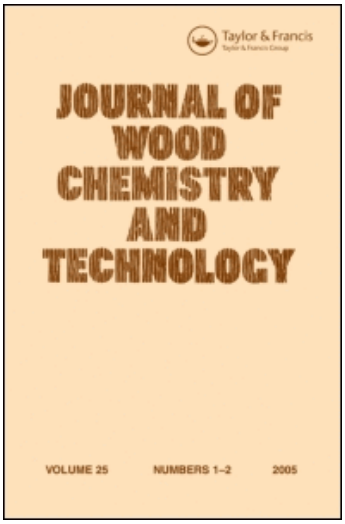

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Abstract: The effects of resin content (10–30%), cellulose fiber length (120 and 300 micrometer), and molding conditions (press, injection, and extrusion molding) on the thermal expansion of high filler content cellulose/polypropylene composites were evaluated. Other physical properties such as densities, bending strengths, and water absorption of composites were also determined. The results indicated that thermal expansion of composites is dependent on the above conditions. Small thermal expansion was observed for composites with low resin (high cellulose) content. Composites with long fibers (300 micrometer) showed smaller thermal expansion than those of 120 micrometer, except for injection-molded composites with 20% resin content. Composites prepared by injection and extrusion molding showed anisotropy of thermal expansion depending on the parallel and perpendicular directions of molding. The results of thermal expansion are discussed in the light of other physical properties and the interaction of fibers in the composites.

Keywords: Cellulose, composite, interaction of fibers, polypropylene, thermal expansion

INTRODUCTION

Wood plastic composite (WPC), which uses wood flour or cellulosic fiber as a filler and polyolefin as a resin, has features of both wood and plastic depending on its composition, and is now increasingly in demand for use in exterior construction materials such as decks.[1] WPC usually shows some thermal expansion, a characteristic feature of plastic materials,[2–17] which can

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be disregarded for wood materials since cellulose has a strong reinforcing effect as a fiber. Developing materials with high dimensional stability is an important issue in construction application. Using the original reinforcing effect of cellulose fiber in composites is an important subject of study. Yano et al.[18–20] and Nishino et al.[21] reported high dimensional stability of thin films prepared from a cellulose/plastic composite and all cellulose composites, respectively, utilizing the high thermal stability property of cellulose. Many reports on fiber reinforcement and thermal expansion can be found in the field of plastics reinforced by inorganic fibers such as glass.[22,23] However, research on the fiber reinforcement effect on the thermal expansion of biomaterial/plastic composites prepared by conventional molding procedures such as press, injection, and extrusion molding is limited.[24] Furthermore, there is no report on the thermal stability of molded composites with a high cellulose content, although it is assumed that the effect of fiber would be conspicuous for composites with high fiber content. WPC with a high content of cellulose or wood has advantages in the appearance and feeling of wood, the effect of moisture control, and thermal dimensional stability, and is a useful material for interior use.

Previously, we discussed the effect of fibers on the melt fluidity of compounds, and the mechanical properties of molded composites for a high content cellulose/plastic composite using a viscoelastic procedure.[25–27] We also studied the contribution of fiber tangling[28,29] on flow characteristics, although the supporting evidence was inconclusive. In this study, the effects of resin content and cellulose fiber length on the thermal expansion of composites were examined for high cellulose content plastic composites using several molding procedures such as press, injection, and extrusion molding. The allowable range of variation in molding conditions is limited for composites of high filler content because appropriate flow of the compound during the molding process is required for injection and extrusion molding.

EXPERIMENTAL

Materials

The following raw materials were used: cellulose fiber (Arbocel supplied by J. Rettenmaeyer and Soene; Rosenbreg, Germany (Table 1)), polypropylene (PP; PM930V/random, MFR 30; Sun Alomar; Tokyo, Japan), and a compatibilizing agent, PP resin modified with maleic anhydride (MAPP, Umex 1010; Sanyokasei; Kyoto, Japan).

Compounding Process

Formulations of compounds (a mixture of raw materials) are shown in Table 2. Preparation of each compound was carried out using a conical twin screw

extruder (Taitan 80; Cincinnati Extrusion; Vienna, Austria) to give pellets 4 mm in diameter and 5mm long at a molding temperature of 230°C.

Table 1. Dimensions of cellulose fibers used

Cellulose	Product number	Average fiber (micrometer)	Average fiber diameter (micrometer)
Arbocel	BE00	120	20
	BC200	300	20

Molding

Press Molding

The material for press molding was prepared by smashing the compound pellets to pass through a 4 mm-mesh using a grinder (Wood grinder; Inoue Densetu; Nagoya, Japan). Press molding was carried out under a constant pressure of 11 MPa with a mold of 80 mm × 100 mm at a temperature of 220°C, and press time of 5 min, using a hydraulic press (H.P.-1 B-P; Seibu; Nagoya, Japan).

