

EFFECTS OF INJECTION TEMPERATURE ON MECHANICAL PROPERTIES OF BAGASSE/POLYPROPYLENE INJECTION MOLDING COMPOSITES

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Effects of injection temperature on thermal degradation and porosity of the bagasse/polypropylene injection molding composites were studied. Above 185 °C, incomplete filling occurred. The incomplete filling increased with increase of injection temperature. It was found that the gas generated by thermal degradation of bagasse fibers was so accumulated in the injection cylinder that the injected composites ended up with incomplete filling. A modified injection method with the venting of gas increased the complete filling percentage. Mechanical properties decreased with increase of injection temperature from 165 °C to 260 °C. This was due to increase of porosity and fiber shortening. The calculated flexural modulus, which incorporated the effect of porosity and fiber length, agreed well with the experimental results. Composites with maleic acid anhydride grafted polypropylene (MAPP) were also investigated. Flexural strength and impact strength were improved by 45% and 35%, respectively, by addition of 20wt% MAPP. In the MAPP composites, fiber breakages at their roots were observed in the fracture surface after an impact test, while pulled-off fibers were observed in those without MAPP.

Keywords: Bagasse; Natural fiber; Composites; Injection molding; Thermal degradation; Flexural modulus; Mechanical properties

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INTRODUCTION

Natural fiber reinforced composites, such as bagasse, kenaf, jute, hemp, and polyolefin polymer composites, have a potential to replace traditional glass fiber reinforced composites due to their strength and light weight (Huda et al. 2005; Kuo et al 2009; Wollerdorfer et al. 1998; Karmaker et al. 1996; Bogoeva-Gaceva et al. 2007; Gassan et al. 2002; Keller et al. 2003; Herrera-Franco et al. 2004; Luz et al. 2007; Pickering et al. 2007; Mohanty et al. 2004; Chow et al. 2007). In fact, a number of auto components are presently being fabricated from natural fiber composites. The authors have been studying natural fiber composites in view of mechanical properties. In previous papers (Shibata et al 2005, 2006, 2008), it was found that the flexural modulus in natural fiber composites is well predicted by modified Cox's model (Cox et al. 1952; Hull 1982; Fukuda et al. 1981; Fukuda et al. 1982, Thomson 1996; Thomson 2002; Bos et al. 2006; Ganster et al. 2006), which incorporates fiber compression in addition to well known parameters, such as fiber length, Young's modulus, fiber orientation distribution.

The cross sectional structure of natural fiber is porous and compressed by compressive pressure. As a result, the compression ratio was revealed as 1.2-1.5 for bamboo, 1.4-1.7 for kenaf, and 2.8-3.5 for bagasse.

On the other hand, many studies on natural fiber composites have been performed in view of mechanical properties. These composites were mainly fabricated with a conventional hot pressing. Although injection molding is a productive method, injection molding parameters have not received enough study. Kuo et al. (2009) examined the effect of the material compositions of wood-plastic composite by injection molding. Wollerdorfer et al. (1998) reported that the fiber length in natural fiber composites was shortened after injection molding. Karmaker et al. (1996) revealed that 3-3.5 mm original fiber length was shortened to 0.39 mm by injection molding processing due to high fiber attrition. The fiber length as well as fiber weight fraction and orientation distribution is an important factor affecting flexural modulus and strength in fiber reinforced composites. In particular, Bos et al. (2006) indicated that with a mean fiber length between 0.1mm and 3mm in natural fiber composites, the Young's modulus increased 1GPa to 4GPa at 20 fiber vol% and 1GPa to 7GPa at 40 fiber vol%.

The injection molding of natural fiber composites can be performed at rather high injection temperature due to low melt flow index (MI), especially at high values of fiber fraction (Kuo et al. 2009). According to Fung et al. (2003), at a high injection temperature of more than 200 °C, thermal degradation of natural fiber occurred and the composites were found to be darkened. Also, an odour could remain in the long term in the composites specimen. Thus, in the present study the effects of injection temperature on mechanical properties of natural fiber composites were examined. Moreover, a modified injection procedure by gas vent was performed to improve the complete filling. To improve flexural strength and impact strength, MAPP (0.5wt% maleic acid anhydride grafted polypropylene) was added to the composites before injection molding.

EXPERIMENTAL

Materials

Bagasse that had been obtained as the residue after liquor extraction in a sugar mill was dried for 72h at 80°C. The bagasse was sieved, and the pith was removed. The average fiber diameter was 0.390 mm. Polypropylene (PP, AZ864-N, Mw 162000, Mw/Mn 6.1) as a matrix was gratefully received from Sumitomo Chemical Co., Ltd, Japan. The MAPP, which was 0.5wt% maleic acid grafted to polypropylene, was used in order to improve the surface adhesion between polypropylene and bagasse fibers. The mechanical properties of these fiber and PP, which were measured by the authors, are shown in Table 1.

