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## The Morphological, Mechanical and Physical Properties of Wood Flour-Poly Lactic Acid Composites under Various Filler Types

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**Abstract:** The morphological, mechanical and physical properties of composites of PLA (Poly Lactic Acid) with or without MPLA (maleic anhydride (MAH)-modified-PLA) compatibilizer under various filler types and testing conditions had been studied. The tensile properties of the composites varied with the filler types. The tensile properties of the composites with Cellulose Powder (CP) based filler are greater than those with WF (Wood Flour) and CS (Corn Starch) based fillers. The order is CP>WF>CS. The Scanning Electron Microscopy (SEM) showed that addition of MPLA compatibilizer to the composites improved the dispersion and the adhesion of the filler and the matrix. It was found that after immersion in cold water for 24 h and drying at 60°C for 7 days or 105°C for 3 days, the tensile properties of the composites based on CP and WF fillers were unchanged. The composites based on CP and WF fillers were more resistant to water than the CS-filled composite. After storage in a room adjusted to 20°C and 60% R.H. for 3 months, the Moisture Content (MC) of the composites with CP/WF/CS fillers was less than 6%. The CP and WF-filled composites with MPLA compatibilizer tended to give lower MC than compatibilizer-free composites.

**Key words:** Poly lactic acid, composites, compatibilizer, wood flour, cellulose powder, corn starch

### INTRODUCTION

In recent years, preparation of composites products using biodegradable synthetic polymer and woody fillers is becoming an interesting approach for the developing new specialty products, because they could allow complete degradation in soil or by composting process and do not emit any toxic or noxious component (Gatenholm and Mathiasson, 1992, 1994; Rossa *et al.*, 2003; Lee and Wang, 2006). Poly lactic acid is one of biodegradable polymer having a good potential for biodegradable packaging and consumer goods because of the very good thermoplastic behavior, such as mechanical strength, transparency, compostability and safety (Sinclair, 1996; Whiteman *et al.*, 2002).

The possibilities of using wood fiber-reinforced thermoplastics are enormous, for example, automobile, construction components, frames, shipping crates, pallets, storage bins, toys, games, household goods, outdoor furniture and recreational boats etc. Unfortunately, the use of cellulose fibers in thermoplastic composites has

been limited because of compatibility problems between hydrophilic cellulose fibers and hydrophobic thermoplastic. Other problems encountered are dimensional instability due to the absorption of moisture in a humid environment and phase separation under stress, especially at subzero and high temperature (Flodin and Zadorecki, 1986; Maldas and Kokta, 1990). However, good interfacial contact that is, bonding of the fiber and the PLA polymer at the interface can eliminate these problems, at least partially.

In our earlier publication (Febrianto *et al.*, 2005) we had proved that grafting by esterification had been attained between the OH-groups of Wood Flour (WF) and acid anhydride groups of maleic anhydride (MAH)-modified PLA (MPLA) during kneading at 180°C, 30-90 rpm for 10 min. Furthermore, by titration analysis, the MAH content of MPLA prepared from 10-20% MAH with 0.3-1.5% dicumylperoxide (DCP) was 0.83-1.66%. The tensile strength varied with the amount of MAH content. The composites of PLA-WF, with or without MPLA compatibilizer, were easily degraded by termites

(*Coptotermes formasanus*) and bacteria (*Bacillus sp.*) but they were hardly attacked at all by the brown-rot (*Tyromyces palustris*) and white-rot (*Coriolus versicolor*) fungi within the experimental conditions adopted.

In the present study, the physical, mechanical and morphological properties of composites of PLA with or without MPLA compatibilizer under various filler types and testing conditions were investigated.

## MATERIALS AND METHODS

**Materials:** PLA used was LACTY # 9000 which was supplied by Shimadzu co., with Mn and Mw based on GPC analysis were  $7.12 \times 10^4$  and  $13.68 \times 10^4$ , respectively. Dicumyl peroxide (DCP), n-pentane (EP), benzyl amine (GR) and maleic anhydride (MAH) were purchased from Nacalai tesque inc., while acetone was purchased from Wakenyaku Co. All these polymers and solvents were used as received. The fillers used were Cellulose Powder (CP), Wood Flour (WF) and Corn Starch (CS).

**Preparation of MPLA:** PLA and 10% MAH were reactively blended in the melt state in the kneader (Toyo Seiki Labo-Plastomill LPM 18-125). When PLA and MAH were homogeneously mixed (torque value stable) initiator (DCP) was added. The amount of initiator was 15% based on the MAH weight. The kneading temperature, kneading time and rate of rotation of the kneader were 160°C, 30-70 rpm, for 15 min., respectively.

