Waste-Newspaper-Polypropylene Thermoplastic Composites: Research- and Plant-Scale Studies of Commercial Feasibility

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Introduction

Wastepaper and paperboard are a major fraction of municipal solid waste in the United States—approximately 37 percent by weight in 1990 (1). Unfortunately, the majority of these wastes are still landfilled. In addition to the costs of landfilling, such disposal constitutes a loss of a valuable resource. One promising use of this resource is as a reinforcing tiller in thermoplastics, where it reduces material costs and enhances numerous properties, for example, strength, stiffness, heat distortion temperature, and creep resistance (2). Compared to inorganic fillers, paper fibers as fillers will also lead to composites of lower density, hence lower volumetric cost.

However, one problem with employing wastepaper fiber in composites is its low bulk density when refiberized, which makes it difficult to handle and feed into compounding equipment such as extruders. This problem was resolved on a laboratory scale at the University of Toronto (Toronto, Ontario) where compounding of waste newspaper (ONP) and polypropylene (PP) was achieved using the paper as ground particles in a 1-L K-mixer, a high intensity, short cycle, batch blender (2,3). At 50 weight percent paper fiber, the resultant blend exhibited large increases in some properties compared with those of the unfilled polypropylene, for example, flexure strength (increased 1.5 times), flexure modulus (increased more than 3 times), and heat distortion temperature (increased 60°C). Early tests at the USDA Forest Service, Forest Products Laboratory (FPL) (Madison, WI) showed that ONP/PP composites possessed better strength, stiffness, and impact properties than similar composites made from wood flour (WF) and PP, a blend that has been commercially extruded into sheets by American Wood-Stock for more than a decade. Although the results of these early tests were highly promising for potential employment of wastepaper in composites, further research and development efforts were clearly necessary before wastepaper fiber could be accepted for such use.

In 1991, we began a cooperative program to answer some remaining questions regarding the commercial use of wastepaper fiber as a reinforcing agent in thermoplastic composites. The program was partially funded by a Waste Recycling and Reduction Demonstration Grant from the Wisconsin Department of Natural Resources. Participants included the FPL, the University of Wisconsin-Madison, the University of Toronto, and several industries. The overall program objectives were to assess the commercial feasibility of melt-processed ONP/PP composites and estimate the potential of these composites for diverting wastepaper from Wisconsin landfills. Secondary technical and technological objectives were as follows:

- develop composite formulations with the desired balance of mechanical and processing characteristics,

- establish that laboratory success in compounding waste newspaper and polypropylene with a 1-L K-mixer can be scaled to industrial-sized K-tiers,

- establish whether other industrial-scale compounding equipment can successfully prepare the same composite blends, and

- establish whether composite blends can be melt processed in existing industrial extrusion and injection molding equipment to fabricate useful articles.

Program Organization

The program was carried out in three phases that integrated research, pilot plant, and full plant efforts. Throughout the program, ONP/PP blends were compared to similar WF/PP blends, because the latter were already in commercial production.

The three phases of the program were as follows:

Phase I–A range of ONP/PP composites were blended in a laboratory-scale K-mixer (1 L), and melt viscosity and mechanical properties were tested to establish guidelines for selection of formulations in Phases II and III.

Phase II–Selected ONP/PP composites were commercially blended in a 40-L K-mixer in tens-of-kilogram quantities, and mechanical properties were tested to verify the capability of the larger K-mixer for blending and to provide additional guidelines for selection of scale-up formulations.

Phase III–Selected ONP/PP composites were commercially blended in a 40-L K-mixer in thousands-of-kilogram quantities, mechanical properties were laboratory tested, and industrial extrusion and injection molding operations were evaluated. Blending by a pilot-plant extruder was also investigated to test capabilities for blending ONP/PP.

Figure 1 shows the project organization, names of cooperators, and cooperators' roles.

Experimental

Materials

Polypropylene was supplied by Solvay Polymers, inc. (Deer Park, TX) as nominal 3, 12, and 30 melt flow index (g/10 min at 230°C) material (that is, Fortilene 9101[l] , 1602, and 1902, respectively). The ONP was production overrun material and was ground to 6- to 12-mm particles as feed to the K-mixer. For tests of blending by extrusion, the ONP was obtained as hammermilled fiber from Custom Fibers International (Elkwood, VA) (CF 30500, approximately 70% larger than 80 mesh (0.177 mm)). The WF was obtained from American Wood Fiber Co. (Schofield, WI) as -40+80 mesh western pine. Epolene E-43 and G3002 (maleated polypropylenes) were supplied by Eastman Chemical Products, Inc. (Kingsport, TN).

Compounding Procedure

The 1-L K-mixer blends were prepared at the University of Toronto. Epolene E-43 and the paper particles were mixed at 3,300 rpm (blade tip speed 22 m/s) for approximately 90 s, after which the polymer was added. During continued mixing for approximately 60 s, the polymer melted, the fibers were dispersed into the molten polymer, and the resultant mixture was heated to 185°C, at which point the blend was automatically discharged Approximately twenty 250-g batches of each formulation were prepared and blended after granulation.