Table 1. Mechanical Properties of Bagasse and Polypropylene.

	Young's modulus (MPa)	Tensile strength (MPa)	Specific gravity (kg/m ³)	Diameter (mm)	Fiber distribution η_0
Bagasse	4517	89	344	0.39	0.75
Polypropylene	1315	24	902	-	-

Composite pellets were prepared as in the following experimental procedure. Each fiber and PP was compounded with a double arm kneading mixer (type S5-2, Moriyama Co. Ltd., Japan) at 190°C for 1h. Then, the compound was pulverized with the mixer for 6h into pellets (2-5 mm in diameters) during cooling. These compounds were prepared at 10, 20, 30, 40, and 50wt % fiber weight fractions in order to investigate the effect of fiber weight fraction on the mechanical properties in the composites.

The pellets were molded into specimens by a 10-ton injection molding machine (type PS10E1ASE, Nissei Co. Ltd., Japan). The injection was carried out at injection temperature (at nozzle temperature) of 165 to 260°C, a hold pressure 11M Pa, a mould temperature 90°C, a cooling time 7 sec, a screw rotation speed 50 rev./min, and an injection speed 0.5 sec/shot. The prepared composites were a cylindrical shape, 60 mm in length and 5.8 mm in diameter, and subjected to the following mechanical tests.

Methods

Mechanical tests

Three-point flexural tests were conducted in accordance with ISO 178 specifications for at least five specimens per condition. The dimensions of specimens and span length were ϕ 6x5.8 mm and 48 mm. The cross-head speed was 1 mm/min. The flexural modulus was determined by the initial derivative of the load-displacement curve. The derivative part was from 0 to 15% of the maximum load. The flexural strength was calculated by maximum load.

The Charpy impact test was carried out with a U-F Impact Tester (Ueshima Seisakusyo Co. Ltd, Japan), using the standard method (ISO 179) with a hammer weight of 0.6 kgf and a falling height of 0.325 m. The unnotched specimen was mounted on the span of 20 mm. More than five specimens were conducted in each injection condition. The impact strength was defined as an absorption energy which was calculated by,

$$E = Wr (\cos \alpha - \cos \beta) \quad (1)$$

where E , W , r , α , and β denote the impact absorption energy, the weight of the hammer, the distance between the center of the rotational axis of the hammer and the center of mass of the specimen, the angle of rise without the specimen, and the angle of rise with the specimen.

The morphologies of fibers and the fracture surface of composites were observed by SEM, using a WS-250 (Topcon Co., Japan). All samples were sputter-coated with gold to provide enhanced conductivity, and they were studied with the microscope operating at 25kV.

Calculation of flexural modulus

It is well known (Pickering et al. 2007; Mohanty et al. 2004) that Young's modulus of a short fiber reinforced composite, E_{comp} , is determined by

$$E_{comp} = \eta_s \eta_f V_f \cdot E_f + (1 - V_f) \cdot E_m \quad (2)$$

where E_f , E_m , and V_f denote the Young's modulus of the fiber, the matrix, and the volume fraction of the fiber in the composite. The terms η_f and η_θ denote efficiency factors of fiber length and orientation. The factor η_f is given (Fukuda et al. 1981, 1982) by

$$\eta_f = 1 - (\tanh \frac{1}{2} \beta L) / \frac{1}{2} \beta L \quad (3)$$

$$\beta = \left(\frac{2 G_m}{E_f r_f^2 \ln(R / r_f)} \right)^{\frac{1}{2}} \quad (4)$$

Here, the fiber length is L , where r_f and R denote the radius of the fiber and the interval among fibers, and G_m is the shear modulus, with the assumption that the fibers is homogeneous in an ideal packing square composite.

The value of η_θ has been determined as 0.75 in a previous article (Shibata et al. 2006). K is the fiber compression ratio and indicates the fiber concentration of bulk density by,

$$K = V'_f / \left[V - \left(\frac{W - W_f}{\rho_m} \right) \right] \quad (5)$$

where V'_f , V , W , W_f , and ρ_m denote the original fiber volume, the volume of the composite, the weight of the composite, the weight of the fiber, and the density of the matrix, respectively. Therefore, the final equation that predicts the flexural modulus of the composite is

$$E_{comp} = K \eta_\theta \eta_f V_f \cdot E_f + (1 - V_f) \cdot E_m \quad (6)$$

In a published article (Shibata et al. 2006), it has been revealed that the calculated flexural modulus in natural fiber composites according to Eq. (6) agrees well with experimental results.