**Compounding PLA, MPLA and WF:** Compounding PLA and CP/WF/CS as well as PLA, MPLA and CP/WF/CS were carried out by using a kneader at 180°C, 30-90 rpm, 10 min. A prescribed amount of PLA with or without MPLA and CP/WF/CS were hand-mixed and placed into the kneader at 180°C, 30 rpm for 2 min. After charging, the kneading was continued for 5 min at 90 rpm. The amount of matrix and filler were 24 g, being enough to fill up the mixing chamber and causing torque while kneading.

**Preparation of composite sheet:** Kneaded samples were molded into sheet by hot pressing with Toyo seiki 10 t bench hot-presses for testing. The prescribed amount of kneaded samples (5-7 g) were placed between a pair of terephthalate sheet with 0.3 mm thick spacer. The temperature of the hot press was 200°C and the samples were subjected to 0-50 and 150 kgf cm<sup>-2</sup> pressure for 2 and 0.5 min, respectively. After subsequent cold pressing at the same pressure for 30 sec. The sheets were then cooled at room temperature.

**Tensile test:** Strip samples 80.0×5.0×0.3 mm were prepared from composite sheets. Tensile tests were made on these strip samples with a Shimadzu Autograph DCS-R-500. The measurements were made with a span length of 40 mm and cross head speed of 10 mm min<sup>-1</sup> in a room adjusted to 20°C and 60% RH. The average values of tensile strength, breaking elongation and Young's modulus were obtained automatically from ten repeated measurements. Before tensile testing, samples of each type of composite were divided into 9 different groups.

**Group 1:** Tensile properties were measured at room temperature (20°C and 60% RH.)

**Group 2:** Samples were immersed in cold water for 24 h (1 day) in a room adjusted to 20°C and 60% R.H. Samples were then wiped and stored in the same room for 2 days prior to measurement.

**Group 3:** Samples were immersed in cold water for 48 h (2 days) in a room adjusted to 20°C and 60% RH. Samples were then wiped and stored in the same room for 2 days prior to measurement.

**Group 4:** Samples were immersed in cold water for 168 h (7 days) in a room adjusted to 20°C and 60% RH. Samples were then wiped and stored in the same room for 2 days prior to measurement.

**Group 5:** Samples were immersed in boiling water for 3 h. Samples were then wiped and stored in a room adjusted to 20°C and 60% RH. for 2 days prior to measurement.

**Group 6:** Samples were immersed in 60°C water for 3 h. Samples were then wiped and stored in a room adjusted to 20°C and 60% RH. for 2 days prior to measurement.

**Group 7:** Samples were dried in the oven at 105°C for 3 days. Samples were then wiped and stored in a room adjusted to 20°C and 60% RH for 2 days prior to measurement.

**Group 8:** Samples were dried in the oven at 60°C for 7 days. Samples were then wiped and stored in a room adjusted to 20°C and 60% RH for 2 days prior to measurement

**Group 9:** Samples were dried in the oven at 105°C for 7 days. Samples were then wiped and stored in a room adjusted to 20°C and 60% RH for 2 days prior to measurement.

**Water absorption test:** Square samples of (50×50×0.3) mm size were prepared from the composite sheets groups 2, 3, 4 and 6. Water absorption test was made on this square samples in three replications. The samples were dried over night in the vacuum oven at the temperature of 60°C and then stored in a desiccator. The weights (W1) of the samples were measured in a room adjusted to 20°C and 60% RH. Subsequently, the samples were dipped for 24 h to water that had been conditioned at a room adjusted to 20°C, 60% RH for 48 h. Then, the samples were wiped and the weight (W2) is measured. Finally, the samples were dried in the 60°C oven in vacuo to obtain the constant weight (W3). Water absorption was calculated (Eq. 1).

$$\text{Water absorption (\%)} = \frac{W2-W3}{W1} \times 100$$

**Moisture content test:** Square samples of (50×50×0.3) mm size were prepared from the composite sheets. Moisture content was measured every week for 3 months in a room adjusted to 20°C and 60% RH.

**Scanning Electron Microscopy (SEM) observation:** The morphology of the tensile fracture of the composites was studied by using a JEOL JSM-T330A Scanning microscope. The samples were mounted on brass tubs and were coated with a thin layer of gold using a JEOL JFC-1100E ion sputter coater. The samples were dried before coating to remove all the sorbed water. SEM photographs were taken under the following condition: working distance 15 mm and accelerating voltage of 10 kV.

