mechanical properties*

The flexural properties of the wood fiber reinforced PLA composites are given in Table I. There is an increasing trend of flexural strength for all composites after adding wood fibers. Neat PLA has a flexural strength and modulus of 98.8 MPa and 3.3 GPa, respectfully, whereas PLA/wood fiber (60 wt %/40 wt %) composite has a flexural strength and modulus of 114.3 MPa and 5.6 GPa, respectfully. The flexural strength increases significantly from 98.8 to 118.3 MPa with the presence of 20 wt % wood fibers, and there after flexural strength decreases to 114.3 MPa despite increasing the fiber content to 40 wt%. This reduction of flexural strength could be the result of less than adequate adhesion between the cellulose fibers and the PLA matrix. The flexural modulus also increases significantly with the addition of the wood fibers. The large

increase in modulus suggests an efficient stress transfer between the polymer and fiber [8, 14]. In addition, the good dispersion also contributes to the large increase in modulus.

**Table I.** *Flexural properties of the composites.*

| Polymer/Fibers (wt %) | Flexural Strength (MPa) | Flexural Modulus (GPa) | Improvement (modulus) (%) |
|---|---|---|---|
| Neat PLA | 98.8 ± 1.0 | 3.3 ± 0.1 | - |
| PLA/Wood fiber (80/20) | 118.3 ± 2.1 | 7.1 ± 0.2 | 115 |
| PLA/Wood fiber (70/30) | 116.6 ± 2.4 | 8.9 ± 0.8 | 169 |
| PLA/Wood fiber (60/40) | 114.3 ± 5.6 | 10.2 ± 0.9 | 209 |
| Neat PP | 32.9 ± 1.8 | 1.5 ± 0.2 | - |
| PP/Wood fiber (80/20) | 43.0 ± 1.2 | 2.7 ± 0.1 | 80 |
| PP/Wood fiber (70/30) | 51.4 ± 4.3 | 3.4 ± 0.9 | 126 |
| PP/Wood fiber (60/40) | 55.1 ± 2.4 | 4.6 ± 0.3 | 206 |

Table I also give the mechanical properties of the PP and PP based composites. Both flexural modulus and flexural strength of PP increase significantly with the addition of wood fibers. Table II gives the mechanical properties of the wood fiber reinforced PLA- and PP-based composites in the presence of MAPP. The flexural strength and modulus of PLA/MAPP (95 wt %/5 wt %) composite increased significantly with the addition of MAPP. Similar increment was observed in the case of PP/MAPP(95%/5%) composite. According to Keener et al.[15], as MAPP polymers chemically link to the surface of the reinforcement/filler, it also co-crystallizes with the base matrix, creating a composite with increased thermal and physical properties. Table II also shows the effect of MAPP on the mechanical properties of wood fiber composites. It can be seen from Table II that flexural strength of PLA/TC 1004/MAPP (65 wt %/30 wt %/5 wt %) composite decreased with the addition of 5% MAPP when compare with PLA/TC 1004 (70 wt %/30 wt %) composite. Both flexural modulus and flexural strength of PP/wood fiber/MAPP (65/30/5) composite increased significantly after the addition of 5 wt % MAPP as seen in Table II. The addition of MAPP improved the PP/wood fiber interfacial bond dramatically. Generally, interactions between the hydroxyl groups of natural fibers and the anhydride groups of maleated coupling agents can overcome the incompatibility problem to increase the mechanical properties of natural fiber reinforced thermoplastic composites [8]. The formation of covalent linkages between hydroxyl groups of cellulose fiber and maleic anhydride was indicating by Hedenberg and Gatenholm [16] though IR and ESCA analysis. In this study, we observed greater improvement in flexural properties of the wood fiber composites.

The tensile properties of the PLA matrix and PLA based composites are shown in Table III. Tensile modulus increases significantly with the addition of the 20 wt % and 30 wt % wood fibers, and tensile strength remains nearly same. This reveals that the incorporation of the wood fibers into the matrix provides effective reinforcement. Further addition of wood fibers increased the modulus, but the tensile strength of this 40 wt % wood fiber reinforced PLA composite decreased when compare with neat PLA. The decrease in tensile strength with an increase in the wood fiber weight fraction is probably due to either the aggregation of the wood fibers or insufficient hydrogen bonding between the PLA matrix and the wood fiber [2, 5]. Usually, the

orientation of wood fibers in the composites influences the tensile strength of fiber reinforced composite.

