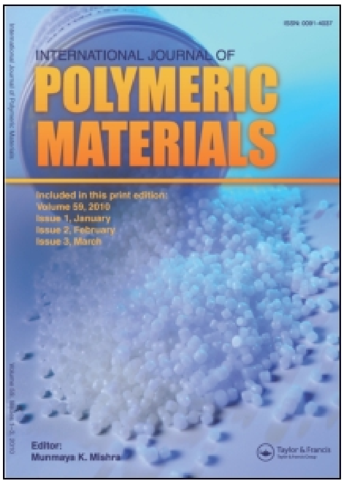

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Wood Flour and Polypropylene or High Density Polyethylene Composites: Influence of Maleated Polypropylene Concentration and Extrusion Temperature on Properties

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Wood Flour and Polypropylene or High Density Polyethylene Composites: Influence of Maleated Polypropylene Concentration and Extrusion Temperature on Properties

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The effects of the concentration of a maleated polypropylene additive (0 to 5 percent by weight) and of extrusion blending temperature (190°C to 250°C) on the mechanical properties of extruded and injection-molded polypropylene-wood flour composites were investigated. The effects of maleated polypropylene additive on similarly processed polypropylene-wood flour and high density polyethylene-wood flour composites were also compared. Both the additive and the high extrusion temperature led to some wood degradation and to a less polar wood surface. The additive led to greater reinforcement of the composites, as indicated by moderate but useful increases in heat deflection temperature, strength, and modulus. The major portion of those improvements was achieved by adding 1 to 2 percent additive. However, both the additive and the high extrusion temperature decreased impact resistance, presumably as a consequence of increased reinforcement by the filler particles and wood degradation. Heat deflection temperature, strength, and modulus of the polypropylene-wood flour system were marginally better than that of the high density polyethylene-wood flour system; impact resistance was marginally poorer.

KEY WORDS Polypropylene, polyethylene, wood flour, composite, coupling agent, maleated polypropylene

INTRODUCTION

Composites made by extruding mixtures of polypropylene or polyethylene and wood flour or wood fiber are of interest for high volume applications because of their low cost, their low density relative to inorganic-filled systems, and their thermoformability into shaped products (e.g., automobile door panels and trunk liners, storage bins). However, the manufacture of these composites is associated with two problems: (1) the incompatibility of polar wood filler and nonpolar polymer leads to difficulties in obtaining good dispersion of the wood particles and to poor reinforcement of the polymer by the filler and (2) the high temperatures required for melt processing (e.g., extrusion and injection molding) approach those where wood thermolysis begins to occur at a significant rate.

Several laboratories have investigated the use of maleated polypropylene to improve the compatibility and bonding of wood and polypropylene, either as a high molecular weight substitute for the polypropylene[1-3] or as a low molecular weight additive (coupling agent).[4-7] We recently described the effects of several variables on the mechanical properties of an extruded polypropylene-wood flour (PP-WF) system; the variables included one concentration of Epolene E-43,[†] a commercial low molecular weight maleated polypropylene.[6] At 2.5 weight percent E-43 (total dry system basis) and at 200°C extrusion temperature, increases in strength and modulus approached 30 percent relative to the control without E-43. However, notched impact energy was slightly decreased at that E-43 level. Moreover, extrusion at 200°C in the presence of E-43 caused darkening of the composite, indicating that the acidity of E-43 catalyzed wood degradation.

In this paper, we report the results of a follow-up investigation to determine the effects of different Epolene E-43 concentrations and extrusion temperatures on the mechanical properties of PP-WF composites (Experiment A). The results have implications for balancing the mechanical property mix against the added costs of higher E-43 levels and extrusion temperatures. We also compare the effects of E-43 on composites made with wood flour in polypropylene to those made with wood flour in high density polyethylene (HDPE) (Experiment B). Composites made with HDPE are of interest for low cost, high volume applications because HDPE has greater low temperature toughness than polypropylene and it is available in large volume as waste milk containers.

EXPERIMENTAL

Experiment A

Test matrix. We replicated a full factorial matrix of two variables at four levels, as follows:

[†] The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Department of Agriculture of any product or service.

Variable	Value
Epolene E-43 concentration	0, 0.5, 2.0, and 5.0 percent by weight of total dry system
Extrusion blending temperature	190°C, 210°C, 230°C, and 250°C barrel and die

Each replicate of the matrix was run in a different random order. The ratio of wood flour to total polymer (polypropylene + E-43) was held constant at 50/50 by weight (dry basis).