RESULTS AND DISCUSSION

Effects of Injection Temperature on the Complete Filling Percentage of Bagasse/Polypropylene Composites

Figure 1 shows the relationship between flexural modulus, flexural strength, impact strength, and bagasse fiber weight fraction. The injection molding was performed at 185 °C. The results in the figure were calculated by Eq. (6) using the modified Cox's model, which incorporates fiber compression ratio. Fiber compression ratios in fiber

weight fractions of 10, 20, 30, 40, and 50 wt% were 2.76, 3.174, 3.38, 3.36, and 3.54, respectively. These values were calculated by Eq. (5), the specific gravities of the composites. Other parameters are shown in Table 1; these were measured in the already published articles (Shibata et al. 2006, 2008). As shown in the Fig. 1, flexural modulus increased with fiber weight fraction according to the rules of mixture. The maximum flexural modulus at 50 wt% reached 3500 MPa. As shown in Fig. 1, the experimental and calculated flexural modulus agreed well.

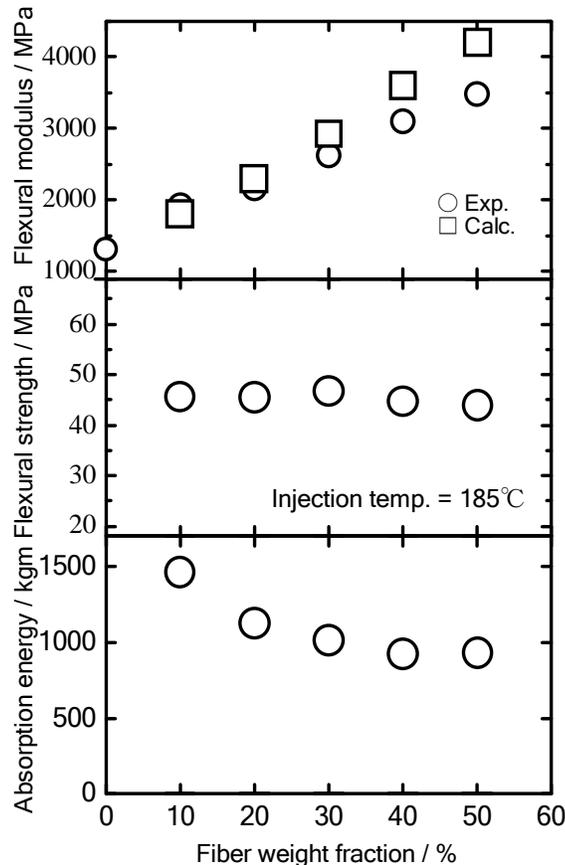


Fig. 1. Relationship between mechanical properties and fiber weight fraction

Figure 2 shows the appearances of injected bagasse/polypropylene composites at 185°C and 225°C injection temperatures. In Fig. 2(a), the perpendicular part in the Fig. is the injection sprue, and the horizontal part is the specimen for mechanical tests. The end of the specimen is the air vent part. As shown in the figure, in the case of Fig. 2(b) at 225°C, the specimen was incompletely filled. The complete filling percentage dramatically decreased with increasing injection temperature, as shown in Fig. 3. Here, the complete filling percentage was defined as the percentage of specimens with the air vent part in the total number of specimens, as shown in Fig. 2(a). At 260°C, the percentage was extremely low, approximately 1%.

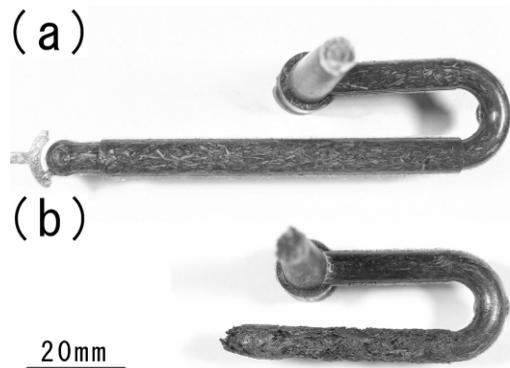


Fig. 2. Appearances of injection molding composites (a) at 185 °C, and (b) at 220 °C. The perpendicular part is the sprue and the horizontal part is the specimen part.

Figure 4 shows the relationship between injected weight and injection temperature. The injected weight was measured from 165°C to 260°C at five attempts without touching the cylinder nozzle to the mould. All of the injected weights were more than 4.5 g, which is the horizontal line in the Fig., at all the injection temperatures. The weight of the specimen in Fig. 2 (a) was 4.5 g. Thus, judging from the result of the injected weight measurement, all specimens at the temperatures should be completely filled. However, it was found that generated gas was continuously blown off at the injection nozzle during the measurement of injected weights. Therefore, we modified the injection procedure from Fig. 5(a) to 5(b). Normal injection molding steps are as follows, (1) mould clamp, (2) injection, (3) holding, (4) cooling and filling up the cylinder, and (5) mould release. In the modified procedure, filling up the cylinder was performed after the mould release after ejection of the filled composite in the mould. In this procedure, the generated gas was exhausted by emptying the specimen in the mould. The complete filling percentage dramatically increased in the modified procedure, as shown in Fig. 3. The complete filling percentage above 225°C was still less than 100%. This might be attributed to the fact that the gas was so increased and not completely exhausted. After this, all the specimens were fabricated with the modified procedure, and only complete specimens were provided for mechanical tests.