**Table II.** *Flexural properties of the composites.*

| Polymer/Fibers (wt %) | Flexural Strength (MPa) | Flexural Modulus (GPa) | Improvement (modulus) (%) |
|---|---|---|---|
| Neat PLA | 98.8 ± 1.0 | 3.3 ± 0.1 | - |
| PLA/MAPP (95/5) | 111.8 ± 0.6 | 6.1 ± 0.0 | 84 |
| PLA/Wood fiber /MAPP (65/30/5) | 75.3 ± 1.8 | 8.6 ± 0.2 | 161 |
| Neat PP | 32.9 ± 1.8 | 1.5 ± 0.2 | - |
| PP/MAPP (95/5) | 44.0 ± 0.8 | 2.3 ± 0.0 | 53 |
| PP/Wood fiber/ (65/30/5) | 64.6 ± 1.3 | 3.9 ± 0.4 | 160 |

**Table III.** *Tensile properties of the PLA based composites.*

| Polymer/Fibers (wt %) | Tensile Strength (MPa) | Tensile Modulus (GPa) | Improvement (modulus) (%) |
|---|---|---|---|
| Neat PLA | 62.8 ± 4.9 | 2.7 ± 0.4 | - |
| PLA/Wood fiber (80/20) | 65.7 ± 1.3 | 4.8 ± 0.6 | 77 |
| PLA/Wood fiber (70/30) | 63.3 ± 5.8 | 5.3 ± 0.8 | 96 |
| PLA/Wood fiber (60/40) | 58.7 ± 3.1 | 6.3 ± 0.9 | 133 |

The notched Izod impact strength results for tested materials are shown in Table IV. As seen in Table IV, the impact strength of the PLA/wood fiber (60 wt %/40 wt %) composite decreased slightly after addition of higher fiber content when compare with the PLA/wood fiber (70/30) composite, which has impact strength of 23.2 J/m. It can also be seen that the impact strength of both PLA/wood fiber (70 wt %/30 wt %) and PLA/wood fiber/MAPP (65 wt %/30 wt %/5 wt %) composites remain nearly same even after the addition of 5 wt % MAPP. Table IV shows that after addition of 20 wt % wood fibers the impact strength of the PP composite improved significantly. The impact strength of the PP/wood fiber (60 wt %/40 wt %) composite decreased slightly after addition of higher fiber content when compare with the PP/wood fiber (70 wt %/30 wt %) composite, which has impact strength of 46.4 J/m. The practical effect of fiber on impact strength of composites depends on the factor, such as fibers increase the impact strength due to the fact that fibers reduce the creak propagation rate by forcing creaks around the fibers and bridging creaks through fiber pull out [17]. The impact strength of the PP composite also improved after addition of 5 wt % MAPP as seen in Table IV. The impact strength of the PP/wood fiber/MAPP (65 wt %/30 wt %/5 wt %) composite increased after addition of 5 wt % MAPP, where MAPP plays a role of coupling agent and improved the adhesion between the fiber and the matrix.

**Table IV.** *Notched Izod Impact Strength of the composites.*

| Polymer/Fibers (wt %) | Notched Izod Impact Strength (J/m) | Improvement (%) |
|---|---|---|
| Neat PLA | 25.7 ± 1.3 | - |
| PLA/Wood fiber (80/20) | 23.9 ± 0.7 | - |
| PLA/Wood fiber (70/30) | 23.2 ± 2.9 | - |
| PLA/Wood fiber (60/40) | 21.9 ± 3.3 | - |
| PLA/MAPP (95/5) | 26.5 ± 2.7 | 3 |
| PLA/Wood fiber/MAPP (65/30/5) | 24.5 ± 0.6 | - |
| Neat PP | 29.7 ± 3.1 | - |
| PP/Wood fiber (80/20) | 57.6 ± 5.9 | 93 |
| PP/Wood fiber (70/30) | 46.4 ± 1.8 | 56 |
| PP/Wood fiber (60/40) | 37.8 ± 3.9 | 27 |
| PP/MAPP (95/5) | 25.5 ± 4.4 | 6 |
| PP/Wood fiber/MAPP (65/30/5) | 47.3 ± 4.5 | 59 |

