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Date Published: January 2000

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Polymer Processing Additives

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Susan S. Woods
Dyneon LLC
Alphonsus V. Pocius
3M Company

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## ABSTRACT

Polyolefin Plastomer films formulated with slip and antiblock were blown on a wide die gap with and without two Dynamar™ polymer processing additives (PPAs). A wide die gap was used so that melt fracture-free film could be obtained with no PPA present for comparison purposes. The films were analyzed for the following properties: surface tension (on treated films), gloss, haze, clarity, transmittance, hot tack, heat seal, COF and block. In addition, the surface of films was examined using ESCA (Electron Spectroscopy for Chemical Analysis) and SSIMS (Static Secondary Ion Mass Spectrometry) to determine the surface chemical composition.


## INTRODUCTION

The influence of PPAs (Polymer Processing Additives) on the surface, mechanical and optical properties (Ref. 1) and also the hot-tack properties (Ref. 2) of LLDPE (linear low density polyethylene) blown film has been previously documented. More recently the questions of the effect of PPAs on polyolefin plastomers ('plastomers') and the effect of PPAs on surface, optical, hot-tack and heat seal properties in the presence of slip and antiblock were raised. This study addresses these concerns.

The use of PPA in blown film applications can result in many enhancements for the film producer. In addition to eliminating surface melt fracture, PPAs enable one to run longer between die cleanings (Ref. 3) and reduce certain types of gels formed in the extruder (Ref. 4, 5). For LLDPE films, it has already been documented (Ref. 1,2) that PPAs have no negative effects on film properties. In fact, in many instances, they have a slight positive effect.

Although it has been previously documented that PPAs do not bloom to the surface of films (Ref. 1), it seems, for whatever reason, the question still lingers in many peoples' minds. Questions about minor issues such as matrix effects and the presence of other additives (such as slip and antiblock) continue to arise. We hope that this study puts these issues to rest. Perhaps the confusion exists because, in the molten state, PPAs are incompatible with polyethylene. As shown schematically in Figure 1, the fluoropolymer exists as insoluble droplets in the polymer melt;.however, this insolubility in the molten state does not result in blooming in the solid state. Rather, in the solid state it results in frozen 'islands' of fluoropolymer existing in a 'sea' of polyethylene. Figure 2 is an optical photomicrograph of a PPA masterbatch which illustrates this point. It is also appropriate to cite previous work where microscopic cross sections of samples containing PPA showed that there is no concentration gradients even at levels as high as 20,000 ppm. (Ref. 7)

## EXPERIMENTAL

### Materials

The resin used in this study was a commercially available polyolefin plastomer (1.0 MI, 0.902 density, well-stabilized, formulated with moderate levels of slip and antiblock). The PPAs used in this study were fluorocarbon-based polymers commercially known as Dynamar FX 9613 (PPA-1) and Dynamar FX 5920A (PPA-2).

Masterbatches of PPA-1 and PPA-2 were prepared in a 6.0 MI, 0.911 density plastomer at a 3% level. The fluoropolymer levels were verified by Parr Bomb analysis (Ref. 8) and dispersion testing (Ref. 9). The fluoropolymer levels in the films were also verified by Parr Bomb analysis. (Dispersion at let down levels is not practical to ascertain so this testing was not done.)

### Film Extrusion and Treating

PPA-1 and PPA-2 were evaluated at 0, 600 ppm, 1200 ppm and 2000 ppm. Film was fabricated at these PPA levels in a commercial resin formulated with slip and antiblock. To summarize: 2 mil film was blown with a monolayer blown film line with a 2.5 inch extruder, 24:1 L:D, a 6 inch rotating film die with a 100 mil die gap, dual lip air ring, chilled air, continuous gravimetric blending and a NDC on-line gauging system. An MC-2 single flight screw was used and a 20/40/60/80/100/200/20 mesh screen pack. Set and actual conditions are detailed in Table I. Constant output was maintained as much as possible throughout the film trial, as were other extrusion parameters.

Films were treated using an industrial 'covered-roll' corona treater from Sherman Treaters Ltd., Model GT.09. Films were treated at 3.1 amps, at ambient air temperature and humidity. Treatment parameters were chosen such that a nominal surface energy of 41 dynes/cm was achieved as measured by ASTM D 2579-94.

### Film Testing

The film physical and optical properties were tested according to the methods listed in Table II. Because of the variation possible in heat seal and hot tack set conditions, these are listed in some detail as two separate paragraphs at the end of the Table II.

### Surface Analysis

The surfaces of the films generated in this study were examined by two surface analytical methods, X-ray photoelectron spectroscopy (also known as ESCA) and static secondary ion mass spectrometry. We used a Physical Electronics Model 5100 X-ray Photoelectron Spectrometer and a Physical Electronics Model 6000 Secondary Ion Mass Spectrometer. The base pressure in the vacuum system was less than $1x10^{-7}$ torr during the analysis.

## RESULTS AND DISCUSSION

### Film Test Results

The film physical and optical test results are detailed in Table III, summarized in Table IV and plotted in Figures 3-16.

To magnify any effects, the PPAs were added at levels up to 2000 ppm. This is in excess of the typical use levels. Previous work on this film line has shown 400 ppm of PPA-2 (or 500 ppm of PPA-1) was required to completely eliminate melt fracture from the formulated plastomer used in this study.

For most of the test results, one can see from the plots, there is no difference in the films with and without PPA. There is little to comment on about these tests. Some points of interest follow.

We chose to test kinetic COF (inside/inside) over the other variations of COF because plastomer resins are often used as sealant layers in films for vertical form fill and seal applications and so, this test made sense to illustrate end use performance. We also chose a second COF test – static (outside/outside) to correlate to the property of 'stackability' of bags.

We chose to do induced block because we were hoping to spread apart any differences in the films a bit more, if there were any differences to be seen. The differences in block between the various formulations are not statistically significant.

If one is unfamiliar with the color test, please see Appendix 1 where the meaning of the various color indices is illustrated.

Clarity was measured with the ASTM – pending method using the Hazeguard Plus instrumentation. This specific property has not been reported in previously published work on PPA effects on film optics. The clarity test itself measures narrow angle (< 2.5 degrees) light scattering and is a way of quantifying the ability to resolve detail through films (Ref. 10). Although the trend was for PPA to improve clarity, the improvements are not visible to the naked eye and are thus not significant in any practical application. For those interested in theoreticals, perhaps the PPA is acting in some kind of nucleating role to make any minor improvement seen.

Gloss measures light scattered back at the viewer and is a way of quantifying how shiny films appear. The test method typically has a ± 1 unit variability and the typical trained technician can see ± 2 units. PPA-2 was previously found to significantly enhance gloss in LLDPE films (Ref. 1). This positive effect was not seen here in plastomer film perhaps because of the superior inherent gloss of plastomer films making it difficult to bring about any incremental improvement. In addition to this inherent difference in optics between LLDPE and plastomers, perhaps with the LLDPE, the PPA is eliminating some type of 'micro' melt fracture that one does not see to the same extent with the plastomers we used in this study due to a different molecular architecture.

Again, for all the following tests: clarity, gloss, transmittance, color (a*, b*, L*, YI), hot tack, heat seal, block, surface tension, COF - the presence of PPA , even at levels far exceeding normal use levels, was found to have essentially no effect.

Haze measures wide angle (>2.5 degrees) light scattering and is a way of quantifying how well we can see contrast through films (Ref. 10). With PPA-2, one sees a trend of increasing haze with increasing PPA. The differences are not statistically significant at 600 ppm based on 95% confidence interval calculations. However, at 1200 ppm and 2000 ppm PPA-2, they are statistically significant. The PPA-1 at all levels showed an impact for haze. (Although the difference at 600 ppm was statistically significant based on 95% confidence interval calculations, the differences are barely noticeable with the naked eye.) For PPA-1, at levels of 1200 ppm and 2000 ppm, PPA-1 showed statistically significant differences in haze. These levels exceed the normal use levels for this resin. From previously unpublished work, the authors know the eye of a well-trained technician can just start to see differences at 0.5% haze. Perhaps the reason for the haze is that the fluoropolymer component of the PPA has a different refractive index than the polyethylene and is causing some wide angle scatter of light. Previous work (Ref. 1) on PPA in LLDPE on a narrow die gap had not concluded this effect of PPA on haze. However, the current study was done on a wide die gap. It should be noted that

one of the primary advantages in using PPAs is to enable one to run on narrow die gaps. Narrow die gaps are generally known to provide enhanced optics (Ref. 1) due to the faster quenching of the melt which results from its initial thinness. Thus, PPA indirectly provides a positive effect on optics although it was not highlighted by this particular study. Another point worth noting is that the plastomer film used in this study, when formulated with antiblock and slip, had haze numbers that are quite low (5.0) when compared to the haze of LLDPE films. So perhaps it is the excellent haze values of the plastomer film that highlight the minor increase in haze. With LLDPE the haze is generally not so good and so any minor increase would not be noticeable, or, if noticed, would not be of practical significance.

**Surface Chemistry**

The surface chemistry of a polymer film controls many aspects of its performance. The primary effect that surface chemistry controls is the wettability of the film. Adhesion of coatings to the film as well as adhesion of the film to itself are controlled by its surface chemistry. There are numerous examples of this effect in the literature.

One technique that we used for this analysis for surface chemistry analysis is **XPS** or **X**-ray **P**hotoelectron **S**pectroscopy. The technique is known also as **E**lectron **S**pectroscopy for **C**hemical **A**nalysis or **ESCA**. A sample is exposed to an X-ray source in this high-vacuum surface-analysis method. The atoms in the surface of the sample absorb the X-rays and electrons are emitted by the Einstein effect. The mean free path of an electron is very short, usually on the order of tens of Angstrom units. The shortness of the electron mean free path provides the surface sensitivity of ESCA. That is, only those electrons emitted from atoms within one mean free path of the surface can escape from the surface, thus providing a detectable signal. ESCA is sensitive to all the elements of the periodic table with the exception of hydrogen and helium. ESCA provides three types of data. Having a set of previously determined sensitivity factors allows one to determine the surface elemental composition of the so-interrogated surface layer. This composition is the percentage of each element on the surface less hydrogen and helium. The next type of data is a limited amount of chemical information. By examination of higher resolution spectra, extra peaks are observed for each element. The position of the extra peaks is indicative of the chemical state in which the electron-emitting atom finds itself. The more electronegative the neighboring atoms, the higher the binding energy of the electron. This effect, however, is limited to nearest neighbors. The final type of data that ESCA can provide is a depth profile of elemental composition. The mean free path effect discussed above and the geometric arrangement of sample and detector provides a means to determine a rough surface chemical composition determination as a function of distance from the surface into the bulk of the material. The Take-off Angle is roughly the distance in Angstroms that the technique interrogates. Thus a Take-off Angle of 45° corresponds roughly to a depth of analysis of 45 Angstrom units ($45 \times 10^{-8}$ cm.)