Extrusion Molding

Extrusion molding was carried out using a conical twin screw extruder (Taitan 65; Cincinnati Extrusion) with a mold of 100 mm × 9 mm and the compound pellets at a starting temperature of 215°C.

Injection Molding

An injection molder (Fanac alpha 100C; Fanac; Fuji, Japan) was used to mold the compound pellets into a dumbbell-shaped test piece at a starting molding temperature of 200°C.

Table 2. Formulation of compound (by weight)

Cellulose			
BE00	BC200	PP	MAPP
	90	8	2
	80	18	2
	70	28	2
80		18	2

Physical Properties of the Molded Composites

Density and Mechanical Strength

The density of each molded composite was measured according to the JIS K7112 standard using four test pieces of 25 mm × 80 mm × 5 mm. Bending strength was determined in accordance with the JIS A5908 standard.

Composite Thermal Expansion Coefficient

Thermal expansion was evaluated in a temperature range from –10°C to +30°C by a thermo-mechanical analyzer (TMA SS6100; Seiko instrument; Chiba, Japan) for four test pieces of 5 mm × 5 mm × 15 mm by measuring the dimensional change per °C for 15 mm in the lengthwise direction.

Water Absorption

Water absorption was tested by immersing the composite in water for 0 to 24 h under atmospheric pressure at room temperature and calculated using the following equation:

$$\text{Water absorption } (\%) = ((W_{ab} - W_{dry})/W_{dry})) \times 100$$

where W_{ab} shows the weight of the wiped sample after water absorption and W_{dry} is the dry weight of the sample before the water absorption test.

RESULTS AND DISCUSSION

Press Molding

Since press molding does not require much compound flow, the applicable range of conditions for this molding procedure was wide and compounds with 10–30% resin contents and fibers of two lengths (120 and 300 micrometer) were successfully processed.

Correlation between Resin Content and Thermal Expansion

Thermal expansion coefficients of press molded composites with several resin contents were examined using cellulose fibers 300 micrometer-long, and the results are shown in Figure 1. There was a tendency for thermal expansion to increase with resin content. The physical properties of press molded composites are shown in Table 3. The low densities of the composites show that compression was relatively low in the press molding compared with other

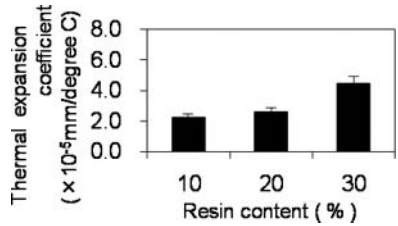

Figure 1. Effect of resin content on thermal expansion of a composite with 300 micrometer cellulose fibers (press molding).

molding procedures. Void space, which is estimated from the difference between measured density and theoretical density, decreased with resin content. Theoretical density is calculated from the composition in Table 2, and the net density of each component (cellulose 1.4 and PP 0.9g/cm^3). Void space decreased proportional to the resin content, and there was a linear relationship between void space and water absorption, as shown in Figure 2. However, thermal expansion was not proportional to the resin content, and showed smaller change in expansion from 10–20% of resin content than the corresponding change from 20–30% (Figure 1). These results suggest that void space is an important factor for determining water absorption, but not a crucial factor for thermal expansion. Some other factor(s), such as the interaction of fibers in the composite, may play a crucial role in thermal expansion of composites. We examined bending strengths of high filler content cellulose/PP composites in previous papers,[25,26] and proposed that fiber tangling may contribute to the strength of the composite.[26]

Correlation between Fiber Length and Thermal Expansion

Thermal expansion coefficients of press molded composites with 120 and 300 micrometer-long fibers and 20% resin content are shown in Figure 3. Thermal expansion of composites with 300 micrometer-long fibers was smaller than that of 120 micrometer fibers. Since the two composites had the same proportion of cellulose/PP, the theoretical densities are the same (1.26). The difference

Table 3. Effect of resin content on physical properties (press molding)

Resin Content (R/C)		10%	20%	30%
Density	g/cm^3	1.05	1.06	1.08
(Theoretical density)	(g/cm^3)	(1.33)	(1.26)	(1.20)
Estimated void space	%	21.1	15.9	10.0
Bending strength (dry stage)	MPa	34.2	33.4	31.6

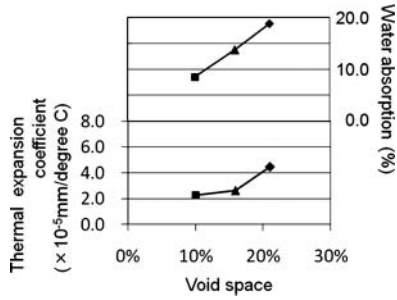

Figure 2. Correlation between void space and thermal expansion or water absorption in press molding. Symbols: solid square, 30% resin content; solid triangle, 20% resin content; solid diamond, 10% resin content.

in observed density corresponds to the difference in void space. The tendency observed in the previous section was reproduced here, such that the composite with more void space (composite with 300 micrometer fibers) absorbed more water, and showed a higher bending strength, but smaller thermal expansion. Since press molding does not require much compound flow and it is estimated that the states of fibers in the compound are largely transferred to the composite in press of molding, the present results observed for press molded composites can be expected to reflect the states of fibers in the compound.