### *3.2. Thermo-mechanical properties*

Figures 1(A) and 1(B) show respectively the dynamic storage modulus and tan delta of the PLA based composites, as a function of temperature. As seen in Figure 1(A) and Table V, the moduli increase in the presence of wood fibers in the composite, i.e., the storage modulus of PLA based composites is higher than that of PLA matrix. This is due to the reinforcement imparted by the wood fibers that allows stress transfer from the matrix to the wood fiber [16, 18]. This study found that both the storage and loss moduli increased with the incorporation of wood fiber in PLA. As seen in Figure 1(B), incorporation of fibers reduces the tan delta peak height by restricting the movement of the PLA polymer molecules. The addition of MAPP has decreased the storage modulus of PLA/wood fiber/MAPP (65 wt %/30 wt %/5 wt %) composite.

**Table V.** *The storage modulus of the composites.*

| PLA/Fibers (wt %) | Storage Modulus (GPa) at 25 °C | Storage Modulus (GPa) at 40 °C | Storage Modulus (GPa) at 60 °C | Reinforcement imparted by the fibers at 25 °C (modulus) (%) |
|---|---|---|---|---|
| Neat PLA | 3.2 | 3.1 | 1.8 | - |
| PLA/Wood Fiber (80/20) | 9.3 | 9.1 | 8.5 | 190 |
| PLA/Wood Fiber (70/30) | 10.2 | 10.0 | 9.2 | 218 |
| PLA/Wood Fiber (60/40) | 12.6 | 12.1 | 11.3 | 293 |
| PLA/Wood Fiber/MAPP (60/30/10) | 9.3 | 9.1 | 8.4 | 190 |
| PLA/MAPP (95/5) | 7.5 | 7.3 | 4.8 | 134 |

