

The Effects of Antiblocking Agents on the Performance of Polymer Process Aids

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ABSTRACT

Fluoroelastomer processing aids can improve the melt processibility of polyolefins only by depositing on the internal dies surfaces. It has been shown previously that other additives in the compound can interfere with deposition on the die surface. This present work expands on this knowledge by examining the effectiveness of several commercial processing aids when paired with various antiblocking agents in a LLDPE resin run on a blown film line.

The results show that fluoroelastomer processing aids can perform efficiently in the presence of antiblocking agents if they contain an interfacial agent. The interactions from most antiblocks can be effectively overcome by the use of such technology, however antiblocks that incorporate synthetic silica can be very interactive and reduce the overall efficiency of the polymer process aids.

INTRODUCTION

Fluoroelastomer processing aids are widely used in polyolefin applications to eliminate melt fracture, reduce die deposits, improve throughput, and reduce extrudate surface defects. These processing aids function by depositing a thin coating of the fluoroelastomer on the metal surfaces of the die, thereby promoting slip at the interface of this coating and the polyolefin. To effectively eliminate melt fracture in a blown film application, the polymer process aid (PPA) must uniformly coat the exit region of the die [1]. It has been shown that antiblock additives in blown film applications can adversely affect PPA performance by interacting with the PPA [2–5]. There are two main categories of interaction between PPA and antiblocking agents: by surface area interactions (adsorption), and by abrading the PPA coating from the die surfaces [6]. The type of interaction is dependent upon the type of antiblock being used.

The goal of this present work is to expand on current knowledge by determining the effectiveness of several commercial processing aids when paired with various antiblocking agents in a LLDPE resin when run on a blown film line. Five different antiblocking agents are studied in combination with three different commercial PPAs. The effectiveness of the PPA was determined by the length of time it took to eliminate melt fracture.

EXPERIMENTAL

Materials

A complete listing of the materials used in this study is shown in Table 1.

The three polymer process aids (PPA) used for this study are manufactured by DuPont Performance Elastomers and sold commercially. Each contains a fluoroelastomer (FKM) that is a co-polymer of vinylidene fluoride and hexafluoropropylene in a 60/40 weight ratio. The commercial types are Viton® FreeFlow™ 40, Viton® FreeFlow™ Z 100, and Viton® FreeFlow™ Z 200. Viton® FreeFlow™ 40 is a pure fluoroelastomer in pellet form. Viton® FreeFlow™ Z 100 is a blend of fluoroelastomer (slightly more than 50 percent by weight), dusting agent, and polyethylene glycol (PEG), slightly more than 40 percent by weight, as an interfacial agent. Viton® FreeFlow™ Z 200 is a blend of fluoroelastomer (slightly more than 30 percent by weight), dusting agent, and a polyester polyol (polycaprolactone, or PCL), slightly more than 60 percent by weight, as an interfacial agent. In the present work, these PPAs are referred to as PPA-1, PPA-2, and PPA-3, respectively. The PPA masterbatches were produced with a 1% concentration.

The resin used for this study was an ethylene-butene LLDPE, produced by ExxonMobil Chemical Co. (LL 1001.59), having a melt index (MI) of 1.0 and a density of 0.918. The product is produced in a gas phase reactor with no additives other than a minimal antioxidant level. The resin used for PPA masterbatch production was a similar resin, melt index 2.0, in powder form (LL 1002.09).

Ingenia Polymers, Inc kindly supplied the antiblocking agents in masterbatch form used for this study. AB-1 is diatomaceous earth (silica or DE) with a median particle size of 9 microns. AB-2 is a surface treated talc (magnesium silicate) with an average particle size of 2.3 microns. AB-3 is an untreated talc (magnesium silicate) with an average particle size of 2.8 microns. AB-4 is a mineral blend (sodium-potassium aluminum silicate) with an average particle size of 6.7 microns. AB-5 is a synthetic amorphous silica with an average particle size of 5 microns. All the antiblock masterbatches were produced with a 2.0 MI ethylene-butene LLDPE, at a 25% concentration, except for AB-5, which has a 10% concentration. These masterbatches are summarized in Table 2.

Table 1 Materials Listing

PPA-1	Pure fluoroelastomer polymer process aid
PPA-2	Blended PPA of fluoroelastomer, dusting agent, PEG
PPA-3	Blended PPA of fluoroelastomer, dusting agent, PCL
AB-1	Antiblock, diatomaceous earth (DE), natural silica
AB-2	Antiblock, surface treated talc
AB-3	Antiblock, untreated talc
AB-4	Antiblock, mineral blend
AB-5	Antiblock, synthetic amorphous silica

Table 2 Description of Antiblock Masterbatches

Label	Content	Particle Size	Concentration
AB-1	Diatomaceous Earth (DE)	9 microns	25%
AB-2	Treated Talc	2.3 microns	25%
AB-3	Untreated Talc	2.8 microns	25%
AB-4	Mineral Blend	6.7 microns	25%
AB-5	Synthetic Silica	5 microns	10%

Extrusion Equipment

Masterbatches were produced on a 28 mm co-rotating, fully intermeshing, 3-lobe twin screw extruder. The speed was held at 150 rpm, and the barrel temperature setpoints, from the feed zone forward, were 140/160/180/200°C. With these operating conditions, the output was approximately 9 kg/hr.