Another technique that we used for this analysis is known as **S**tatic **S**econdary **I**on **M**ass **S**pectroscopy or **SSIMS**. This is a high vacuum surface analysis method in which a sample is bombarded with a high energy (3.5 kV), low current (1.5 nA) Xenon ($Xe^+$) ion beam. The beam current is low so that the sample is kept under "static" conditions, i.e., conditions under which minimal sample surface damage occurs. At 1.5 nA, damage occurs at something around 10 minutes of bombardment. None of the data that we generated had this level of sample bombardment. An electron beam also bombards the sample. As material is removed from the sample, the surface charges and ion removal is blocked by a space charge effect. The electron beam mitigates this phenomenon so ions can be extracted from the surface. Of the material removed from the surface, about 5% is ionized. The ions are accelerated by a series of lenses through a sector analyzer and finally through a quadrapole mass spectrometer. The ion mass/charge ratio is measured as well as the ion current at that mass-to-charge ratio. The data in the spectra show the fragments of the materials that come from the surface. The ions that are detected are the most stable ions that result either from removal of intact portions of the material from the surface or from rearrangement in the space above the sample. Both positive and negative ions are measured. The (-)SSIMS is particularly useful for examining surfaces which contain electronegative elements such as oxygen or fluorine. We operate at a resolution of about 0.25 amu. The technique is very sensitive, the detection limit is sub-monolayer. The technique is not quantitative in that peak intensities are very sensitive to the sample charging conditions that could change from sample to sample. The fragments that can be removed from a certain sample are quite reproducible. The fragmentation pattern can be used as a means of identification (if library spectra are available) or as a means of comparing samples (the same surface will yield the same fragmentation pattern of masses).

Figure 17a is a XPS survey spectrum for a fully formulated plastomer while I7b is for that same formulated plastomer but containing 2000 ppm PPA additive. The XPS spectra show the presence of carbon, nitrogen and oxygen. The plastomer is not expected to contain any heteroatoms. The nitrogen and oxygen are undoubtedly due to the slip agent additive. The reader's attention is drawn to the region around 690 eV binding energy. This is the region for fluorine. Spectrum I7b shows no detectable fluorine for

the formulated plastomer containing the PPA. Table V provides a complete listing of the XPS surface elemental composition results taken from higher resolution spectra. No measurable fluorine is detected for any sample at any Take-off Angle.

Figures 18a and l8b display the corresponding (-)SSIMS spectra. The reader's attention is drawn to the region at approximately 19 amu. This peak corresponds to the F ion. Approximately the same sized fluorine peak is detected for the sample containing PPA and that not containing PPA. We conclude that this small peak must be due to a background signal for fluorine.

Surface analysis indicates that the surfaces of plastomer generated with and without the presence of PPA are virtually identical. We conclude that the adhesion of coatings to these materials or of these materials to themselves would not be affected by the presence of the PPA.

## CONCLUSION

In general, one can say that PPAs when used at typical dose levels have essentially no effect on plastomer film surface or optical properties. The results of this study did indicate some detraction from haze at high levels of PPA exceeding normal dose levels with PPA-2 being not quite as detrimental at high levels as PPA-1. Surface analysis indicates that the surfaces of plastomer generated with and without the presence of PPA are virtually identical. We conclude that the adhesion of coatings to these materials or of these materials to themselves would not be affected by the presence of the PPA additive.

## ACKNOWLEDGEMENTS

The authors thank Kelly Frey and Doug Foubister of Dow Chemical Canada Inc. for assisting with film fabrication, Lori Carroll of Dow Chemical Canada Inc. for coordinating the film testing and Jeff Wooster, Laura Mergenhagen (The Dow Chemical Company) and Deb Walker (Dow Chemical Canada Inc.) for discussions on the results of this study. The authors would also like to thank Joyce Kunde of Dyneon LLC for the optical microscopy photo of PPA in LLDPE.

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**Table I**
**Macro Blown Film Line Fabrication Conditions**

| Run Date | | 2/12/98 | 2/13/98 | 2/13/98 | 2/13/98 | 2/16/98 | 2/16/98 | 2/16/98 |
|---|---|---|---|---|---|---|---|---|
| Formulation | | 0% PPA | 600 ppm | 1200 ppm | 2000 ppm | 600 ppm | 1200 ppm | 2000 ppm |
| | | | FX5920A | FX5920A | FX5920A | FX-9613 | FX-9613 | FX-9613 |
| Layflat (inches) | | 24 | 24 | 24 | 24 | 24 | 24 | 24 |
| Extruder Temperature Profile (F) | Set | | | | | | | |
| Zone 1 (Feed) | 249 | 250 | 250 | 250 | 250 | 250 | 250 | 250 |
| Zone 2 | 269 | 270 | 270 | 270 | 270 | 270 | 270 | 270 |
| Zone 3 | 359 | 360 | 360 | 360 | 360 | 360 | 360 | 360 |
| Zone 4 | 399 | 360 | 360 | 360 | 360 | 360 | 360 | 360 |
| Adaptor | 399 | 403 | 399 | 399 | 399 | 401 | 399 | 399 |
| Rotator | 399 | 396 | 401 | 401 | 399 | 399 | 401 | 401 |
| Die 1 | 399 | 406 | 401 | 399 | 399 | 399 | 399 | 401 |
| Die 2 | 399 | 397 | 399 | 399 | 399 | 399 | 399 | 401 |
| Die 3 | 399 | 387 | 399 | 399 | 399 | 401 | 399 | 399 |
| Melt Temperature 1 (F) Screen Pack | | 406 | 403 | 403 | 403 | 403 | 403 | 403 |
| Melt Temperature 2 (F) Die | | 406 | 412 | 412 | 414 | 412 | 412 | 414 |
| Extruder Pressure | | | | | | | | |
| P1 | | 1 | 10 1 | 10 | 11 | 13 1 | 14 | 16 |
| P2 | | 3171 1 | 2647 1 | 2764 | 3371 | 2877 1 | 2996 | 2890 |
| P3 | | 4440 1 | 3940 1 | 3990 | 4040 | 3870 1 | 3940 | 3810 |
| P4 | | 4255 1 | 3840 1 | 3860 | 3870 | 3690 1 | 3675 | 3550 |
| Air Ring Temperature (F) | | 52 | 54 | 54 | 55 | 52 | 52 | 52 |
| Air Ring Pressure (inches $H_2O$) | | 4.2 | 4.3 | 4.4 | 4.4 | 4.4 | 4.4 | 4.6 |
| Blower RPM | | 1939 | 1942 | 1941 | 1940 | 1952 | 1930 | 1985 |
| Screw Speed (rpm) | | 50 | 52 | 52 | 59 | 48 | 48 | 48 |
| Screw Volts (volts) | | 170 | 173 | 173 | 190 | 166 | 168 | 163 |
| Screw Load (%) | | 63 | 53 | 52 | 40 | 54 | 48 | 55 |
| Film Speed (ft/min) | | 59 | 59 | 59 | 62 | 59 | 59 | 59 |
| Frost Line Ht. (inches) | | 23 | 22 | 24 | 24 | 22 | 22 | 22 |
| Output Rate (lb/hr) | | 139 | 142 | 141 | 140 | 138 | 142 | 139 |
| Drive Current (amps) | | 80.0 | 67.3 | 66.0 | 50.8 | 68.6 | 61.0 | 69.9 |
| HorsePower (hp, calc) | | 18.2 | 15.6 | 15.3 | 12.9 | 15.3 | 13.7 | 15.3 |
| Output Rate | | 139 | 142 | 141 | 140 | 138 | 142 | 139 |
| Die Rate (lb/hr/inch die circumference) | | 7.4 | 7.5 | 7.5 | 7.4 | 7.3 | 7.5 | 7.4 |
| Pumping Efficiency (lb/hr/rpm) | | 2.79 | 2.73 | 2.71 | 2.37 | 2.88 | 2.95 | 2.90 |
| Energy Efficiency (lb/hr/hp) | | 7.6455 | 9.0971 | 9.2067 | 10.8206 | 9.0430 | 10.3218 | 9.1075 |
| Screen Pack | 20/40/60/80/100/200/20 | | | | | | | |
| Other: 6" die, 2.5 inch screw, 24:1 length: diameter, dual lip air ring, no IBC, chilled air, 100 mil die gap | | | | | | | | |

**Table II**
**Test Methods Used in This Study**

| **Test** | **Method** |
|---|---|
| Gauge | Winzen International Inc bench model capacitance thickness profiler with a 2 x 1/8 inch head |
| Surface Tension | ASTM D 2578-94 |
| Color | Colortech Spectrophotometer |
| 45 Degree Gloss | ASTM D 2457 |
| Haze | ASTM D 1003 |
| Clarity | ASTM pending (Hazeguard Plus Instrumentation) |
| Transmittance | ASTM D 1003 |
| COF | ASTM D 1894 |
| Block | ASTM D 3354-89 |
| | |
| **Hot Tack Test Conditions:** | |
| DTC Hot Tack Tester Model No. 52D | |
| Specimen Width: | 24.4 mm (1 inch) |
| Sealing Time: | 0.5 seconds |
| Sealing Pressure: | 0.27 N/mm/mm |
| Delay Time: | 0.5 seconds |
| Peel Speed: | 150 mm/sec |
| No. of Samples/Temperature: | 5 |
| Temperature Increments: | 5°C |
| Min. Force Chosen for Hot Tack | |
| Initiation: | 1.0 N/inch |
| | |
| **Heat Seal Test Conditions:** | |
| Specimen Width: | 24.4 mm (1 inch) |
| Sealing Time: | 0.5 seconds |
| Sealing Pressure: | 0.27 N/mn/mm |
| No. of Samples/Temperature | 5 |
| | |
| Seals conditioned for 24-48 hours at 50% RH and 23°C. | |
| | |
| Seals pulled using an Instron Tensile Tester Model No. 1122 as follows: | |
| Direction of Pull: | 90 degrees to seal |
| Crosshead Speed: | 500 mm/min |
| Full Scale Load: | 5 kg |
| Threshold: | 1% of FSL |
| Break Criterion: | 80% |
| Gauge Length: | 2.0 inches |
| Sample Width: | 1 inch |
| Min. Force Chosen for Heat Seal | |
| Initiation: | 1 lb/inch |