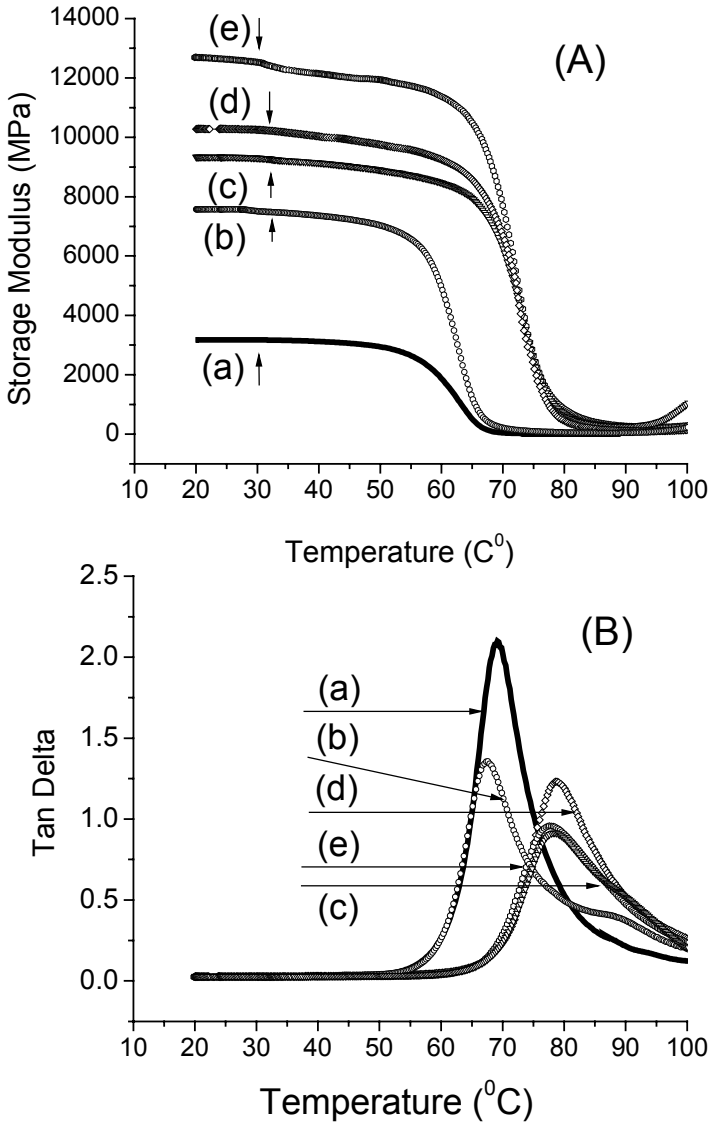


**Figure 1.** Temperature dependence of (A) storage modulus, and (B) Tan delta of PLA and PLA based composites: (a) neat PLA (□), (b) PLA/MAPP (95/5) (–), (c) PLA/Wood fiber/MAPP (65/30/5)(X), (d) PLA/Wood fiber (70/30)(M), and (e) PLA/Wood fiber (60/40)(v).

Figures 2(A) and 2(B) show the dynamic storage modulus and tan delta of the PP based composites, as a function of temperature, respectively. As seen in Figure 2(A) and Table VI, the moduli increase in the presence of wood fibers in the composite. The storage modulus of PP based composites is higher than that of the PP matrix; which indicates that stress transfers from

the matrix to the wood fiber. Figure 2(B) shows that the tan delta values of the PP/wood fiber composite are lower than those for the PP matrix at low temperatures. The tan delta values for the composite increased with increasing temperature. The incorporation of fibers reduces the tan delta peak height by restricting the movement of the PP polymer molecules as seen in Figure 2(B). This study found that both the storage and loss moduli increased significantly with the incorporation of wood fiber into polypropylene when no coupling agent was added. As seen in Figure 2(B), the addition of MAPP has increased the storage modulus of PP/wood fiber/MAPP (65 wt %/30 wt %/5 wt %) composite, where MAPP shows positive effects on the thermo-mechanical properties of the wood fiber-reinforced PP composites.

