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Transverse properties of hemp/PLA composite fabricated with micro-braiding technique

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In the present study, transverse mechanical properties of hemp fiber reinforced poly (lactic acid) (PLA) composites were investigated. Three molding conditions were selected according to a previous study. To discuss the effects of matrix properties on composite properties, monolithic PLA specimens were also prepared at molding temperature of 170, 190, 210, and 230 °C. It is confirmed that the weight-averaged molecular weight affected on the mechanical properties of monolithic PLA and also on the transverse properties of PLA.

Keywords: green composite; transverse property; micro-braiding

1. Introduction

Natural fiber reinforced plastics (NFRP) have attracted much attention as alternatives of glass fiber reinforced plastics, because of their sustainable resource-based materials. At the same time, since mechanical properties of natural fiber are lower, continuous fiber reinforced composites is recommended. In addition, from the view point of shorter processing time, thermoplastics are preferred. In thermoplastics, poly(lactic acid) (PLA), which is naturally derived, is desirable because natural fiber reinforced PLA becomes a carbon-neutral material.

Investigations have been widely conducted on natural fiber composites.[1–12] The authors have investigated unidirectional natural fiber [13,14] and natural fiber textile [15] reinforced PLA composites using micro-braiding technique to improve matrix impregnation to natural fiber yarn. For the actual usage of such kind of composites, mechanical properties of not only longitudinal but also transverse direction are necessary, because of structural design. However, transverse properties of NFRP are not reported frequently.

As investigations using thermoset matrix, de Weyenberg et al. investigated surface modification of natural fiber and processing method for flax/epoxy composites and obtained transverse bending strength 38 MPa and bending modulus 4.1 GPa.[16] Baley et al. focused on transverse damage mechanism of unidirectional flax/polyester composites and reported tensile modulus 3.62 GPa and strength 12.6 MPa.[17]

As investigations using thermoplastics, Madsen et al. reported transverse modulus and strength for flax fiber/poly(propylene) as 1.54 GPa and 6.50 MPa [18] and for hemp/poly (ethylene terephthalate) as 3.5 GPa and 19 MPa,[19] respectively. Herrera-Franco et al.

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investigated henequen/high-density polyethylene and reported transverse flexural strength 20 MPa and modulus 0.35 GPa.[20] However, investigations using PLA as a matrix material have been limited.

The objective of the present study is to clarify the effects of molding condition on transverse mechanical properties of hemp/PLA composites molded with micro-braiding technique. At first, mechanical properties of monolithic PLA, which largely affect transverse properties of the composites, were investigated. Then, transverse properties of the composites are investigated and discussed.

2. Experimental method

2.1. Molding of hemp/PLLA composites and monolithic PLA

In the present study, we use hemp spun yarn (590tex) and PLA yarn (55tex, ecodear 560T-96-NK02, Toray) as a reinforcement and matrix, respectively. Micro-braided yarn (MBY) as an intermediate material was fabricated using a standard braider (Kokubun Ltd). In this case, we use one hemp yarn at the center and eight PLA yarns around the hemp yarn. MBYs fabricated are shown in Figure 1. The diameter of the MBY was about 1 mm. Hemp fiber volume fraction was about 48%.

Fabrication of continuous hemp fiber reinforced PLA thermoplastic composites was twofold process. The first step was performed by initially winding intermediate material 160 times in a parallel configuration onto a metallic frame. The second step involved the placement of the metallic frame containing intermediate materials in a preheated molding die for consolidation by compression molding to produce composite specimens using a hot-press system (IMC-1837, Imoto). After prescribed time elapse, platens of the hot-press system were cooled to 40 °C by the flow of water through the pipes embedded in the platens. In our previous study, enough resin impregnation to hemp fiber yarns was not obtained at 170 °C; severe decomposition of hemp fiber and PLA was observed at 230 °C, and the optimum molding condition for unidirectional hemp/PLA composites was decided as molding temperature 190 °C, molding pressure 1 MPa, and molding time 4 min.[14] In this study, we selected three conditions, such as 190 °C-1 MPa-4 min as a reference, 190 °C-3 MPa-4 min as higher molding pressure, and 210 °C-1 MPa-4 min as lower matrix viscosity. Composite plates obtained were 200 mm long and 160 mm wide.

