Weathering characteristics of fiber-polymer composites

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Abstract

This paper summarizes a 2-year, extensive testing program designed to assess the environmental degradation of fiber-filled polypropylene and polyethylene. The fiber material used was wheat straw. The visual and mechanical characteristics of the plastics were tested after accelerated weathering (ultraviolet and humidity at elevated temperatures), thermal cycling, water saturation, and fungal attack to assess the stability of these organically filled agroplastics. The effect of fiber size, fiber loading, and matrix type (polypropylene or polyethylene) and different treatments were tested. Overall, the agroplastics showed significantly less weathering degradation after 1,000 hours than unfilled plastics. The 50 percent short fiber composite exhibited more degradation than the other fiber composites. Without any treatment, oxidation is a major problem. The trends were similar for both polypropylene and polyethylene. As expected, water saturation levels in-

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The authors would like lo thank USDA's Small Business Innovation Res. Prog. Grant No. 97-33610-4471 for support for this project. In addition, AlliedSignal Federal Manufacturing and Technology Center, operator of the US DOE's Kansas City plant, did the thermal data testing for the company.

creased and saturation time decreased with increased fiber loading. Increasing moisture content caused significant degradation in the physical properties of the plastic. Freeze-thaw cycles did not significantly alter the physical characteristics.

Introduction

When fiber-plastic composites are used outdoors they are exposed to ultraviolet radiation, moisture from rain, snow, and humidity, freezing/thawing, and fungal attacks. When this study was initiated, we did not know whether the agroplastics could withstand these influences without degrading mechanical properties. Therefore, an extensive testing program was done to test the agroplastics' response to accelerated environmental tests when chemically and physically modified and without any modifications. After the tests, the material was inspected visually and then subjected to tensile, flexural, and notched and unnotched Izod impact tests to determine any degradation.

The open literature contains little data on the environmental degradation of organic composites. Simonsen (3) found that composites of wood or other biofillers in thermoplastics are not impervious to the effects of outdoor exposure. Degradation was noted especially in stiffness. English and Falk (2) found that woodfiber-plastic composites absorb very little water. They also found that increasing fiber content substantially decreased the linear coefficient of thermal expansion. Coomarasamy and Boyd (1) examined the effect of freeze-thaw cycles on the mechanical properties of plastic lumber and found that at the end of the temperature cycling, none of the samples showed any signs of cracking, spalling, or other forms of deterioration, but several samples showed a reduction in strength.

Accelerated weathering

Accelerated weathering exposure conformed to ASTM G 53-96 and ASTM D 4329-92. A Q-Panel (Cleveland, Ohio) QUV/se Accelerated Weathering Tester was used for all the tests. Specimens for exterior exposure in the QUV were exposed to a cycle of 4 hours condensation at 50°C, and 8 hours irradiation by UVA-340A lamps at 0.77 $W/m^2/nm$ at 60°C. The specimens and equipment were checked daily, except weekends, for uniform condensation and correct irradiation. The flourescent lamps were calibrated every 400 hours, as per manufacturer's instructions. The two end panels on each side of the QUV/se were not used for exposures per ASTM D 4329. Specimens were rotated weekly per manufacturer's instructions; outside panels were rotated to the interior position in the QUV/se once per week.

Specimens were removed at the end of an irradiation cycle, rinsed in cold tap water for 3 to 5 seconds, and patted dry. They were then weighed after 3 to 4 hours of exposure at room conditions and the weight was recorded. Visual observations were made and any unusual observations were recorded. After conditioning at room temperature for at least 72 hours, tensile (ASTM D 638), flexural (ASTM D 790), or unnotched Izod (ASTM D 5941) tests were run. For notched Izod, samples were conditioned as above, except the samples were notched after QUV exposure and 3 to 4 hours of exposure at room conditions and then conditioned to room temperature for at least 40 hours and tested per ASTM D 5941-96.

Baseline results

A complete set of baseline tests were done for the agrocomposites. These included polypropylene, polyethylene, 30 percent long fiber, 50 percent, 30 percent short fiber, and 50 percent short fiber in both polypropylene and polyethylene. Tests included tensile, tensile modulus, flexural, flexural modulus, notched Izod, and unnotched Izod. An example of a typical run is shown in Figure 1.