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[l]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.

The 40-L K-mixer blends were prepared at Synergistics Industries Ltd. (St. Remi de Napierville, Quebec). For Phase II, approximately five 5-kg batches of each formulation were blended and subsequently pelletized. The K-mixer was operated at 6,100 rpm (tip speed 40 m/s), and residence times were approximately 60 s. Two discharge temperatures were used for each ONP/PP formulation, differing by 10°C for each formulation but ranging from 200°C to 215°C for all formulations.

For Phase III, numerous batches were blended semicontinuously in the K-mixer to produce approximately 5,000 kg of each formulation Maximum tip speeds were approximately 41 m/s, and maximum discharge temperatures were 215°C to 224°C. Werner & Pfleiderer (Ramsey, NJ) used a twin-screw corotating intermeshing extruder (40-mm screw diameter) with downstream feeding of the paper fiber to prepare several hundred kilograms of several formulations from paper fiber feed and polypropylene at a variety of screw configurations and speeds.

Mechanical Properties

Specimens for mechanical property measurements on pelletized or granulated blends were prepared by injection molding, using a Frohring Minijector Model SP50 plunger-type injection molder at 204°C, a ram pressure of 3 to 9 MPa, and a mold temperature of 30°C. For sheets extruded at American Wood-Stock (Sheboygan, WI), specimens were machined. Specimens were stored over a desiccant for at least 3 days before testing. Specimens and testing procedures followed ASTM specifications (4).

Melt Viscosity

Apparent viscosities at apparent sheer rates were calculated from volumetric throughput rate and die pressure drop during steady state extrusion at 190°C with a Brabender Plasticorder using a 1.8-mm capillary die at L/D 45.

Plant-Scale Evaluation

American Wood-Stock extruded blends in a production twin-screw extruder (170 mm, counter rotating, intermeshing) to prepare 2-mm-thick by 600-mm-wide sheet at approximately 600 kg/h. Thermoformability of the sheet was investigated using proprietary laboratory tests plus production of a full automotive interior panel. Tee1 Plastics Co. (Baraboo, WI) extruded blends in **a** production single-screw extruder (90 mm) to prepare 75-mm ID tubing, using a cooled vacuum sleeve to control diameter. Flambeau Corp. (Baraboo, WI) used a production mold and injection molder (680-t DeMag) to make an ammunition packaging support (a complex cylinder approximately 420 mm long and 75 to 125 mm in outer diameter with 8-mm-thick walls).

Laboratory Studies (Phases I and II)

Phase I was statistically designed and was conducted strictly in accordance with the design. Phases II and III were originally planned as statistical studies but in actual practice did not follow the designs because limitations arose at industry cooperators because of schedule and supply problems. Consequently, we present a statistical analysis only for Phase I. For purposes of discussion, we assume that change in properties must be at least 20% to be considered of practical importance. Also, portions of Phase I were summarized earlier (5).

TABLE 1. Phase I Matrices[a]

Variable	Level
Main matrix[b]	
PP melt flow index	3, 12, or 30 g/10 min
ONP/PP weight ratio	32, 37, or 42 weight percent
E-43 coupling agent content	5 weight percent of filler content
Supplemental matrix	
PP melt flow index	3, 12, or 30 g/10 min
WF/PP weight ratio	42/58
E-43 coupling agent content	5 weight percent of filler content

[a]Blends compounded in 1-L K-mixer.
[b]Full factorial, replicated.

TABLE 2. Phase II Matrices[a]

Matrix	PP MFI (g/ 10 min)	Filler	PP/filler (weight ratio)	Additive (weight % filler)	Discharge temperature[b]
Main[c]	12	ONP	58/42 or 68/32	G3002 (0 or 3)	Low or high
Supplemental					
A	12	WF	58/42	G3002 (3)	Low or high
B	12	ON-P	58/42	E43 (3)	Low or high
C	30	ONP	68/32	G3002 (3)	Low or high

[a]Blends compounded in 40-L K-mixer.
[b]Total temperature range 195°C to 215°C; for each pair (low or high), range was 10°C.
[c]Full factorial, partially replicated.

Test Matrices

Test matrices are summarized in Tables 1 and 2. As noted, the goals were to define formulations for scale-up and to verify the ability to Scale from the 1-L to the 40-L K-mixer. In addition to K-mixer size, the variables included PP melt flow index (MFI), ONP content, filler (ONP or WF), coupling agent, and K-mixer discharge temperature, which slightly prolonged fluxing time.

Effects of PP MFI, ONP Content, and Filler Type

Phase I focused on the effects of PP MFI, ONP content, and filler type. Table 3 summarizes the mechanical properties tested, Table 4 summarizes the influences of several variables on mechanical properties, Table 5 presents the results of the analysis of variance, and Figures 2 and 3 illustrate observations. Neither the tenfold change in PP MFI (Table 4, item 1) nor the increase in ONP content from 32% to 42% (Table 4, item 2) produced changes in properties having practical significance. However, compared with unfilled plastic, adding 42% WF (Table 4, item 3) more than doubled flexural modulus, increased flexural strength nearly 50%, and nearly doubled the heat distortion temperature; simultaneously, however, it drastically reduced

unnotched impact energy. Substituting ONP for the WF provided even greater increases in strength, modulus, and heat distortion temperature while somewhat mitigating the loss inunnotched impact energy (Table 4, items 4 and 5).