## RESULTS AND DISCUSSION

**The tensile properties of composites under various filler types:** Table 1 reveals the effect of filler types on the tensile strength, breaking elongation and Young's modulus of composites with or without MPLA compatibilizer. The fillers used were CP, WF and CS. The composites consisting of 100 parts of filler, 80-100 parts of PLA and 0-20 parts of MPLA. The MPLA was prepared from PLA with 10% MAH and 15% DCP (based on MAH weight) in the kneader at 160°C, 30-70 rpm, for 15 min. The kneading conditions for obtaining the composites were set at 180°C, 30-90 rpm, for 10 min. The pressing conditions were 200°C, 0-50 kgf cm<sup>-2</sup> for 2 min and 150 kgf cm<sup>-2</sup> for 30 sec.

It is clear that the role of MPLA in strengthening the composites at various filler types is similar (Table 1). Addition of MPLA improved the tensile properties of the obtained composites. The tensile strength, breaking elongation and Young's modulus of the PLA-MPLA-CP

Table 1: Tensile properties of PLA-CP/WF/CS composites with or without MPLA compatibilizer

Composite	T. Strength (Mpa)	B. Elongation (%)	Y. Modulus (×10 <sup>-3</sup> MPa)
PLA-CP (50-50)	45.41	2.09	2.99
PLA-MPLA-CP (40-10-50)	83.49	3.83	3.81
PLA-WF (50-50)	41.33	1.64	3.00
PLA-MPLA-WF (40-10-50)	68.70	3.11	3.57
PLA-CS (50-50)	32.16	1.65	2.20
PLA-MPLA-CS (40-10-50)	61.60	3.27	2.54
PLA film	73.87	5.41	2.19

composite is higher than the tensile strength, breaking elongation and Young's modulus of PLA-MPLA-WF and PLA-MPLA-CS composites. The order is PLA-MPLA-CP composite > PLA-MPLA-WF composite > and PLA-MPLA-CS composite. The difference in strength values obtained can presumably be attributed to the difference in properties among CP, WF and CS. Cellulose powder composed of 100% cellulose. Wood flour is composite material mostly composed of cellulose (40-50%), hemicellulose (20-30%) and lignin (20-30%) with small quantities of inorganic compound and extractives. Cellulose consists simply of D-glucose units linked by the 1,4-β-glycoside bonds. Several thousand glucose units are linked to form one large molecule and different molecules can then interact to form a large aggregate structure held together by hydrogen bonds. On the other hand, starch is a polymer of glucose in which monosaccharide units are linked by the 1,4-α-glycoside bonds. Starch can be separated into two fractions: a fraction soluble in cold water, called *amylopectin* and a fraction insoluble in cold water, called *amylose*. Amylose, which accounts for about 20% by weight of starch, consists of several hundred glucose molecules linked together by 1,4-α-glycoside bonds (Mc Murry, 1992). Hence, filler with a higher degree of polymerization can result in greatly strengthened composites. Similar results had been reported by Han (1990) in the WF-PP composites using MPP as a compatibilizer and Dissolving Pulp (DP), bleached Kraft Pulp (KP), Refiner Ground Pulp (RGP) and Wood Flour (WF) as fillers; Febrianto *et al.* (2001) in the WF-*trans*-1,4-isoprene rubber (TIR) composites using maleated *trans*-1,4-isoprene rubber (MTIR) as a compatibilizer and Cellulose Powder (CP), Corns Starch (CS) and Wood Flour (WF) as fillers.

**Morphology of composites:** Evaluation of the fracture surfaces of the composites by Scanning Electron Microscope (SEM) can provide information on how MPLA compatibilizer improves the adhesion between the matrix and the filler. In the composites without MPLA compatibilizer, it is shown that the fillers embedded in the polymer matrix are well dispersed. The inter phase region between the fillers and the polymer matrix shows that the interface is clean, the particle are not broken and there are distinct gaps/voids between the fillers and the PLA matrix.