The trends shown in Figure 1 are very consistent between tests and the polymer matrix. In general, there is a slow degradation of the virgin plastic. The 30 percent long and short fibers are very consistent and are similar to the 50 percent long fiber. The 50 percent short fiber composites usually exhibit characteristics between the other fibers and unfilled polymer. For comparison, the results of tensile strength for agrofibers in a polyethylene matrix are shown in Figure 2.

Visually, all the plastic material showed signs of oxidation with weathering. Unfilled polypropylene showed noticeable crazing and yellowing, and by the end of 3 weeks the material had degraded to the point of sloughing of the sample sticks. Unfilled polyethylene exhibited crazing by the end of 5 weeks, but did not slough material.

The agroplastics exhibited noticeable fading but the oxidation. was powdery and adhered fairly well to the surface.

Treatment results

Four different treatments were tested to determine if there was a treatment that would improve the mechanical properties of the agroplastics with respect to exposure and also reduce the amount of visible oxidation. The effect of the four treatments is shown in Figure 3.

At first glance, it appears that only one treatment, B, showed any significant impact over the control group. However, when the data is plotted as percent improvement from the control (Fig. 4), it shows that three treatments–B, C, and D–all have at least a 25 percent improvement in flexural strength from the control group. Treatment B was the only treatment that substantially reduced the amount of oxidation on the outer surface of the agroplastic.

Fungal resistance

Injection-molded flexural test specimens were cut into 1/2-inch lengths and ovendried overnight at 50°C to remove any residual surface moisture

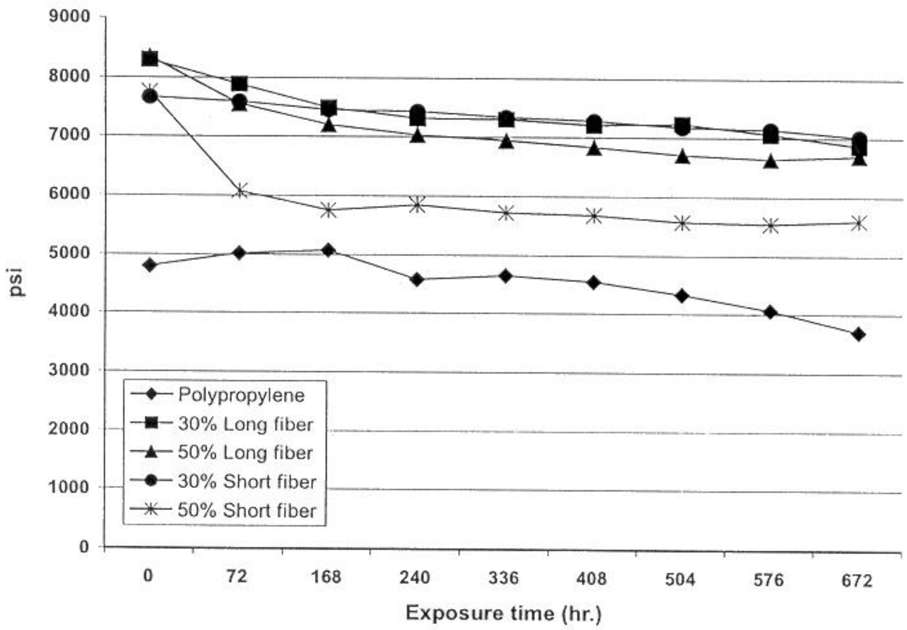

Figure 1. ~ *Flexural strength versus exposure for four types of agroplastics and polypropylene.*