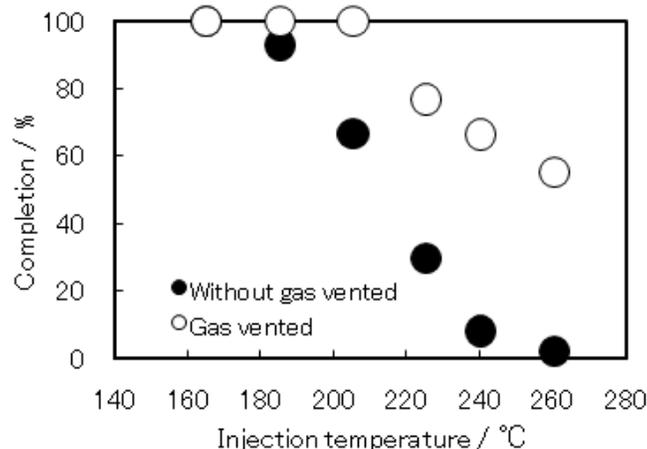


Fig. 3. Relation between injection temperature and completion percentage of the filling of the bagasse/polypropylene composites

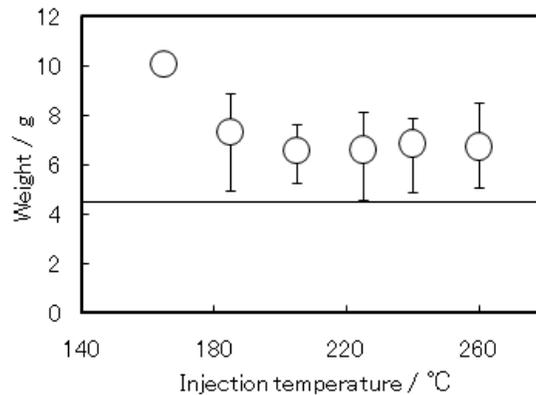


Fig. 4. Relation between injection temperature and weight of the injected composite from the injection nozzle

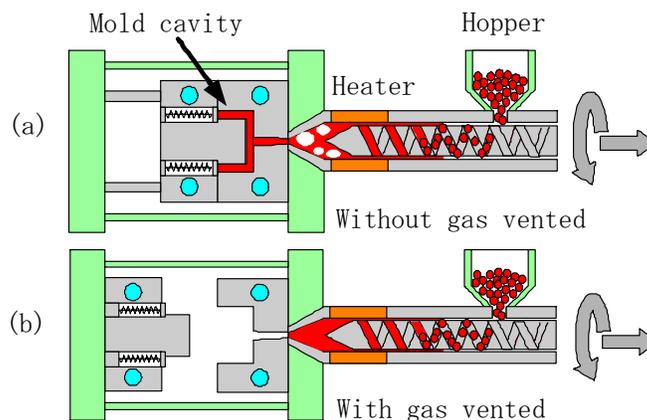


Fig. 5. Injection procedure (a) without gas vented, and modified procedure (b) with gas vented. The view of (b) shows the modified process that let gas vent during filling up the composite in the cylinder for the next injection after ejection of the specimen, while the view of (a) filling up the composite with keeping the specimen in the mold cavity.

Effects of Injection Temperature on Mechanical Properties of Composites

Figure 6 shows the relationship between mechanical properties of composites and injection temperature. The injection temperatures ranged from 165°C to 260°C, which corresponded to the range recommended by the supplier of the equipment. All the mechanical properties decreased with increase of injection temperature. The intervals in the figure were injection interval time from shot to shot. During the interval, melted composite had been kept in the injection cylinder. The cylindrical parts of the specimens injected at 185°C and 225°C are shown in Fig. 7. The specimen injected at 225°C was clearly darkened, and its fibers lengths were shorter than those injected at 185°C. The difference in color indicates that thermal degradation of bagasse fiber had occurred. Figure 8 shows the hot pressed specimens by bagasse/polypropylene composites. Item (a) shows the original pellet specimens, and (b) through (d) are the hot-pressed specimens after injection at 185°C to 260°C. It was found that the fibers lengths (b) through (d) were apparently shorter than those in original pellets (a). Moreover, at higher temperature, the

fiber length decreased. Thus, it is considered that the bagasse fibers were shortened by fiber attrition and mechanical shearing in the cylinder, as indicated by Karmaker et al. (1996).