In order to discuss effects of matrix properties on transverse properties of hemp/PLA composites, monolithic PLA plates were also molded. PLA yarns were cut to less than 5 cm. Then, 45 g PLA yarns were put in a matched die. The mold die was heated to melt PLA yarns for 20 min and was pressed for 5 min. Clamping force was 1.4 kN. After prescribed time, the platens were cooled by flow of water. The molding temperature was 170, 190, 210, and 230 °C. Unfortunately, we cannot conduct the same molding pressure with the composites, because of the different die shapes. Further, we

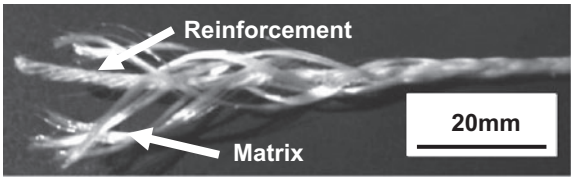

Figure 1. Micro-braided yarn.

cannot conduct the same molding time with the composites as 4 min, which is due to lower thermal conductivity for PLA than for hemp/PLA composites. Though the differences in molding condition, we can roughly evaluate the effect of molding temperature on matrix properties.

2.2. Tensile tests

The composite plates obtained were machined to rectangular shapes using a diamond cutter. Aluminum tabs were glued on both end of the specimen to prevent stress concentration at the grips. The specimen geometries are shown in Figure 2. Note that the thickness of the specimen is different in Figure 2 depending on the molding condition. Tensile tests were conducted using a universal testing machine (AGS-1000A, Shimadzu). Cross-head speed was 1 mm/min. Load and strain were measured using a load cell (capacity: 10 kN) and unidirectional strain gauges (KFG-5, Kyowa) glued on both surfaces of the specimen using cyanoacrylate-based cement (CC-33A, Kyowa), respectively. The signals of load and strain were measured using a digital data logger (GL-220, GRAPHTEC), simultaneously.

2.3. Shear tests

On monolithic PLA specimens, shear tests were also conducted with the Iosipescu method.[21] An Iosipescu apparatus was set on a universal testing machine (AGS-1000A, Shimadzu). The specimen geometry was 18 mm × 60 mm with isosceles notches of 4.5 mm × 4.5 mm in both sides of the center of a specimen, as shown in Figure 3. The notches were inserted using a diamond saw. Then, the edges were polished using a #1000 polishing paper. Compressive loading was applied, which resulted in in-plane shear loading at minimum cross section. Shear stress was calculated from load measured using a load cell (capacity: 10 kN) and minimum cross-sectional area. Similar to the tensile tests, the signals of load were measured using a digital data logger (GL-220, GRAPHTEC), simultaneously. The test speed was 1.0 mm/min.

2.4. Molecular weight measurement

Molecular weight (M_n) distribution of a monolithic PLA specimen was measured using a gel permeation chromatography (GPC) system. The system was composed of column (SHIMPACK GPC-804C, Shimadzu GLC Ltd), column oven (CTO-20A, Shimadzu Co., Ltd), and differential reflective index detector (RID-10A, Shimadzu Co., Ltd).

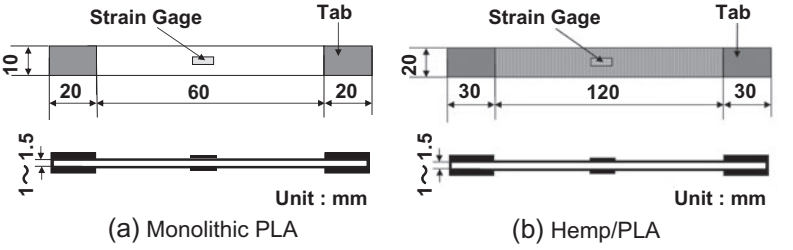

Figure 2. Specimen geometries for tensile test.

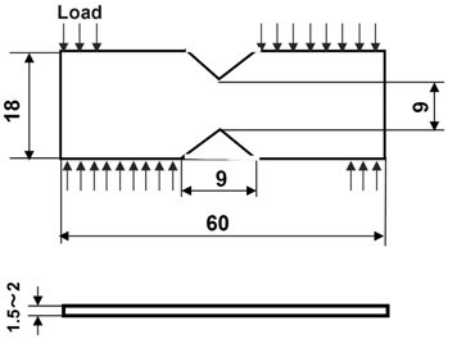

Figure 3. Specimen geometry for shear test.