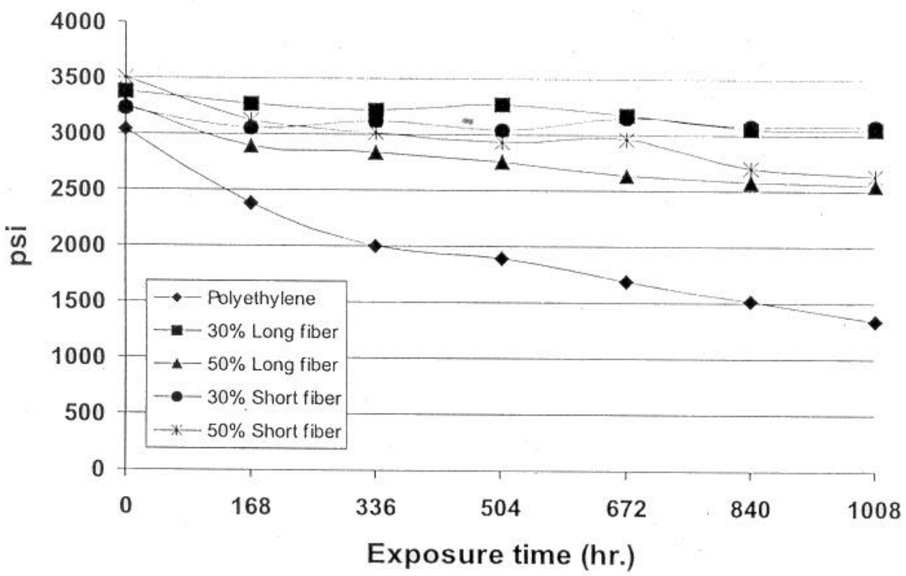

Figure 2. ~ *Tensile strength versus exposure for agrocomposites and polyethylene.*

that may have absorbed onto the composites during storage in plastic bags. The samples were removed from the oven and their initial weights were measured to four decimal places and cooled to room temperature. The samples were then placed into fungal decay culture bottles for a 12-week study on the effects of fungi on 50 percent wheat straw-polypropylene and polyethylene composites.

Five test samples were used for each composition for a total of 40 fungi tests to determine the weight loss of the composite samples and the visual effects on the surface quality of the composites. A modified version of ASTM D 1413-76 (Standard Method of Testing Wood Preservatives by Laboratory Soilblock Cultures) was followed for preparation of the fungi test bottles. All procedures were done in accordance with ASTM D 1413, but instead of a l-inch soil block test specimen a 1/2-inch-long section of an ASTM flexural test specimen was placed in culture bottles for fungi decay testing. Test culture bottles were steam sterilized at 103 kPa (15 psi) for 30 minutes. Soil for the culture bottles was sifted, and water-holding capacity of the soil was tested. Approximately 90 g of soil was placed in each culture bottle. One feeder strip per culture bottle (3 by 28 by 35 mm) was placed on top of the soil. A *Gloeophyllum trabeum* fungi culture was inoculated onto the feeder strip and the culture bottles were set in an incubation room for the mycelium to cover the feeder strip.

After the fungi cultures were growing and established on the feeder strips, the ovendried and cooled composites were placed on top of the established fungi cultures. The culture bottles were then placed in an incubation room (80°F 70% relative humidity) and left for the 12-week study. After removal from the test bottles, the test samples were brushed clean of fungi and placed in an oven at 50°C for 24 hours. Intermediate weight loss measurements were taken. The samples were

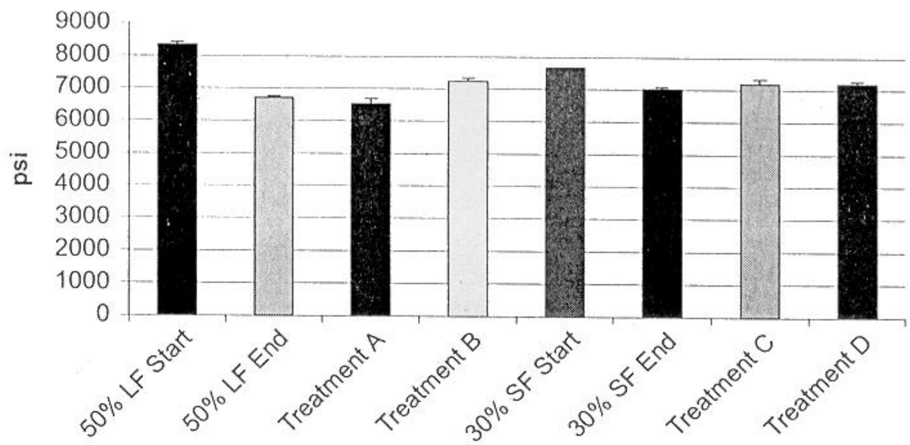

Figure 3. ~ *Effects of four different treatments on flexural strength of polypropylene composites with 672 hours exposure. LF = long fiber; SF = short fiber.*