On the basis of these Phase I findings, Phase II focused on effects of coupling agents, scale-up to the 40-L K-mixer, and K-mixer discharge temperature.

Effects of Coupling Agent and K-Mixer Variables

Flexure and impact properties for composites made with the 40-L K-mixer are summarized in Table 6. Figures 4 and 5 illustrate the coupling agent effects on mechanical properties for flexure strength and unnotched impact energy, respectively. Addition of Epolene coupling agents, particularly G3002, resulted in very usefull increases in strength (62%) and unnotched impact energy (115%). The combination of relatively large simultaneous improvements in strength and unnotched impact energy appears to suggest that Epolene acts more as a dispersion aid than as a true coupling agent. Surprisingly, neither the change from 1-L to 40-L K-mixer nor the 10°C difference in K-mixer discharge temperature had a significant effect on properties (data not presented), which indicates that neither change appreciably altered the degree of fiber dispersion.

Effects on Composite Melt Viscosity

Apparent viscosities were measured at several shear rates (extruder throughput rates) below 1,000 s^{-1}. Table 7 summarizes the values obtained by interpolating linear log-log plots (Fig. 6) to 100 and 500 s^{-1} and extrapolating to 1,000 s^{-1}; the last value approaches the shear rates existing during commercial extrusion. Unfortunately, with the 3-MFI PP, equipment limitations permitted measurements only with the unfilled polymer, which is the polymer employed by American Wood-Stock in their commercial WF/PP product. At 1,000 s^{-1} shear rate, the viscosity of the 42/58 ONP/12-MFI PP system was approximately 25% more than that of the similar 32/68 ONP/PP system and nearly twice that of the 42/58 WF/PP system. Clearly, however, the greater viscosity of the ONP systems compared with the WF systems could be overcome by substituting a higher MFI PP without large property reduction.

TABLE 3. Mechanical Property Summary

PP MFI (g/10 min)	Filler type	Filler content[a]	Tensile[b] Strength (MPa)	Tensile[b] Modulus (GPa)	Flexure[b] Strength (MPa)	Flexure[b] Modulus (GPa)	Cantilever[c] Strength (MPa)	Cantilever[c] Modulus (GPa)	Impact energy (J/m) Notched	Impact energy (J/m) Unnotched
3	ONP	32	[illegible]	4.17	76.4	3.14	88.6	2.15	22.3	160
		37	[illegible]	4.33	78.9	3.33	84.5	2.35	22.5	149
		42	48.9	4.44	78.2	3.52	87.9	2.40	27.2	141
12	ONP	32	47.9	4.31	72.9	3.13	82.2	2.31	18.0	134
		37	48.5	4.32	75.2	3.35	83.0	2.33	22.7	158
		42	49.8	4.65	76.1	3.47	81.9	2.49	21.1	106
30	ONP	32	48.7	4.26	71.0	3.09	81.1	2.30	17.7	159
		37	47.7	4.46	73.5	3.36	78.3	2.48	23.6	117
		42	49.9	4.79	74.2	3.51	83.7	2.59	21.1	124
3	WF	42	39.3	4.05	62.7	3.22	77.3	2.26	23.2	105
12		-	37.6	4.53	61.6	3.53	71.3	2.38	20.5	83
30		-	38.1	4.50	59.8	3.42	67.8	2.37	24.6	73

[a]Filler/polymer weight ratio; composites contain 5% E43 by weight of filler.

[b]Maximum strength and initial modulus.

[c]Maximum strength and 9° secant modulus.

TABLE 4. Phase I Mechanical Property Changes (Ratio of Mean Property Values)

Change in composition[a]	Tensile modulus	Tensile strength	Flexural modulus	Flexural strength	Izod impact energy notched (unnotched)	Heat distortion temperature at 1.83-MPa load
3 to 30 g/10 min	1.04	0.96	1.02	0.94	0.87 (0.88)	1.00
32% to 42% ONP	1.09	1.02	1.12	1.04	1.20 (0.92)	1.16
0% to 42% WF	-	1.22	2.40	1.49	0.95 (0.13)	1.88
0% to 42% ONP	-	1.57	2.45	1.85	0.97 (0.19)	2.13
42% WF to 42% ONP	1.06	1.29	1.04	1.24	1.02 (1.43)	1.12

[a]Values for effect of filler type and content were averaged over all three PP MFI values; values for effect of MFI were averaged over all three ONP contents.