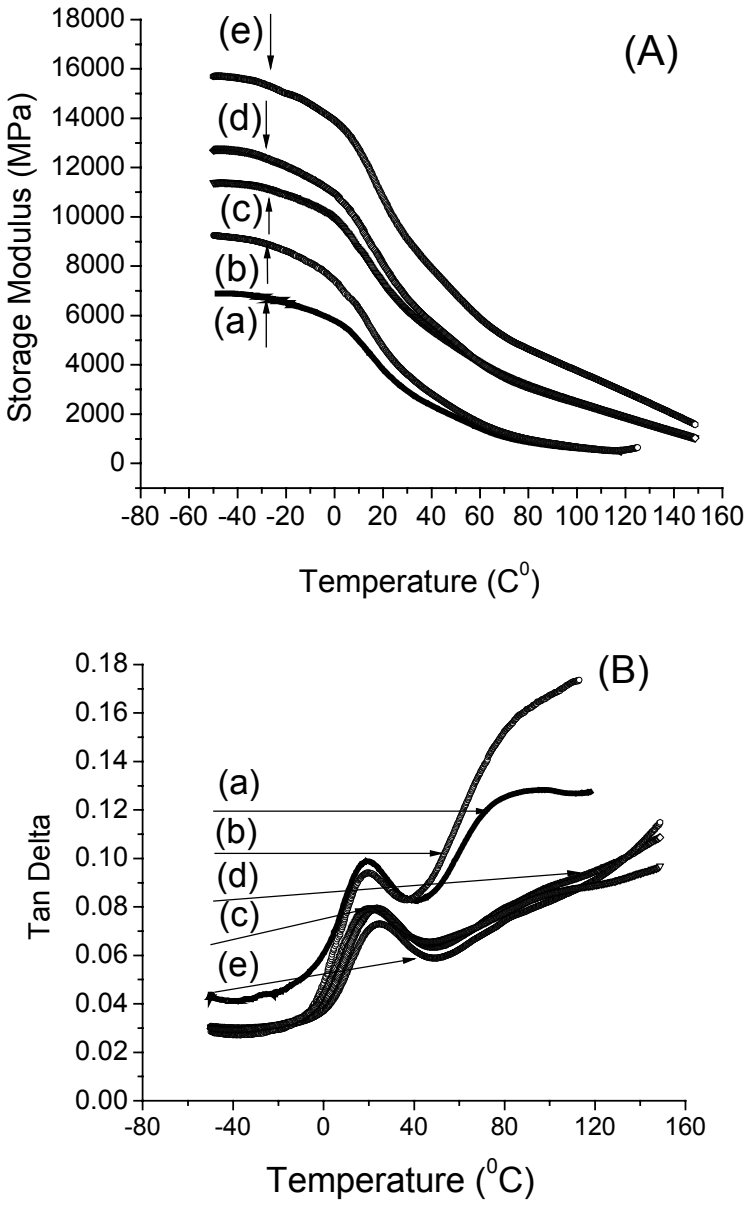


**Figure 2.** Temperature dependence of (A) storage modulus, and (B) Tan delta of PP and PP based composites: (a) neat PP (□), (b) PP/Epolene (95/5) (–), (c) PP/Wood fiber/Epolene (65/30/5)(X), (d) PP/Wood fiber (70/30)(M), and (e) PP/Wood fiber (60/40)(v).

**Table VI.** *The storage modulus of the composites.*

| Polymer/Fibers (wt %) | Storage Modulus (GPa) at 25 °C | Storage Modulus (GPa) at 40 °C | Storage Modulus (GPa) at 60 °C | Reinforcement imparted by the fibers at 25 °C (modulus) (%) |
|---|---|---|---|---|
| Neat PP | 3.3 | 3.1 | 1.8 | - |
| PP/Wood fiber (80/20) | 5.6 | 4.4 | 3.1 | 69 |
| PP/Wood fiber (70/30) | 7.3 | 5.7 | 4.0 | 121 |
| PP/Wood fiber (60/40) | 9.8 | 7.9 | 5.8 | 196 |
| PP/Wood fiber/MAPP (65/30/5) | 6.6 | 5.3 | 4.0 | 100 |
| PP/MAPP (95/5) | 4.0 | 2.8 | 1.6 | 21 |

### *3.3. Morphology of the composites*

In Figure 3, SEM micrographs illustrate the individual separation and dispersion of the wood fibers in the form of single fibers, which indicates that the wood fibers have been separated during the extrusion process and well dispersed in the PLA matrix. The closer observation in micrograph shows that the matrix is an indication of good fiber-matrix adhesion (Figure 3b). In Figure 4, coupling agent does not cause wood fiber particle dispersion to change the PLA matrix. MAPP particles are visible on the composite surface, which indicates that MAPP does not locate in the interface between the fiber and the matrix, and hence MAPP does not work as coupling agent in the in the PLA/wood composite system. The morphology of the impact fracture surfaces of PP/wood fiber (70/30) composite was investigated by SEM (in Figure 5). As seen in Figure 5, the dispersion of the wood fibers in the form of single fibers, where no large aggregates are present and this morphology is optimal for toughening to occur. Figure 6 shows that coupling agent does favor a better PP-fiber interaction and the wood fiber has been covered with a thin layer of the matrix, which lead to better stress transfer between the matrix and the reinforcing fibers.

## 4. Conclusions

The mechanical and thermo-mechanical properties of the wood fiber reinforced PLA composites were found to compare favorably with the corresponding properties of PP composites. Compared to the neat resin, the flexural modulus and strength of PLA composites were significantly higher as a result of reinforcement by the wood fibers. From the DMA results, incorporation of wood fibers gives rise to a considerable increase of the storage modulus and a decrease in the tan delta values. These results demonstrate the reinforcing effect of wood fibers on both PLA and PP matrices. The morphology evaluated by SEM indicated that there were less voids on the fracture surface, which indicates that the wood fibers are well trapped by the PLA matrix as well as PP matrix. Maleated polypropylene coupling agent (MAPP) favors a better PP-wood fiber interaction. Wood fibers are more effectively serve as reinforcement instead of filler with the MAPP. PLA composites containing wood fiber may be as good as or even better than polypropylene reinforced with wood fiber. Overall, the advantages of using a biodegradable

polymer like PLA as a matrix was proved since the environmentally friendly composites prepared with this material present good thermal and mechanical properties. Furthermore, the use of wood fibers, as reinforcements in PLA, gives interesting alternatives for production of low cost and ecologically friendly composites.