## Table III - Detailed Results

| Formulation | slip,A/B-no PPA | | slip,A/B-600ppmFX9613 | | slip, A/B-1200ppmFX9613 | | slip,A/B-2000ppmFX9613 | | slip,A/B -600ppmFX5920A | | slip,A/B -1200PPMFX5920A | | slip,A/B 2000ppmFX5920A | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Avg. | Std. Dev. | Avg. | Std. Dev. | Avg. | Std. Dev. | Avg. | Std. Dev. | Avg. | Std. Dev. | Avg. | Std. Dev. | Avg. | Std. Dev. |
| **Gauge (Winzen, capacitance)** | | | | | | | | | | | | | | |
| Average (mil) | 1.88 | | 1.89 | | 1.94 | | 1.92 | | 1.87 | | 1.91 | | 1.8 | |
| High (mil) | 2.37 | | 2.18 | | 2.38 | | 2.39 | | 2.37 | | 2.34 | | 2. 1 | |
| Low (mil) | 1.65 | | 1.51 | | 1.72 | | 1.69 | | 1.65 | | 1.69 | | 1.53 | |
| **Surface Tension Inside (dynes)** | 41 | | 41 | | 41 | | 41 | | 41 | | 41 | | 41 | |
| **Surface Tension Outside (dynes)** | 41 | | 41 | | 41 | | 41 | | 41 | | 41 | | 41 | |
| **Color (Colortech Spectrophotometer)** | | | | | | | | | | | | | | |
| L* | 96.54 | | 96.52 | | 96.53 | | 96.50 | | 96.57 | | 96.52 | | 96.58 | |
| a* | 0.02 | | 0.02 | | 0.02 | | 0.01 | | 0.02 | | 0.02 | | 0.02 | |
| b* | 0.02 | | 0.02 | | 0.02 | | 0.01 | | 0.04 | | 0.03 | | 0.01 | |
| Yellowness Index* | -0.04 | | -0.04 | | -0.04 | | -0.07 | | -0.01 | | -0.02 | | -0.06 | |
| **45 Degree Gloss (ASTM D 2457)** | 74.8 | 0.1 | 75.9 | 0.7 | 74.0 | 1.4 | 74.0 | | 75.3 | 0.5 | 75.3 | 1.0 | 76.7 | 1.1 |
| Haze (%) (ASTM D 1003) | 5.0 | 0.0 | 5.6 | 0.1 | 7.5 | 0.2 | 7.8 | | 5.3 | 0.1 | 5.8 | 0.2 | 6.1 | 0.4 |
| Clarity (%) (ASTM pending) | 94.4 | 0.0 | 94.5 | 0.0 | 94.7 | 0.0 | 94.7 | | 94.5 | 0.0 | 94.7 | 0.1 | 94.7 | 0.2 |
| Transmittance % (ASTM D 1003) | 94.0 | 0.0 | 93.9 | 0.0 | 93.9 | 0.0 | 93.8 | 0.1 | 93.9 | 0.0 | 93.9 | 0.1 | 93.9 | 0.0 |
| **COF (ASTM D1894)** | | | | | | | | | | | | | | |
| Inside/Inside | | | | | | | | | | | | | | |
| Kinetic | 0.09 | 0.01 | 0.08 | 0.01 | 0.08 | 0.00 | 0.10 | 0.02 | 0.11 | 0.02 | 0.1 | 0.01 | 0.11 | 0.01 |
| Outside/Outside | | | | | | | | | | | | | | |
| Static | 0.15 | 0.02 | 0.11 | 0.01 | 0.12 | 0.01 | 0.12 | 0.02 | 0.16 | 0.01 | 0.15 | 0.02 | 0.13 | 0.00 |
| **Block (ASTM 3354-89)** | | | | | | | | | | | | | | |
| Induced Block | | | | | | | | | | | | | | |
| Test Temperature (50°C) | | | | | | | | | | | | | | |
| Load (g) | 22 | 1 | 22 | 2 | 26 | 1 | 32 | 3 | 32 | 4 | 36 | 3 | 37 | 5 |

## Table III - Detailed Results

**Hot Tack Strength (N/25 mm)**

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 70°C | 0.19 | 0.00 | 0.19 | 0.01 | 0.22 | | 0.18 | 0.01 | 0.19 | 0.01 | 0.207 | 0.01 | 0.20 | 0.01 |
| 75°C | 0.49 | 0.05 | 0.50 | 0.01 | 0.48 | | 0.47 | 0.02 | 0.52 | 0.03 | 0.5 | 0.03 | 0.44 | 0.04 |
| 80°C | 0.98 | 0.08 | 0.99 | 0.05 | 0.97 | | 0.87 | 0.04 | 0.93 | 0.05 | 0.943 | 0.02 | 0.90 | 0.07 |
| 85°C | 1.87 | 0.07 | 1.74 | 0.05 | 1.82 | | 1.72 | 0.20 | 1.75 | 0.09 | 1.738 | 0.06 | 1.75 | 0.07 |
| 90°C | 3.07 | 0.11 | 3.11 | 0.22 | 3.24 | | 3.02 | 0.54 | 3.11 | 0.19 | 2.98 | 0.21 | 3.15 | 0.28 |
| 95°C | 3.67 | 0.16 | 3.65 | 0.17 | 3.88 | | 3.43 | 0.11 | 3.92 | 0.17 | 3.851 | 0.09 | 3.40 | 0.14 |
| 100°C | 3.96 | 0.23 | 3.98 | 0.61 | 4.28 | | 3.86 | 0.29 | 4.24 | 0.22 | 3.96 | 0.12 | 4.07 | 0.37 |
| 105°C | 3.63 | 0.26 | 3.89 | 0.12 | 3.69 | | 3.80 | 0.25 | 3.81 | 0.30 | 3.678 | 0.34 | 3.65 | 0.67 |
| 110°C | 3.49 | 0.18 | 3.58 | 0.12 | 3.92 | | 3.60 | 0.22 | 3.52 | 0.19 | 3.354 | 0.33 | 3.77 | 0.17 |
| 115°C | 3.26 | 0.11 | 3.20 | 0.39 | 3.85 | | 3.48 | 0.19 | 3.24 | 0.25 | 3.012 | 0.71 | 3.24 | 0.57 |
| 120°C | 2.41 | 0.23 | 2.87 | 0.25 | 3.05 | | 3.24 | 0.27 | 3.13 | 0.24 | 1.882 | 1.08 | 2.95 | 0.43 |
| 125°C | 2.27 | 0.37 | 2.85 | 0.23 | 2.84 | | 2.89 | 0.30 | 2.81 | 0.16 | 2.234 | 0.30 | 2.22 | 0.65 |
| 130°C | | | | | | | | | | | | | | |

**Heat Seat Failure Load (lb/125 mm)**

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 80°C | 0.09 | 0.07 | 0.32 | 0.39 | 0.33 | 0.42 | 0.14 | 0.02 | | | 0.13 | 0.03 | 0.08 | 0.01 |
| 85°C | 1.10 | 0.07 | 0.87 | 0.38 | 1.03 | 0.02 | 0.96 | 0.06 | 1.07 | 0.04 | 1.03 | 0.05 | 0.95 | 0.04 |
| 90°C | 1.97 | 0.11 | 1.91 | 0.08 | 1.89 | 0.03 | 1.85 | 0.07 | 1.82 | 0.18 | 1.92 | 0.06 | 2.16 | 0.41 |
| 95°C | 2.72 | 0.17 | 3.02 | 0.65 | 2.68 | 0.04 | 2.76 | 0.07 | 2.70 | 0.17 | 2.686 | 0.09 | 2.24 | 0.38 |
| 100°C | 4.26 | 0.60 | 4.42 | 0.15 | 4.11 | 0.04 | 4.03 | 0.09 | 4.11 | 0.16 | 4.35 | 0.37 | 3.97 | 0.16 |
| 105°C | 4.01 | 0.68 | 4.37 | 0.14 | 4.87 | 0.46 | 4.91 | 0.19 | 4.72 | 0.06 | 4.427 | 0.18 | 4.44 | 0.55 |
| 110°C | 4.01 | 0.22 | 4.83 | 0.55 | 4.74 | 0.44 | 4.86 | 0.48 | 4.46 | 0.38 | 4.971 | 0.27 | 4.49 | 0.50 |
| 115°C | 4.61 | 0.54 | 4.83 | 0.37 | 4.68 | 0.56 | 5.20 | 0.59 | 4.93 | 0.13 | 5.0006 | 0.21 | 4.92 | 0.31 |
| 120°C | 4.44 | 0.19 | 5.50 | 0.25 | 5.50 | 0.47 | 5.34 | 0.34 | 5.07 | 0.62 | 5.133 | 0.19 | 5.12 | 0.30 |
| 125°C | 5.01 | 0.26 | 5.18 | 0.27 | 5.24 | 0.23 | 5.19 | 0.16 | 5.08 | 0.45 | 5.037 | 0.42 | 5.07 | 0.43 |
| 130°C | 5.53 | 0.27 | 5.29 | 0.60 | 5.36 | 0.26 | 5.21 | 0.40 | 5.05 | 0.95 | 5.299 | 0.35 | 4.90 | 0.24 |

**Table IV**
**Summary of Results**

| Property | PPA-1 | PPA-2 | Comments |
|---|---|---|---|
| Surface Tension | 0 | 0 | |
| 45 Degree Gloss | 0 | 0 | |
| Haze | 0/- | 0 | PPA-1: low levels had no effect, high levels had slight negative effect<br>PPA-2: no effect |
| Clarity | 0 | 0 | |
| Transmittance | 0 | 0 | |
| COF (Kinetic, I/I) | 0 | 0 | |
| Block | 0 | 0 | |
| Hot Tack | 0 | 0 | |
| Heat Seal | 0 | 0 | |
| Color (a*,b*,L*, and yellowness index) | 0 | 0 | |

Legend: 0 = no impact; - = negative impact; + = positive impact

**Table V**
**Surface Elemental Composition Plastomer Containing Slip, Anti-block Additives and PPA.**

| Sample Designation | Take-Off Angle (Degrees) | Carbon | Oxygen | Nitrogen | Fluorine |
|---|---|---|---|---|---|
| Slip, anti-block only | 20 | 94.9 | 2.3 | 2.7 | ND |
| | 45 | 92.7 | 3.5 | 3.8 | ND |
| | 90 | 92.2 | 3.8 | 4 | ND |
| Slip, anti-block + 2000 ppm PPA-2 | 20 | 95.8 | 1.9 | 2.3 | ND |
| | 45 | 94.5 | 2.7 | 2.9 | ND |
| | 90 | 94.6 | 2.7 | 2.7 | ND |
| Slip, anti-block + 2000 ppm PPA-1 | 20 | 94.4 | 2.4 | 3.2 | Nd |
| | 45 | 93 | 3.7 | 3.4 | ND |
| | 90 | 92.9 | 3.6 | 3.5 | ND |