TABLE 5. Phase I Analysis of Variance Summary[a]

		Critical F ratio[b]	Cantilever Strength (MPa)	Cantilever Modulus (GPa)	Flexure Strength (MPa)	Flexure Modulus (GPa)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Impact energy (J/m) Notched	Impact energy (J/m) Unnotched
Main matrix										
Main effect	PP MFI (3, 12, or 30 g/10 min)	4.3	22.4	32.9	32.8	0.0	3.9	3.2	2.7	1.0
	ONP content (32%, 37%, or 42%)	4.3	0.3	76.6	11.9	14.8	1.5	13.2	3.4	1.9
Interactions	Filler content x MFI	3.6	1.7	4.5	0.4	0.1	2.6	0.9	1.0	1.2
Supplemental matrix										
Main effect	Filler type (WF or ONP)	6.0	253.6	10.8	17.6	4.8	8.9	12.8	0.4	27.1
	PP MFI (3, 12, or 30 g/10 min)	5.1	99.5	3.4	2.1	2.2	0.8	11.4	1.7	6.2
Interactions	Filler content x MFI	5.1	27.0	0.5	1.0	4.2	1.0	1.1	0.8	1.3

[a]Values are F ratios, which are variances of response attributable to particular variable divided by variances resulting from experimental error.
[b]Experimental F ratios must be greater than critical values for effect to be significant at 95% confidence level.

TABLE 6. Phase II Mechanical Properties[a]

Variable	Flexural modulus (GPa)	Flexural strength (MPa)	Izod impact energy (J/m) Notched	Izod impact energy (J/m) Unnotched
K-mixer discharge temperature[b]				
Low	3.68	68.0	26	127
High	3.68	67.2	26	124
Coupling agent				
None	3.37	50.8	24	75
E43	3.92 (16)	66.4 (31)	24 (0)	98 (31)
G3002	4.12 (2)	82.5 (62)	29 (21)	161 (115)

[a]42/58 ONP/12-MFI PP; values in parentheses are percentage increase compared with system without coupling agent.
[b]Values averaged over several variables.

TABLE 7. Viscosity of Composite Blends[a]

PP MFI (g/10 min)	Filler	Filler/PP (weight ratio)	Vicsocity at shear rate (s^{-1}) 100	500	1,000[b]
3		0	1,230 (3.4)		
12		0	359 (1.0)	149 (1.0)	102 (1.0)
	WF	42/58	452 (1.3)	195 (1.3)	136 (1.3)
	ONP	32/68	618 (1.7)	227 (1.5)	148 (1.5)
	ONP	37/63	728 (2.0)	269 (1.8)	175 (1.7)
	ONP	42/58	881 (2.5)	301 (2.0)	190 (1.9)
30	ONP	42/58	572 (1.6)	208 (1.4)	135 (1.3)

[a]Values in Pa-s; parenthetic values are ratios of filled system to unfilled PP at 10 MFI and particular shear rate; E-43 present at 5 weight percent of filler.
[b]Extrapolated from log-log plot of data measured between 100 and 500 s^{-1}.

TABLE 8. Phase III Matrices

Matrix	PP MFI (g/10 min)	Filler feed	PP/filler (weight ratio)	G3002 (weight % filler)	Compounding
Main	12	ONP particles or WR	58/42	0 or 3	40-L K-mixer
Supplemental					
A	3	WF	58/42	0 or 3	Twin-screw extruder[a]
B	12	ONP fibers	58/42	0 or 3	ZSK-40 twin-screw extruder[b]

[a]American Wood-Stock direct extrusion of WF/3-MFI PP.
[b]Werner and Pfleiderer tests of ability of extruder to compound ONP fiber feed and PP.

Implications for Scale-Up and Industry Evaluation

We arrived at the following conclusions for scale-up and industry evaluation in Phase III, based on the Phase I and II results and the experience of American Wood-Stock in producing a 50/50 WF/3-MFI PP extruded panel:

- The 40-L K-mixer can be employed for compounding.
- The Epolene G3002 warrants further study because it improved mechanical properties and it did not influence melt viscosity.
- A 42/58 ONP/PP ratio with 12-MFI PP and G3002 appears to offer acceptable melt processability and improved mechanical properties compared with the current WF/PP system.

Scale-Up and Industrial Evaluation (Phase III)

Test Matrices

The Phase III matrices described in Table 8 had several objectives: (a) to confirm the ability to scale the K-mixer compounding to ton quantities, (b) to evaluate the ability of plastic fabricators to melt-process the blends into commercial articles using existing equipment, and (c) to confirm that commercial extrusion could be used to compound ONP/PP blends with ONP fiber as feed.

Compounding Procedure

Synergistics compounded and pelletized more than 20,000 kg total of the main matrix blends (Table 8) using the 40-L K-mixer. American Wood-Stock encountered difficulties in extruding the WF/10-MFI PP blends and therefore substituted their own WF blends with 3-MFI PP (Supplemental blend A). Werner and Pfleiderer prepared several hundred kilograms of ONP/PP blend (Supplemental blend B), and established that their pilot plant twin-screw extruder could be used to compound ONP/PP with ONP fibers as direct feed instead of the ONP particles employed as feed for the K-mixer.