The data were analyzed by analysis of variance (ANOVA) to determine the significant main effects of the variables and the interactions between the variables. Because the ANOVA only determines whether at least two of the results are statistically different at a certain confidence level, Duncan's multiple range test was also applied to determine which levels of E-43 and extrusion temperature produced significantly different properties.[8]

Materials. The polypropylene consisted of Soltex Fortilene 9101 homopolymer spheres with a density of 0.900 g/mL and a melt flow index of 2.5 g/10 min (230°C/2,160 g). The stabilizers Irganox-1010 (a tertiary butyl hydroxyhydrocinnamate) (Ciba-Geigy, Hawthorne, NY) and Ionol (a butylated hydroxy toluene) (Uniroyal, Middlebury, CT) were added at 0.10 and 0.20 percent of the polypropylene, respectively. Epolene E-43 (Eastman Chemical Products, Kingsport, TN) has a density of 0.934 g/mL, an acid number of 47, and an approximate molecular weight of 4,500. The wood flour was no. 402 yellow pine, nominal 40 mesh (American Wood Fibers, Inc., Schofield, WI).

Processing. The wood flour was dried at 105°C in an air-circulating oven for about 3 h to a moisture content of <1 percent and then stored in plastic bags within sealed metal cans containing desiccant. All other ingredients for a given trial were added to the dried wood flour and dry blended immediately before extrusion. The 16 trials for each matrix were extruded in random order in a Killion single-screw laboratory extruder at 190°C, 210°C, 230°C, or 250°C and at flow rates from 26 to 31 g/min. The extruded strand was pelletized and stored in sealed cans containing desiccant. Test specimens were prepared in the same random order using a Frohring Mini-Jector model SP50 (Newbury Industries Inc., Newbury, OH) plunger injection molding machine at 215°C with a residence time of about 20 s, ram pressure of 9 MPa, and mold temperature of about 30°C. After molding, the specimens were stored over desiccant at room temperature for at least 3 days before testing.

Testing. Each mechanical property was measured on six specimens from each trial. The desiccated specimens were measured at 23°C with minimum exposure to ambient humidity. Maximum tensile strength was measured in conformance with ASTM D 638-84[9] with specimen type IV at a nominal strain rate of 0.15 mm/mm-min. Specimens for flexural (three-point bending and cantilever-beam) tests were 127 by 12.7 by 3.2 mm. Three-point bending maximum strength and modulus were measured in conformance with ASTM D790-84a,[9] using a support span of 102 mm and a crosshead rate of 5 mm/min. Maximum flexural strength of cantilever-beam specimens was determined in conformance with ASTM D747[9] using a Tinius Olsen stiffness tester with a bending span of 50 mm and a rate of about 60°/min; a secant modulus was calculated from the load at 9°. Notched and unnotched impact energy was measured with an Izod impact tester; specimens were 64 by 12.7 by 6.4 mm, and tests conformed to ASTM D256-84.[9] Densities were determined using a neutral buoyancy method in a series of aqueous salt solutions.

Wood flour was obtained from the processed composites by xylene extraction of 1- to 2-g samples taken from selected injection molded specimens. The composite samples were continuously eluted for several hours at the boiling point of xylene, and the undissolved wood flour was rinsed with petroleum ether and dried. Infrared spectra were measured on the recovered wood flour and on extracted, unprocessed wood flour using several techniques—transmittance spectra on KBr pellets, photoacoustic spectra on flour, and attenuated total reflectance and diffuse reflectance spectra on thin sheets made by pressing the flour at 140°C and 850 MPa for 5 min.

The extracted wood flour samples were re-extracted with boiling xylene and, following Takase and Shiraishi,[2] were tested for their qualitative relative ability to be wet by water and diethyl ether. Aliquots from both singly and doubly extracted materials were placed in test tubes containing water and diethyl ether; the test tubes were evacuated to remove air, shaken overnight, re-evacuated briefly, and allowed to stand. They were then examined to determine whether the flour had fallen to the bottom of the water layer or remained at the water-ether interface.

Experiment B

A simple unreplicated matrix was tested in Experiment B. The polymers were either the same polypropylene as in Experiment A or high density polyethylene (Soltex Fortilene A60-70-119, density 0.960 g/mL, melt index 0.70 g/10 min at 190°C/2,160 g). Epolene E-43 concentration was 0, 0.75, and 1.5 weight percent of the total system. Wood flour was the 40 mesh material used in Experiment A. All processing procedures were as in Experiment A. Cantilever beam strength and modulus and notched impact were determined as in Experiment A. Heat deflection temperature was also measured, in conformance with ASTM D 648.[9]

RESULTS AND DISCUSSION

Experiment A

Table I summarizes the means and standard deviations of the mechanical properties at each experimental condition. The means are plotted against E-43 concentration or extrusion temperature in Figures 1 to 7. Table II presents the results of the analysis of variance for main effects and interactions; Table III shows the results of applying Duncan's multiple range test to determine the significance of observed property differences among the different extrusion temperatures and E-43 concentrations.