ND=Not Detected

**Figure 1**

## Schematic of PPA in the Polymer Melt

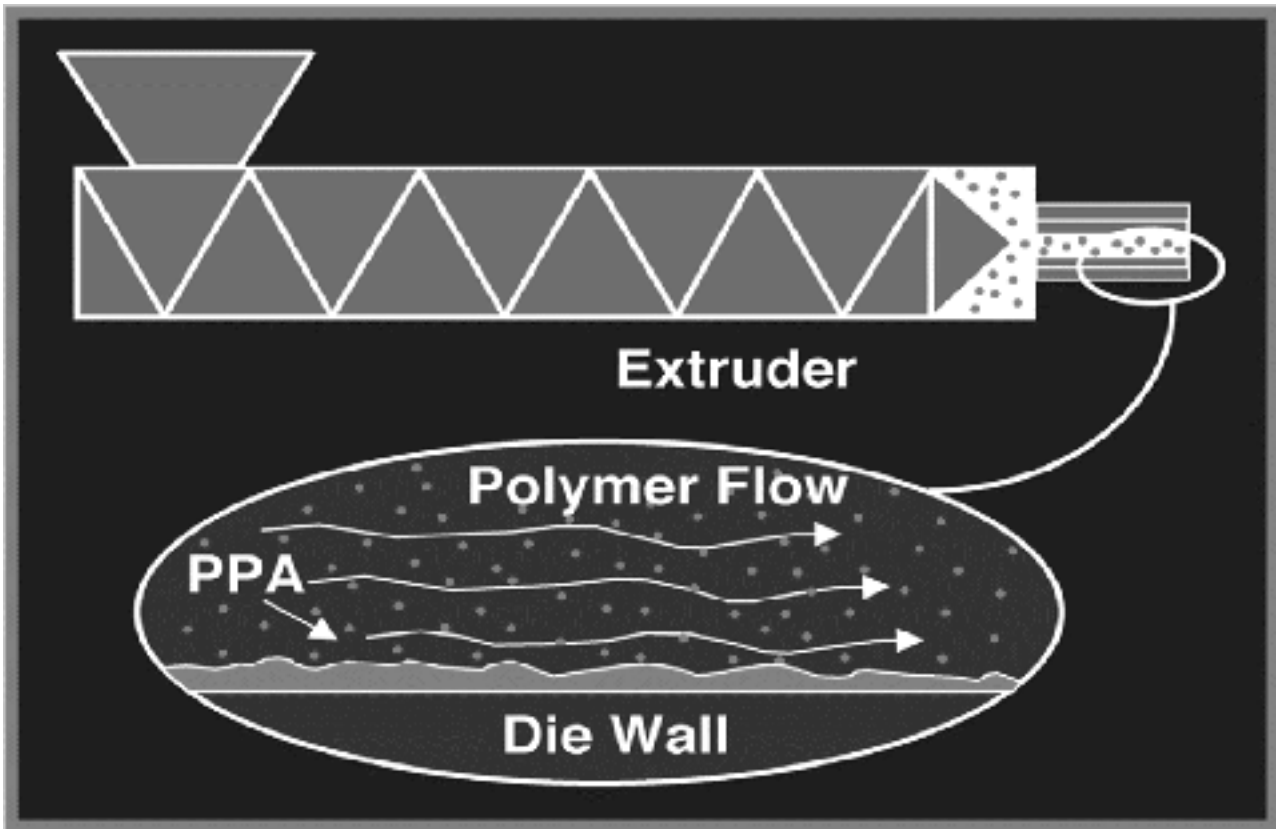


**Figure 2**

## Optical Micrograph of a PPA-1 Masterbatch in LLDPE

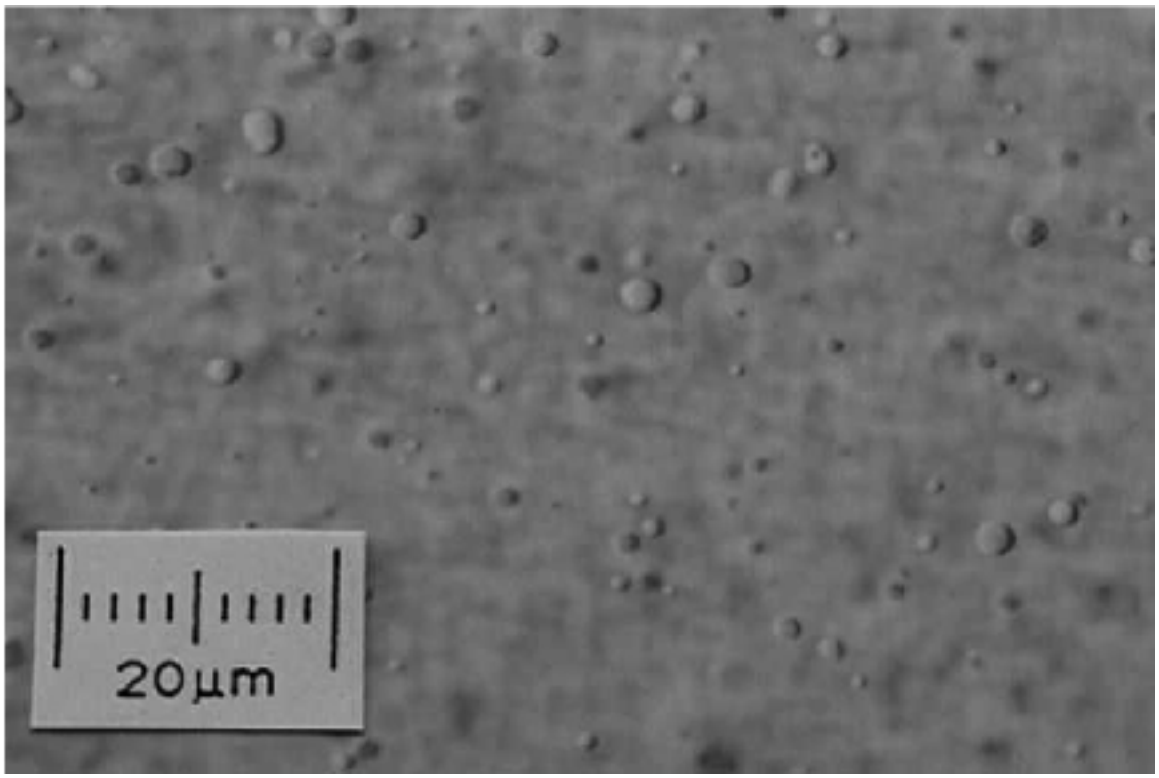


**Figure 3**

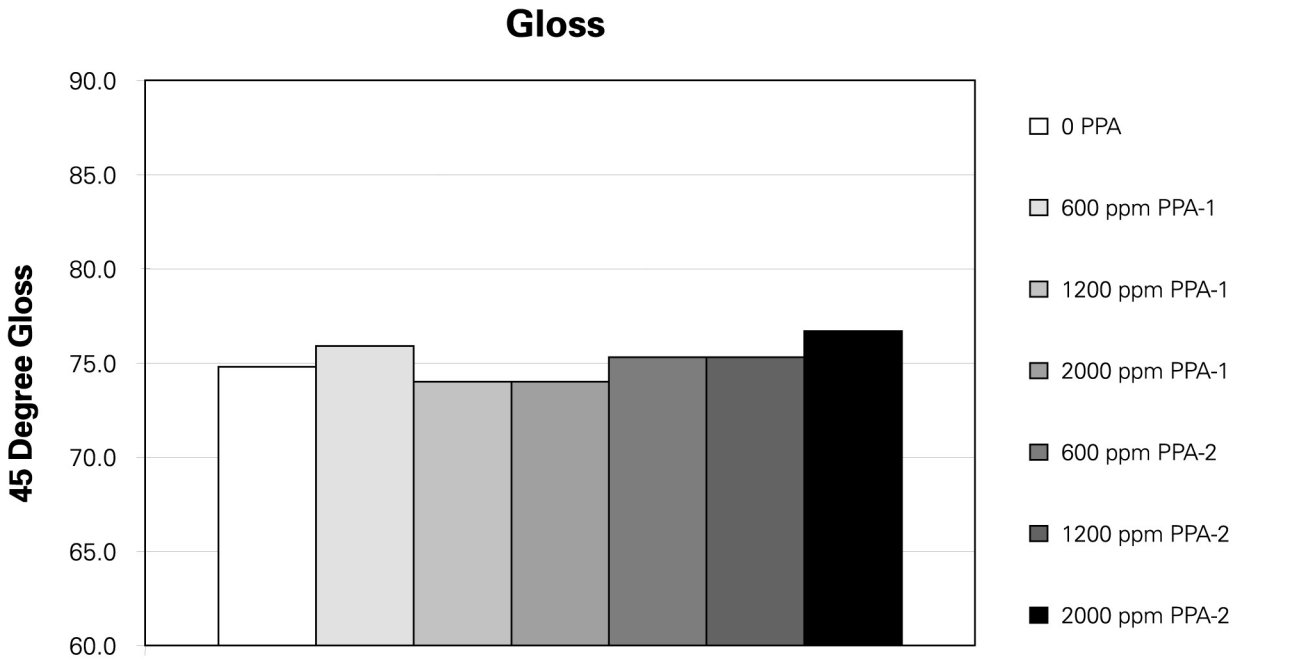



**Figure 4**

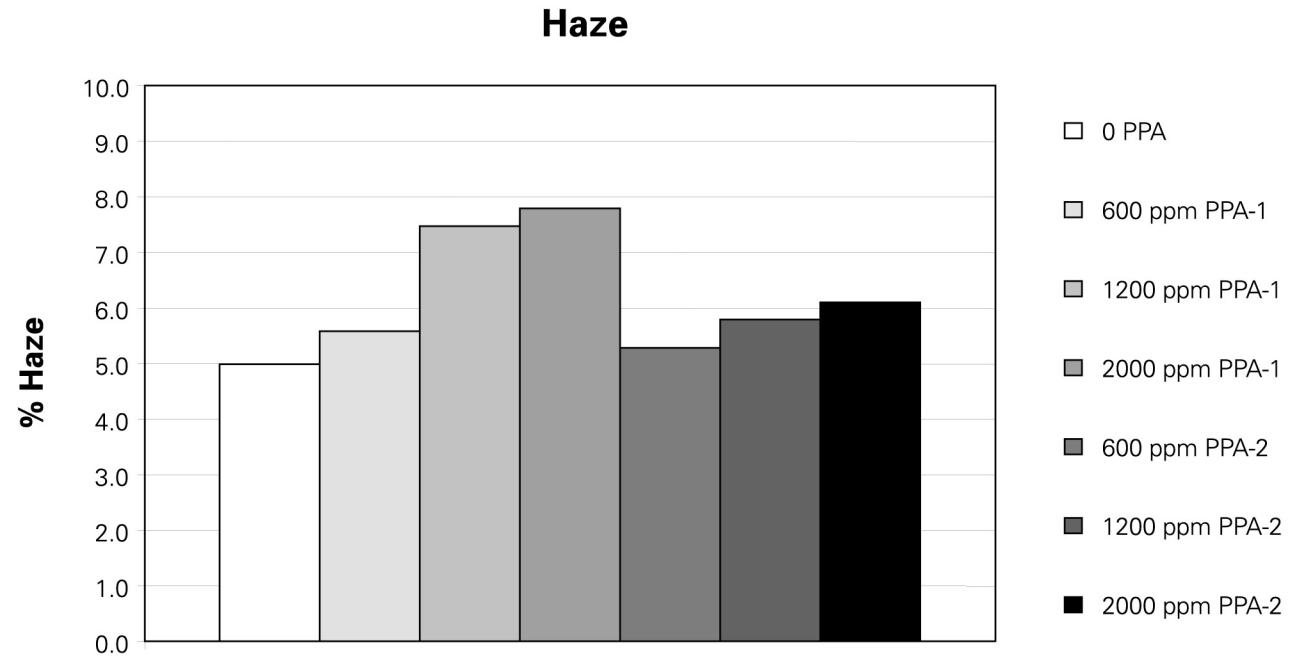


**Figure 5**

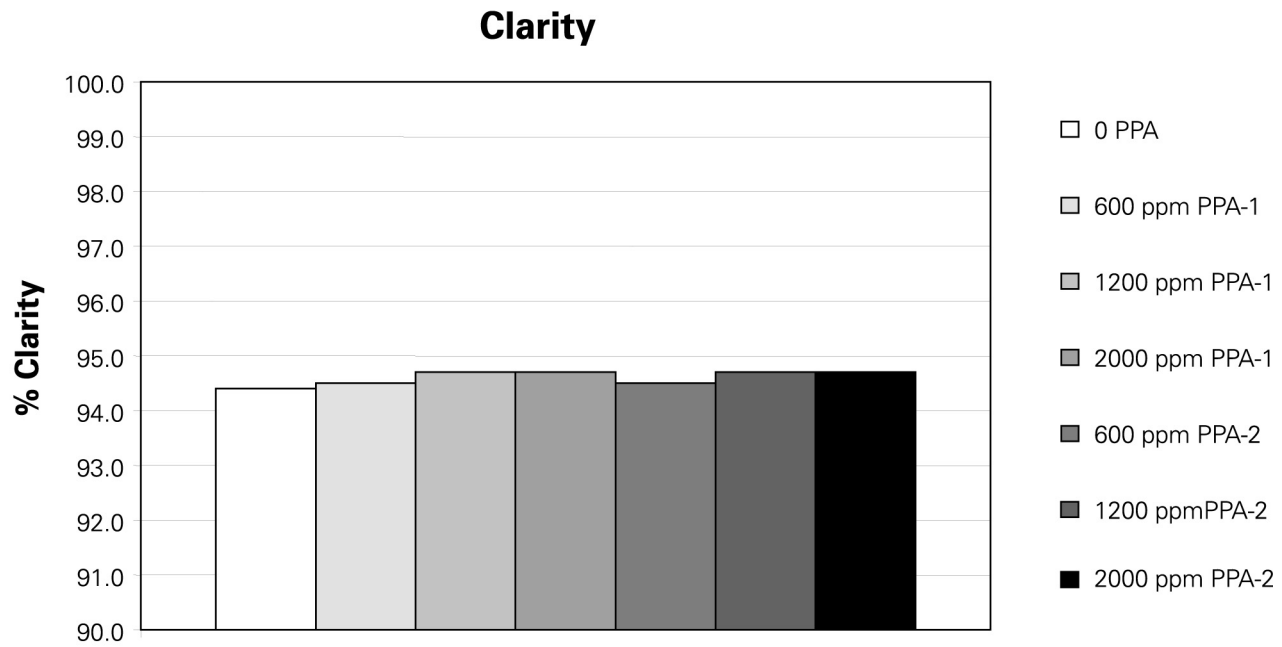



**Figure 6**

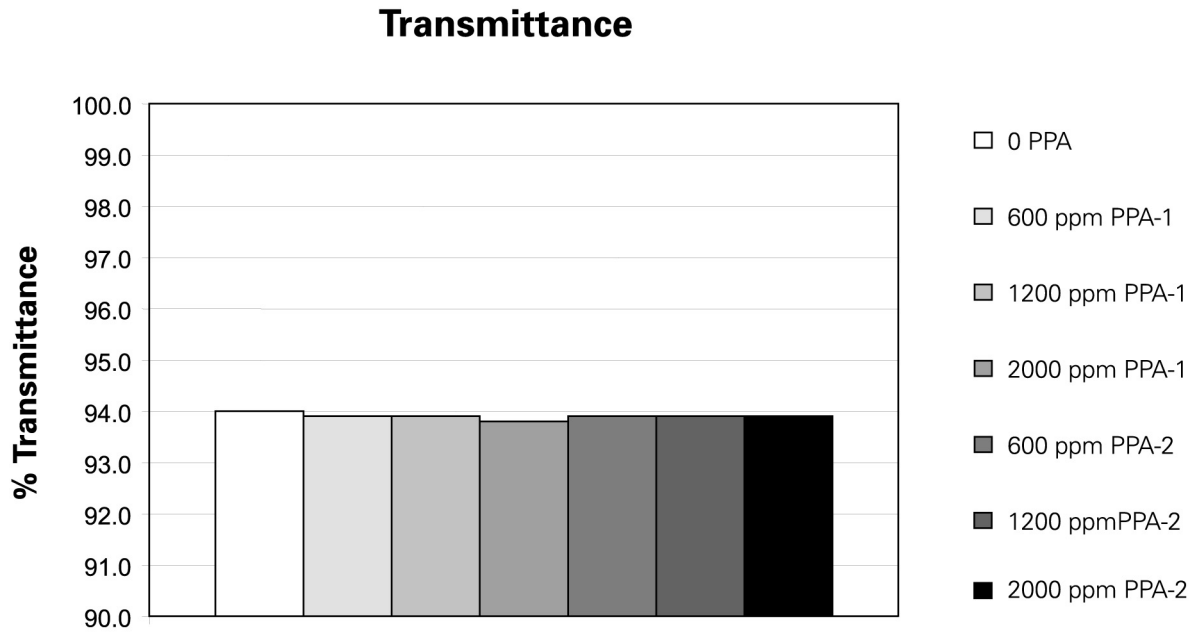