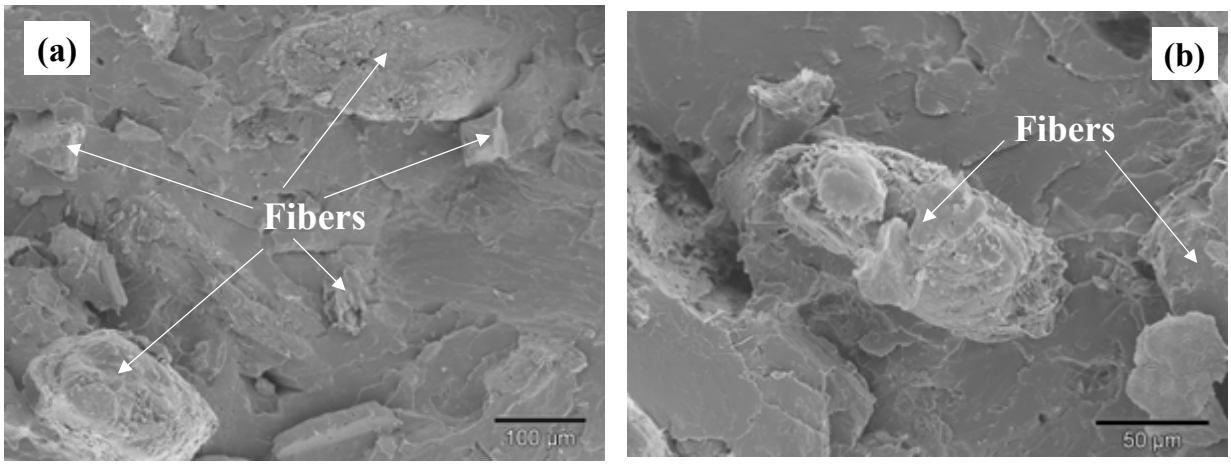


**Figure 3.** SEM micrographs of PLA/wood fiber (70/30) composite: (a) 100 µm (200x) and (b) 50 µm (500x).

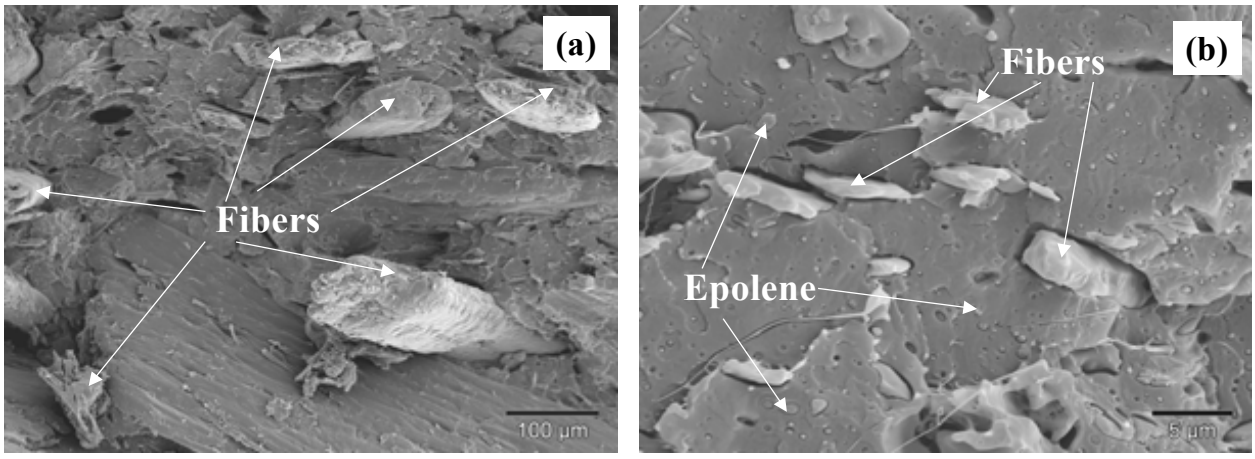


**Figure 4.** SEM micrographs of PLA/wood fiber/MAPP(Epolene) (65/30/5) composite: (a) 100 µm (200x) and (b) 50 µm (3500x).