Mechanical Properties

We observed some small, marginal differences in mechanical properties among blends compounded with the 1-L K-mixer (several-kilogram scale in Phase I), the 40-L K-mixer (tens-of-kilogram scale in Phase II and thousands-of-kilogram scale in Phase III), and the Werner and Pfleiderer extruder (several-hundred-kilogram scale). We attribute the differences to variations in fiber length and dispersion and in compounding conditions, such as temperature, shear, and residence time. Figures 7 and 8 compare flexure strength and unnotched impact energy, respectively, for extruded American Wood-Stock panels made from ONP or WF, with and without G3002. The data confirmed expectations. For a given blend, flexure strength and unnotched impact energy increased in the following order: cross machine direction (XMD), machine direction (MD), and reground/injection molded (inj). Moreover, G3002 provided superior properties, and particularly with G3002, the ONP system was superior to the WF system.

Industry Evaluation

Overall, American Wood-Stock was very positive about their ability to replace their current WF/PP formulation with an ONP/PP formulation. They encountered no difficulties in extruding the ONP blends into their standard sheet using existing production facilities and acceptable conditions, and power requirements and material output rates were approximately equal for ONP/PP and WF/PP blends. In addition, the 2.0-mm ONP/PP sheet appeared equivalent in mechanical properties to the presently marketed 2.50-mm WF/PP sheet, presenting the possibility of lighter weight and lower cost panels. American Wood-Stock reached the following conclusions from subsequent tests of thermoformability:

- Relative to the WF system, the ONP system has a wider operating temperature range, is more easily handled when hot, and performs better in shallow die-draw moldings.

- The ONP system produces satisfactory production moldings for an automobile rear deck (Fig. 9).

In contrast to American Wood-Stock the other plastic fabricators in the project had little previous experience with highly filled plastics, and their processes and equipment were oriented towards lightly filled or unfilled plastics. As a result, evaluations of these other fabricators were encouraging but not conclusive. Teel Plastics, for example, was able to extrude tubing with both the WF and ONP composites (Fig. 10), but neither type completely met specifications. Flambeau was unable to completely fill the mold for their injection molded ammunition packaging support with the 42/58 ONP/PP blend but were successful after dilution to 21/79 ONP/PP (Fig. 10). Modifications to the mold probably could have resolved the problem, but those changes could not be made as part of this project,

Conclusions

This project achieved its major goal of proving technical viability of old newspaper-thermoplastic composites on a commercial scale. This project was a rather unique example of research and development cooperation between diverse groups with a common goal. Specific conclusions follow:

- Relative to unfilled plastic products, ONP-filled products possess several advantages (for example, higher strength (50% to 80%), stiffness (145%), and heat distortion temperature (113%)).

- Epolene G3002 imparts very useful property improvements to ONP/PP composites (for example, strength increased 62% and unnotched impact energy increased 115 %).

- ONP/PP composites can be compounded on a commercial scale using either the K-mixer with ONP particles as feed or using a twin-screw extruder with ONP fibers fed separately from the plastic.

- ONP/PP sheet containing 42 weight percent ONP can be prepared by extrusion on a commercial scale. This sheet meets existing specifications for automobile panels and can be thermoformed into a variety of shapes.

- Given proper design of melt processing equipment, a wide variety of commercial products other than automobile panels can be manufactured from ONP/PP composites with similar ONP content.

- Firm estimates of production costs for ONP/PP composite products must await the following:

  - additional examination of compounding methods to define the optimum balance of dispersion ability, throughput rate, and cost;

  - improvement in methods to deliver wastepaper in a form and at a cost acceptable to a compounder or a manufacturer of plastic products, keeping in mind that wood flour is available in large quantity at approximately $0.22 per kilogram.

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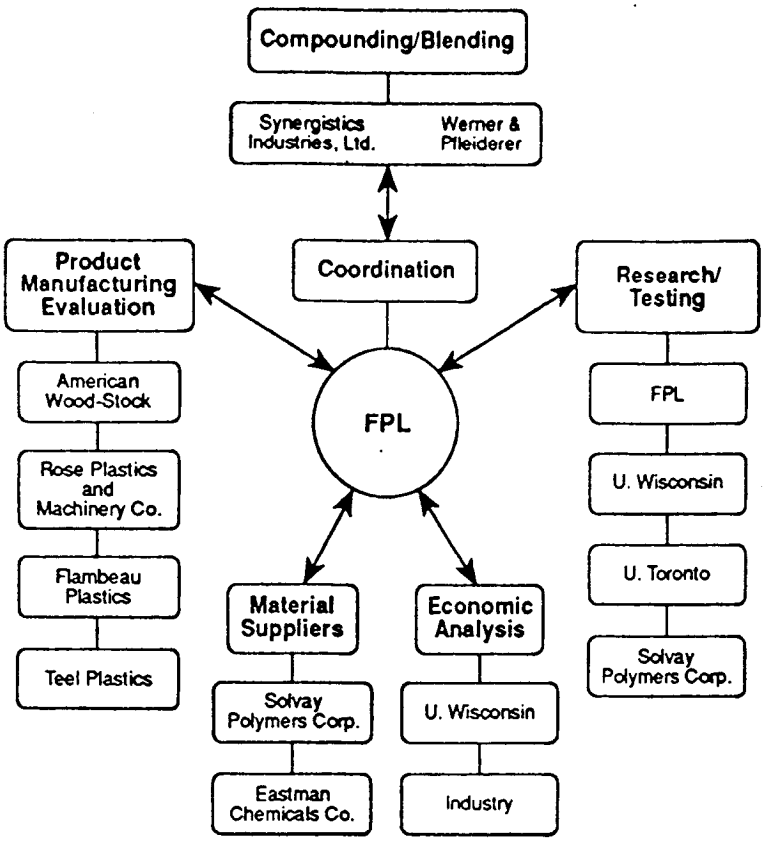