Composite strength and modulus. The ANOVA (99 percent confidence level) indicated that the overall effect of adding E-43 was to increase all the strengths and moduli (Table II, Figures 1 to 5). However, the Duncan's multiple range test

TABLE I

Mechanical properties observed in Experiment A[a]

Epolene E-43 (percent)[b]	Extrusion temperature (°C)	Flexural max. strength (MPa)	Cantilever beam max. strength (MPa)	Tensile max. strength (MPa)	Flexural modulus (GPa)	Cantilever beam secant modulus (GPa)	Notched impact energy (J/m)	Unnotched impact energy (J/m)
0	190	46.0 (1.4)	54.8 (1.6)	26.6 (0.6)	1.51 (0.04)	2.33 (0.12)	28.1 (1.4)	65.4 (5.0)
	210	47.0 (2.3)	54.4 (2.8)	26.6 (1.0)	1.71 (1.0)	2.52 (0.10)	27.7 (1.2)	59.8 (5.8)
	230	45.7 (2.4)	53 .8 (2.5)	25.1 (1.5)	1.61 (0.20)	2.46 (0.26)	26.2 (1.6)	57.5 (8.3)
	250	46.0 (1.2)	53.6 (1.7)	26.5 (0.9)	1.72 (0.13)	2.58 (0.13)	23.5 (0.7)	55.2 (4.8)
0.5	190	48.6 (1.2)	57.4 (2.5)	28.6 (0.9)	1.77 (0.18)	2.51 (0.08)	26.6 (1.2)	60.9 (3.5)
	210	51.5 (1.6)	58.6 (3.5)	30.0 (2.4)	1.85 (0.15)	2.66 (0.19)	24.9 (1.3)	59.2 (5.2)
	230	52.1 (1.7)	58.1 (4.0)	28.8 (1.2)	1.85 (0.09)	2.74 (0.16)	24.5 (1.0)	55.3 (4.6)
	250	50.1 (1.1)	55.2 (1.3)	27.8 (1.0)	1.71 (0.13)	2.72 (0.09)	22.8 (1.0)	54.0 (5.3)
2	190	54.1 (1.4)	61.0 (2.2)	28.5 (1.1)	1.81 (0.09)	2.75 (0.09)	25.3 (0.9)	65.5 (4.5)
	210	55.8 (1.9)	62.3 (2.4)	30.5 (0.8)	1.76 (0.07)	2.91 (0.14)	24.2 (0.8)	58.2 (2.9)
	230	55.5 (1.2)	64.6 (2.8)	31.6 (1.4)	1.94 (0.09)	2.86 (0.08)	23.5 (0.7)	61.7 (6.4)
	250	55.3 (3.6)	61.4 (4.6)	31.5 (1.7)	1.98 (0.26)	2.94 (0.11)	20.9 (0.7)	57.2 (3.4)
5	190	54.4 (1.1)	61.4 (4.6)	30.7 (0.6)	1.86 (0.08)	2.86 (0.13)	24.0 (0.8)	61.0 (5.6)
	210	56.8 (1.3)	65.6 (2.9)	32.3 (0.8)	1.97 (0.17)	3.14 (0.14)	23.5 (0.9)	58.2 (3.6)
	230	57.7 (0.8)	63.4 (2.1)	31.7 (1.3)	2.06 (0.09)	3.11 (0.12)	22.3 (1.0)	57.9 (3.7)
	250	56.1 (2.5)	59.2 (0.9)	30.3 (0.6)	1.92 (0.16)	3.00 (0.10)	21.2 (0.6)	59.5 (6.2)
Overall mean		52.0	59.1	29.4	1.81	2.76	24.3	59.2

[a] Mean values from duplicate matrices; 6 specimens per matrix. Values in parentheses are standard deviations, reflecting random and matrix-to-matrix errors.

[b] Weight percent of total dry system.

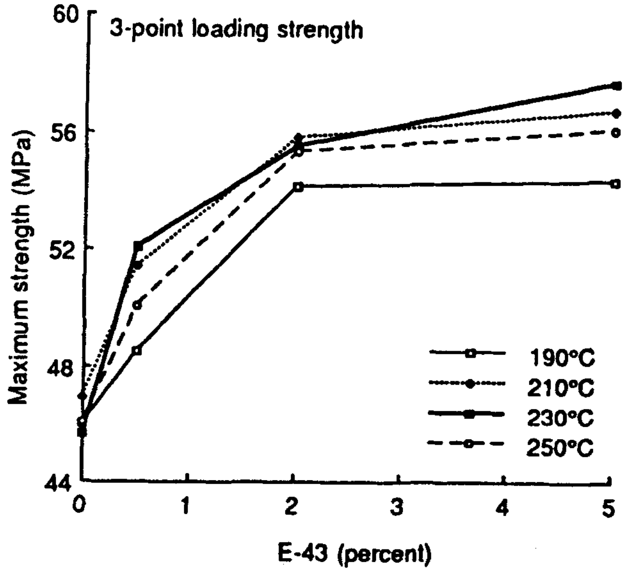

FIGURE 1 Flexural (three-point loading) strength of polypropylene-wood flour composites as a function of Epolene E-43 concentration at different extrusion temperatures.