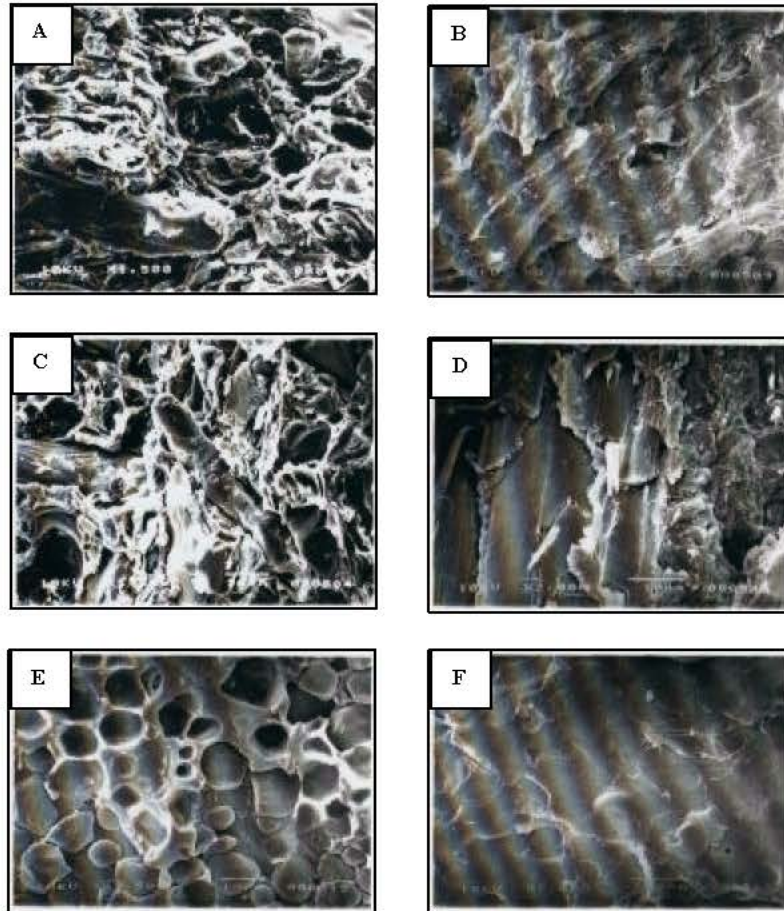


Fig. 1: SEM micrograph of tensile fracture of the composites. (A) PLA-CP; (B) PLA-MPLA-CP; (C) PLA-WF; (D) PLA-MPLA-WF; (E) PLA-CS and (F) PLA-MPLA-CS

This indicates poor adhesion or interaction between the surfaces. A large number of holes appear, especially in Fig. 1E, which result from the fiber pull out from the matrix. Further evidence for this is that the filler particles have smooth surfaces and it can be easily seen that the fracture passes through the weak interface (Fig. 1A, C and E).

On the contrary, Fig. 1B, D and F show the microstructure of the composites with MPLA as a compatibilizer. The microstructure is different from that of Fig. 1A, C and E. Generally, it is more difficult to differentiate the filler particles from the PLA matrix. This may suggest that the filler particles are coated probably by the matrix and that the failure most commonly occurs in the matrix rather than at the filler surfaces. There are places where the adhesion between PLA and CP/WF/CS is good, as broken filler particles can be seen with no gap between the matrix and the fillers. The filler surfaces are

not as clean and smooth as in the previous figures and the MPLA is believed to be located at the filler surfaces. When the adhesion is not so good, there are voids around the filler particles and places where filler particles have been pulled out. Good adhesion between the CP/WF/CS fillers and the PLA matrix was expected because the composites had improved tensile strength, which means that loads can be transferred from the matrix to the fillers (Table 1). This suggests that there is some kind of interfacial contact between fillers and matrix. The MPLA may also act as a dispersing agent between polar fillers and non-polar matrix resulting in better dispersion of the fillers.

**Tensile properties of composites under various conditions:** Keeping the amount of CP/WF/CS fillers at 50%, the ratio between PLA and MPLA was set at 40:10. Table 2 and Fig. 2 to 5 show the change in tensile

**Table 2: Tensile properties of composites of PLA-CP, WF and CS fillers under various conditions**

Groups		Cellulose Powder (CP)			Wood Flour (WF)			Corn Starch (CS)		
		TS (Mpa)	BE (%)	YM 10 <sup>-3</sup> (Mpa)	TS (Mpa)	BE (%)	YM 10 <sup>-3</sup> (Mpa)	TS (Mpa)	BE (%)	YM 10 <sup>-3</sup> (Mpa)
G1	A	45.41	2.09	2.306	41.33	1.64	1.839	32.16	1.65	1.599
	B	83.49	3.56	3.788	68.70	3.11	3.641	61.60	3.27	2.536
G2	A	38.81	1.85	2.684	30.27	1.66	2.207	24.09	1.95	1.627
	B	74.01	3.77	3.642	57.44	2.34	3.389	41.14	2.54	2.071
G3	A	40.78	2.01	2.626	30.38	2.04	2.086	23.45	1.85	1.575
	B	69.10	4.12	3.360	57.65	2.49	3.201	38.37	2.47	2.006
G4	A	32.59	2.05	2.111	24.42	1.78	1.849	22.24	1.94	1.582
	B	69.35	3.22	3.439	58.33	2.25	3.510	35.65	2.58	1.848
G5	A	26.83	2.55	1.585	13.78	2.01	0.692	ND	ND	ND
	B	47.56	2.34	3.242	22.44	1.61	1.937	ND	ND	ND
G6	A	30.25	2.19	2.076	17.30	1.97	1.132	4.42	2.01	0.375
	B	59.18	3.17	3.156	44.88	2.52	2.625	11.39	2.55	0.647
G7	A	47.51	2.50	2.822	30.92	1.70	2.283	26.72	1.73	2.120
	B	83.24	3.60	3.915	64.48	2.54	3.664	56.16	2.54	3.003
G8	A	40.22	1.85	2.833	18.98	0.96	2.448	25.62	1.63	2.094
	B	75.72	3.43	3.580	70.32	3.21	3.540	63.06	3.49	2.559
G9	A	40.98	1.77	2.654	31.22	1.69	2.206	27.06	1.72	2.042
	B	56.25	2.47	3.179	44.16	2.16	3.040	34.76	2.16	2.480