Blown film melt fracture testing was carried out on a 63.5 mm, 24:1 L/D extruder with a 101.6 mm die and a set gap of 0.76 mm. The die has a spiral mandrel design with four inlet ports. The screw was a typical barrier design with a Maddock mixing section, and the speed was held at 45 rpm, which resulted in an extruder output of approximately 45 kg/hr. The nominal shear rate was 540 1/s in the die gap. Temperature setpoints, from the feed zone forward, were 160/180/194/190 °C, and the adapter and die were set at 190°C. Melt temperature at the extruder exit was typically about 225°C. Additives were fed via separate hoppers in a weight-loss feed system to the extruder inlet hopper.

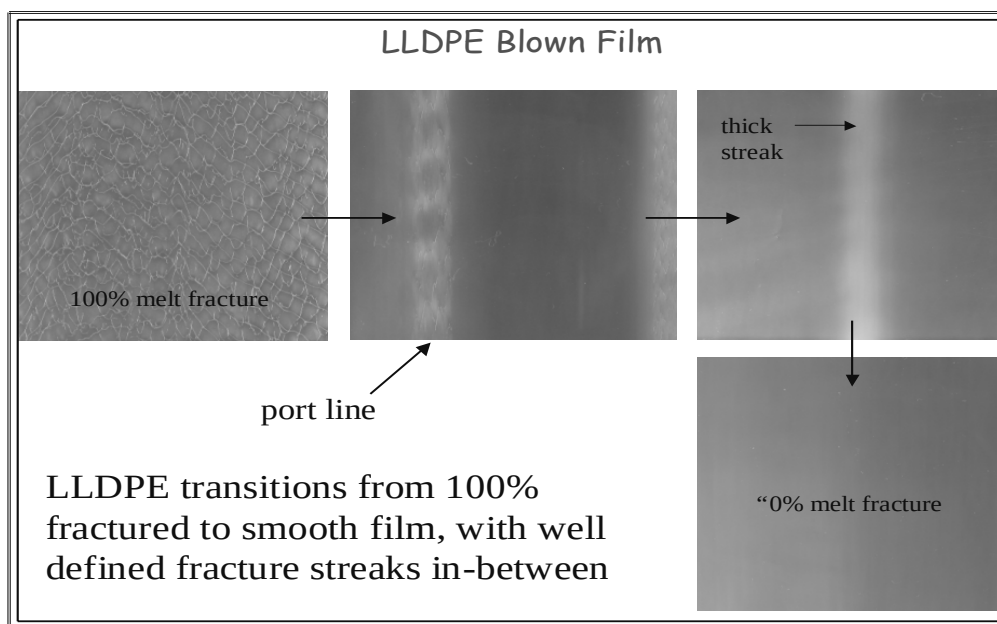
Melt Fracture Test Procedure

Before initiation of a melt fracture run on the film line, the line was purged with a commercial purge compound (Ampacet 807193) containing synthetic silica in polyethylene for 30 - 45 minutes to remove the

PPA from the extruder and die. The line was then started up with the pure LLDPE base resin (no additives) mentioned previously.

To determine the response curves of the PPAs in the absence of antiblock, control runs were made on the blown film line by stabilizing the bubble using the base resin only, without additives. Runs with the antiblocking agents began by stabilizing the film bubble with the addition rate of the antiblock masterbatch. An initial five minute running period ensured the film exhibited 100 percent melt fracture at the beginning of each run, and that no residual PPA remained from the previous run. A rate check was also performed during this period to record the extruder output with no PPA being added. The appropriate PPA masterbatch was then introduced to the feed throat of the extruder and a timer was started. At ten-minute intervals film samples were taken, and the operating parameters of the equipment were recorded. The percent of melt fracture on the film was determined by summing the width of the remaining melt fracture streaks on the film layflat, and dividing by the total film layflat width. This method does not distinguish between melt fracture on the inner and outer surfaces on the film. From an overall standpoint, any fracture remaining is considered to be present on both sides of the film. The test run was stopped when the melt fracture is completely cleared (0 percent), or after 80 minutes of running time, whichever occurred first. A final rate check was made before terminating the test run. Some pictures of melt fracture in the film layflat at various stages throughout a melt fracture film test run are shown in Figure 1.

Figure 1 Melt fracture test run on a blown film line



RESULTS AND DISCUSSION

Control Runs – PPA Only

Control runs were conducted on the blown film line using the base resin with no additives, and feeding PPA masterbatch only. Each of the PPAs were fed at various dosage levels, ranging from 200 ppm to 1200 ppm, to determine the PPA effectiveness (Figure 2). PPA-1 exhibited residual melt fracture at the two lower levels (200 ppm and 400 ppm). A dosage level of 1200 ppm was required to approximate the performance of the other two PPAs, eliminating the melt fracture in 40 minutes. For this reason, a dosing level of 1200 ppm was selected for PPA-1 in the film runs containing antiblock.

The performance of PPA-2 was better than that of PPA-1, as expected since it uses a fluoroelastomer with different rheology and polyethylene glycol (PEG) as an interfacial agent. At 400 ppm dosage level, it eliminated the melt fracture in the control run at 31 minutes. The use of PEG as an interfacial agent in process aids has been shown to improve the processability of LLDPE resins [7]. Based on the results of the control runs, a dosage level of 400 ppm was selected for most runs that included antiblock formulations.

PPA-3 also exhibited improved performance compared to PPA-1, and it showed slightly better performance compared to PPA-2. In the control run, it eliminated the melt fracture in 28 minutes at 400 ppm dosage level. This PPA uses polycaprolactone (PCL) as an interfacial agent. This technology has been well documented as a high performance interfacial agent [3]. Based on the results of the control runs, a dosage level of 400 ppm was selected for the film runs containing antiblock to provide a direct comparison to PPA-2.