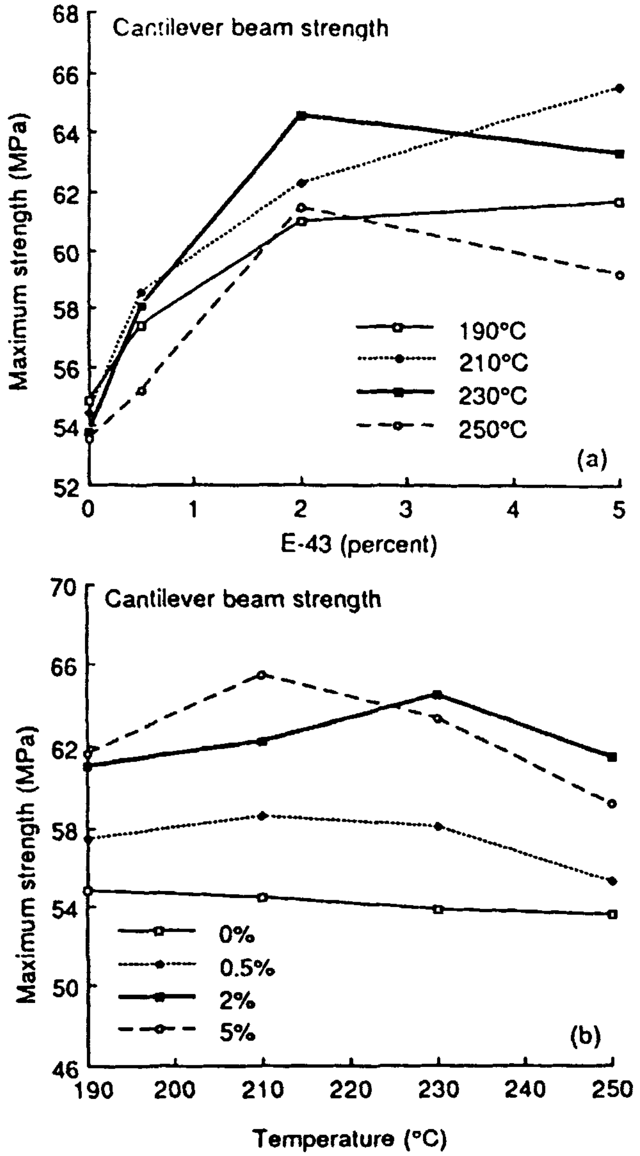

FIGURE 2 Cantilever beam strength of polypropylene-wood flour composites as a function of (a) Epolene E-43 concentration at different extrusion temperatures and (b) extrusion temperature at different Epolene E-43 concentrations.

(95 percent confidence, Table III) indicated that the gains in strength and modulus were all achieved by the time the E-43 level reached 2.0 percent.

Increasing extrusion temperature from 190°C to 250°C had a slightly positive overall effect on three of the five strength and modulus properties (Table II, Figures 1 to 5), but in all cases the effect was much less than that of E-43. Because of the greater influence of E-43, Duncan's test was not applied to the temperature data.

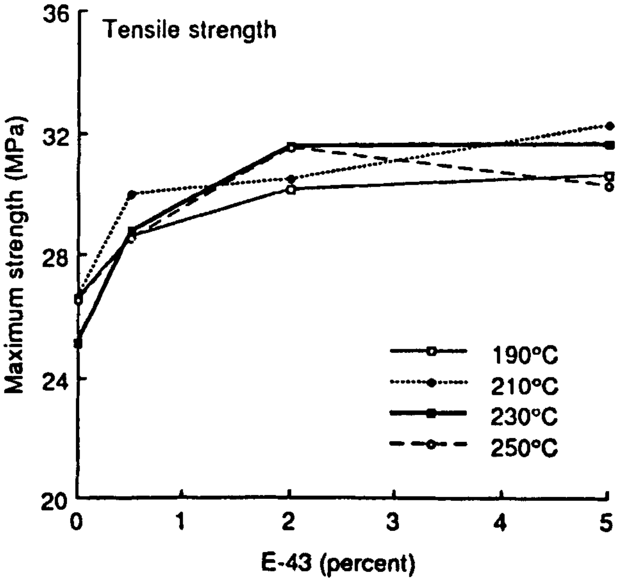

FIGURE 3 Tensile strength of polypropylene-wood flour composites as a function of Epolene E-43 concentration at different extrusion temperatures.

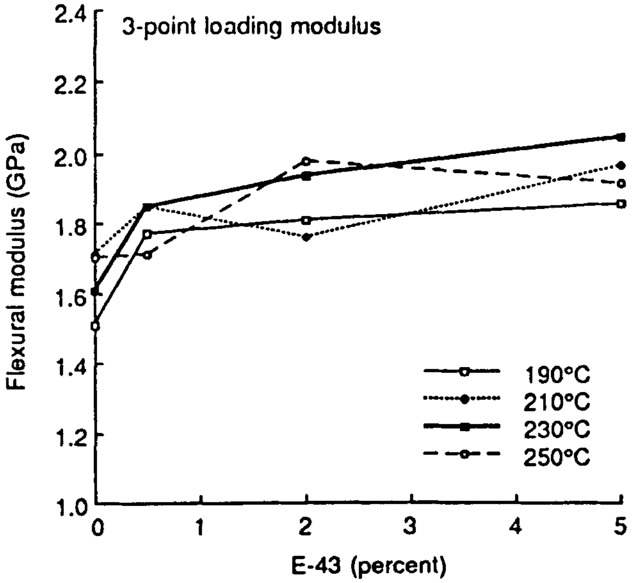

FIGURE 4 Flexural (three-point loading) modulus of polypropylene-wood flour composites as a function of Epolene E-43 concentration at different extrusion temperatures.