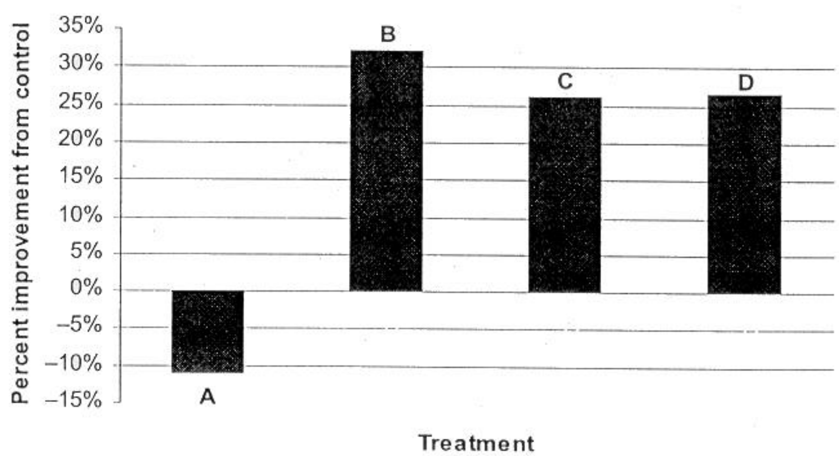

Figure 4. ~ *Percent improvement of treatments versus controls.*

placed back in the oven at 105°C for 24 hours and final weight loss measurements were obtained.

The results of the 3-month fungal decay test are summarized in Figure 5. The polypropylene treatment E was less effective than the polyethylene treatment F. In the control samples there was no difference between the short fibers but there was a significant difference between the long fiber controls. Surface degradation was significant in all samples except when treatment F was used.

Water absorption

Three samples of each type of agroplastic were dried for 24 hours at 50°C. These were then submerged in distilled water. The water was changed once per week. Each week, samples were removed from the water, patted dry, and weighed.

The rate of water absorption was different depending upon the loading level and matrix type (Fig. 6). As expected, the composites with 30 percent fiber had less water absorption then those with 50 percent fiber. A surprising difference was exhibited between polypropylene and polyethylene composites. The polyethylene composites were still absorbing water at the end of the test. The sticks were slimy to the touch-evidence of fungal and bacterial growth.

The next test was to determine the effect of water absorption on mechanical properties. Tensile coupons were immersed in water for 1,562 hours, patted dry, allowed to sit for 24 hours, and tested for tensile properties. The results are in Figure 7. There was a significant decrease in all composite mechanical properties with saturation. This is