A comparison of the control runs are shown in Figure 3 at the dosage levels discussed above. PPA-2 and PPA-3 began to clear the melt fracture more quickly, and are significantly more efficient than PPA-1.

Figure 2 PPA effectiveness at various dosage levels

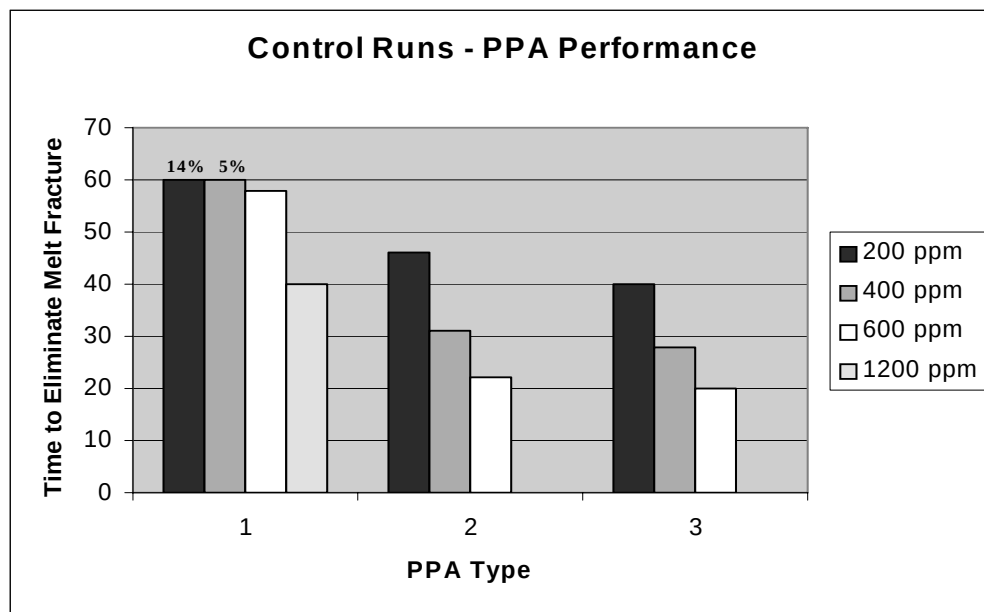
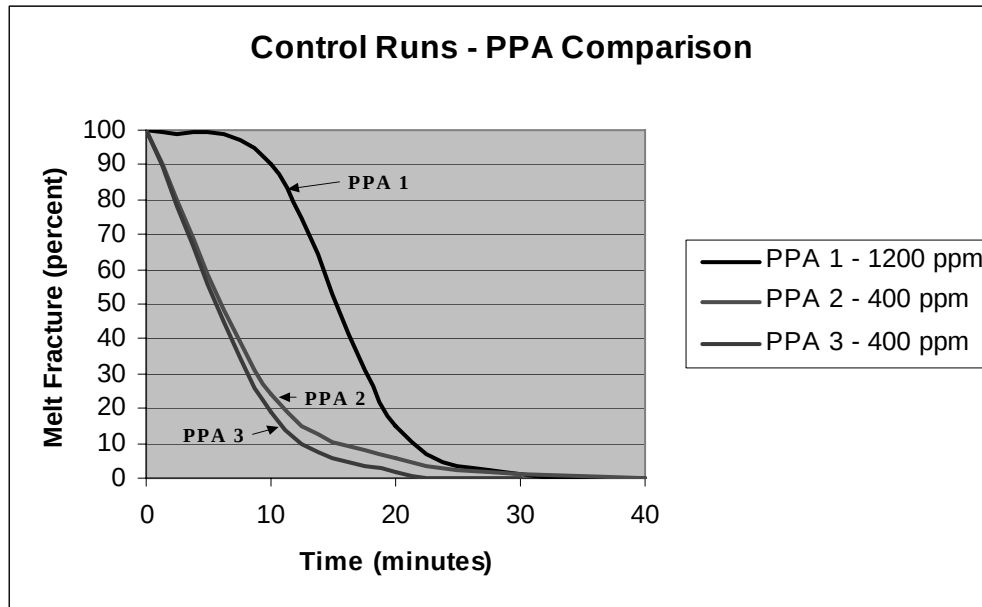


Figure 3 Control Runs – response curves for PPA –1 –2 –3



PPA-1 Performance with Antiblocking Agents

The results from the blown film runs using PPA-1 in combination with the various antiblocks are summarized in Table 3, and shown in chart form in Figure 4. For the film runs, PPA-1 was dosed at 1200 ppm, and the antiblock was dosed at 4000 ppm.