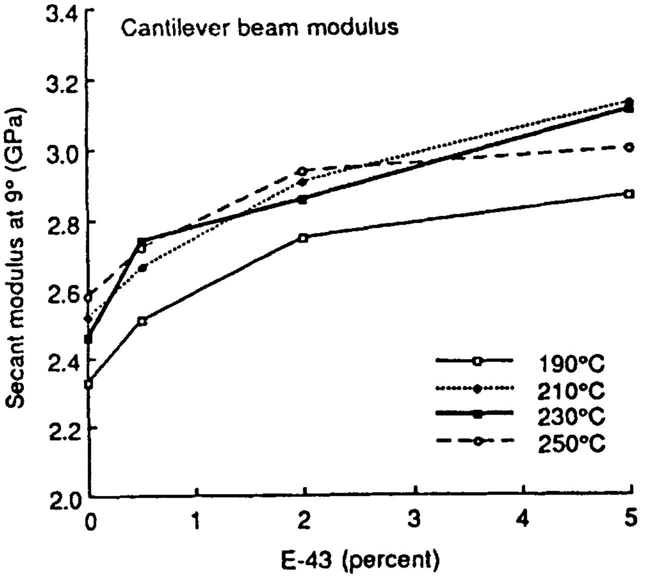

FIGURE 5 Cantilever beam secant modulus of polypropylene-wood flour composites as a function of Epolene E-43 concentration at different extrusion temperatures.

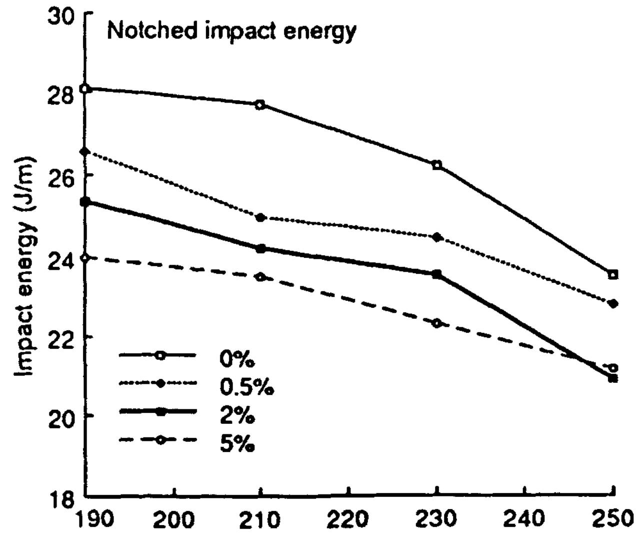

FIGURE 6 Notched impact energy of polypropylene-wood flour composites as a function of extrusion temperature at different Epolene E-43 concentrations.

As Figure 2b indicates, however, there was some indication of an optimum in properties in the 210°C to 230°C extrusion range.

For the strength and modulus properties, reasonable choices for extrusion temperature and E-43 concentration appear to be 200°C to 210°C and 1 to 2 percent,

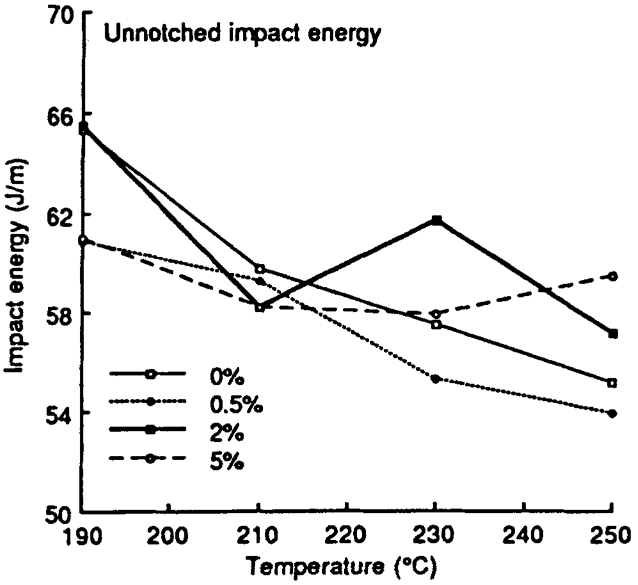

FIGURE 7 Unnotched impact energy of polypropylene-wood flour composites as a function of extrusion temperature at different Epolene E-43 concentrations.