FIGURE 1. Project organization.

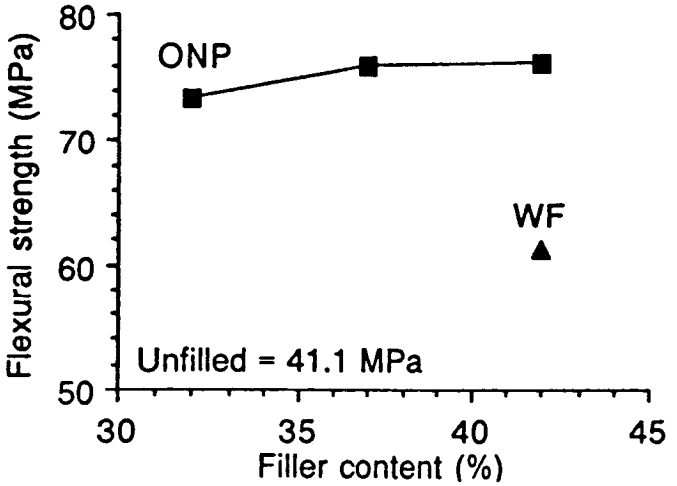

FIGURE 2. Effect of filler content and type on flexural maximum strength (averaged over 3-, 12-, and 30-MFI PP) (ONP is waste newspaper; WF is wood flour).

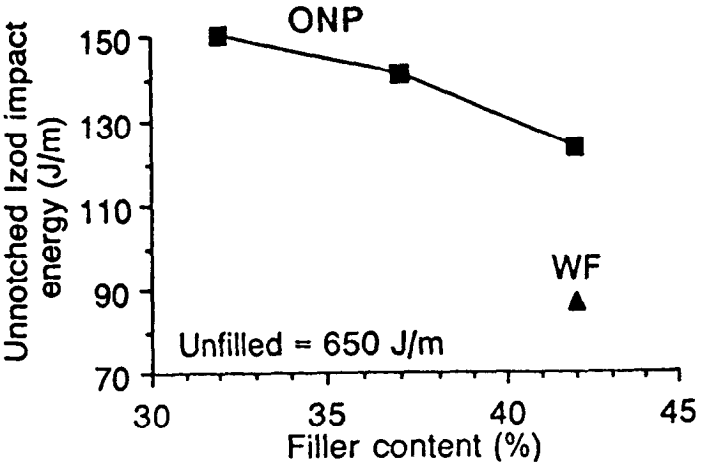

FIGURE 3. Effect of filler content and type on unnotched Izod impact energy.

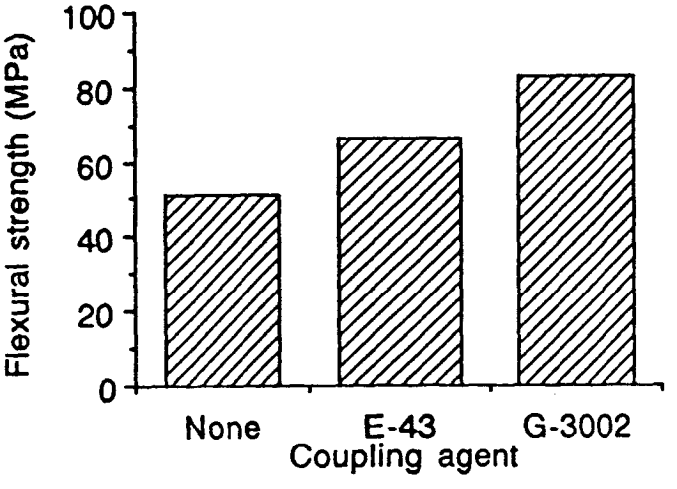

FIGURE 4. Effect of coupling agents on flexural maximum strength of ONP/PP composite (12-MFI PP, 42% filler).