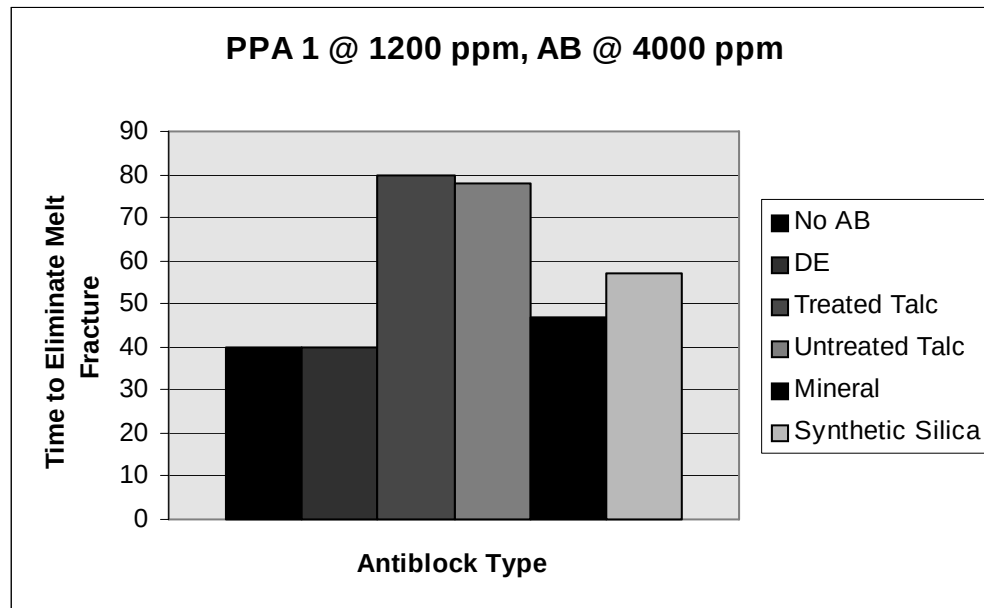
In terms of a relative rating of the effects of the various antiblocks, diatomaceous earth (DE) is the least interactive, followed by the mineral blend, synthetic silica, and the two talcs (untreated and treated). The talc runs gave essentially the same results, with the treated talc clearing melt fracture in 80 minutes, and the untreated talc clearing in 78 minutes. The two minute time difference is within the normal variation of a melt fracture film run.

It has been shown that DE has little effect on the performance of a fluoroelastomer process aid [8], and that talcs interact very strongly due to a chemical affinity for the fluoroelastomer [9]. It was interesting that the results were essentially no different for a treated talc versus an untreated talc, implying that there is no need for a treated talc to reduce interaction with the process aid. The mineral blend antiblock had some slight interactions with the process aid and a higher dosage level of the PPA would be required to maintain melt fracture performance. The same is true for the synthetic silica, although to a greater extent.

Table 3 Time to eliminate melt fracture on a blown film line using PPA-1 in combination with various antiblocks

Antiblock	Time to eliminate melt fracture (minutes)
none	40
AB-1	40
AB-2	80
AB-3	78
AB-4	47
AB-5	57

Figure 4 PPA-1 Performance with various Antiblocks in LLDPE



PPA-2 Performance with Antiblocking Agents

The results from the blown film runs using PPA-2 in combination with the various antiblocks are summarized in Table 4, and shown in chart form in Figure 5. For the film runs, PPA-2 was dosed at 400 ppm, and the antiblock was dosed at 4000 ppm.

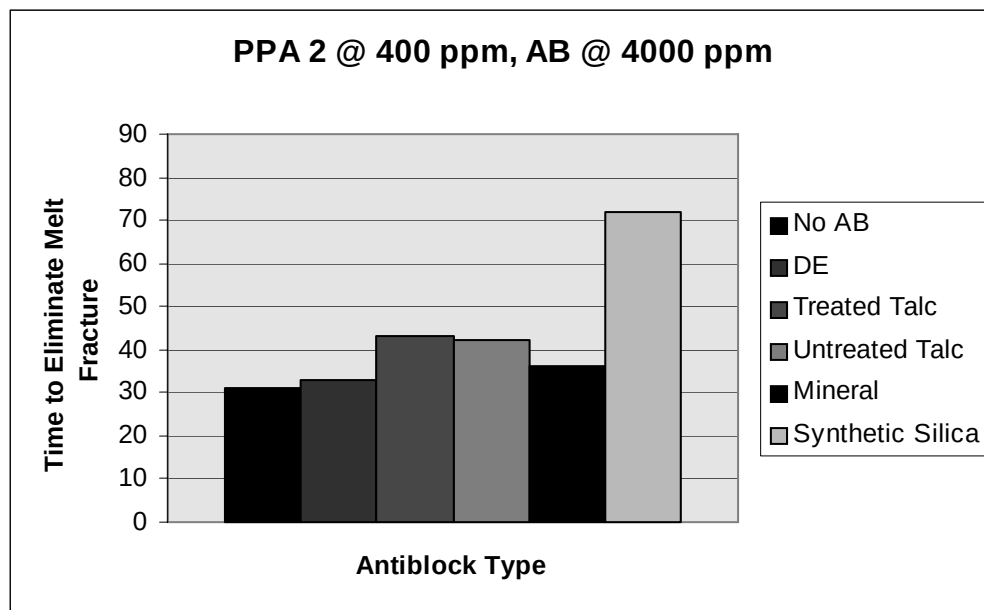
In terms of a relative rating of the effects of the various antiblocks, the diatomaceous earth (DE) is the least interactive, followed by the mineral blend, the two talcs (untreated and treated), and the synthetic silica. The talc runs gave essentially the same results, with the treated talc clearing melt fracture in 43 minutes, and the untreated talc clearing in 42 minutes. The one-minute time difference is well within the normal variation of a melt fracture film run.

It has been shown that talc interact very strongly due to a chemical affinity for the fluoroelastomer [9], however, in the case of PPA-2, the performance did not degrade as much as was seen with PPA-1. Using PEG as an interfacial agent helps to protect the fluoroelastomer from dispersion [3], and although the performance drops, it is still economical to use this technology in the presence of talc antiblock. It was interesting that the results were essentially no different for the treated talc versus the untreated talc, implying that there is no need for a treated talc to reduce interaction with the process aid. The synthetic silica film run showed a significant interaction when used in combination with PPA-2. Since the synthetic silica is amorphous, the surface interferes with the process aid by adsorption (including adsorption of the interfacial agent) allowing the fluoroelastomer to be more finely dispersed, which reduces the effectiveness due to a smaller particle size [3]. The only way to overcome this interaction is to use a higher dosage of the process aid.

