High Performance Mineral Reinforcement Concentrate for LLDPE & HMW-HDPE Blown Film Extrusion

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Abstract

LLDPE-based pelleted calcium carbonate ($CaCO_3$) concentrates utilizing two different viscosity base resins were used to add up to 20wt.% fine-ground, surface-treated mineral to LLDPE and HMW-HDPE Films. Film was extruded and converted into institutional can liners on production equipment.

Concentrates based on higher viscosity resins yielded superior impact and tensile performance compared to those based on lower viscosity resins. No problems with dispersion were noted. Slightly higher melt pressure and motor current were observed but were still well within typical extrusion process conditions for the type of base resins used.

Introduction

In a previous paper[1] the author described the different modifications of film properties observed utilizing mineral reinforcement concentrates based on three different polyolefins. The concentrate base resin had a major effect on the film properties obtained with each of four film resin types. Although certain combinations of film resin and concentrate produced outstanding film characteristics, no one concentrate uniformly delivered a substantial increase in performance.

This paper details the results obtained by increasing the viscosity and molecular weight of the carrier resin, yielding a concentrate of higher viscosity and lower melt index. Improvements in compounding technology have allowed the commercial production of highly loaded mineral concentrates with moderately viscous carrier resins. In addition, a calcium carbonate concentrates of 0.5 MI has an I_{21} of 20. This is close to the typical 1.0 MI film grade LLDPE resin I_{21} of 20 – 25, and above the 8 – 12 typical I_{21} values of film grade HMW-HDPE resins. The high-load melt index, I_{21}, is more representative of the shear rate experienced by the extrudate during film processing.

Other papers[2,3,4,5] have discussed the mineral factors (particle morphology, particle size distribution, particle surface chemistry, and chemical purity) and polymer factors (molecular weight, molecular weight distribution, branching type and distribution, density/crystallinity, and polymer chemistry, e.g. polar/non-polar) which affect the processing and product properties with mineral addition. Proper mixing and dispersion of the mineral into the polymer matrix is a critical processing factor in the complete realization of the benefits of this technology. Commercial film extrusion equipment in good condition with modern screw designs has proven satisfactory in achieving the necessary level of homogenization, even with concentrates as low as 0.3 MI.

Discussion

Mineral and Polymer Selection

Two LLDPE resins of 0.920ρ were chosen as carriers for the preparation of 75% calcium carbonate concentrates. The first was an LLDPE chosen to yield a concentrate MI of 3.0. The second was chosen to yield a concentrate MI of 0.5. These two concentrates are manufactured by Heritage Plastics and HM10® and HM10HP, respectively

A wet-ground calcium carbonate with a 1.0μ mean particle size (MPS) and 8μ top-cut (maximum particle size) was selected as the reinforcing mineral. The calcium carbonate was treated with a fatty acid by the mineral supplier to form a hydrophobic coating on the surface of the mineral. This allows the polyethylene to "wet" the mineral surface, greatly improving the dispersion of the mineral into the polymer matrix and processability of the mineral/PE composite.

A total of four film trials were conducted, two each with LLDPE and HMW-HDPE. The details of the processing conditions employed and results obtained are detailed below.

LLDPE Film Extrusion and Conversion

The first LLDPE extrusion run was conducted using these two concentrates, utilizing a 1.0MI/0.920ρ ethylene/hexene copolymer as the film resin at 1.0 mil thickness and 1.5:1 BUR. A 114mm (4.5") extruder, 406mm (16") die and in-line bag machine were used to produce films under the conditions listed in Table 3. Films were produced at loadings of both 12% calcium carbonate ($CaCO_3$)(16% concentrate) and 20% $CaCO_3$ (27% concentrate).

Table 1 Processing Conditions HBC LD#5, 114mm (4.5") 24/1 L/D Extruder, 406mm (16") die

Concentrate Type/MI	3.0	0.5	3.0	0.5
% $CaCO_3$	12	12	20	20
Melt Temperature, °C	216	216	216	216
Screw RPM	32	32	32	32
Head Pressure, MPa	33.4	36.4	33.0	36.1
Motor Current	192	216	185	212
Output, kg/hr	144	144	144	144

Both concentrates ran within the normal process operating parameters of this extrusion line. As expected, the higher viscosity concentrate required slightly higher motor current and head pressure at the same rates.

Table 2. Film Properties at 1.2 mil, 1.5:1 BUR

Concentrate Type/MI	3.0	0.5	3.0	0.5
% $CaCO_3$	12	12	20	20
Dart, g	435	615	446	>635
Elmendorf Tear MD	285	335	335	335
g TD	600	630	650	680
Tensile @ Yield, MD	8.8	10.4	8.5	9.2
MPa, TD	9.5	10.2	9.3	10.1
Tensile @ Break, MD	48.6	57.4	42.7	42.7
MPa, TD	31.4	36.5	26.8	29.6

The films made with the higher viscosity concentrate yield stronger films at both mineral loadings, especially in impact performance.