**Figure 7**

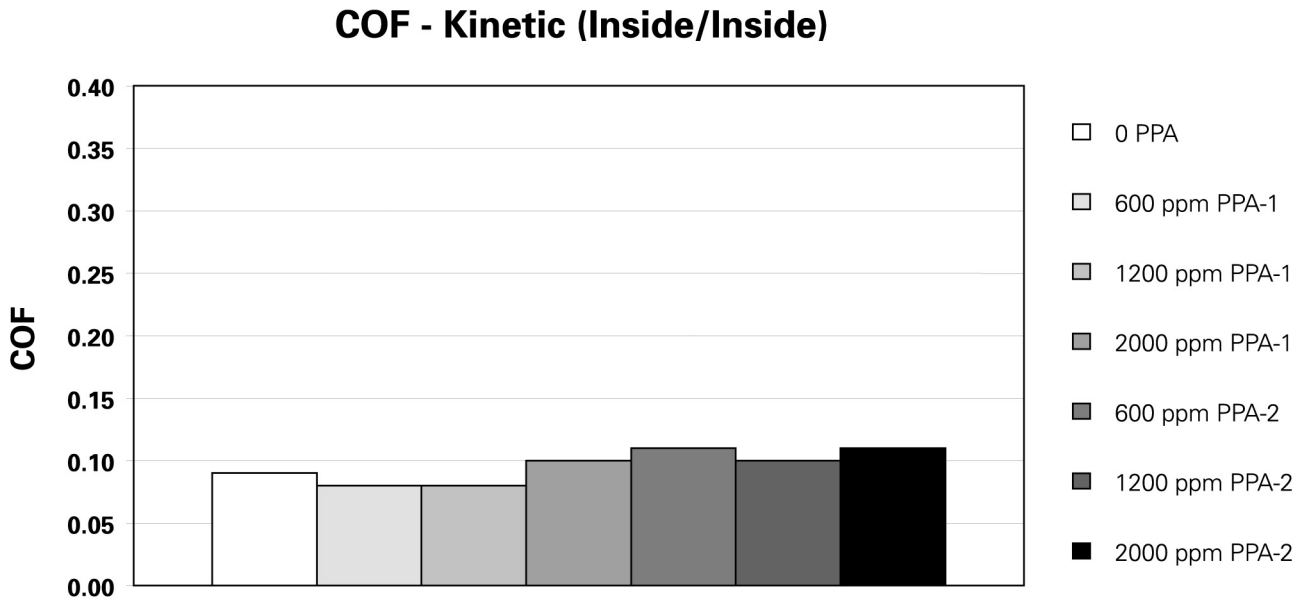



**Figure 8**

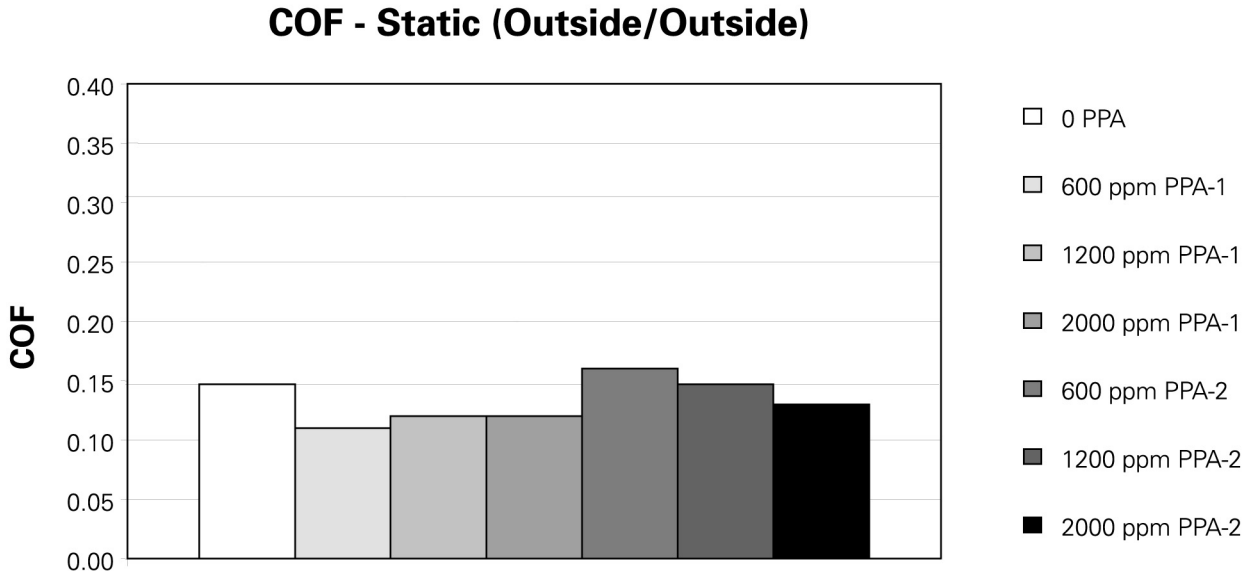


**Figure 9**

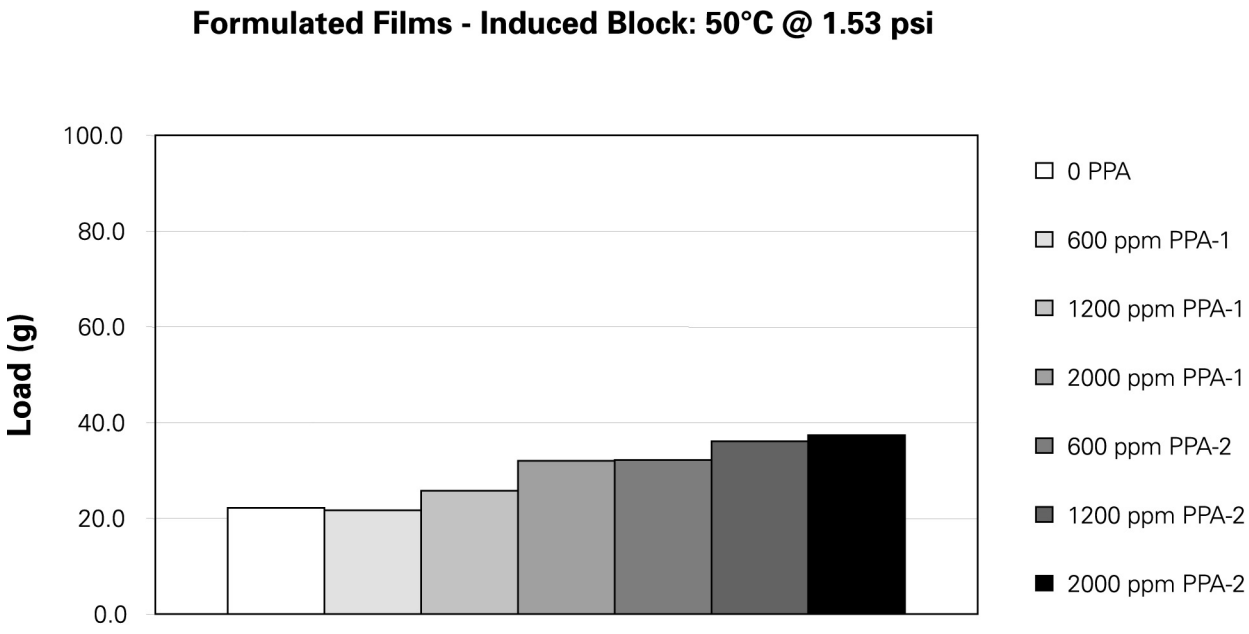



**Figure 10**

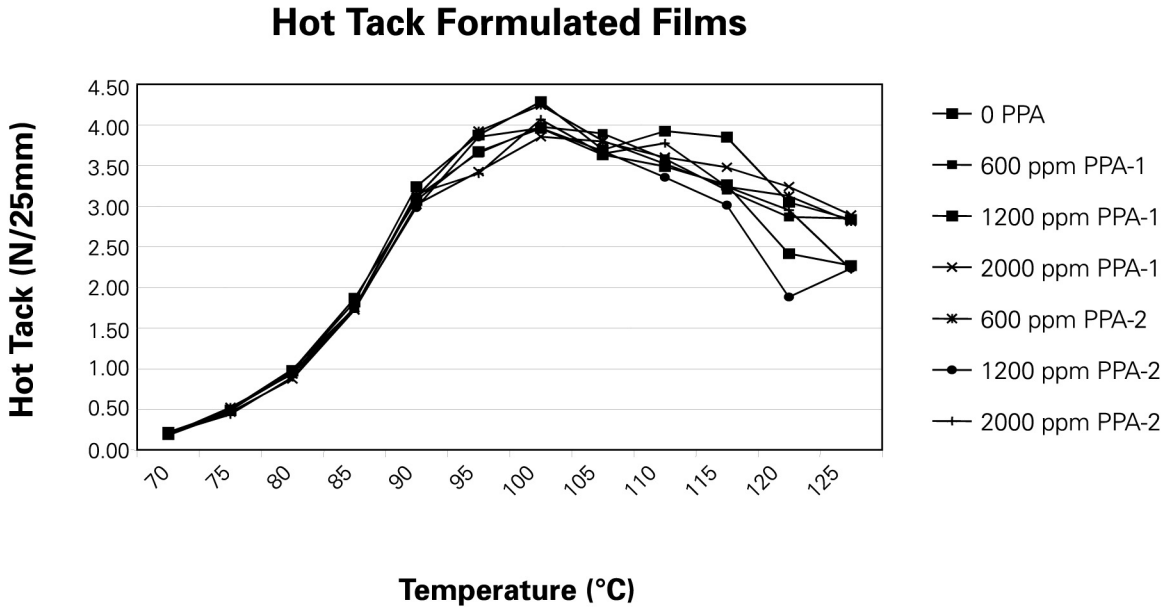


**Figure 11**

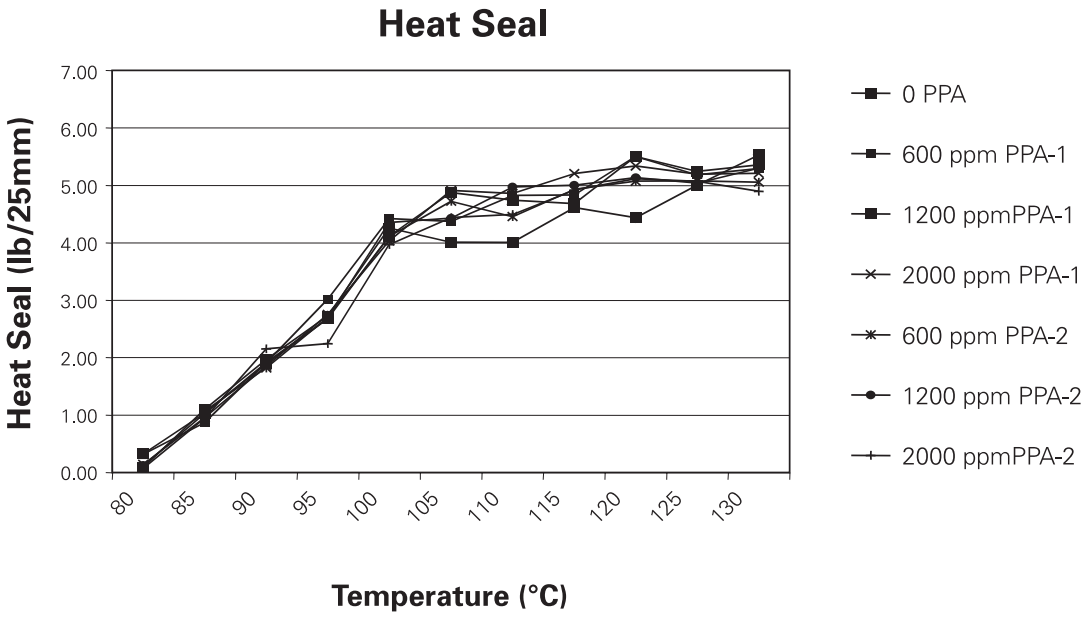



**Figure 12**

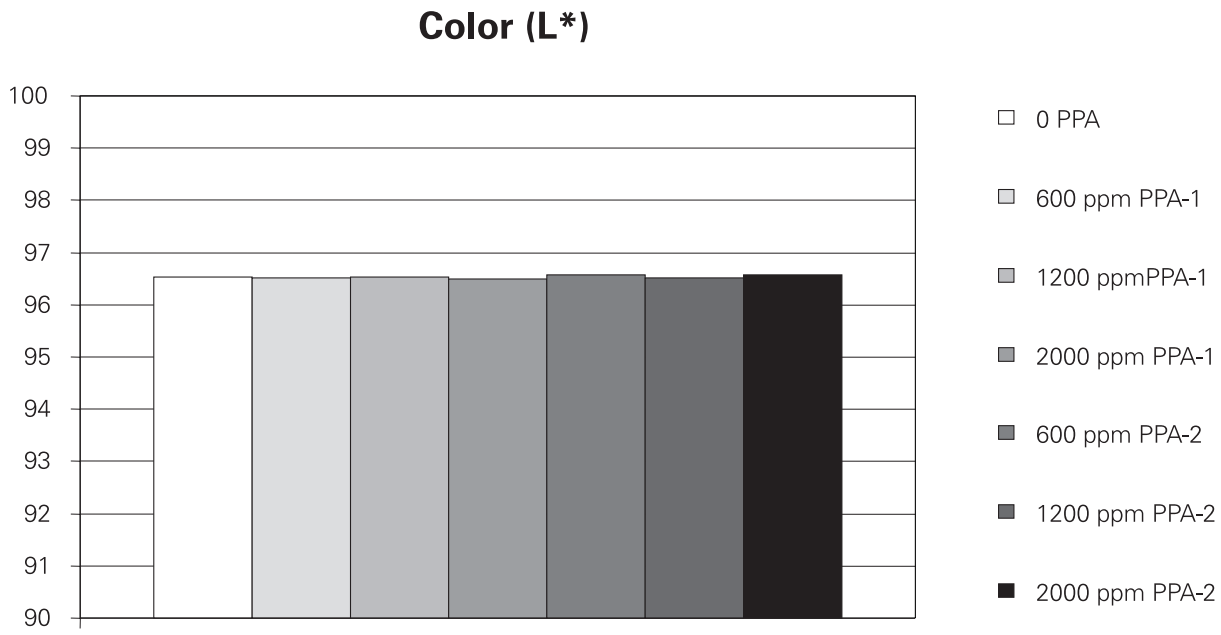


**Figure 13**

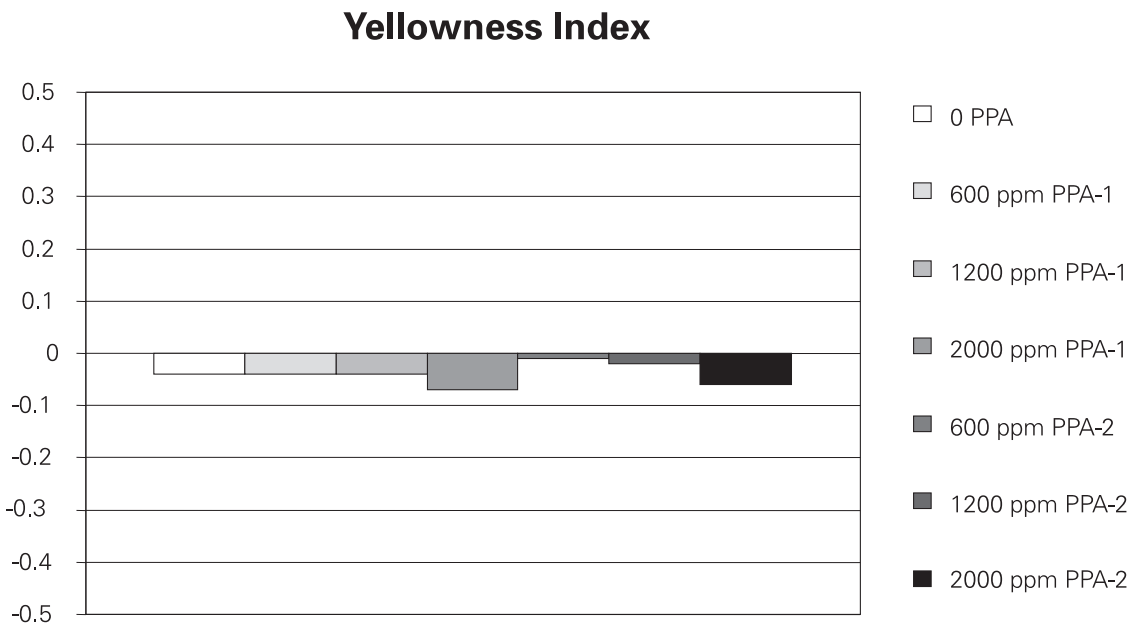



**Figure 14**

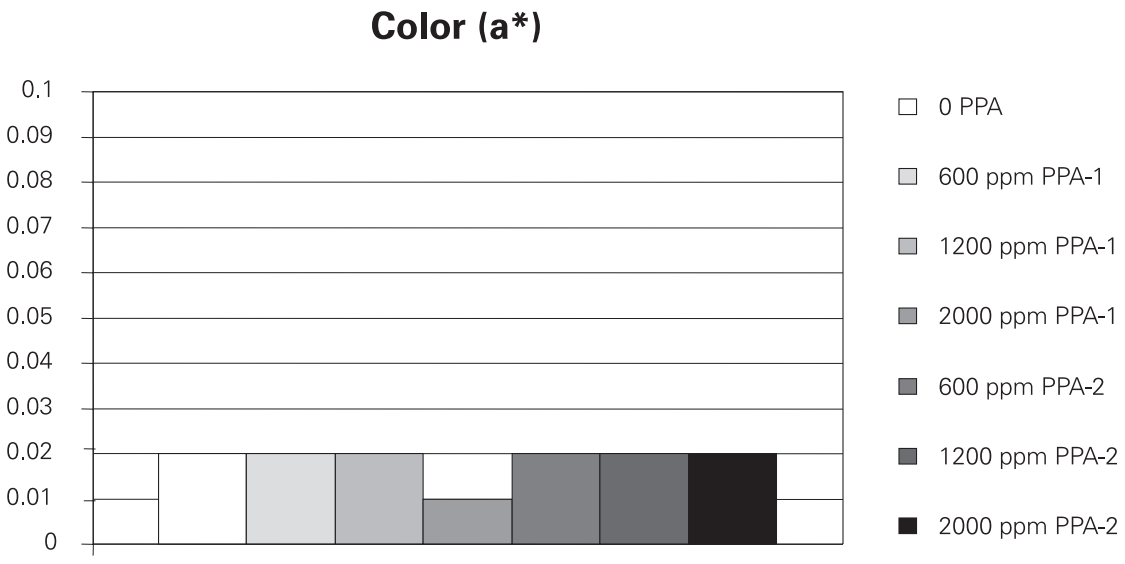