Table 4 Time to eliminate melt fracture on a blown film line using PPA-2 in combination with various antiblocks

Antiblock	Time to eliminate melt fracture (minutes)
none	31
AB-1	33
AB-2	43
AB-3	42
AB-4	36
AB-5	72

Figure 5 PPA-2 Performance with various Antiblocks in LLDPE



PPA-3 Performance with Anti-blocking Agents

The results from the blown film runs using PPA-3 in combination with the various antiblocks are summarized in Table 5, and shown charted in Figure 6. For the film runs, PPA-3 was dosed at 400 ppm, and the antiblock was dosed at 4000 ppm.

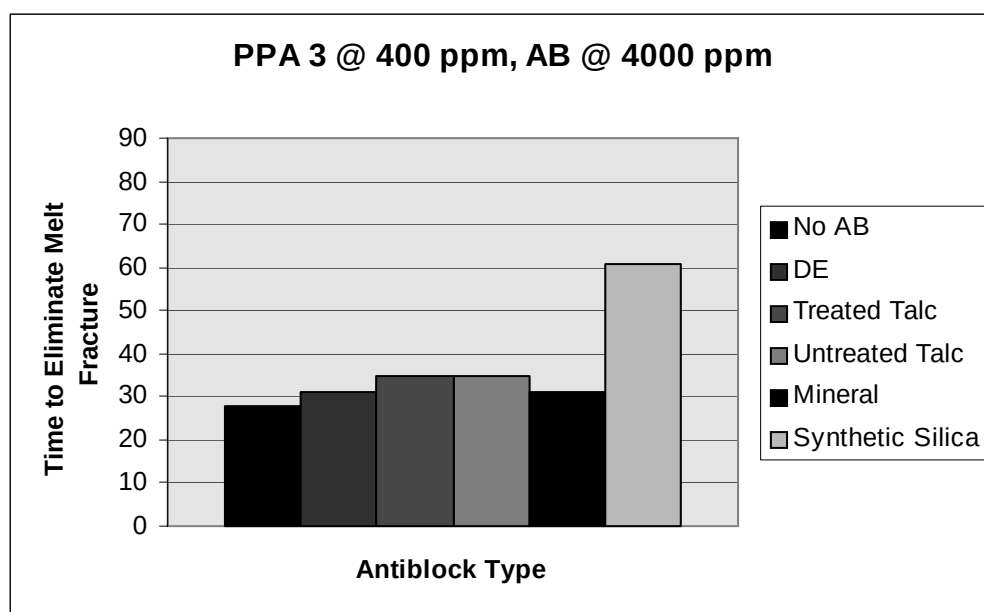
In term of a relative rating of the interaction of the various antiblocks, the diatomaceous earth (DE) and the mineral blend both cleared in 31 minutes, the two talcs (untreated and treated) both cleared in 35 minutes, and the synthetic silica cleared in 61 minutes. In all cases, PPA-3 exhibited the best performance of the PPAs tested, regardless of the antiblock.

Once again, the interaction with talc was significantly reduced by the use of a high performance interfacial agent (PCL), and the type of talc (treated or untreated) had no effect on the PPA performance. The effects of the mineral blend antiblock were also minimized with this PPA. And once again, the synthetic silica film run showed a significant interaction when used in combination with PPA-3. Since the synthetic silica is amorphous, the surface interferes with the process aid by adsorption (including adsorption of the interfacial agent) allowing the fluoroelastomer to be more finely dispersed, which reduces the effectiveness due to a smaller particle size [3]. The only way to overcome this interaction is to use a higher dosage of the process aid.

Table 5 Time to eliminate melt fracture on a blown film line using PPA-3 in combination with various antiblocks

Antiblock	Time to eliminate melt fracture (minutes)
none	28
AB-1	31
AB-2	35
AB-3	35
AB-4	31
AB-5	61

Figure 6 PPA-3 Performance with various Antiblocks in LLDPE



PPA Comparison by Ant-block Type

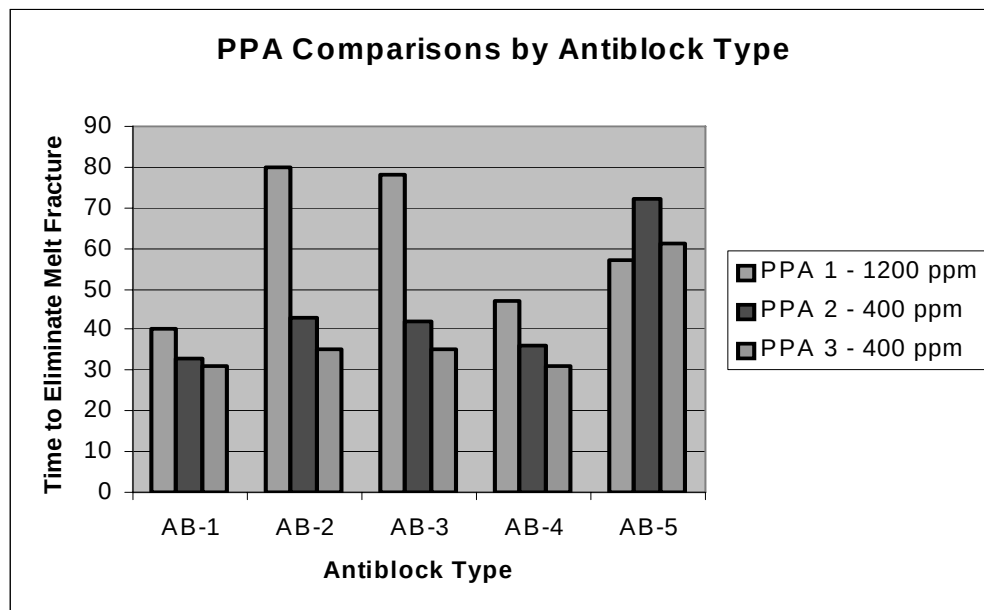
It is useful to look at the results from the viewpoint of a PPA comparison. The results from the blown film runs are summarized in Table 6, and shown in chart form in Figure 7. For the film runs, PPA-1 was dosed at 1200 ppm, PPA-2 and PPA-3 were dosed at 400 ppm, and the antiblock was dosed at 4000 ppm.