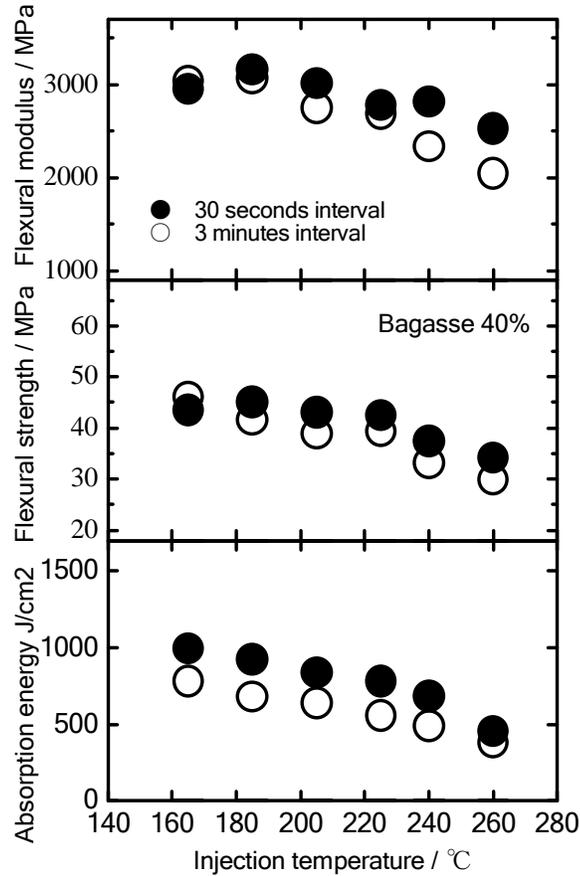


Fig. 6. Relationship between mechanical properties and injection temperature

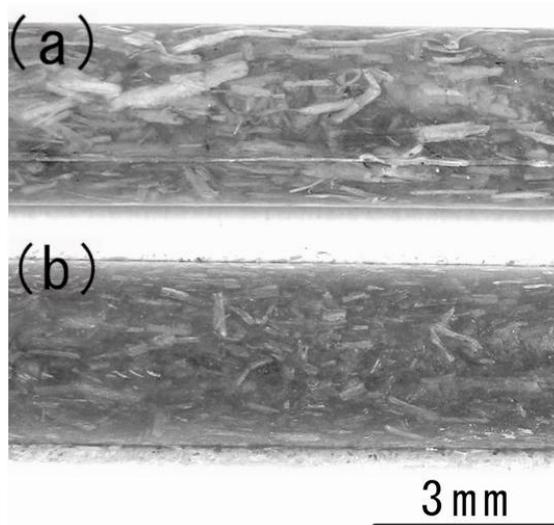


Fig. 7. Appearances of bagasse/polypropylene composites (a) 185 °C and (b) 225 °C

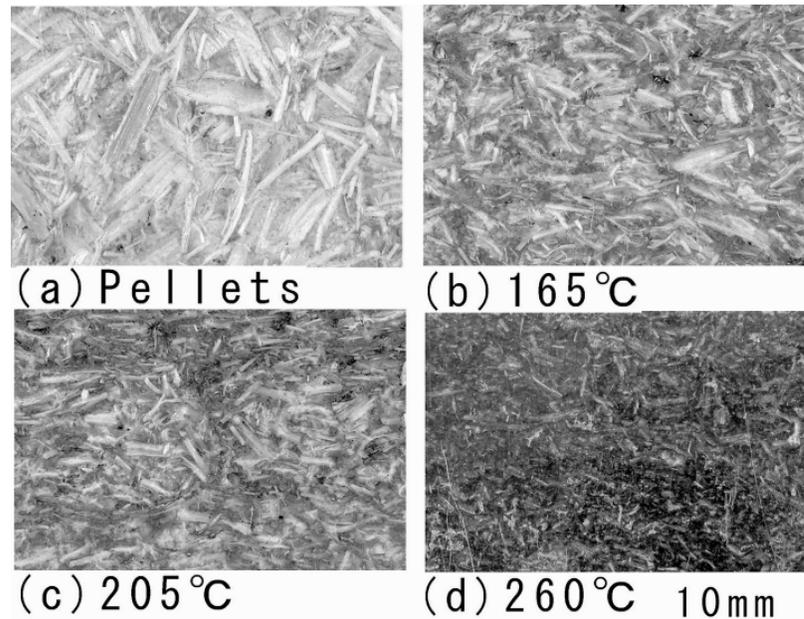


Fig. 8. Microphotographs of bagasse/polypropylene composites after hot pressing

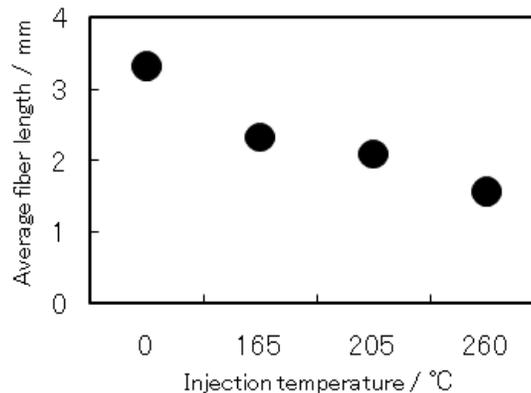


Fig. 9. Relationship between average fiber length in composites and injection temperature