**Figure 15**

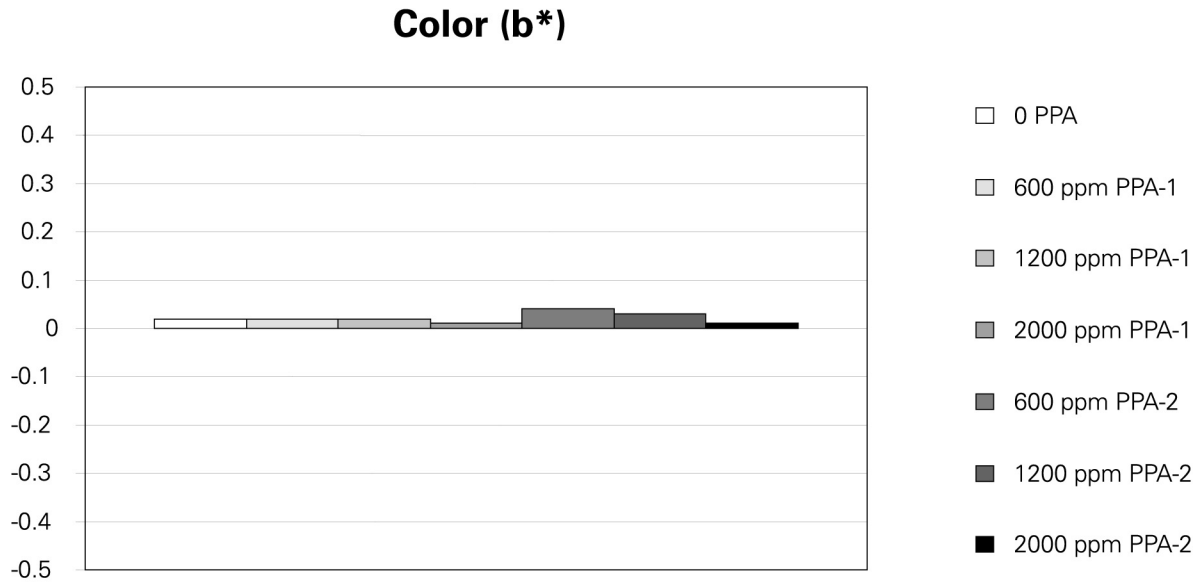



**Figure 16**

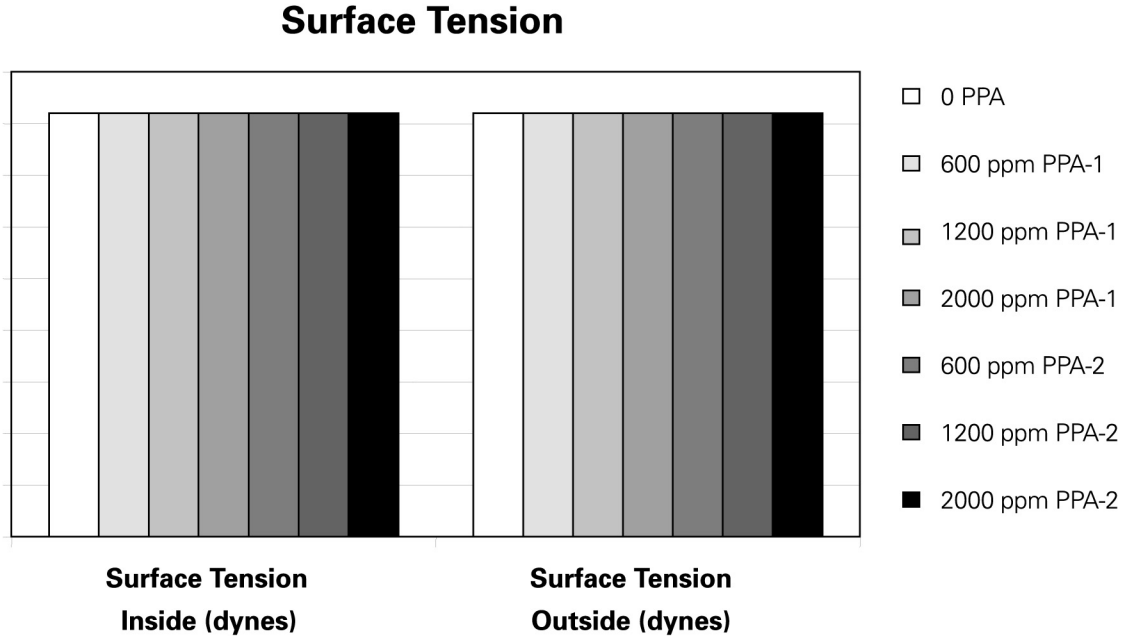


**Figure 17a**
**ESCA Survey Spectrum for Plastomer containing slip and anti-block agent.**

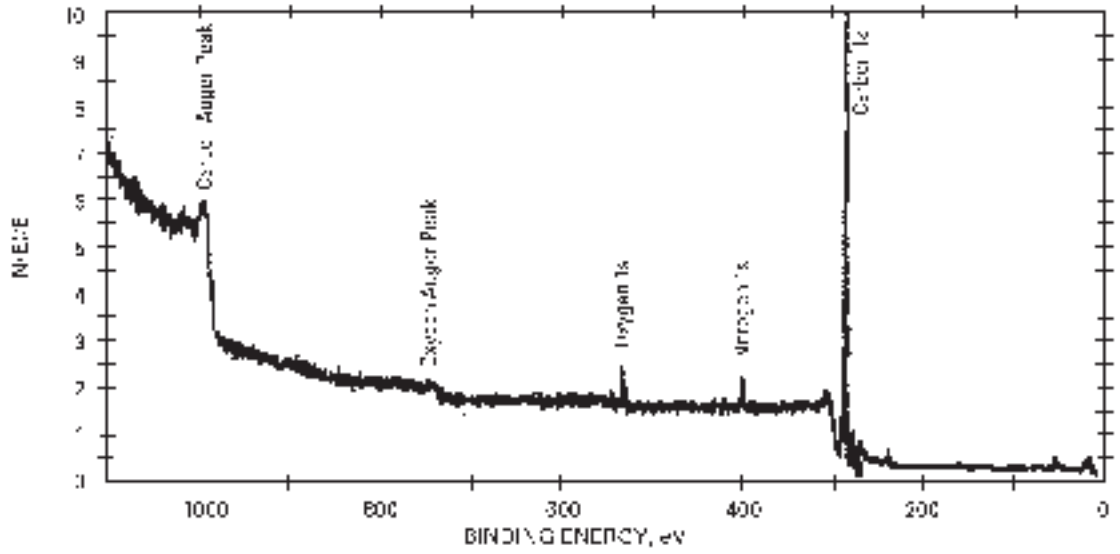



**Figure 17b**
**ESCA Survey Spectrum for Plastomer containing slip, anti-block agent and 2000 ppm FX 5920A. Note the absence of a peak at about 690 dV indicating the absence of fluorine at the sample surface.**

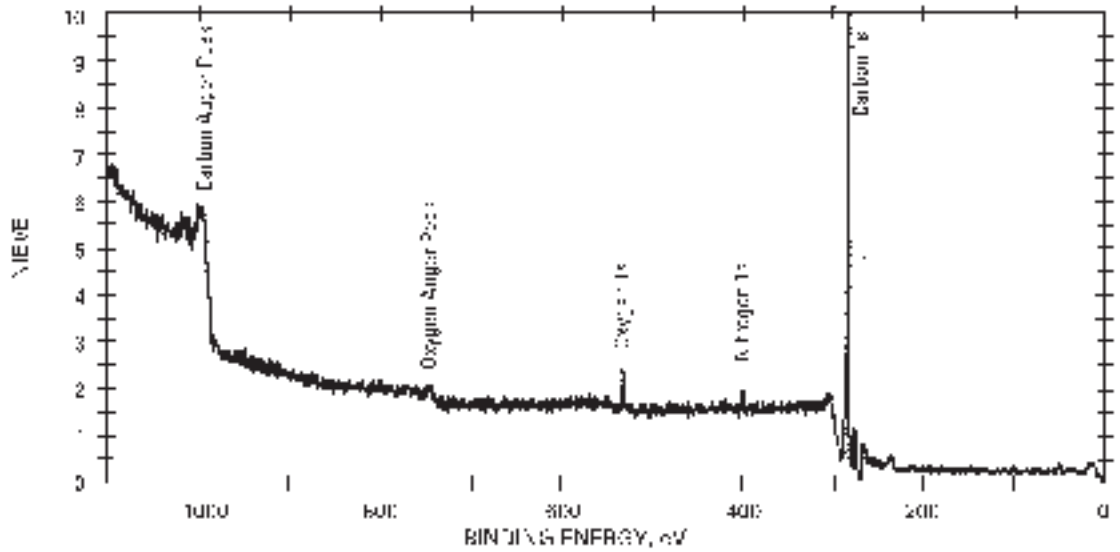