In term of a relative performance ranking of the process aids, PPA-3 gave the best performance of the process aids tested in all cases. Even when used in combination with the synthetic silica, its performance was better than PPA-1 due to a much lower dosage level (400 ppm versus 1200 ppm). PPA-3 also outperformed PPA-2 when used in combination with AB-2, AB-3, and AB-5, producing much better results.

Table 6 Comparison of PPA performance in combination with the various Antiblocks

Antiblock	Time to Eliminate Melt Fracture (minutes)		
	PPA-1 (1200 ppm)	PPA-2 (400 ppm)	PPA-3 (400 ppm)
none	40	31	28
AB-1	40	33	31
AB-2	80	43	35
AB-3	78	42	35
AB-4	47	36	31
AB-5	57	72	61

Figure 7 PPA Comparison by Antiblocks in LLDPE

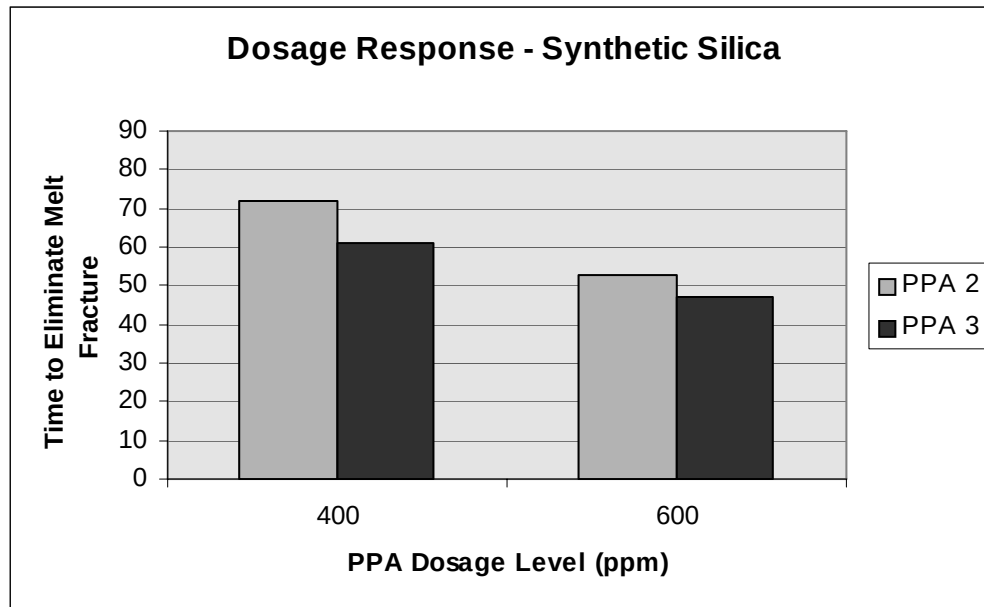


Synthetic Silica Dosage Response

The one antiblock that stands out as being highly interactive with processing aid is synthetic amorphous silica (AB-5). Additional testing with this antiblock was conducted to examine the dosage response of the PPA at higher levels. PPA-2 and PPA-3 were chosen for this work, and two additional film runs were made with the PPA dosed at 600 ppm and the antiblock dosed at 4000 ppm. The results from the higher dosage level are shown in Figure 7, as a comparison to the lower dosage level (400 ppm) for each PPA.

The results show that PPA-2 had the most improved performance at the higher dosage level. As the PPA dosage level increases the performance gap between PPA-2 and PPA-3 narrows. The level of interaction is still high, but the performance improves by adding a higher level of process aid, regardless of the PPA type. PPA-3 still provides the best overall performance with this highly interactive antiblock.

Figure 8 PPA Dosage Response with Synthetic Silica



CONCLUSIONS AND SUMMARY

This present work examines the effects of antiblocking agents on the performance of fluoroelastomer process aids by determining how effectively the PPA eliminated melt fracture in a LLDPE on a blown film line. Five different antiblocking agents were studied in combination with three different commercial PPAs.

The results indicate that fluoroelastomer processing aids can perform efficiently in the presence of antiblocking agents if they contain interfacial agent technology. The most effective interfacial agent for reducing these interactions is a polyester polyol, polycaprolactone (PCL). The interactions from most antiblocks can be effectively overcome by use of such technology. However antiblocks that contain synthetic silica can be very interactive and reduce the overall efficiency of the polymer process aids. The only way to overcome these interactions is to increase the dosage level of process aid.

The results of this study also show that an untreated talc antiblock has no more interaction with fluoroelastomer process aid than does a treated talc. There is no need to use a treated talc to reduce the interactions with a process aid.