Injection Molding

This molding procedure requires the highest rate of compound flow during processing. Although the molding temperature at the starting point was lower than that of press molding, it was quite possible that the actual temperature in the net molding process was higher than in the other molding procedures. Measurement of the temperature in the net molding stage was not possible with the machine used in this experiment.

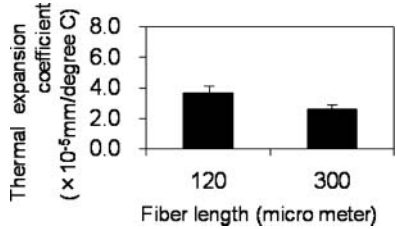

Figure 3. Effect of fiber length on thermal expansion of a composite with a resin content of 20% (press molding).

Table 4. Effect of fiber length on physical properties

Fiber length (micrometer)		120	300
Density	g/cm^3	1.08	1.06
Water absorption	%	12.4	13.8
Bending strength	MPa	31.1	33.4

Correlation between Resin Content and Thermal Expansion

Thermal expansion coefficients of injection molded composites with 300 micrometer cellulose fibers with two types of resin contents are shown in Figure 4. Probably because of a significant compound flow and orientation of fibers during injection molding, anisotropy in the thermal expansion was observed for samples cut in directions parallel and perpendicular to the molding direction of the composite. With this molding procedure, the composite with a 20% resin content showed higher thermal expansion than that containing 30% resin, contrary to the results of other molding procedures. The densities of composites of both types of resin contents were close to the theoretical densities and water absorption was negligible as shown in Table 5. Although there may be some contribution of the surface structure of a molded composite to the observed low water absorption since materials with a high flow rate (such as PP in the present case) tend to come out to the surface of the composite to decrease water absorption, our preliminary experiment with a molded composite with a removed surface did not show any significant change in water absorption behavior in the short time range of this experiment. Therefore, the main factor controlling thermal expansion of an injection molded composite is not the void space, but the state of the reinforcing fibers in the composite. The results suggest that the reinforcing effect due to fibers did not develop in the injection molded composite with a 20% resin content unlike as the composite with a 30% resin content. The results might be acceptable if we consider different states of fibers during

Table 5. Effect of resin content and fiber length on physical properties (injection molding)

Resin content (R/C)		20%		30%
Fiber length (micrometer)		120	300	300
Density	g/cm^3	1.32	1.32	1.26
Water absorption	%	1.3	1.2	0.7
Bending strength (dry stage, parallel to melding direction)	MPa	65.4	67.5	71.2

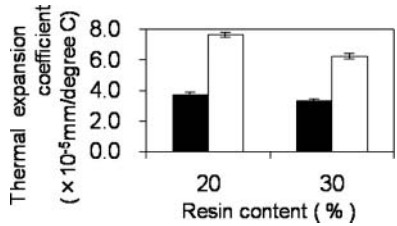

Figure 4. Effect of resin content on thermal expansion of a composite with 300 micrometer cellulose fibers (injection molding). Symbols: black, molding direction; white, width direction.

molding processes between these two cases, such that fiber tangling persisted in the compound with good flow (30% resin content) and remained in the composite, but fibers flocculated or were cut in the compound with poor flow (20% resin content) during the high speed molding process such as injection molding. In the latter case, some of reinforcing effect of fibers would be lost in the molded composite.

Correlation between Fiber Length and Thermal Expansion

Thermal expansion coefficients of injection molded composites with two fiber lengths of 120 and 300 micrometer and a resin content of 20% are shown in Figure 5. Anisotropy of thermal expansion was again observed for both composites of 120 and 300 micrometer fiber lengths, showing the orientation of the fibers. In the effect of fiber length on thermal expansion, the composite containing 300 micrometer-long fibers showed a larger thermal expansion than the 120 micrometer fiber one. This tendency is contrary to the observations for composites of press and extrusion molding. Since the densities of composites of 120 and 300 micrometer fibers are the same, the void space of the two composites should be similar (Table 5). Water absorption was almost the same

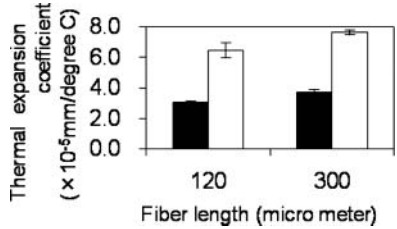

Figure 5. Effect of fiber length on thermal expansion of a composite with a resin content of 20% (injection molding). Symbols: black, molding direction; white, width direction.

