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Compounding and Processing Additives for Woodfiber-Plastic Composites

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Quality Additives for Performance

ABSTRACT

The use of wood and other cellulosic fillers have been used extensively to fill both thermoset and thermoplastic compounds for decades. Recent advances in compounding technology and further research into this area have resulted in the development of injection molding and profile extrusion grades of thermoplastic compounds which provide many benefits to a wide variety of end-users. However the use of these types of fillers, like other fillers, can cause a number of compounding and processing problems associated with the dispersion of the filler and flow characteristics of the resulting mixed composite. Common deficiencies include surface mottling and fracture, poor flow characteristics and compounding problems due to low temperature mixing requirements.

In this presentation we will discuss the use of chemical process additives, viscosity modifiers and dispersion enhancers in wood flour-filled polypropylene composites. It is our goal to show improvements in flow, surface appearance and physical properties at specific levels of filler loading and further that the use of these types of additives may allow for higher levels of filler loading without sacrificing processability, performance properties or surface appearance.

EFFECT MECHANISMS OF THE ADDITIVES:

- ▶ Tribological
 - Adhesives
 - Lubricants
 - Surfactants
- ▶ Molecular

EFFECT MECHANISMS OF THE ADDITIVES:

- ▶ Tribological
 - Adhesives
 - Increased interfacial forces created by surface attachment
 - Increase energy required to break adhesive bonds causing increased shear
 - Lubricants
 - Function to minimize the frictional forces between moving surfaces
 - Can be divided into internal and external
 - Internal is polymer: polymer, polymer: filler interaction
 - External is polymer: hot metal, filler: hot metal interaction
 - Surfactants
 - Create a surface active film via polar and non-polar ends
 - Polar end absorbs/bonds to a surface
 - Wetting of the filler allows for improved low energy dispersion
 - Similar to lubricants effect

DIFFERENCES IN LUBRICANT TYPES:

Taken from PVC based terminology:

▶ **External**

- Typically provide lubrication between the polymer and the metal surface of the processing equipment.

- **Types**

Polyethylene Homopolymers, Paraffins, Esters, Metallic Soaps, Amides, Fatty Acids and Oxidized Polyethylenes

▶ **Internal**

- Typically reduce bulk viscosity by being partially compatible with the PVC, thus helping to open the polymer chain with the lubricants' soluble component, while providing intermolecular lubrication with the less soluble portion of the lubricant molecule.

- **Types**

Fatty Alcohols, Esters, EVA Waxes

SPECIFIC EFFECTS OF LUBRICANTS

■ **Internal Lubricants**

- ▶ Promote flow
- ▶ Exhibit good clarity
- ▶ Promote good weld line strength
- ▶ Minimize sink marks
- ▶ Improve die filling
- ▶ Reduce die swell
- ▶ Allow increased molding speed without shear burn
- ▶ Reduce head and back pressure
- ▶ Do not affect paintability
- ▶ Lower heat distortion

■ **External Lubricants**

- ▶ Provide metal release
- ▶ Help reduce process temperature
- ▶ Can plate out
- ▶ Slow fusion
- ▶ Can cause delamination
- ▶ Can lower weld line strength
- ▶ Can cause surging



EFFECT MECHANISMS OF THE ADDITIVES:

- ▶ Molecular
 - Chemical alteration of molecular weight average and number
 - In polypropylene, addition of peroxide results in chain scission
 - Lower molecular weight results in increased flow rates/low viscosity



TYPICAL PROPERTIES OF HIGHLY FILLED COMPOUNDS:

- ▶ Increased:
 - ✓ Viscosity
 - ✓ Flexural modulus
 - ✓ Heat deflection temperature (HDT)
 - ✓ Dimensional stability

- ▶ Decreased:
 - ✓ Izod Impact
 - ✓ Mold shrinkage
 - ✓ Thermal expansion
 - ✓ Part cost



MATERIALS USED IN THIS PROGRAM:

- ▶ Exxon Homopolymer Polypropylene PD3625 (38 MFI)
- ▶ American Wood Fibers 8010 Hardwood Flour (120 Mesh)
- ▶ Struktol TR016
 - A unique blend of specialty amide and calcium stearate
 - Specialty amide provides sites that can hydrogen bond to filler for improved wetting and dispersion
 - Also a very effective metal release agent
- ▶ Struktol TR251
 - Blend of TR016 and ethylenebis-stearamide (EBS)
 - EBS portion provides improved dispersion to complement the wetting characteristics of TR016
 - Also a very effective metal release agent
- ▶ Struktol TR060
 - Highly branched blend of resinous materials acts as an adhesive (mechanical) dispersion aid
- ▶ Elastochem EF(DBDB)-60 (60% Peroxide on Wax)



FORMULATIONS:

40% Wood Flour Composites

<u>Material</u>	<u>Resin</u>	<u>Cont.</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>
PP Resin	100	60	60	60	60	60	60	60
Wood Flour		40	40	40	40	40	40	40
TR251			1	2				
TR016					1	2	1	1
TR060							1	
60% Peroxide								0.1
TOTAL	100	100	101	102	101	102	102	101.1



FORMULATIONS:

60% Wood Flour Composites

<u>Material</u>	<u>H</u>	<u>I</u>	<u>J</u>	<u>K</u>	<u>L</u>	<u>M</u>	<u>N</u>
PP Resin	40	40	40	40	40	40	40
Wood Flour	60	60	60	60	60	60	60
TR251		1	2				
TR016				1	2	1	1
TR060						1	
60% Peroxide							0.1
TOTAL	102	101	102	101	102	102	101.1



LABORATORY EXPERIMENT RESULTS:

Data generated is based on compounding on a Brabender PL2000 torque rheometer using the bowl mixer equipped with cam blades at 160°C and 90 rpm. The total time of the mixing cycle was 4 minutes.

Capillary rheometer testing was performed on a Shimadzu CFT-500C equipped with a 10 mm by 1 mm diameter die and set at 190°C. A variety of shear rates were used to give a complete viscosity picture.

Physical property data was generated using injection molded test specimens prepared on a Cincinnati Milacron 33 ton lab molding machine. The temperature/pressure profile was:

Barrel	355-365°F
Nozzle	370°F
Mold	100°F
Pressure	1200 psi

The wood flour was dried overnight at 100°C prior to processing.



BRABENDER MIXING DATA:

40% Wood Flour Composites

	<u>Cont.</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>
Peak Torque, Nm	90	68	57	67	62	65	55
Torque @ 2', Nm	11	13	10	12	12	11	9
Torque @ 3', Nm	9	11	8	11	11	10	6
Torque @ 4', Nm	9	11	7	11	10	10	5
Equilibrium Temp, °C	174	172	171	175	172	173	169
Metal Release Rating	+	++++	++++	+++	+++	+++	++

60% Wood Flour Composites

	<u>H</u>	<u>I</u>	<u>J</u>	<u>K</u>	<u>L</u>	<u>M</u>	<u>N</u>
Peak Torque, Nm	100	68	57	59	58	65	64
Torque @ 2', Nm	16	15	15	16	14	14	9
Torque @ 3', Nm	13	13	13	14	13	13	7
Torque @ 4', Nm	11	13	12	13	12	12	6
Equilibrium Temp, °C	184	181	180	181	180	179	171
Metal Release Rating	+	+++	++++	+++	+++	+++	++

Metal Release + = Poor ++ = Fair +++ = Good ++++ = Excellent



PRELIMINARY CONCLUSIONS:

Review of the mixing data indicates that there is very little effect on the viscosity/processability of the compounds even by adding relatively high levels of lubricants (up to 2 phr). The only significant effect was on Peak Torque. Based on the Peak Torque data we have established that the mixing procedure is the primary cause of this drop in torque.

Our current mixing procedure includes dry blending the formulations in a Waring blender (simulation of Henschel mixer) prior to melt compounding on the Brabender. We established that the inclusion of an adhesive, lubricant and/or surfactant significantly increases the bulk density of the resulting powdered dry blend, allowing for easier incorporation of filler into the polymer melt during mixing. Although this did not lead to a reduction in equilibrium torque or the time to the peak torque, the overall energy used to mix was significantly reduced.