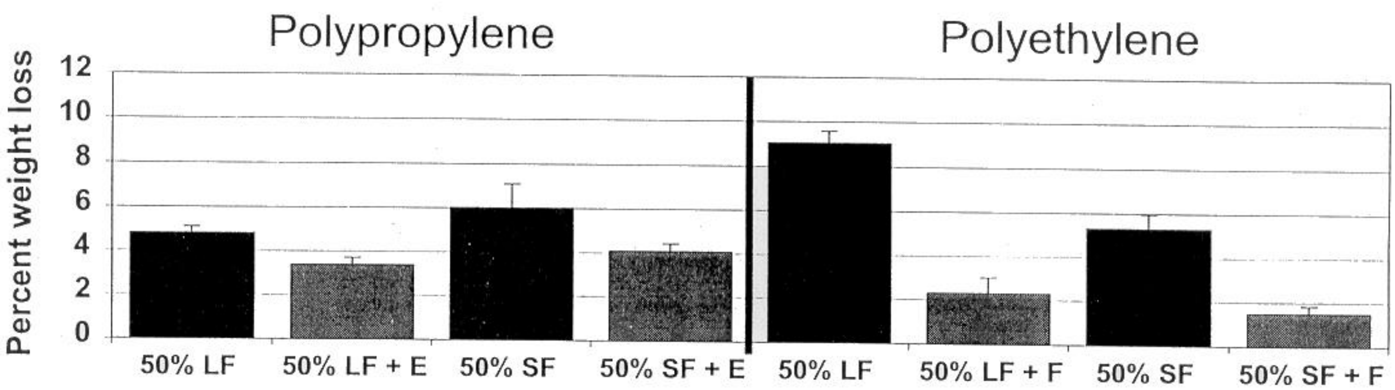

Figure 5. ~ *Percent loss of agrocomposites after 3 months of exposure to fungal decay. Two treatments were used to prevent fungal attack. LF = long fiber; SF = short fiber.*

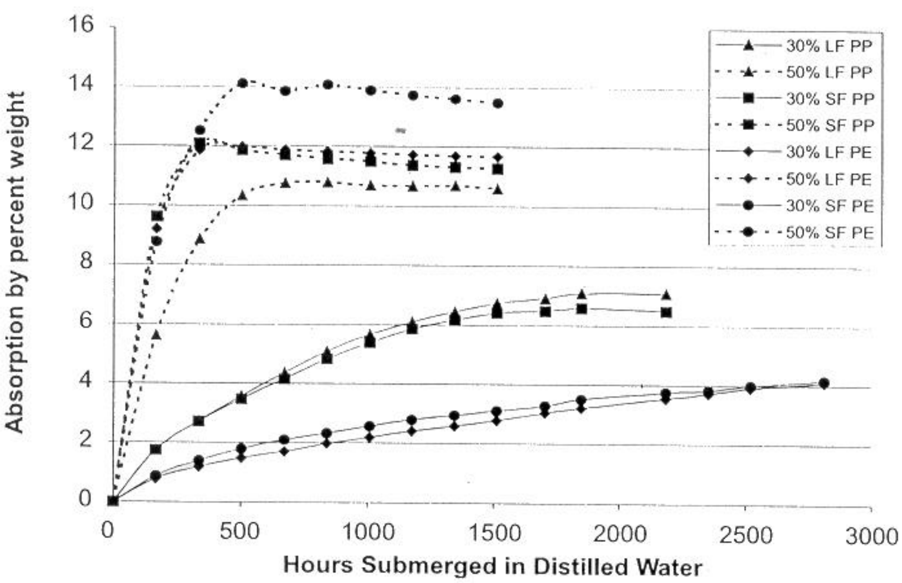

Figure 6. ~ *Rates of water absorption for different types of agroplastics. Polypropylene (PP) and polyethylene (PE) had zero water absorption. LF = long fiber; SF = short fiber.*

probably due to fungal attack of the fibers. Tests are ongoing to examine mechanical properties when fungicides are added.

We determined that drying to ASTM Standard D 570-95 was insufficient for agrocomposite materials. Drying temperature was increased to 70°C and the time to 144 hours, or when the weight change was less than 0.05 percent in 24 hours.

The data were also examined by plotting tensile strength versus water absorption (Fig. 8). Samples were dried and then hydrated by exposure to room conditions or submersion in distilled water, which was changed every 2 days. There is a good correlation between tensile strength and water absorption, and the change is reasonably linear.

Freeze-thaw cycling

In order to examine the effect of freeze-thaw cycles on agroplastics, test coupons of extrusion grade 50 percent long fiber polyethylene were soaked in water for 2 weeks and then subjected to rapid freeze-thaw cycles. Water uptake was 9 percent. 'Temperature cycled from -10°C to 15°C every hour using a Tenny TJR Environmental Test Chamber. There was a lo-minute ramp to freeze and a 7-minute ramp to 15°C. There was a decrease in tensile strength but that was seen in all