for the two composites. It was generally observed in this study that the mechanical strength and thermal expansion showed an inverse tendency, but only in the experiment on fiber size dependence of injection molded composites, were thermal expansion and mechanical strength both large for the composite with 300 micrometer fibers. This result suggests that there is some difference in injection molded composites compared with the press or extrusion molded composites. The results of the effect of fiber length on the thermal expansion of injection molded composites may be explained by a decrease in the reinforcing effect by long fibers, such as breakdown and/or flocculation. However, breakdown of fibers only in the injection molding process does not seem very plausible because it is thought that more breakdown occurs in the high shear compounding process. Flocculation of fibers observed in our previous study when dispersion of fibers was not good[26] may be a reasonable explanation of the present results.

Extrusion Molding

Since extrusion molding is successful only under limited molding conditions with moderate compound flow, it was successful only for the 20% resin content composite in this experiment.

Correlation between Fiber Length and Thermal Expansion

Thermal expansion coefficients of extrusion molded composites with two fiber lengths of 120 and 300 micrometers and a resin content of 20% are shown in Figure 6. Anisotropy was also observed in thermal expansion of the composites, since material flow occurs during extrusion molding. The composite containing 300 micrometer-long fibers showed lower thermal expansion than the composite with 120 micrometer fibers. This result was contrary to the observed tendency for injection molded composites and the same as that for press molded composites. The dependence of water absorption on density was not

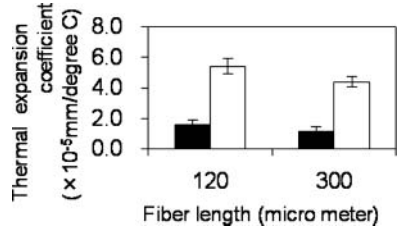

Figure 6. Effect of fiber length on thermal expansion of a composite with a resin content of 20% (extrusion molding). Symbols: black, molding direction; white, width direction.

Table 6. Effect of fiber length on physical properties (extrusion molding)

Figer length (micrometer)		120	300
Density	g/cm^3	1.28	1.30
Water absorption	%	1.5	1.4
Bending strength (dry stage, parallel to melding direction)	MPa	45.1	53.2

significant similar to the injection molded composites, suggesting that void space is not a major factor controlling thermal expansion (Table 6). The largest anisotropy of the thermal expansion coefficient was observed for extrusion molded composites. In addition, thixotropy[27,30] was observed in extrusion molding of the 300 micrometer compound and the extruded amount of material was not proportional to the screw speed, but increased at higher speed. This tendency was not clear with the 120 micrometer compound. These results suggest that the fiber states are better maintained in extrusion molding than injection molding.

CONCLUSIONS

The effects of resin content and fiber length on the thermal expansion, density, bending strength, and water absorption of cellulose/plastic composites with high cellulose fiber content were evaluated for three different molding procedures; press, injection, and extrusion molding. The results are summarized as follows: (1) Thermal expansion and bending strength showed an inverse tendency in that thermal expansion was smaller for composites with larger bending strength, except for the dependence of fiber size in injection molded composites. (2) In a composite with light compression and small compound flow (extrusion molding), thermal expansion of the composite was proportional to the resin content and showed no dependence on the void space, although the void space was proportional to the water absorption. Thermal expansion was large for composites with short fibers. (3) Press molded composites (products of a process of light compression and small flow) and extrusion molded composites (products of a process of moderate compression and moderate flow) showed a similar tendency in the dependence on fiber sizes. (4) Injection molded composites (products of a process of high compression and high flow) showed a different dependence of thermal expansion and bending strength with a change in the fiber size and resin content compared with press and extrusion molded composites. (5) Orientation of fibers was observed as anisotropy of thermal expansion and decreased in the following order: extrusion molding $>$ injection molding $>$ press molding (no orientation). The unique behavior of injection molding may be explained by the idea that the interaction of fibers in

the compound, which are most maintained in the press and extrusion molding, changed during the high-compression and high-flow molding step. As the basis of the reinforcing effects of fibers in composites, the contribution of fiber interaction in the composites is proposed. Although direct supporting evidence is still insufficient, an increase in reinforcement due to fiber tangling and a decrease due to formation of flocculated fiber balls and/or breakdown of fibers are compatible with the present results of thermal expansion of composites.

Finally, this work shows that the interaction of fibers is effective not only for developing the strength of composites, but also for decreasing their thermal expansion, and can be controlled by the flow characteristics of the compound during molding. This conclusion will be useful for the production of composites with high thermal stability, although the basic science using fiber interactions has not yet been established and must be developed. WPC composites with a high content of wood or cellulose filler have a potential as interior construction materials with high thermal dimensional stability.

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