The only other noted difference was metal release, which was improved dramatically, although at varying degrees depending on the additive used.



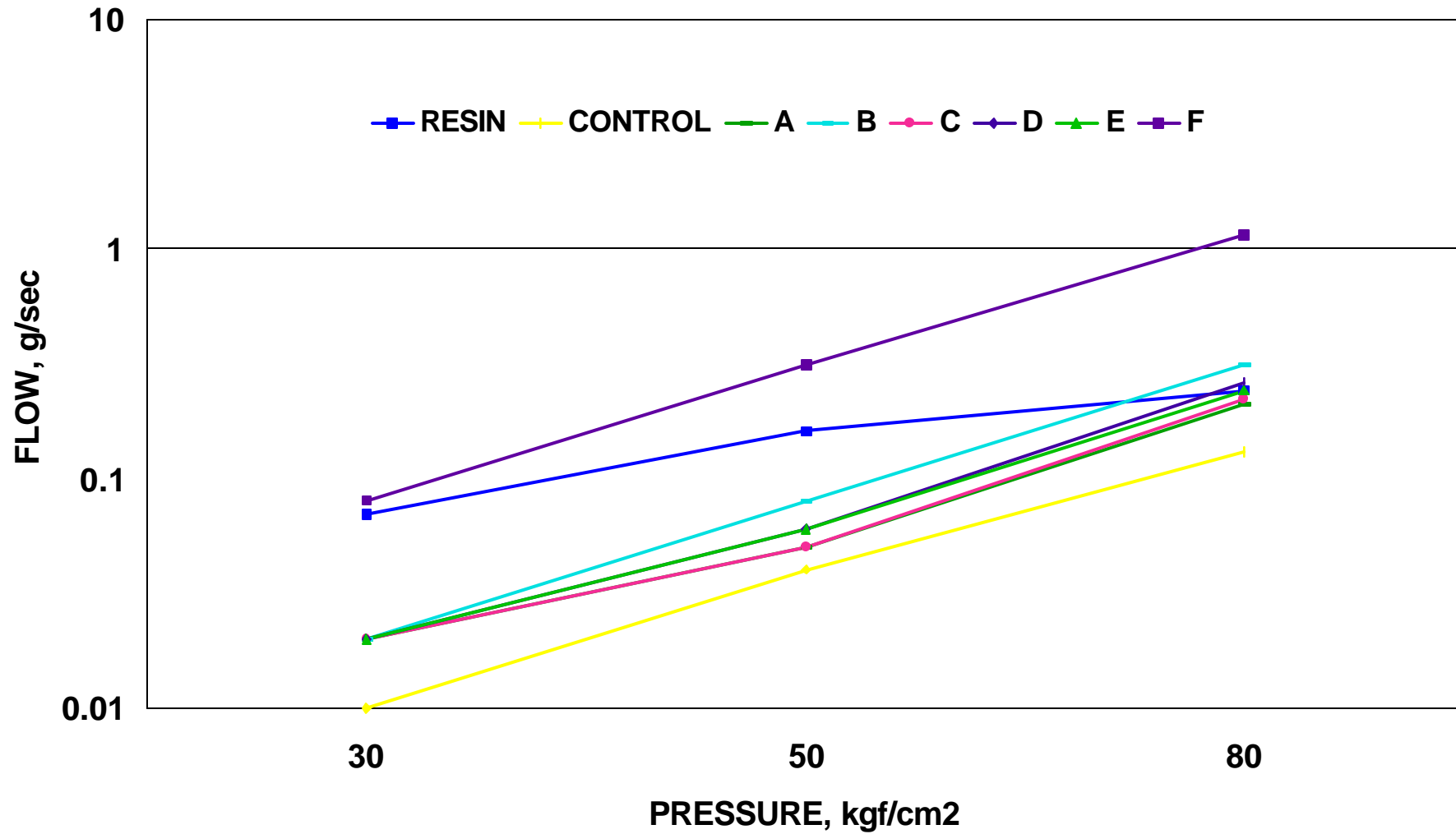
CAPILLARY RHEOMETER DATA:

40% Wood Flour Composites

	<u>Resin</u>	<u>Cont.</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>
30 kgf/cm ² @ 190°C								
Flow, g/sec	0.07	0.01	0.02	0.02	0.02	0.02	0.02	0.08
Viscosity, Pa·s	102	564	423	305	382	350	387	89
Shear, sec ⁻¹	718	130	173	240	192	209	190	822
50 kgf/cm ² @ 190°C								
Flow, g/sec	0.16	0.04	0.05	0.08	0.05	0.06	0.06	0.31
Viscosity, Pa·s	68	274	216	139	382	183	199	39
Shear, sec ⁻¹	1792	446	567	879	192	668	615	3126
80 kgf/cm ² @ 190°C								
Flow, g/sec	0.24	0.13	0.21	0.31	0.22	0.26	0.24	1.16
Viscosity, Pa·s	79	152	90	62	89	74	82	17
Shear, sec ⁻¹	2482	1283	2167	3146	2209	2637	2394	11770



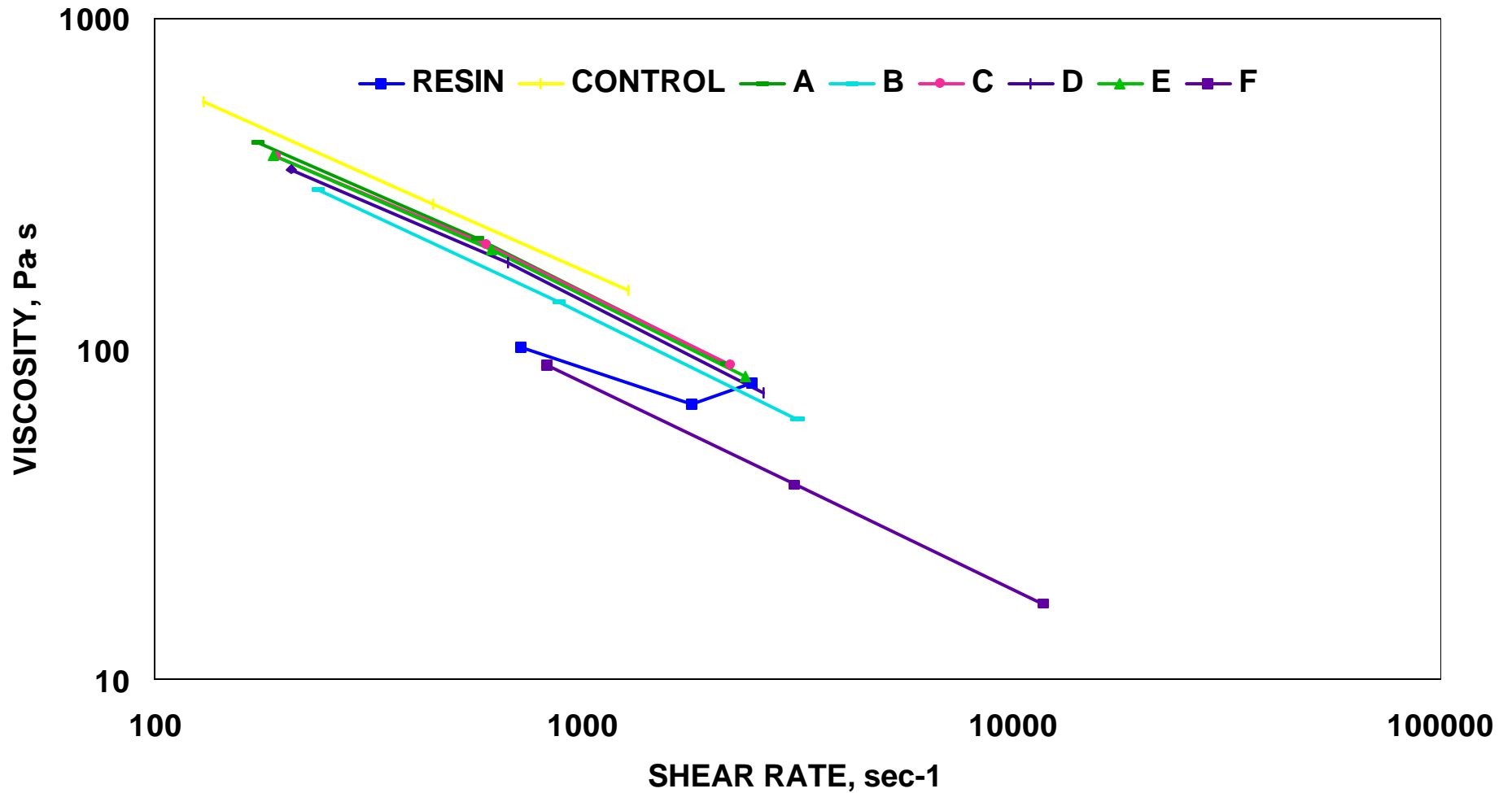
40% WOOD FLOUR COMPOSITE RHEOLOGY LOG FLOW VS. PRESSURE