Furthermore, the matrix color was more darkened, and smoky odors became evident with increasing temperature, as observed by Espert et al. (2005) and Tsai et al. (2006). Figure 9 shows the relationship between average fiber length and injection temperature. 300 fibers on each composite surface were measured by an optical microscope. The average fiber length, which had been 3.3 mm in the original pellets, was shortened to 2.1 mm at 165°C and 1.5 mm at 260°C. Generally, there was a strong correlation between fiber length and flexural modulus in composites. Fig. 10 shows the calculated result for flexural modulus by Eq. (6) as a function of fiber length at 40wt% bagasse/polypropylene composites. According to the obtained result, the flexural modulus decreased from 3000 MPa at 2.1 mm to 2600 MPa at 1.5 mm. Thus, the decrease of flexural modulus by fiber length shortening was estimated at 400 MPa. However, the difference between 165°C and 260°C in Fig. 6 was 700-1200 MPa.

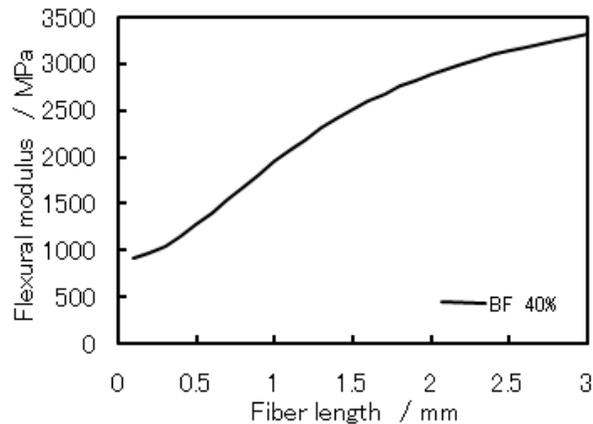


Fig. 10. Calculated flexural modulus by the modified Cox model as a function of fiber length in bagasse composites

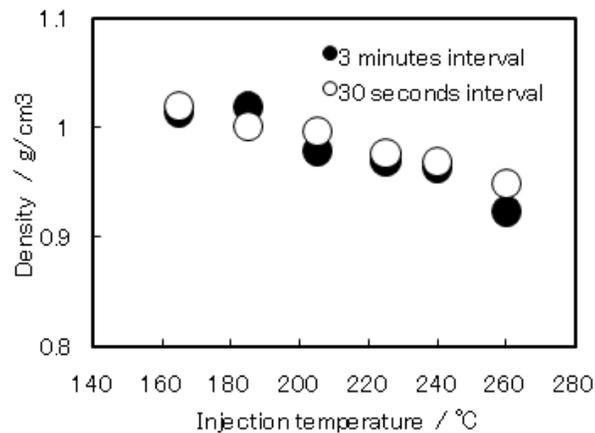


Fig. 11. Relationship between density of bagasse/polypropylene composites and injection temperature

Also, it was expected that the porosity in the composites was generated by the thermal degradation gas when bagasse fibers were heated at thermal degradation temperatures above 200°C. Figure 11 shows the relationship between specific gravity in 40wt% specimens and injection temperature. The specific gravity decreased clearly with increasing injection temperature. The decrease percentage at 260°C was 15% in comparison to that at 165°C. Thus, the fiber degradation is considered to generate the porosity and decrease the specific gravity in the composites.

Figure 12 shows SEM micrographs of the fracture surfaces just after the impact test in 40 wt% specimens at (a) 165°C and (b) 260°C. At 165°C there were some bagasse fibers pulled out, leaving holes behind, as indicated by the white arrows in the figure. On the other hand, at 260°C, no pulled-out fibers were found. The fibers were cut at those roots due to their brittleness, which was a result of thermal degradation. Also, in the fracture surface, there were small cavities that were generated by the gas evolved during fiber thermal degradation. Hence, at high injection temperature, the thermal degradation of fibers not only affects fiber length and brittleness, but also the porosity. This is the reason why the flexural strength, modulus, and impact strength all decreased.