TABLE II

Analysis of variance (observed F ratios) for Experiment A[a]

Effect	Flexural max. strength	Cantilever beam max. strength	Tensile max. strength	Flexural modulus	Cantilever beam secant modulus	Notched impact energy	Unnotched impact energy
Main effect							
Epolene E-43	101.5 (99)	25.6 (99)	36.5 (99)	10.7 (99)	30.2 (99)	35.1 (99)	1.4 (<75)
Extrusion temperature	4.2 (95)	2.7 (90)	0.9 (<75)	1.6 (75)	4.7 (95)	38.4 (99)	6.3 (99)
Interaction							
E-43 x temperature	0.7 (<75)	0.7 (<75)	1.0 (<75)	1.0 (<75)	0.4 (<75)	0.8 (<75)	0.8 (<75)

[a] Numbers in parentheses indicate the confidence level at which the effect is significant.
F is the variance of responses attributable to the particular variable divided by the variance resulting from experimental error.
For the degrees of freedom available in this experiment, F>5.4 indicates significance at the 99 percent confidence level, F>3.3 at the 95 percent confidence level, F>2.5 at the 90 percent confidence level, and F>1.5 at the 75 percent confidence level (8).

respectively. At those conditions, we might expect the increases in strength and modulus shown in Table IV. These improvements from using E-43 are of the same magnitude as we reported previously and indicate greater reinforcement by the wood flour in the presence of the E-43, either because of better dispersion of the wood flour in the polymer or better bonding between the flour and polymer.[6]

Composite impact energy. Both E-43 and extrusion temperature had a significant (99 percent confidence level) negative effect on notched impact energy of the composite (Table II, Figure 6). Data scatter was more pronounced with unnotched impact energy than with notched (Figures 6 and 7), and consequently the F ratios were much lower for the former (Table II). Although no significant effect of E-43 on unnotched impact energy appeared, a negative effect of extrusion temperature

TABLE III

Duncan's multiple range test for effects of Epolene E-4 on nonimpact properties in Experiment A[a,b]

Property	Epolene E-43 (percent)[c] 0	0.5	2.0	5.0
Flexural maximum strength (MPa)	46.2	50.6	55.2	56.2
Cantilever beam maximum strength (MPa)	54.2	57.3	62.3	62.5
Tensile maximum strength (MPa)	26.2	29.0	30.9	31.3
Flexural modulus (GPa)	1.63	1.79	1.87	1.95
Cantilever beam secant modulus (GPa)	2.47	2.66	2.87	3.03

[a] Values averaged over the four extrusion temperatures.

[b] Underlined property values not significantly different at 95 percent confidence level (ref. 8).

[c] Percentage by weight of total system.

TABLE IV

Property changes at selected E-43 levels in Experiment A

Property	Change relative to zero E-43 (percent) 1 percent E-43	2 percent E-43
Strength and modulus[a]		
Flexural strength	13	19
Cantilever beam strength	10	15
Tensile strength	13	16
Flexural modulus	12	14
Cantilever beam modulus	11	16
Impact energy[b]		
Notched	-9	-11
Unnotched	-3	-1

[a] Averaged over the four extrusion temperatures with interpolated property values.

[b] At 200°C with interpolated property values.

was observed (99 percent confidence level). In neither case were there significant interactions between the two variables. As noted previously,[6] loss in impact energy with the addition of E-43 may reflect the greater strength and stiffness of the composites; additionally, lower impact energy may result from increased wood filler brittleness caused by wood decomposition at the high extrusion temperature, catalyzed by the acidic E-43. We inferred that wood decomposition occurred because the composites became darker in color with greater E-43 level and greater temperature.

Because neither temperature nor E-43 had a preponderant influence on impact energy, we did not apply Duncan's test to determine the statistical significance of differences among extrusion temperatures or E-43 concentrations. Assuming that 200°C is a reasonable extrusion temperature, Table IV shows the sacrifice in impact energy that would accompany the improvements in strength and modulus at 1 and 2 percent E-43.

Analytical characterization. At all E-43 levels and extrusion temperatures, the infrared spectra of wood flour extracted from the composites were not significantly different from the spectrum of the extracted, unprocessed wood flour. This apparent lack of any change in wood surface composition probably reflects the low sensitivity of infrared and the complexity of the wood spectrum itself because the wetting experiment (Table V) clearly demonstrated that the wood surface became less polar after the wood flour was processed into the composite. Interestingly, the greatest change in wetting behavior appeared to result not from the addition of E-43 but from exposure of the composite to the melt processing temperature, indicating that the high temperature itself made the wood surface less polar. (Small increases were also observed in the fractional weight of residue after extraction, but these were probably within experimental error. Moreover, the extraction residue values and the wetting behavior were identical after single and double extractions.) The decreased polarity was accompanied by an increasingly darker ap-

TABLE V

Effects of E-43 and melt processing on wood flour wettability in Experiment A

Epolene E-43 (percent)	Extrusion temperature (°C)	Fraction at water-ether interface[a]
0	None[b]	<0.05
	190	~0.8
	250	1.0
5	190	1.0
	250	1.0

[a] Fraction of wood flour that remained at water-ether interface after extraction.