GPC samples were cut out from the specimens with a weight about 0.5 g. The samples were dissolved in chloroform for 24 h. Then, the solution was filtered before injection in the column.

3. Experimental results and discussion

3.1. Properties of PLA

Figure 4 shows molecular weight distributions measured by GPC. Molecular weight distribution became broader and the peak molecular weight decreased with increasing molding temperature. This is due to pyrolysis of molecular chain. Especially at molding temperature of 210 and 230 °C, molecular chains with molecular weight less than 200 were generated. Such molecule consists of two or three lactic acid molecules and, hence, chain scission occurred around molecular chain end. Figure 5 shows number- and weight-average molecular weights obtained from Figure 4. The weight-average molecular weight linearly decreased with increasing molding temperature up to 210 °C and slightly decreased at 230 °C, whereas number-average molecular weight is similar values at molding temperature 190, 210, and 230 °C. This result suggests that pyrolysis starts at around 190 °C.

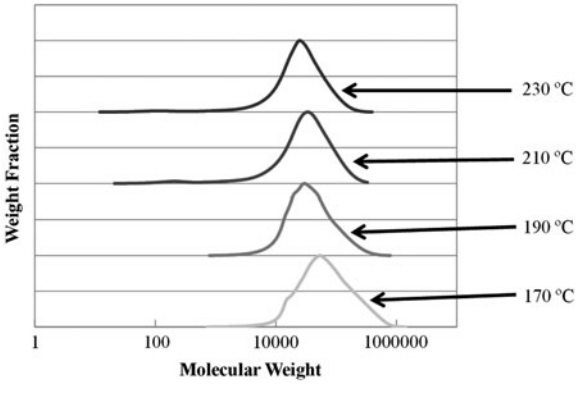

Figure 4. Molecular weight distribution of PLA molded at different temperatures.

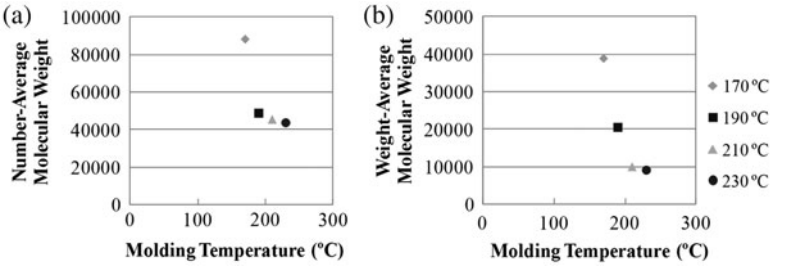

Figure 5. Weight- and number-averaged molecular weight of PLA molded at different temperatures.

Figure 6 shows tensile stress–strain curves for PLA. Stress increased linearly with strain and brittle final fracture occurred. Figures 7–9 show elastic moduli, tensile strengths, and fracture strains for PLA, respectively. Elastic modulus was independent of molding temperature, while tensile strength and fracture strain decreased linearly with molding temperature up to 210 °C and remained constant at 230 °C. This tendency corresponds to that of the weight-average molecular weight. Similar tendency was observed for shear strength as shown in Figure 10. These results suggest that the final fracture of PLA depends on weight-average molecular weight.

3.2. *Transverse properties of hemp/PLA*

Figure 11 shows stress–strain curves for hemp/PLA 90° specimen. All specimens deformed linearly up to 6 MPa. Then, the specimens molded at 190 °C-1 MPa-4 min and 190 °C-3 MPa-4 min exhibit nonlinear deformation, such as plastic deformation, whereas the specimens molded at 210 °C-1 MPa-4 min failed prior to the nonlinear deformation. From the monolithic PLA results which present the linear stress–strain relation until final fracture, the nonlinear response was attributed to hemp fiber yarn and/or hemp/PLA interfacial properties.

Figures 12–14 show elastic moduli, tensile strengths, and fracture strains for hemp/PLA 90° specimen. It seems that elastic modulus have no dependency on the present molding condition. On the other hand, tensile strength and fracture strain decreased with molding pressure and molding temperature. In the case of molding temperature 210 °C,

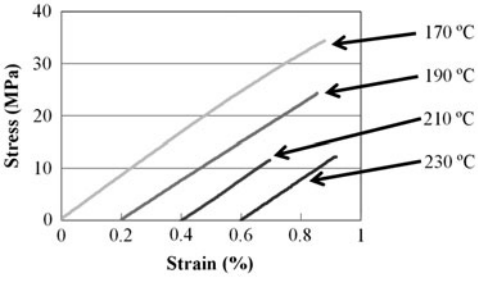

Figure 6. Stress–strain curve of PLA molded at different conditions.