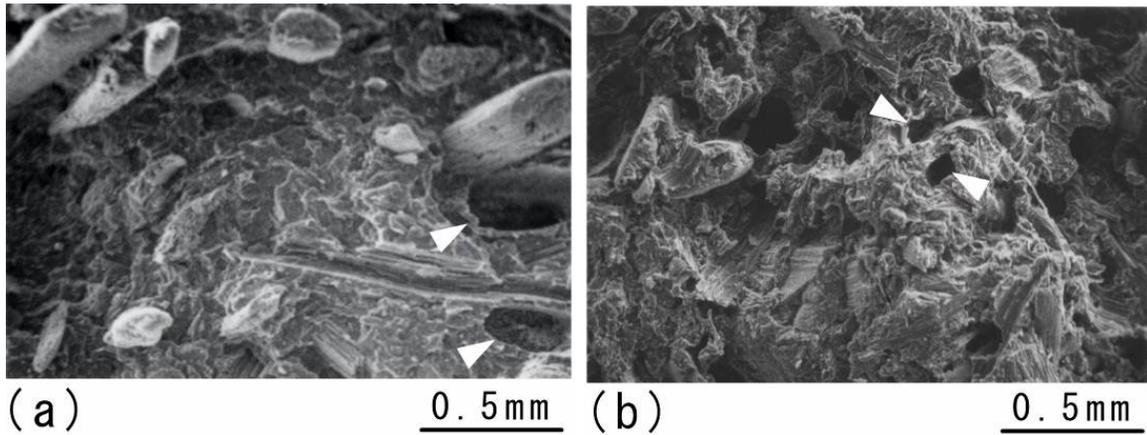


Fig. 12. Fracture surfaces of bagasse composites after impact test (a) 165 °C and (b) 260 °C

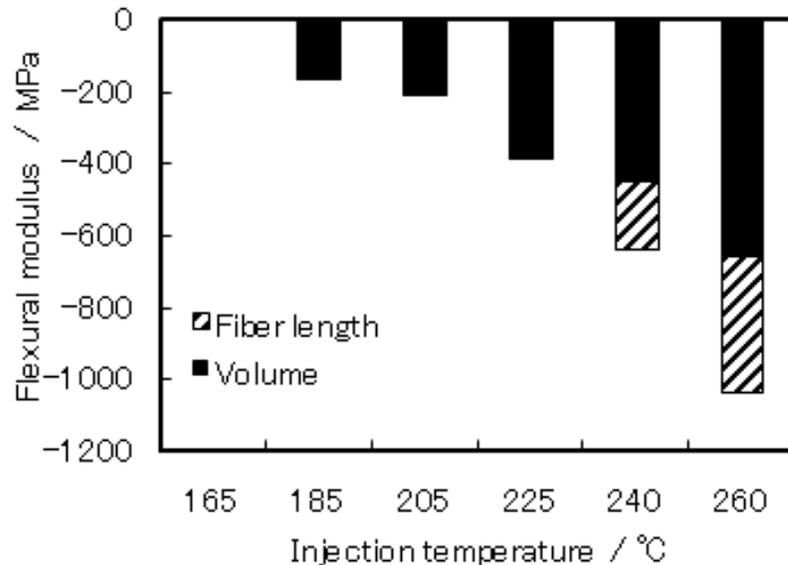


Fig. 13. Relationship between calculated flexural modulus incorporating the effect of both fiber length and composites density as a function of injection temperature

Figure 13 shows the relationship between decrease of flexural modulus by calculation, which incorporates the effects of fiber length, and specific gravity, and injection temperature. The fiber volume in Eq. (6) was reduced by the decrease percentage of specific gravity, as shown in Fig. 12. The decrease of flexural modulus by fiber length was calculated, and results are shown in Fig. 9. As shown, the decrease of flexural modulus reached 1050MPa at 260°C. The decrease in experimental results (Fig. 6) was approximately from 700MPa to 1200MPa at 260°C. Therefore, these results indicate that the prediction of flexural modulus in natural fiber composites needs to consider porosity and fiber length change in injection molding at high temperature.

Effect of MAHPP on Mechanical Properties in Bagasse/Polypropylene Composites

In Fig. 1, the effects of bagasse fiber fraction on flexural modulus, flexural strength and impact strength are shown. Based on results of the experiments, the flexural modulus increased up to 3500MPa at 50wt% fiber fraction. However, flexural strength did not increase, and impact strength decreased with increasing fiber fraction. According to the rule of mixture, flexural strength should increase with fiber fraction because the fiber strength is 89MPa, which is very much higher than that of the matrix, 24 MPa. Hence, the interfacial bond between fiber and matrix was considered not enough to take advantage of the inherent fiber strength.

Therefore, a well known interfacial active agent (Karmaker et al. 1996; Pickering et al. 2007; Ganster et al. 2006), polypropylene grafted with maleic acid anhydride (MAPP) was added to the composites for the purpose of improving the bending strength and impact strength. The MAPP was 0.5wt% maleic acid grafted to polypropylene. This MAPP was mixed and kneaded with polypropylene and bagasse into pellets. Thereafter, the pellets were manufactured to form the cylindrical specimens, as described in the experimental section. Figure 14 shows the results of flexural and impact tests. 10 to 20wt% MAPP addition clearly improved these properties. In particular, flexural strength increased from 45MPa to 63MPa (45%), and impact strength also increased from 820 J/cm² to 1120J/cm² (35%).