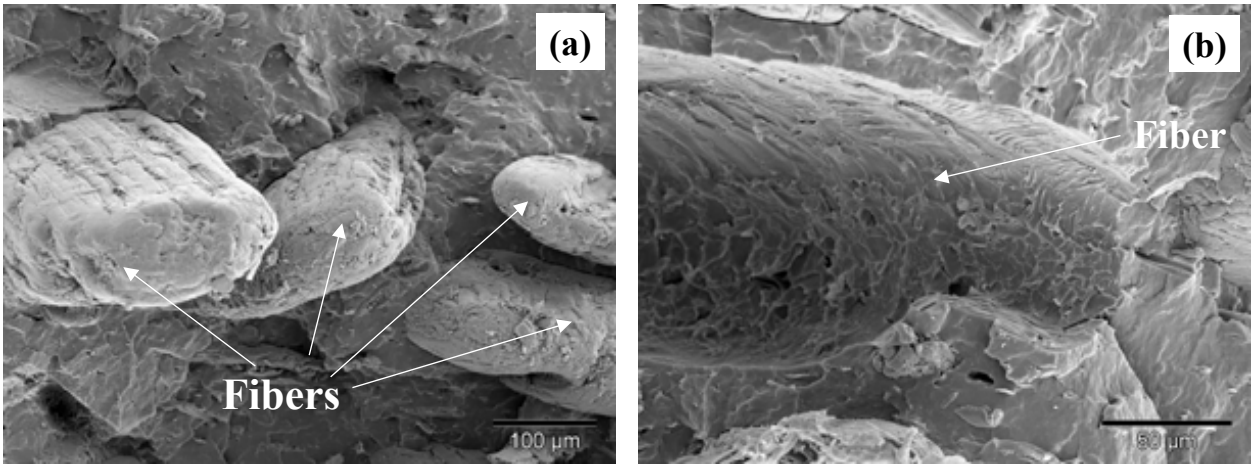


**Figure 5.** SEM micrographs of PP/wood fiber (70/30) composite: (a) 100 µm (200x) and (b) 50 µm (3500x).

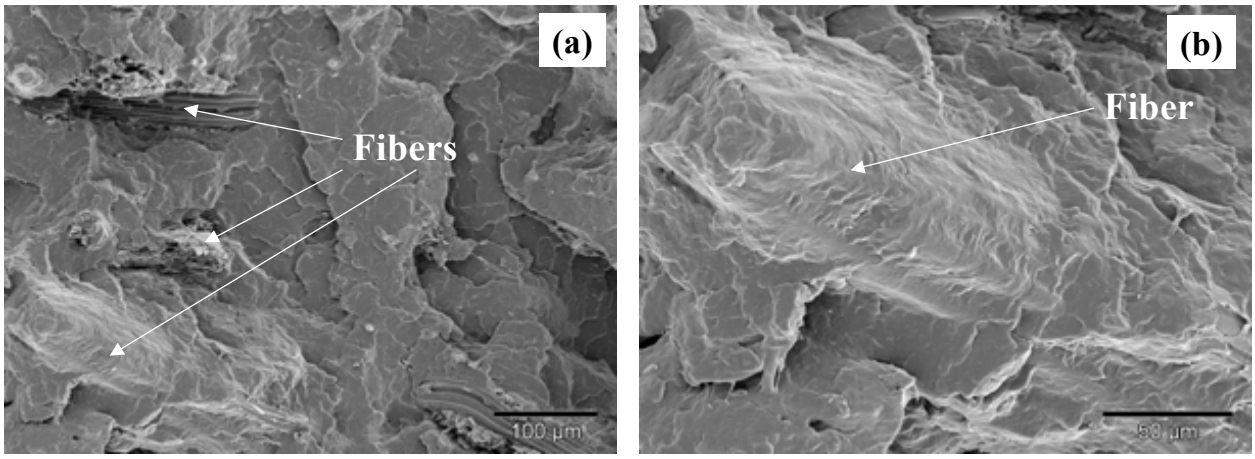


**Figure 6.** SEM micrographs of PP/wood fiber/MAPP (65/30/5) composite: (a) 100 µm (200x) and (b) 50 µm (3500x).

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