[b] Unprocessed, extracted wood flour.

pearance of both the composite and the wood flour residue after exposure to the higher temperature—particularly in the presence of E-43. Because of this complicating extrusion temperature effect on surface polarity and the low sensitivity of the infrared, we are unable to verify that the E-43 was indeed concentrated at and chemically bonded to the wood surface.

Experiment B

In Experiment B, cantilever beam strength and modulus, notched impact energy, and heat deflection temperature were determined for composites containing wood flour in either polypropylene or high density polyethylene. Figures 8 to 11 plot the results of these variables as a function of E-43 level; Table VI gives the percentage increases in the properties at 1 percent E-43 relative to zero E-43 and for polypropylene relative to high density polyethylene (HDPE), both at 1 percent E-43. As noted, no statistical analysis was attempted or warranted in Experiment B, and conclusions must obviously be drawn more cautiously.

In view of the impact data in Figure 6, the rise in impact energy above 0.75 percent E-43 for polypropylene-wood flour (PP-WF) composites (Figure 11) must be viewed as anomalous. The effect of E-43, therefore, was similar for both the PP-WF and the HDPE-WF systems, with the possible exception of cantilever beam maximum strength, where the PP-WF system was increased to a greater extent (Figure 9; Table VI, columns 2 and 3). Because unfilled polypropylene has higher strength and stiffness than does unfilled HDPE, the somewhat greater strength, stiffness, and heat deflection temperature and lower impact energy of the PP-WF

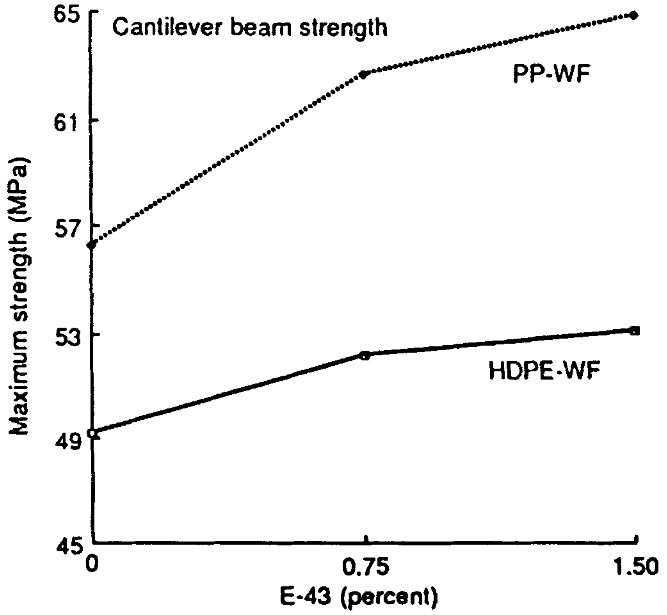

FIGURE 8 Cantilever beam strength of polypropylene-wood flour (PP-WF) and high density polyethylene-wood flour (HDPE-WF) composites as a function of Epolene E-43 concentration.

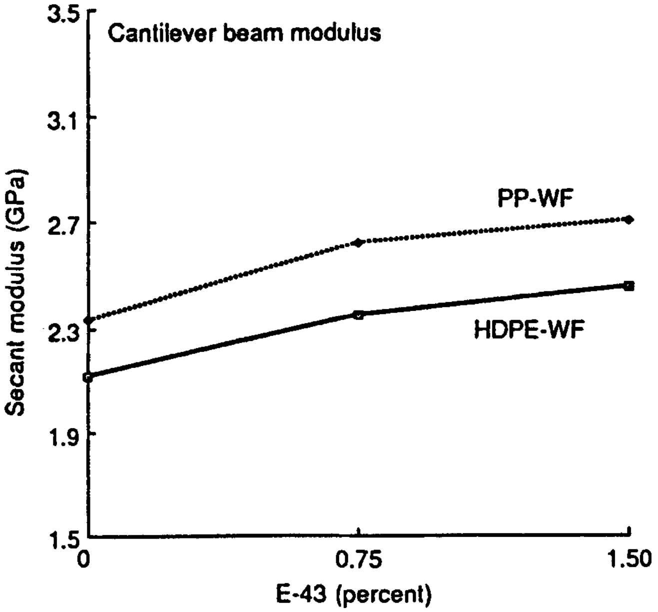

FIGURE 9 Cantilever beam secant modulus of PP-WF and HDPE-WF composites as a function of Epolene E-43 concentration (abbreviations defined in Figure 8).