Quality Additives for Performance



40% WOOD FLOUR COMPOSITE RHEOLOGY LOG VISCOSITY VS. SHEAR RATE





CAPILLARY RHEOMETER DATA:

60% Wood Flour Composites

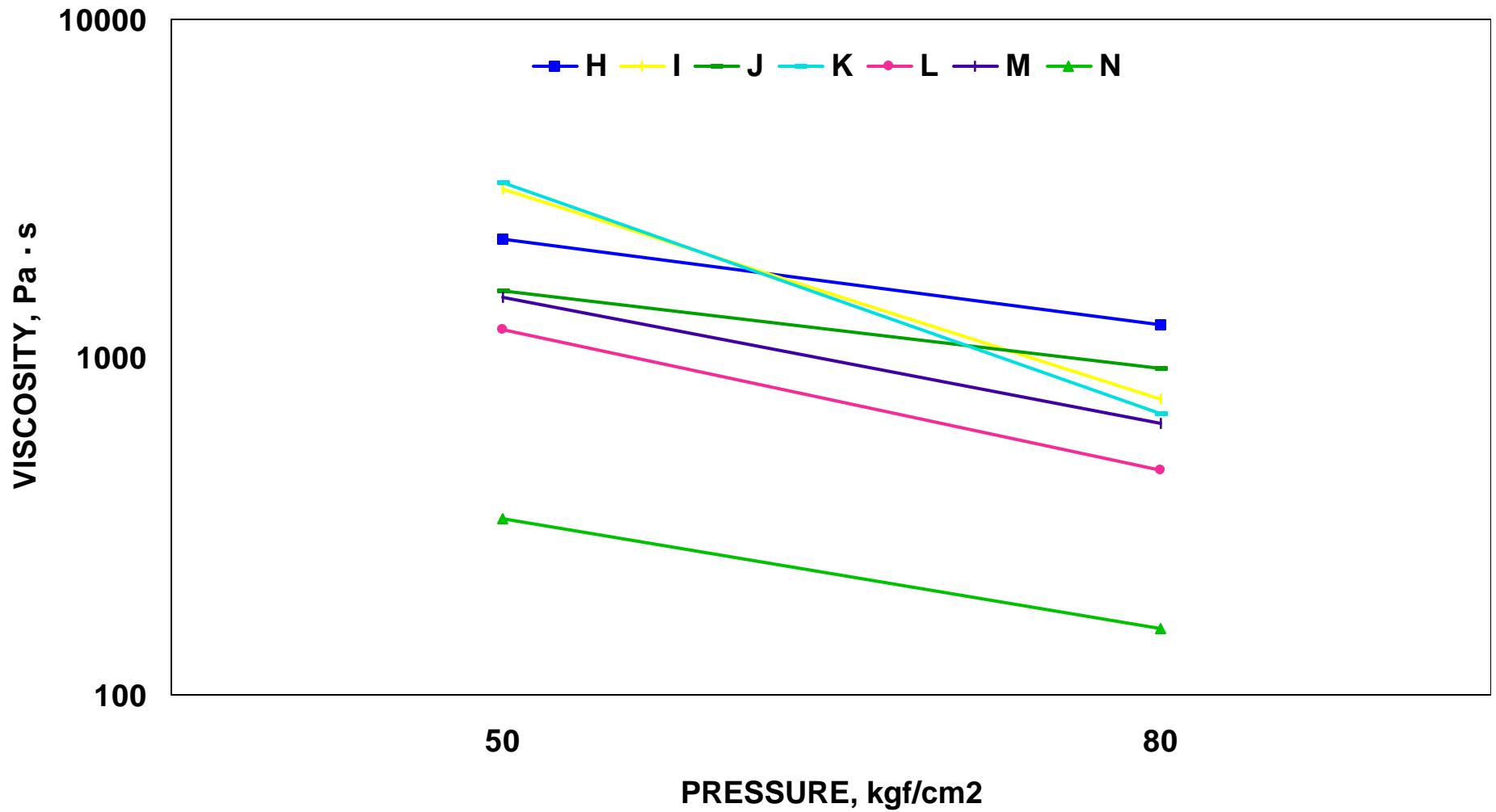
	<u>H</u>	<u>I</u>	<u>J</u>	<u>K</u>	<u>L</u>	<u>M</u>	<u>N</u>
30 kgf/cm ² @ 190°C							
Flow, g/sec	*	*	*	*	*	*	*
Viscosity, Pa·s	*	*	*	*	*	*	*
Shear, sec ⁻¹	*	*	*	*	*	*	*
50 kgf/cm ² @ 190°C							
Flow, g/sec	0.01	0.01	0.01	0.01	0.01	0.01	0.04
Viscosity, Pa·s	2226	3142	1561	3280	1200	1497	332
Shear, sec ⁻¹	55	39	78	37	102	81	368
80 kgf/cm ² @ 190°C							
Flow, g/sec	0.02	0.03	0.03	0.03	0.04	0.03	0.12
Viscosity, Pa·s	1247	758	925	682	464	636	157
Shear, sec ⁻¹	157	258	211	287	422	308	1248