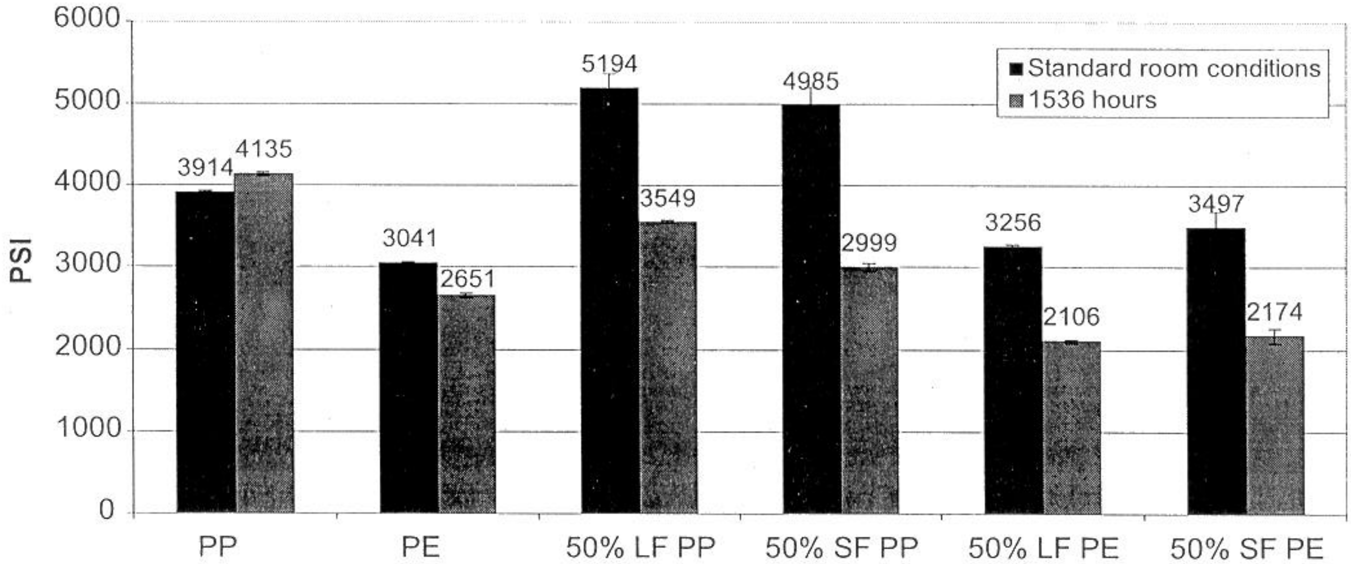

Figure 7. ~ *Effects of water absorption on tensile strength. PP = polypropylene; PE = polyethylene; LF = long fiber; and SF = short fiber.*