Note: TS = Tensile Strength; BE = Breaking Elongation; YM = Young's Modulus; A = PLA/Filler (50/50); B = PLA/MPLA/Filler (40/10/50)  
 ND: Not Determined

**Table 3: Material loss (elution) of composites after immersion in cold water for 7 days**

Composite	Elution (%)
PLA-CP (50-50)	0.58
PLA-MPLA-CP (40-10-50)	0.38
PLA-WF (50-50)	0.39
PLA-MPLA-WF (40-10-50)	0.35
PLA-CS (50-50)	1.55
PLA-MPLA-CS (40-10-50)	3.66

properties (tensile strength, breaking elongation and Young's modulus) of CP, WF and CS based composites with or without MPLA compatibilizer under various conditions.

It has been demonstrated in previous part that addition of small amount of MPLA greatly improved the tensile properties of the CP, WF and CS-filled PLA composites. Figure 2 exhibits the change in tensile strength, breaking elongation and Young's modulus of composites after immersion in hot water (60 and 90°C) for 3 h. It is obvious that the tensile properties, particularly the tensile strength of the composites with or without MPLA decrease after exposure to water at elevated temperatures. The higher the temperature, the greater is the reduction of the tensile strength. It is also clear, that the composites based on CS filler was less resistant to water at elevated temperatures than that containing WF and CP based fillers. A similar phenomenon is observed regarding the Young's modulus.

Figure 3 exhibits the effect of immersion time on the tensile properties of PLA-CP, WF and CS-filled composites with or without MPLA compatibilizer. The samples were immersed in cold water for 1, 2 and 7 days

prior to measurement. All the composites show similar trends regarding the tensile strength. They decrease with an increase in immersion time and remain constant after 2 days of immersion time. Compared to other composites, the CS-based composites show the highest reduction in tensile strength. On the other hand, the Young's modulus of the composites without MPLA compatibilizer remained unchanged after exposure to cold water for prolonged times.

Figure 4 reveals a change in the tensile properties of PLA-CP, WF and CS-filled composites with or without MPLA compatibilizer at elevated drying temperatures. Prior to measurement, the samples were dried at 60°C and 105°C for 7 days. It can be said from this figure that by drying the composites at 60°C for 7 days, the tensile properties of all composites remained unchanged. Increasing the drying temperature to 105°C resulted in a decrease in tensile strength of the composites, particularly the composites with MPLA compatibilizer. Similar phenomena can be seen regarding the breaking elongation. Furthermore, the composites with MPLA compatibilizer show a slightly decrease in the Young's modulus after drying at elevated temperature.

Figure 5 shows a change of the tensile properties of the PLA-CP,WF, CS-filled composites after drying at 105°C for prolonged times. The tensile strength, the breaking elongation and the Young's modulus tend to give similar trends. The tensile properties of the composites with or without MPLA compatibilizer remained unchanged after drying for 3 days at 105°C and further decreased with increasing the drying time.