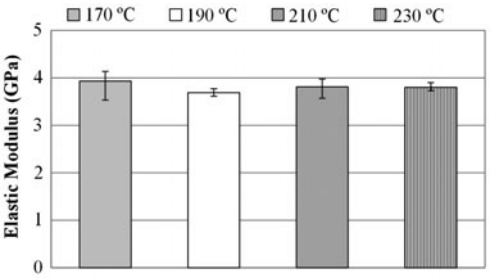

Figure 7. Elastic modulus of PLA molded at different conditions.

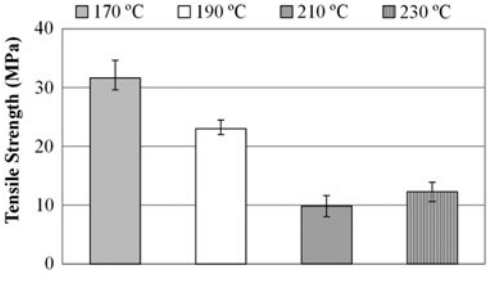

Figure 8. Tensile strength of PLA molded at different conditions.

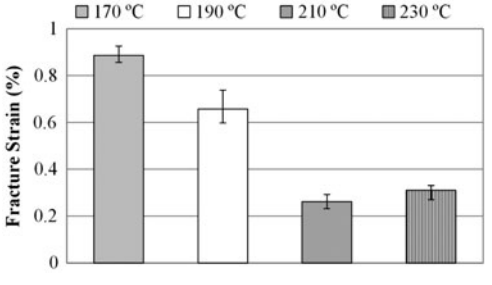

Figure 9. Fracture strain of PLA molded at different conditions.

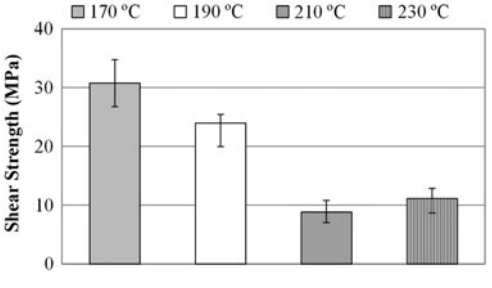

Figure 10. Shear strength of PLA molded at different conditions.

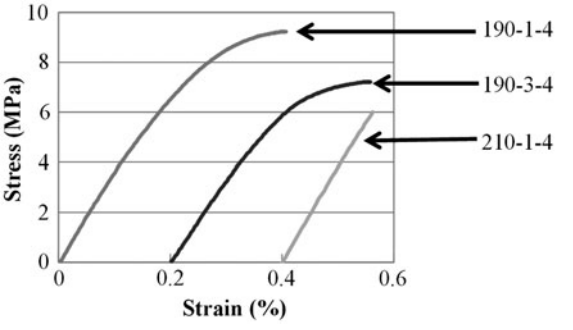

Figure 11. Stress–strain curve of 90° composites molded at different conditions.

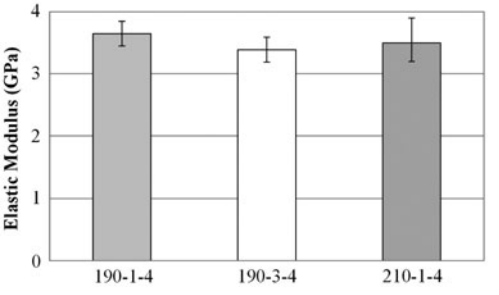

Figure 12. Elastic modulus of 90° composites molded at different conditions.