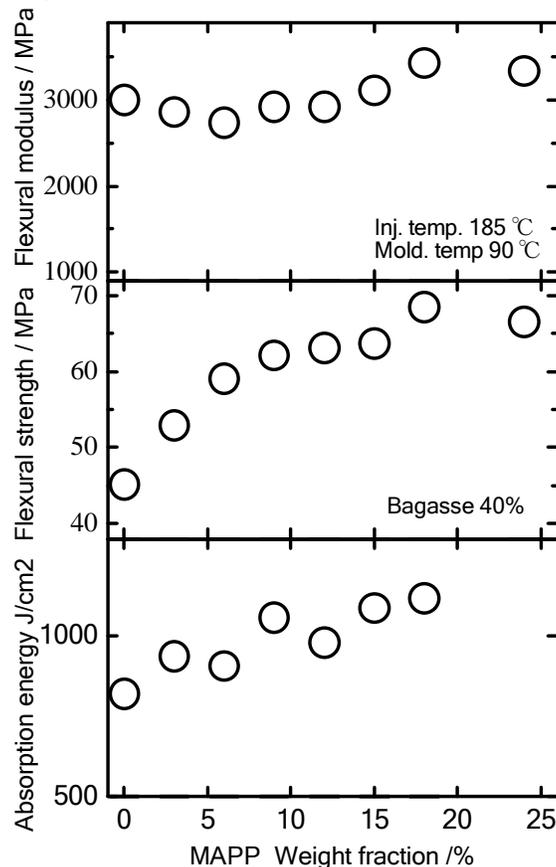


Fig. 14. Relationship between mechanical properties and MAPP weight fraction

Figure 15 shows the fracture surfaces at 40wt% fiber without the MAPP and those with the MAPP 20wt% after an impact test. Pulled-out fibers were found in the case of specimens without MAPP; however, fibers were broken at their roots with MAPP 20wt%. Hence, the interfacial bond between fiber and matrix was improved. This is the reason why the flexural strength and the impact strength increased with MAPP fraction. However, there was not much effect on flexural modulus. This might be attributed to the measurement method of flexural modulus. The flexural modulus was measured by the initial derivative in the load-displacement curve. The initial part was defined as 15% load of maximum load as described in the experimental section. Thus, the initial stage of bending tests, the influence of the interfacial bond on the inclination was probably small. In this reason, the flexural modulus showed only a slight increase with MAPP addition.

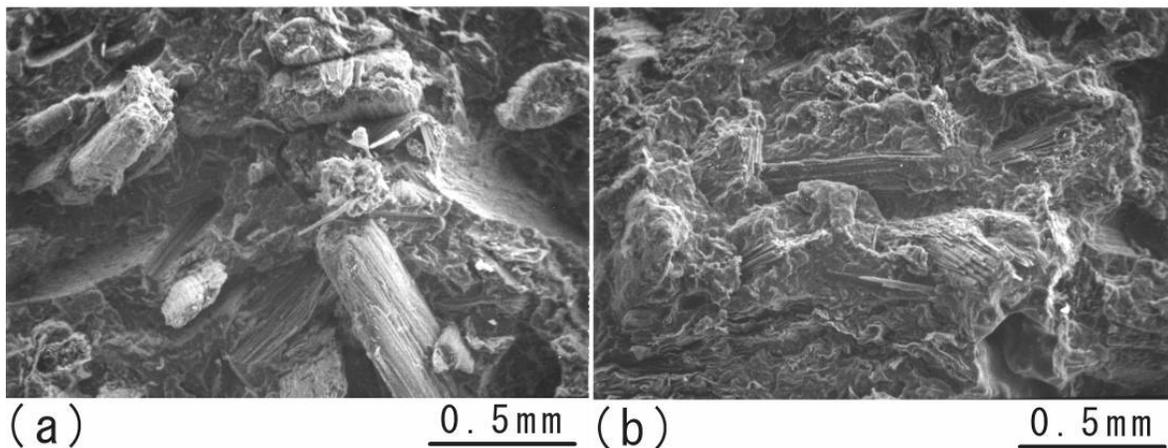


Fig. 15. Fracture surfaces of bagasse composites after impact test (a) BF40 % without MAPP, and (b) BF40% with MAPP 20%

CONCLUSIONS

Effects of injection temperature on fiber degradation and porosity of bagasse/polypropylene composites were examined both in experimental mode and by means of numerical calculations. The following conclusions were obtained.

1. At injection temperatures above 185°C, the complete filling percentage decreased. This was due to the gas generated by fiber thermal degradation. The venting of the evolved gas dramatically improved the completeness of filling during injection molding.
2. Thermal degradation caused fiber shortening and porosity in the composites. These fiber shortening and porosity changes resulted in decreased mechanical properties. The prediction of flexural modulus by modified Cox's model, which incorporates the effect of porosity and fiber length, agreed well with the experimental results.
3. The addition of MAPP at a 20wt% level to bagasse and polypropylene composites led to increases of 45% in flexural strength, and 35% in impact strength, while the

flexural strength was constant and the impact strength decreased in the composites without MAPP. The SEM micrograph showed that fibers were pulled out in the absence of MAPP and that fibers were broken at their roots when MAPP was present in the matrix, at each fracture surface. This was because the interfacial bond between fiber and matrix was improved with the MAPP agent.

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