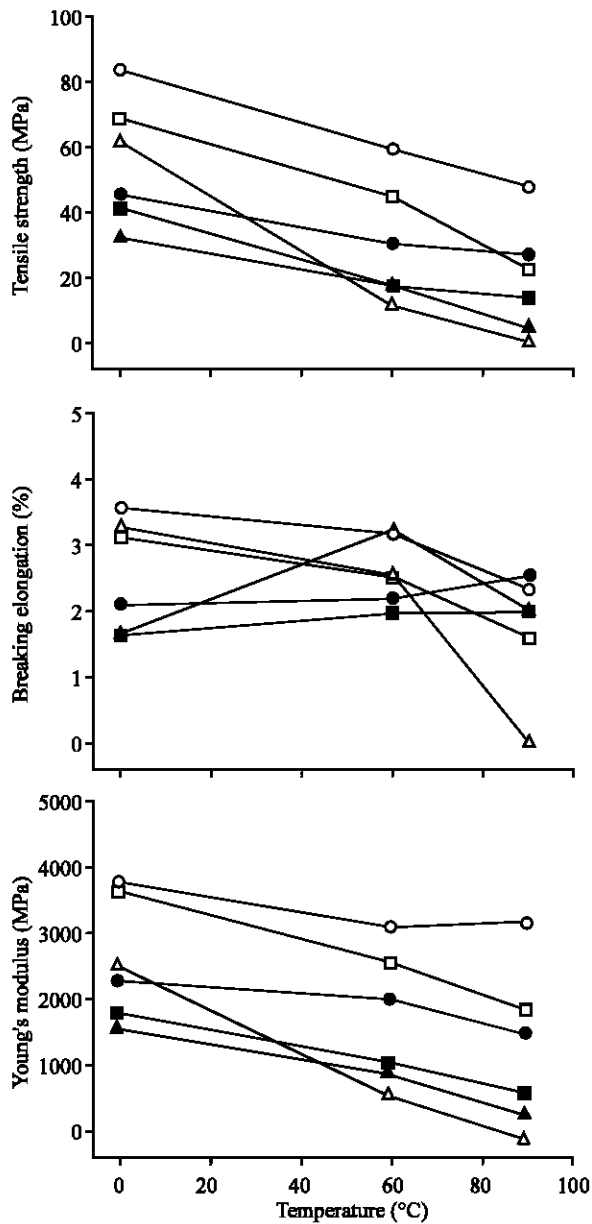


Fig. 2: Tensile properties of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer after immersion in hot water at elevated temperatures (●: PLA/CP; ○: PLA/MPLA/CP; ■: PLA/WF; □: PLA/MPLA/WF; ▲: PLA/CS; △: PLA/MPLA/CS)

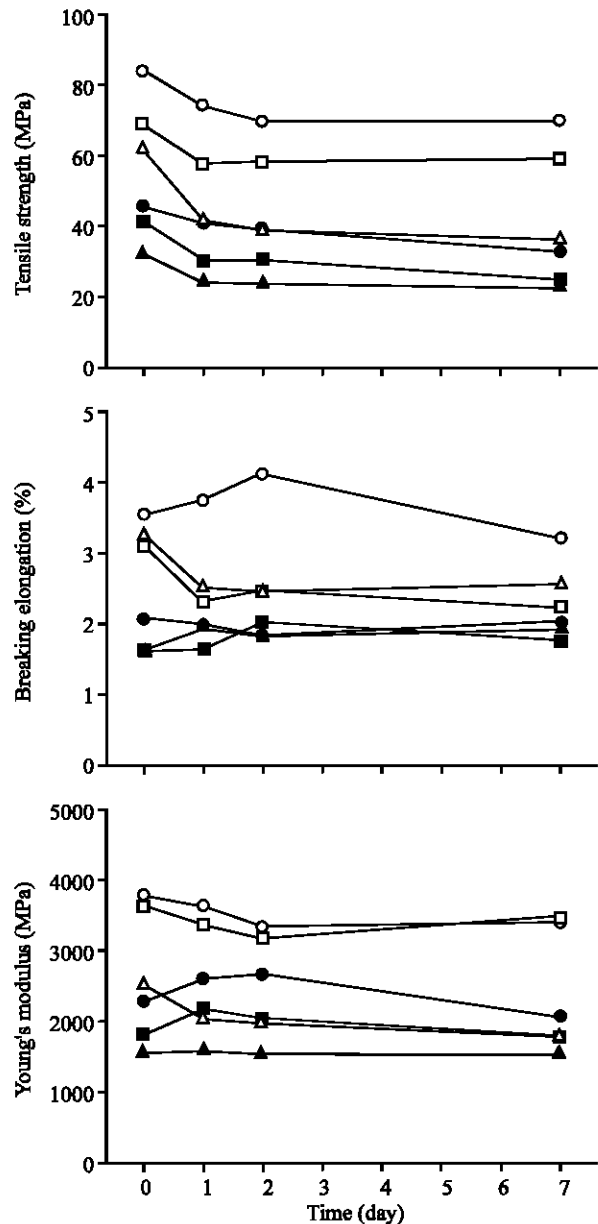


Fig. 3: Tensile properties of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer after immersion in cold water at elevated times (●: PLA/CP; ○: PLA/MPLA/CP; ■: PLA/WF; □: PLA/MPLA/WF; ▲: PLA/CS; △: PLA/MPLA/CS)

**Physical properties of composites under various conditions:** Certain physical properties including water absorption after immersion in cold water for 1,2 and 7 days and the moisture content after 1-12 weeks stored in a room adjusted to 20°C and 60% RH., were also measured.