A second LLDPE extrusion run was conducted using these two concentrates, this time utilizing a 1.0MI/0.920ρ ethylene/octene copolymer as the film resin at 1.2 mil thickness and 2.04:1 BUR. This time an 89mm extruder and in-line bag machine were used to produce films under the conditions listed in Table 3. Films were loaded with 9% $CaCO_3$ (12% concentrate) and 15% $CaCO_3$ (20% concentrate).

Table 3 Processing Conditions HBC LD#7, 89mm (3.5”) 24/1 L/D Extruder, 380mm (15”) die

Concentrate Type/MI	3.0	0.5	3.0	0.5
% $CaCO_3$	9	9	15	15
Screw RPM	64	64	64	64
Melt Temperature, °C	205	206	205	205
Head Pressure, MPa	39.3	41.9	41.3	42.4
Motor Current	116	126	122	125
Output, kg/hr	144	144	144	144

As in the first trial, more torque and pressure were required to process the higher viscosity concentrates, but all parameters were well within the normal operating limits of the equipment.

Table 4. Film Properties at 1.2 mil, 1.5:1 BUR

Concentrate Type/MI	3.0	0.5	3.0	0.5
$CaCO_3$	9	9	15	15
Dart, g	320	350	465	630
Elmendorf Tear MD	435	475	475	470
g TD	960	1050	980	103 0
Tensile @ Yield, MD	7.2	7.7	7.8	8.3
MPa, TD	7.4	8.4	8.7	8.5
Tensile @ Break, MD	41.6	45.2	40.8	42.7
MPa, TD	32.1	34.7	28.3	31.3

Film properties with the lower MI concentrate were again much improved over the values obtained for the higher MI version.

HMW-HDPE Film Extrusion and Conversion

In the first HMW-HDPE trial, 13μ film was produced at a BUR of 3.27:1 and mineral loadings of 15% $CaCO_3$ (20% concentrate) and 22% $CaCO_3$ (30% concentrate) on a 70mm grooved-feed extruder fitted with twin 175mm dies. Processing conditions are summarized in Table 5.

Table 5. HMW-HDPE Film Extrusion, 70mm

Concentrate Type/MI	3.0	0.5	3.0	0.5
% $CaCO_3$	15	15	22	22
Screw RPM	61	58	61	58
Melt Temperature, °C	224	224	224	224
Head Pressure. MPa	42.5	44.4	37.5	40.7
Motor Current	197	199	188	197
Output, kg/hr	245	234	252	242

The higher viscosity concentrate yielded higher head pressures and motor load requirements, but all within typical operating parameters.

Table 6. Film Properties at 13µ (0.5 mil) & 3.27:1 BUR

Concentrate Type/MI	3.0	0.5	3.0	0.5
% $CaCO_3$	15	15	22	22
Dart, g	310	440	280	340
Tensile @ Yield, MD	24.6	28.4	24.2	27.0
MPa, TD	24.9	25.2	23.0	24.0
Tensile @ Break, MD	61.7	70.4	55.7	68.0
MPa, TD	55.2	65.6	44.9	62.7

The higher viscosity concentrate yielded higher impact and tensile strength at both mineral loadings.

A second HMW-HDPE trial was conducted on an identical line (70mm grooved-feed extruder fitted with twin 175mm dies) at a target gauge of 14µ and 3.6:1 BUR. This time a no-mineral control was run for comparison, and all concentrates were run at 20% addition (15% $CaCO_3$).

Table 7. HMW-HDPE Film Extrusion, 70mm

Concentrate Type/MI	None	3.0	0.5
% $CaCO_3$	0	15	15
Screw RPM	54	54	54
Melt Temperature, °C	223	223	223
Head Pressure. MPa	52.0	45.6	45.4
Motor Current	203	191	196
Output, kg/hr	201	224	214

Both concentrates reduced extruder head pressures and motor load requirements, while increasing process output. As expected, the improvements were not as marked with the higher viscosity concentrate.

Table 8. Film Properties at 13µ (0.5 mil) & 3.27:1 BUR

Concentrate Type/MI	None	3.0	0.5
% $CaCO_3$	0	15	15
Dart, g	280	320	380
Tensile @ Yield, MD	30.0	19.3	30.0
MPa, TD	28.5	20.2	26.1
Tensile @ Break, MD	76.6	52.0	72.3
MPa, TD	74.4	47.8	61.7

While addition of either concentrate improves the dart impact of the film, the higher viscosity concentrate yielded greater impact strength gain. Of great importance is that tensile properties remained much higher with the higher viscosity concentrate. These properties are critical in typical thin-gauge HDPE film applications such as T-shirt bags and institutional can liners.

Conclusions

Calcium carbonate mineral reinforcement improves the ductile performance of LLDPE and HMW-HDPE films. Increasing the viscosity/molecular weight of the concentrate carrier resin can improve dramatically the response of film impact and tensile properties to mineral reinforcement. Modern film extrusion equipment in proper operating condition has been shown to be able to mix and disperse concentrates of the rheological properties studied without difficulty.

References

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