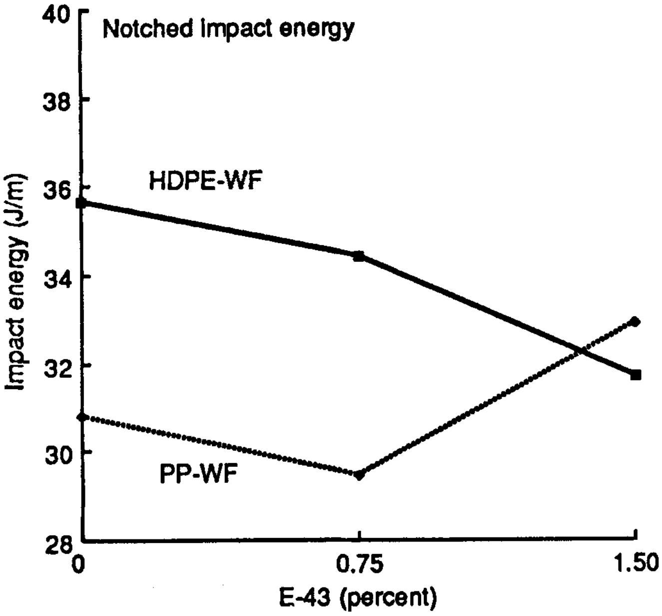

FIGURE 10 Notched impact energy of PP-WF and HDPE-WF composites as a function of Epolene E-43 concentration (abbreviations defined in Figure 8).

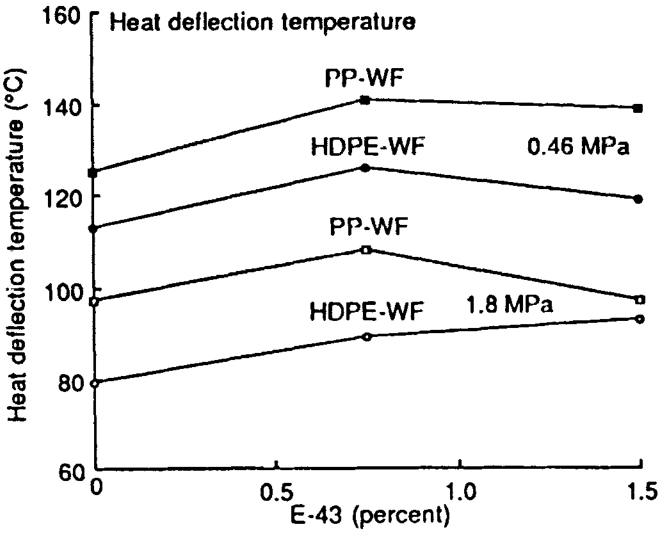

FIGURE 11 Heat deflection temperature of PP-WF and HDPE-WF composites as a function of Epolene E-43 concentration (abbreviations defined in Figure 8).

TABLE VI

Property changes for polypropylene and polyethylene composites at one E-43 level in Experiment B

Property	Change in property (percent)[a]		
	1 percent E-43/ zero E-43		PP-WF/HDPE-WF 1 percent E-43
	PP-WF	HDPE-WF	
Cantilever beam maximum strength	13	5	23
Cantilever beam secant modulus	13	12	11
Notched impact energy	-1	-6	-9
Heat deflection temperature			
At 0.46 MPa	12	9	14
At 1.82 MPa	8	14	17

[a] PP-WF, polypropylene-wood flour composites.
HDPE-WF, high density polyethylene-wood flour composites.

composite is not surprising (Table VI, column 4). The choice between the two matrix polymers for a particular application might well depend less on these property differences than on cost and processing considerations.

CONCLUSIONS

A statistically designed study was performed to determine the influence of Epolene E-43 concentration (0 to 5.0 percent by weight) and extrusion temperature (190°C to 250°C) on the mechanical properties of polypropylene-wood flour (PP-WF) composites (50/50 weight composition). A screening study was also performed to compare the influence of E-43 concentration (0 to 1.5 percent) on both PP-WF and high density polyethylene-wood flour (HDPE-WF) composites.

The major findings and conclusions were as follows:

1. Wood degradation occurred during extrusion of the PP-WF system, particularly at the higher temperatures and in the presence of E-43. This, plus the surface-modifying effect of E-43, led to a less polar surface on the wood particles.
2. Little or no advantage is to be gained by using more than 1 to 2 percent E-43 or extrusion temperatures beyond 200°C to 210°C in the PP-WF system. At those conditions, small but potentially useful increases in the strength, modulus, and heat deflection properties of both PP-WF and HDPE-WF composites (50/50 weight ratios) can be achieved relative to the systems without E-43. These positive effects of E-43 are ascribed to enhanced reinforcement through better dispersion of the wood flour in the polymer or through greater bonding between wood particles and polymer.
3. Impact energy dropped slowly throughout the ranges of E-43 concentration or extrusion temperature examined. We assume this loss in impact resistance resulted because of the greater reinforcement induced by the E-43 and because of greater wood brittleness induced by degradation.
4. At the same composition, the heat deflection temperature, strength, and modulus of the PP-WF system are marginally better than those of the HDPE-WF system, and the impact resistance is marginally poorer. From the available evidence, lower cost recycled HDPE plus E-43 could yield composites competitive in cost and properties with those from virgin polypropylene. This assumption is being studied further.

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