**Figure 18a**
**(-) SSIMS Spectrum for Plastomer containing slip and anti-block agents.**

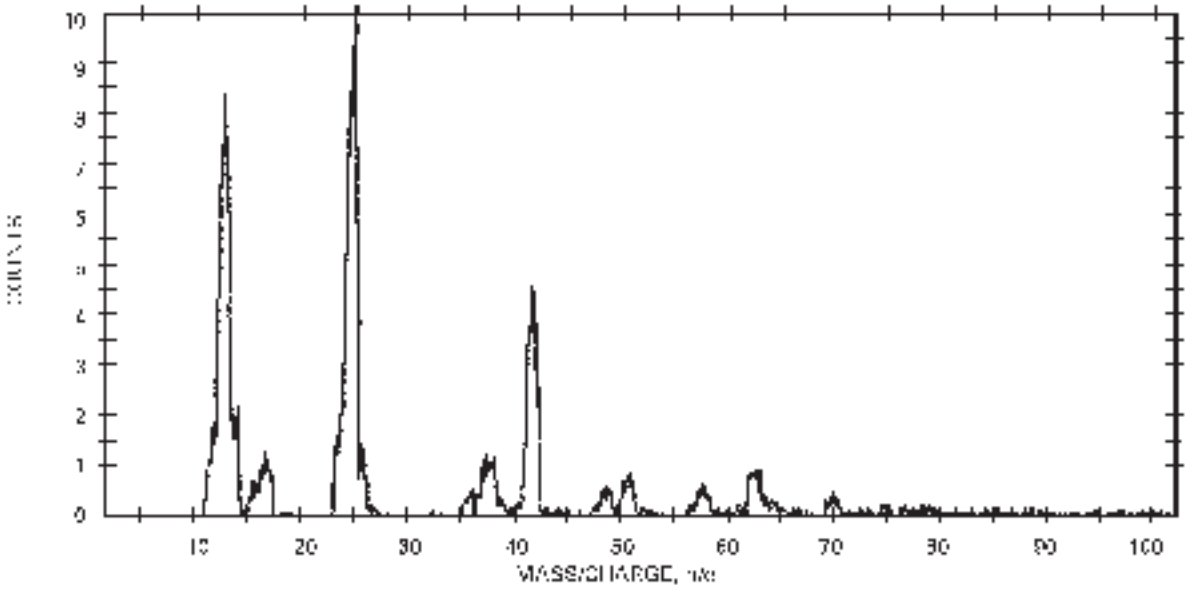
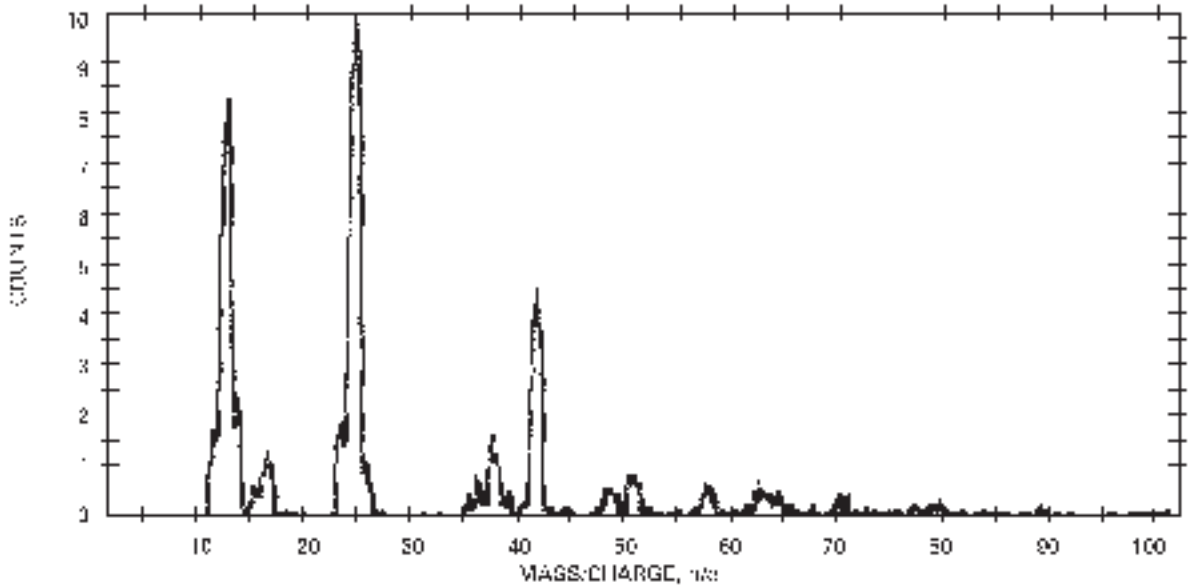
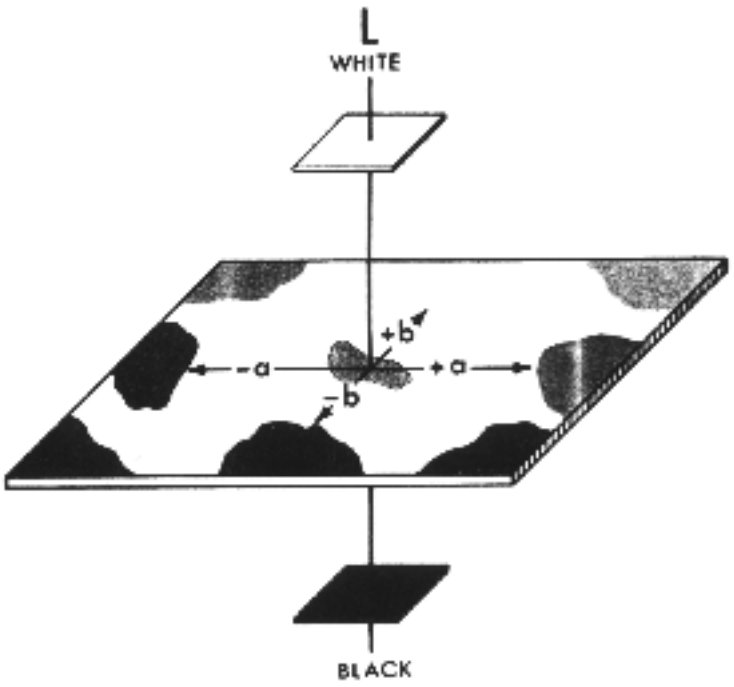



**Figure 18b**
**(-)SSIMS Spectrum for Plastomer containing slip, anti-block agent and 2000 ppm PPA-2. Note that the peak at a mass/charge ratio of 19 (corresponding to fluorine) is the same in Figs 18a and 18b.**



**Appendix 1**

# $L_L$, $a_L$, $b_L$ Color Space



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Issued: 01/00

Dyneon LLC
6744 33rd Street North
Oakdale, MN 55128
+1-651-733-5353
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Printed in USA
98-0504-1008-7


This paper was prepared for presentation at the 1999 Polymers, Laminations & Coatings Conference.

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