ACKNOWLEDGEMENTS

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THE EFFECTS OF ANTIBLOCKING AGENTS ON THE PERFORMANCE OF POLYMER PROCESS AIDS

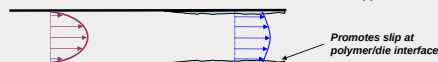
Presented by:
Brian Smith
Viton® FreeFlow™ Technical Services
DuPont Performance Elastomers L.L.C.

Fluoroelastomer Process Aids

Coats the die with a thin slippery film, which allows polymer to pass through the die gap faster and with lower shear stress



- ◆ Typical loading in film resin: 200 - 1000 ppm
- ◆ Continuously replenishes coating on die surface
- ◆ Supplied in resin or as a supplemental masterbatch



- Results in improved processing and final products
 - Expands “processing window” and smoothes operations
 - Lowers die swell and reduces die build-up
 - Eliminates melt fracture

Experimental Design

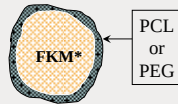
- What is the effect of antiblocks (AB) on the performance of commercial polymer process aids (PPA) ?
- 3 PPAs, 5 AB Masterbatches
- PPA effectiveness was determined by the length of time to eliminate melt fracture in a blown film run
- Antiblock / PPA Interactions
 - Adsorption
 - Abrasion

Material Listing

- Film runs made with 1.0 MI butene-LLDPE
- PPA-1 Pure fluoroelastomer polymer process aid
- PPA-2 Blended PPA of fluoroelastomer, dusting agent, PEG as IA
- PPA-3 Blended PPA of fluoroelastomer, dusting agent, PCL as IA
- AB-1 Diatomaceous Earth (DE), natural silica
- AB-2 Surface Treated Talc, magnesium silicate
- AB-3 Untreated Talc, magnesium silicate
- AB-4 Mineral Blend, sodium-potassium aluminum silicate
- AB-5 Synthetic Amorphous Silica

Interfacial Agent Technologies

- Interfacial agents are used to control size and protect the fluoroelastomer particle as it travels through the melt flow



Polyethylene Glycol (PEG)

- ◆ Traditional interfacial agent
- ◆ Degrades easily at high temperatures
- ◆ Screw slippage at high levels

Polycaprolactone (PCL)

- ◆ Most effective fluoroelastomer complement
- ◆ Thermally stable at higher temperatures
- ◆ No screw slippage

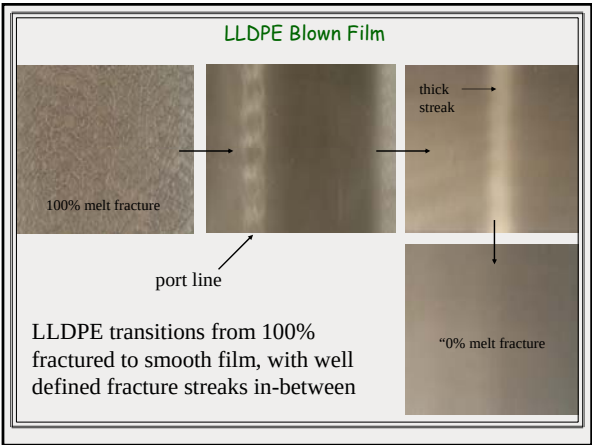
* FKM = Fluoroelastomer (ASTM designation)

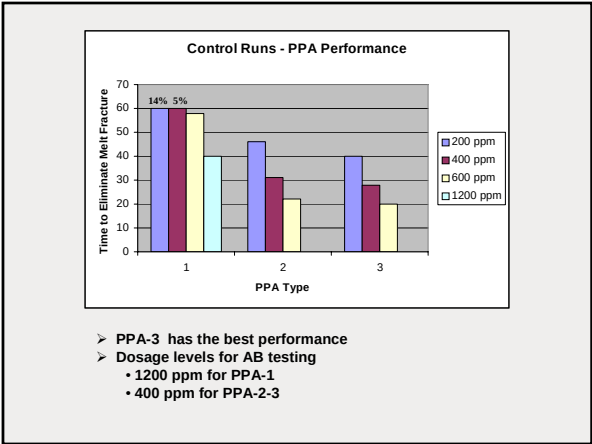
Equipment Description

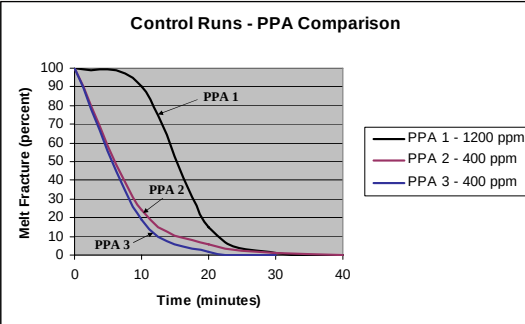
- PPA Masterbatches Produced on 28 mm Twin Screw
- Melt Fracture Testing Done on Blown Film Line
 - 63.5 mm (2-1/2 inch) barrier screw
 - 101.6 mm (4-inch) die
 - Output approximately 45 kg/hr (100 lbs/hr)
 - Nominal shear rate 540 – 1/s

Melt Fracture Test Procedure

- Purge Film Line to Ensure 100% Melt Fracture at Startup
- Initial Run Period to Stabilize Film Bubble
 - Initial rate check
- Begin Test Run
 - 10-minute intervals
 - Determine remaining melt fracture
 - Record process readings
- Run until Fracture is Gone, or 80-minutes
 - Final rate check