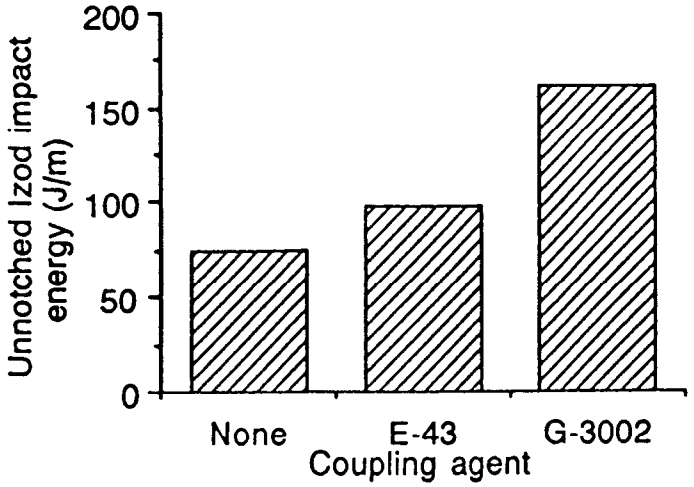

FIGURE 5. Effect of coupling agents on unnotched Izod impact energy of ONP/PP composite (Pig. 4).

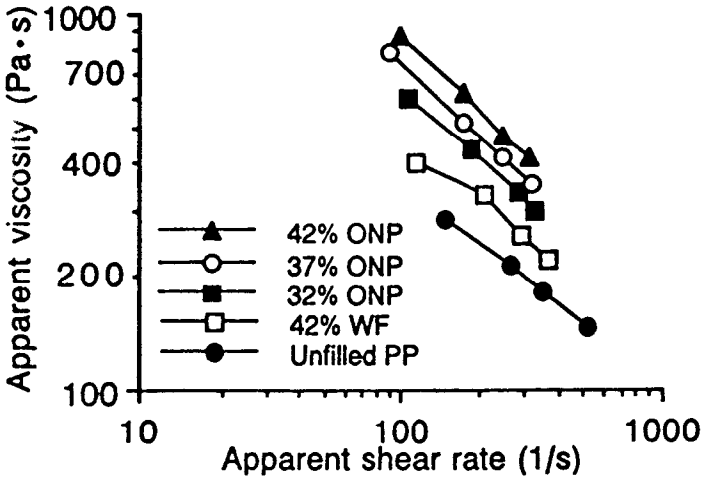

FIGURE 6. Apparent melt viscosity of composites (12-MFI PP).

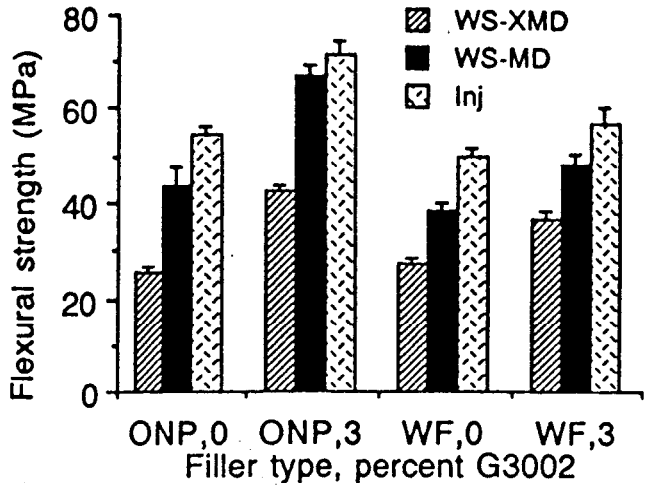

FIGURE 7. Flexural maximum strength for extruded sheet. WS is American Wood-Stock extruded sheet; XMD is across extrusion direction; MD is along extrusion direction; Inj is sheet reground and injection molded.

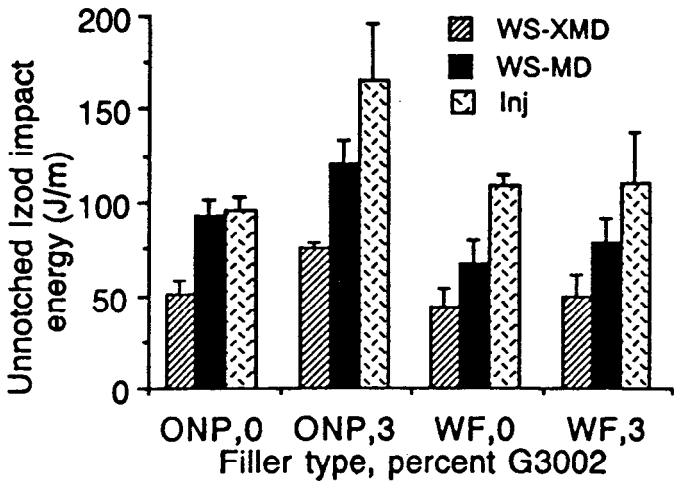

FIGURE 8. Unnotched Izod impact energy for extruded sheet.