Figure 6 shows the absorption of water by the composites of PLA-CP, WF and CS with or without MPLA compatibilizer at elevated times. Increasing the immersion time from 1 to 2 days increases the absorption of water for all composites and remains constant or

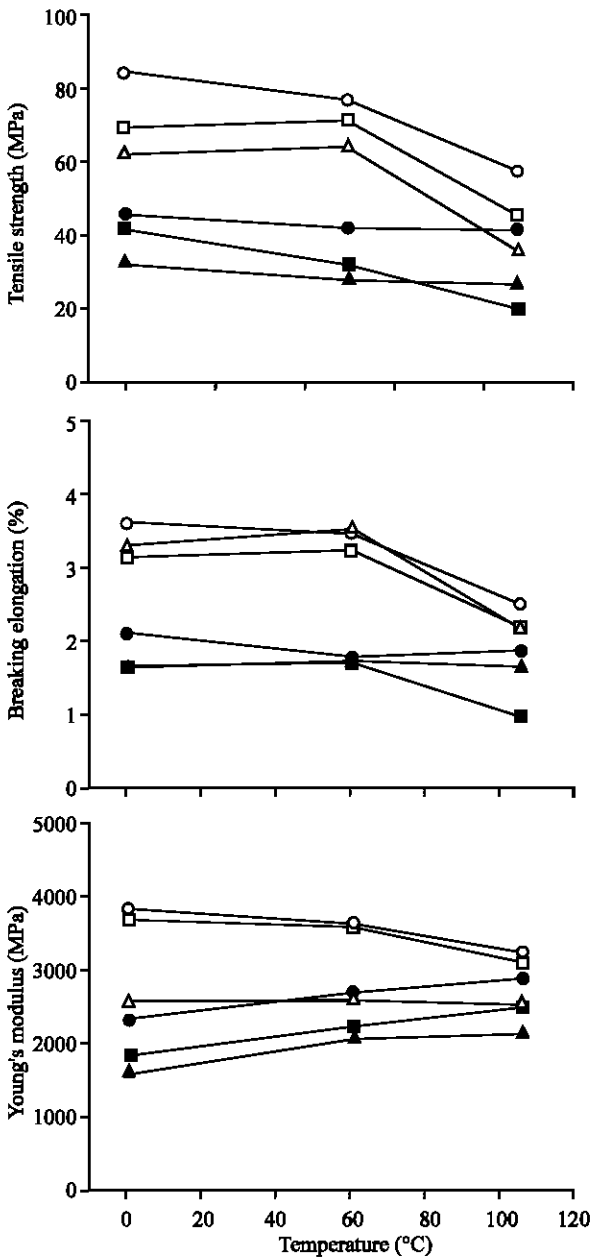


Fig. 4: Tensile properties of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer after drying at elevated temperatures (●: PLA/CP; ○: PLA/MPLA/CP; ■: PLA/WF; □: PLA/MPLA/WF; ▲: PLA/CS; △: PLA/MPLA/CS)

slightly decreases afterwards. The decrease in the water absorption of a part of the samples was due to dissolution of the samples (Table 3). The absorption of water by different fiber-based composites is largely dependent on the availability of free OH-groups on the surface of the fiber. In the WF and CP-based composites with MPLA