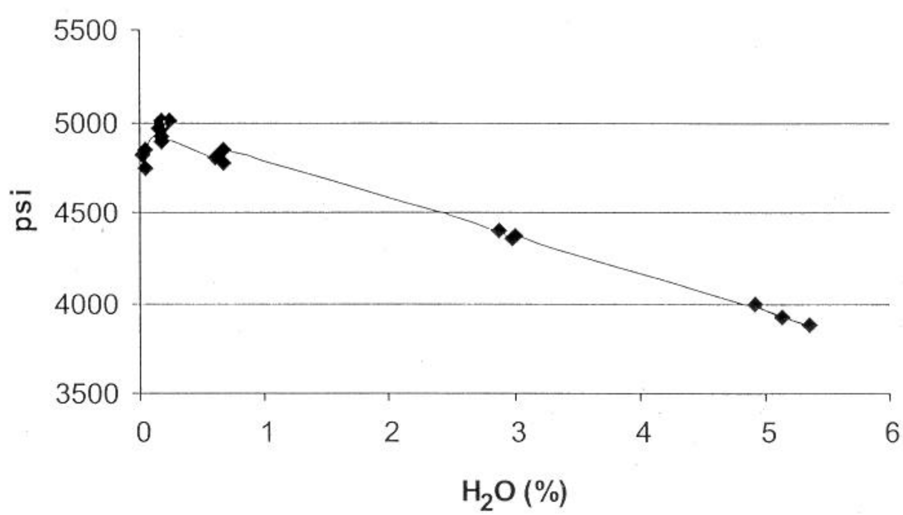

Figure 8. ~ *Correlation of water absorption with tensile strength.*

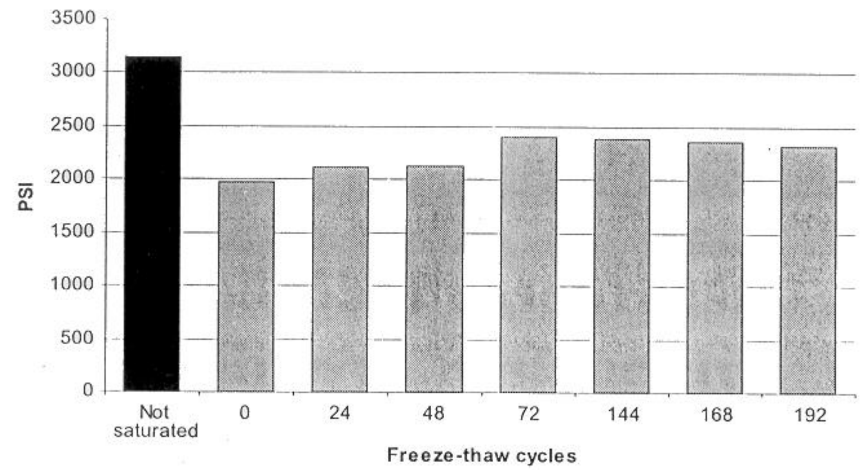

Figure 9. ~ *Effect of freeze-thaw cycles on tensile strength of 50 percent polyethylene long-fiber agroplastics.*

Table 1. ~ *Coefficient of thermal expansion.*

	Coefficient of thermal expansion (°C × 10^6)			
	-40°C to 25°C		25°C to 100°C	
Sample	z-axis	y-axis	z-axis	y-axis
Polypropylene	73.7	80.0	152	161
30% long fiber	72.6	68.3	176	131
50% long fiber	70.0	58.9	155	109
30% short fiber	67.1	69.3	159	136
50% short fiber	63.9	62.9	128	121

Table 2. ~ *Heat of deflection.*

Sample	Heat of deflection
	(°C)
Polypropylene	112
30% long Fiber	153
50% long fiber	149
30% short fiber	137
50% short fiber	138

samples including the ones not subjected to any freeze-thaw cycles (Fig. 9). This decrease was probably due to water absorption and fungal attack versus any effect of freeze-thaw (see the water absorption section). Only one sample was tested so the slight increase seen in tensile strength is probably due to natural variation.

Thermal properties

Thermal expansion

Thermal expansion figures were calculated by taking samples from the center of the 1/2- by 2-1/2-inch bars, and then tested by TMA for the thermal expansion coefficient. The samples were heated from room temperature to 100°C, cooled to -40°C, then heated to 100°C, and finally cooled to room temperature. The expansion coefficients were measured on the second heating and are shown in Table 1. The first number in each group is the z-axis coefficient, measured on the thickness of the bar. The second number in each group is measured on a sample oriented across the width of the bar.

The results are surprisingly close even though some of the samples contain 50 percent filler. As anticipated, the material is anisotropic; this is more pronounced in the longer fibers.

Heat of deflection

Heat of deflection was measured by ASTM D 648 at 66 psi load. The results are shown in Table 2. As anticipated, the filled material is less affected by heat than the unfilled polypropylene. The long fiber material has a higher heat of deflection than the short fiber material.

Literature cited

1. Coomarasamy, A. and S.J. Boyd. 1996. Development of specifications for plastic lumber for use in highway applications. *In:* Woodfiber-Plastic Composites. Forest Prod. Soc., Madison, Wis. pp. 199–208.
2. English, B.W. and R.H. Falk. 1996. Factors that affect the application of woodfiber-plastic composites. *In:* Woodfiber-Plastic Composites. Forest Prod. Soc., Madison, Wis. pp. 189–194.
3. Simonsen, J. 1996. The mechanical properties of woodfiber-plastic composites: theoretical vs. experimental. *In:* Woodfiber-Plastic Composites. Forest Prod. Soc., Madison, Wis. pp. 47–55.

Fifth International Conference on Woodfiber-Plastic Composites

May 26–27, 1999
The Madison concourse Hotel
Madison, Wisconsin

Sponsored by the USDA Forest Service in cooperation with the American Plastics Council, the University of Wisconsin, the University of Toronto, the Cellulose, Paper, and Textile Division of the American Chemical Society, and the Forest Products Society.

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