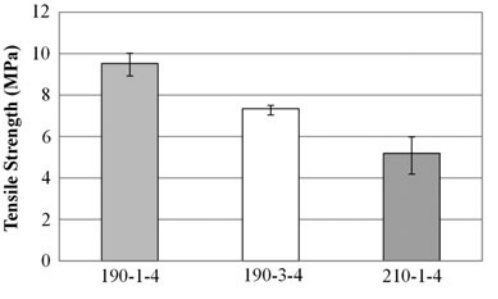

Figure 13. Tensile strength of 90° composites molded at different conditions.

the decrease in molecular weight caused a decrease in strength. At 190 °C, higher molding pressure might have broken the internal structure of hemp fiber. Figure 15 shows the macroscopic images of the composites molded under 190 °C-1 MPa-4 min and 190 °C-3 MPa-4 min. Fiber bridging, which results in higher strength, was observed for the composite molded under the condition of 190 °C-1 MPa-4 min. Figure 16 shows scanning electron microscopic images of a hemp fiber on the fracture surface. Fiber structure was broken in the radial direction in the composite molded at molding pressure 3 MPa. This caused lower stress transfer to hemp fibers and inhibited fiber bridging. In the

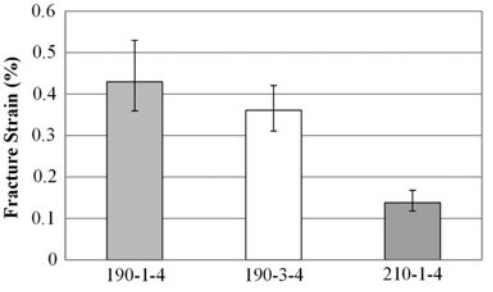

Figure 14. Fracture strain of 90° composites molded at different conditions.

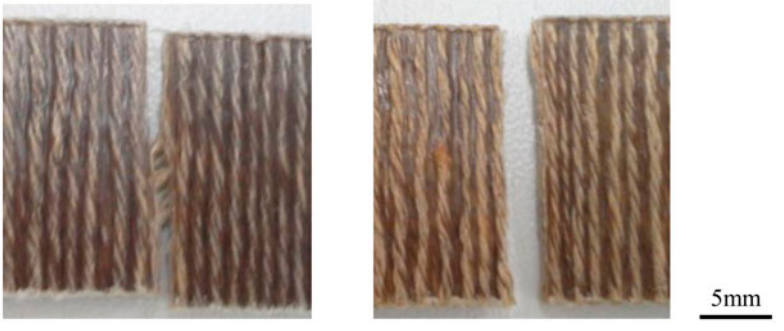

Figure 15. Macroscopic image of 90° composites molded at 190 °C.

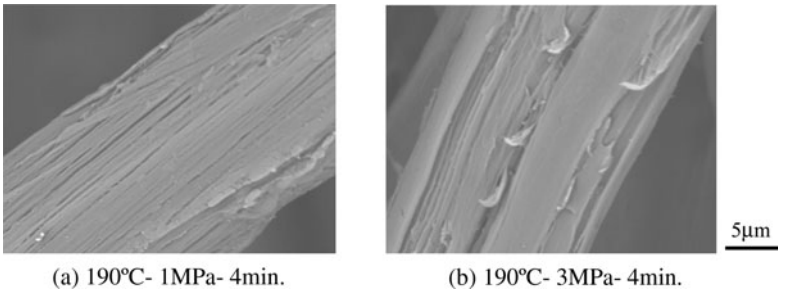

Figure 16. Fiber structure of 90° composites molded at 190 °C observed with a scanning electron microscope.

previous result for longitudinal specimens,[14] no longitudinal strength reduction was confirmed. From the present results, hemp fiber was broken in radial direction but not in longitudinal direction under higher molding pressure.

4. Conclusions

In the present study, in order to clarify effects of molding condition on transverse mechanical properties of hemp/PLA composites, the composites were molded

with micro-braiding technique at molding condition of 190 °C-1 MPa-4 min, 190 °C-3 MPa-4 min, and 210 °C-1 MPa-4 min. Monolithic PLA specimens were also prepared to investigate the effect of matrix properties on transverse properties of composites. It is clarifies that mechanical properties of PLA and associated transverse properties of hemp/PLA composites are largely affected by weight-averaged molecular weight of PLA. In addition, larger molding pressure tends to destruct hemp fiber radially, but not axially. Optimum molding condition for transverse properties is decided as molding temperature 190 °C, molding pressure 1 MPa, and molding time 4 min. Other factors affecting mechanical properties of the composites include cooling rate during molding. The cooling rate plays an important role of crystallization and introduction of residual stress, and the effect of the cooling rate will be our future work.

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