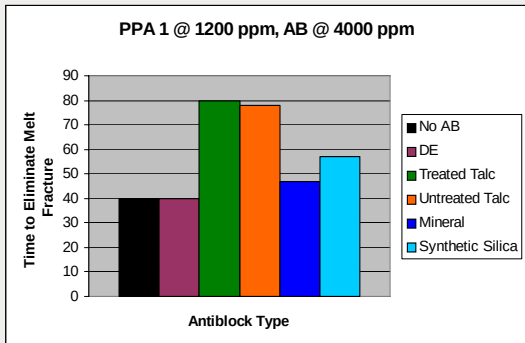




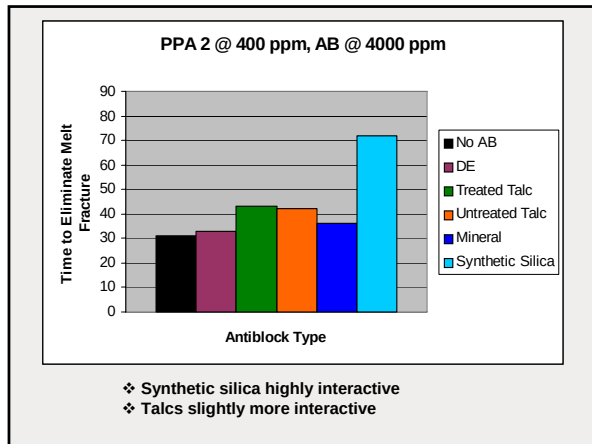
➤ Deposition rates of PPA-2 and PPA-3 are much faster than PPA-1

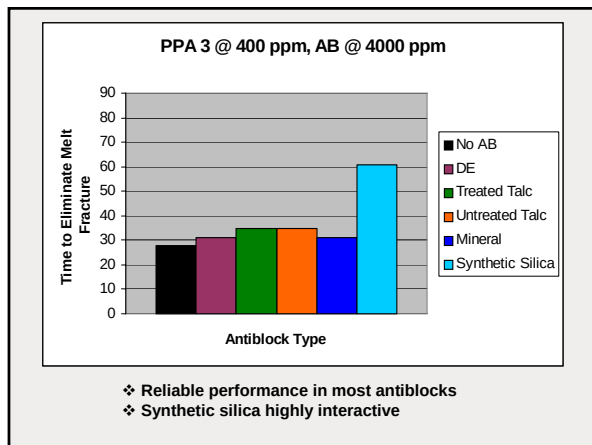
Results – Control Runs

- Relative ranking of PPAs performance
PPA-3 > PPA-2 >> PPA-1
- Dosage level of 1200 ppm for PPA-1
- Dosage level of 400 ppm for PPA-2 and PPA-3



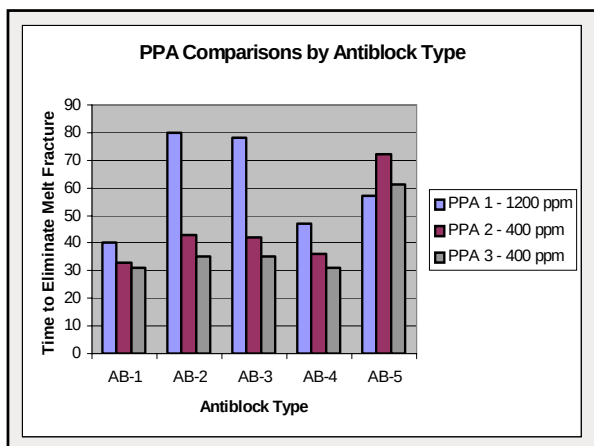
- ❖ Talcs are highly interactive
- ❖ Synthetic silica abrades PPA coating





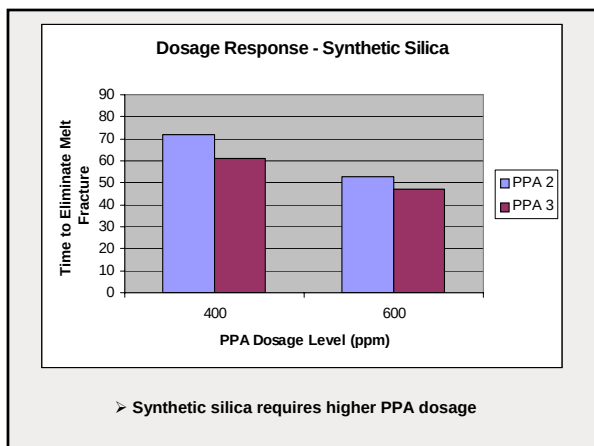
Results by PPA Type

- Relative performance across all runs
PPA-3 > PPA-2 >> PPA-1
- PPA-2 and PPA-3 use different fluoroelastomer
 - Resists abrasiveness of the antiblock
- PPA-3 uses high performance interfacial agent (IA)
 - Reduces adsorption interactions



Results by AB Type

- Diatomaceous earth (DE) has little effect on PPA performance
- There is not a significant difference in the effects of treated talc vs. untreated talc
- Synthetic silica has a highly interactive effect on the PPA, regardless of type



Summary

- By using interfacial agent (IA) technology with fluoroelastomer process aids, the detrimental effects of antiblocks can be minimized in most circumstances
- Synthetic silica antiblock show highly interactive effects, requiring higher PPA dosage levels
- There is not a significant difference in the effects of treated talc vs. untreated talc on the PPA performance

Acknowledgements

- J. Aretz of Veltec Services for compounding and making the film runs for melt fracture testing.
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- My colleague, S. Oriani for his technical assistance and support.

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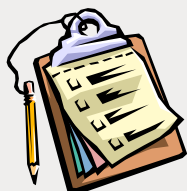
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