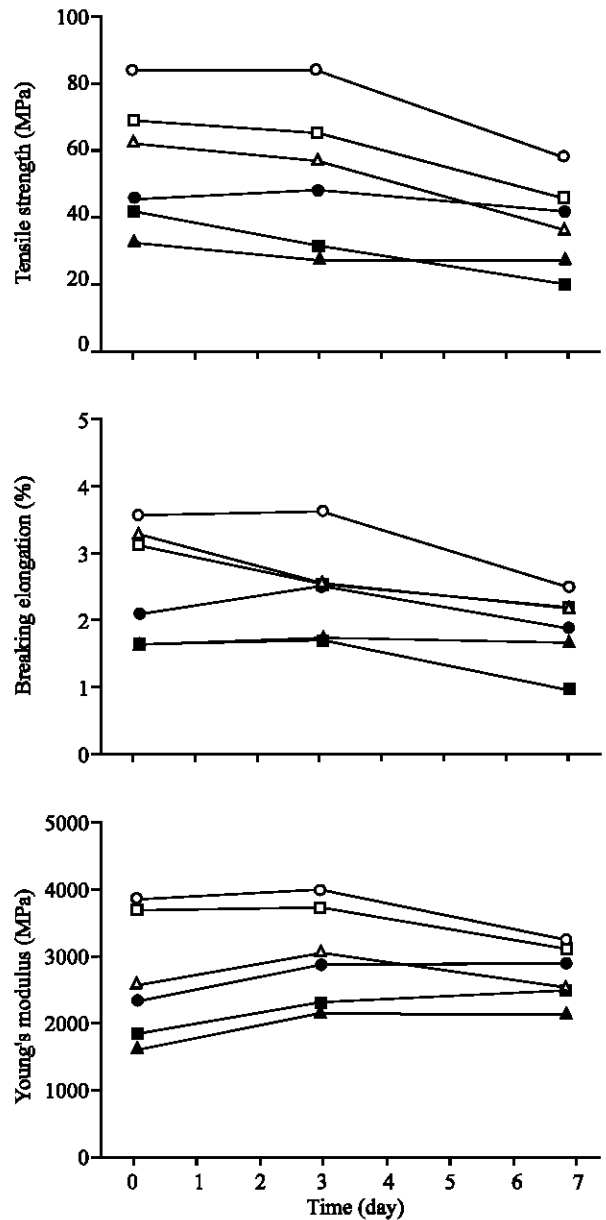


Fig. 5: Tensile properties of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer after drying at elevated times (●: PLA/CP; ○: PLA/MPLA/CP; ■: PLA/WF; □: PLA/MPLA/WF; ▲: PLA/CS; △: PLA/MPLA/CS)

compatibilizer, some of these OH-groups are esterified and due to that, the absorption of water gets restricted. Similar results had been reported by Febrianto *et al.* (1999 and 2006), on the Wood Flour (WF)-*trans*-1.4-isoprene rubber (TIR) composites using maleated *trans*-1.4-isoprene rubber (MTIR) as a compatibilizer and on the WF-recycle polypropylene (RPP) composites using maleic anhydride



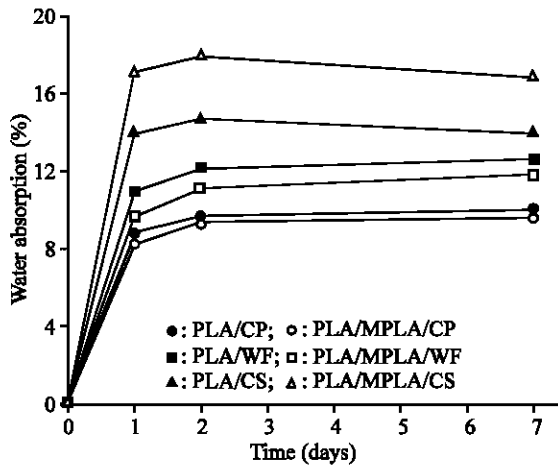


Fig. 6: Water absorption of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer after immersion in cold water at elevated times

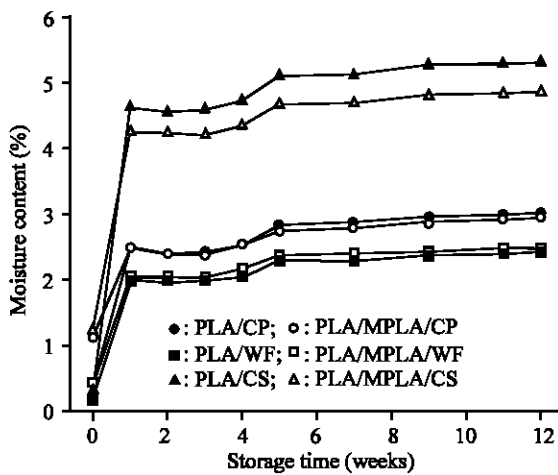


Fig. 7: Moisture content of PLA-CP, WF and CS filled composites with or without MPLA compatibilizer at elevated storage times

(MAH) as a modifier and dicumyl peroxide (DCP) as an initiator, respectively. Furthermore, the absorption of water by CP-composite is lower than WF-based composite. The former is composed of cellulose alone having a larger concentration of OH-groups compared to WF. The larger, the amount of OH-groups, the greater is the possibility of ester linkage formation. On the contrary, the absorption of water by the composites based CS filler with MPLA compatibilizer is higher than its control composites.

Figure 7 shows the Moisture Content (MC) of the composites of PLA-CP, WF and Cs with or without MPLA compatibilizer during prolonged time. After molding, the MC of the composites was very low, that is, around 0-1.5%. Gradually, during storing, the MC increases and

reaches its EMC (equilibrium moisture content) after 5 weeks and remains constant or slightly increases up to 12 week of observation. After 12 weeks of storage time, the MC of composites based on CP-filler is lower than WF and CS based-fillers, respectively. The composites with MPLA compatibilizer tend to give lower moisture content than the compatibilizer-free composites. Although the composites with MPLA compatibilizer have a lower moisture uptake, this value is rather similar to the one exhibited by their control composites, indicating perhaps that this way of chemical modification (esterification) is not able to reduce the water affinity of the fiber fillers to a great extend.

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