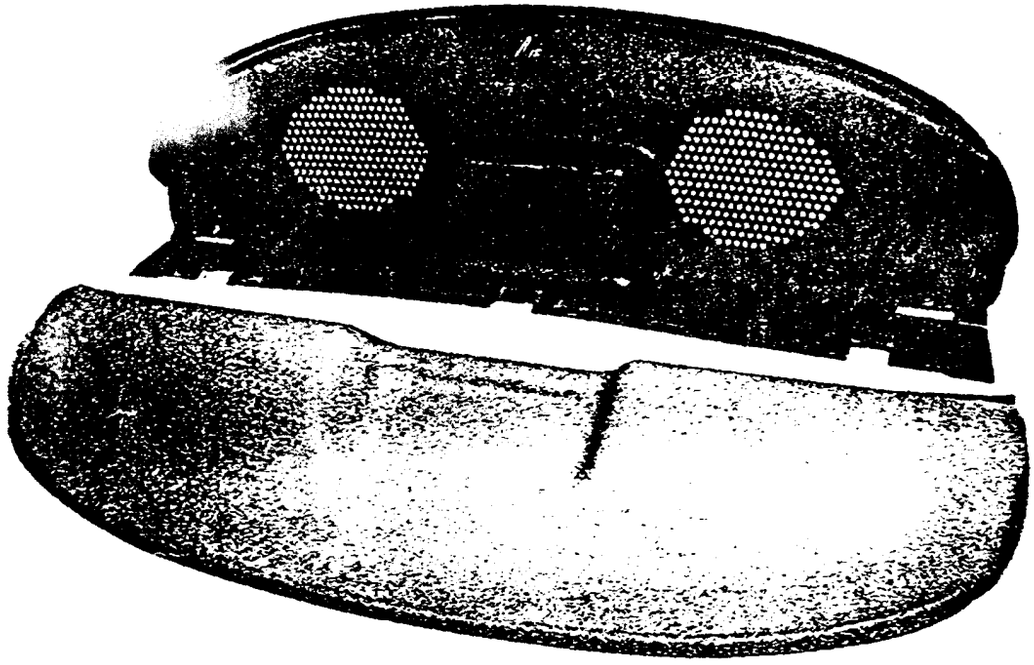

FIGURE 9. Automobile rear deck panel extruded and thermoformed by American Wood-Stock from ONP/PP composite.

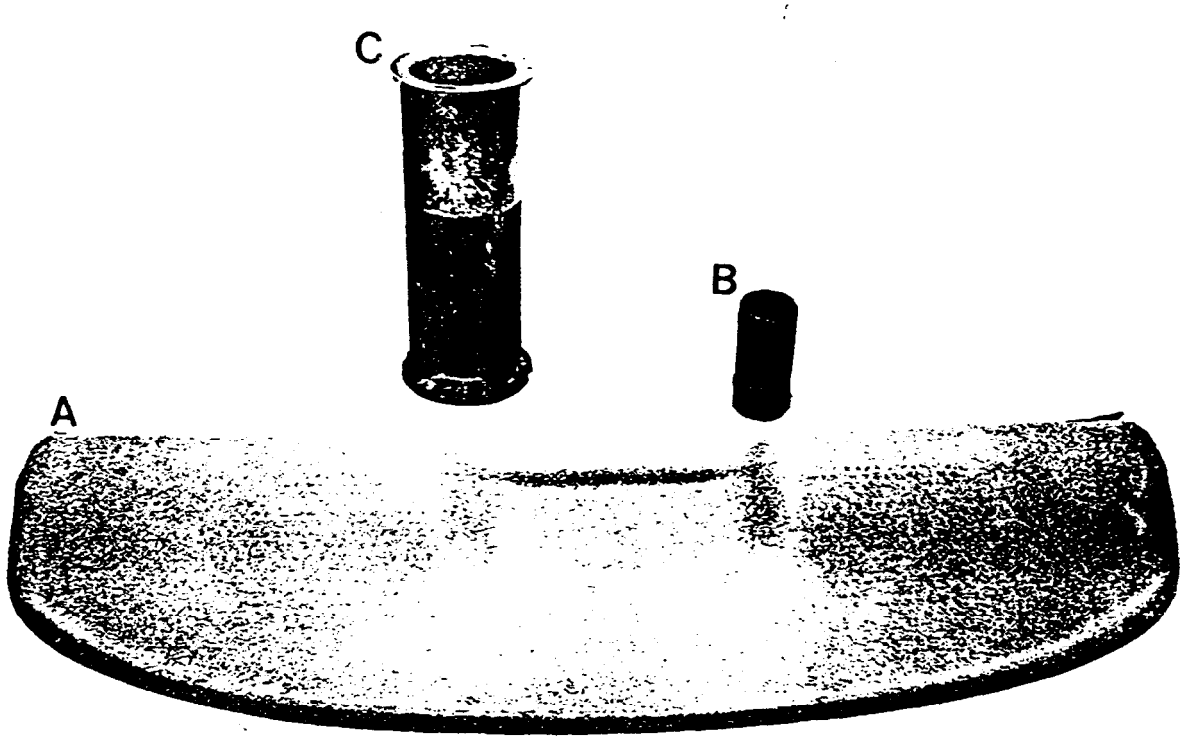

FIGURE 10. Melt-fabricated articles from ONP/PP composites: (A) automobile interior panel made by thermoforming extruded sheet at American Wood-Stock; (B) tube extruded by Tee1 Plastics; (C) ammunition packing support injection molded by Flambeau Corp.