* Data too low to record





60% WOOD FLOUR COMPOSITE RHEOLOGY LOG VISCOSITY VS. PRESSURE



NEW CONCLUSIONS:

The capillary rheometer data tells us that the torque data generated during the Brabender mixing phase is not directly correlative with the viscosity of the composites over a range of pressures. As we expected, the presence of the additives caused significant improvements in flow (reductions in viscosity), with the greatest difference shown in the higher pressure profiles.

The most dramatic viscosity change occurred in compounds "F" and "N". These formulations both contained 0.1 phr of a 60% peroxide on wax concentrate. As shown in the data, this very small amount of peroxide decreased the compounds' viscosity to levels near or lower than the neat polypropylene resin.

At this point we have shown we can formulate a compound with high filler loading that processes with the same viscosity as the neat resin.

But do we get improved physical properties??



PHYSICAL PROPERTY DATA (BEST FLOWING COMPOUNDS):

40% Wood Flour Composites

	<u>Cont.</u>	<u>A</u>	<u>D</u>	<u>E</u>	<u>F</u>
Izod Impact, ft · lbs./in.					
Notched	1.3	1.3	1.3	1.4	1.4
Unnotched	2.3	3.0	2.7	2.7	2.5
Flexural Properties, MPa					
Strength	49.3		46.1		39.6
Modulus	3390		3300		2810

60% Wood Flour Composites

	<u>H</u>	<u>L</u>	<u>N</u>		
Izod Impact, ft · lbs./in.					
Notched	1.3	1.3	1.4		
Unnotched	2.3	2.3	2.3		
Flexural Properties, MPa					
Strength	42.1	37.8	38.6		
Modulus	5050	4790	4550		

FINAL CONCLUSIONS:

- ▶ The use of process adhesives, lubricants and surfactants can significantly improve the processability of woodfiber composites. In addition to improved processability we see:
 - Significant improvements in mold release
 - Improved surface appearance
 - Maintained or improved physical properties

- ▶ The addition of a very small amount of peroxide into the compound allows for an extremely high flow material (higher than the base resin) combined with the physical properties of a highly filled compound. This is important for thin part or high surface area injection molding.

- ▶ Compounds with high levels of filler can be processed the same as compounds with lower level of fillers using the right additive package.

- ▶ The slightly lower flexural modulus values indicate more continuity of the polypropylene phase resulting from better dispersion of the wood flour.



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