ON-LINE COMPOUNDING OF TPO BLENDS FOR LARGE PART THERMOFORMING APPLICATIONS

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ABSTRACT

An effort was initiated to explore the viability of compounding a talc-filled thermoplastic olefin (TPO) formulation directly on a sheet extrusion line. This approach would reduce cost by eliminating a separate compounding step. It was found that it is indeed possible to perform on-line TPO compounding on a sheet extrusion line. A simple single-flighted general purpose screw works as well as a high performance dual-wave screw for on-line compounding when using a talc-concentrate. The use of polypropylene-based talc concentrate is preferred over that of an elastomer-based talc concentrate as the former results in sheet with improved tensile modulus. The sag rate of on-line compounded TPO extruded sheet can also match that of pre-compounded TPO extruded sheet if one uses a polypropylene-based talc concentrate. Based on this work, it has been shown that it should be possible to reduce the manufacturing cost of TPO extruded sheet designed for large part thermoforming applications by eliminating a separate compounding step and blending the formulation directly on a sheet extrusion line through the use of a highly-filled talc concentrate.

INTRODUCTION

TPO formulations typically consist of olefin-based thermoplastics, elastomers, and fillers which are blended in twin-screw extruders, continuous mixers, or batch mixers. The focus of this work was to explore the viability of compounding such a formulation directly on a sheet extrusion line. This would reduce cost by eliminating a separate compounding step. In this study, TPO sheet samples were prepared via on-line compounding on a sheet line equipped with a single screw extruder. Experiments were performed with three different screw designs. To further understand on-line compounding of this formulation, experiments were also performed on a shear refiner to study the influence of applied shear stress on mixing quality. The mechanical properties and thermoforming behavior of the TPO sheet samples prepared in this study were quantified and compared to TPO sheet prepared from the same formulation compounded on continuous mixer.

EXPERIMENTAL

Materials: This on-line compounding investigation was performed using a TPO formulation developed for large part thermoforming applications. This TPO formulation consists of 55% polypropylene (PP), 15% polyolefin elastomer (POE), and 30% talc. The PP used in this formulation is an impact copolymer PP with a 0.5 dg/min melt flow rate (230°C/2.16kg) and the POE used in this formulation is an ethylene-octene copolymer with a 0.868 g/cc density and 0.5 dg/min melt index (190°C/2.16kg).

It would be difficult to compound raw talc on a single-screw extruder sheet line so talc was added as a talc concentrate. Two different talc concentrates were prepared. The first talc concentrate (60% talc) used the PP resin as the carrier resin and the second talc concentrate (67% talc) used the POE resin as the carrier resin. Each of these talc concentrates was prepared on a CP-250 Farrel Continuous Mixer (FCM) using a set point temperature of 210°C.

One of the main questions to be answered in this investigation was to identify which concentrate was best suited for on-line compounding. The main benefit of the first approach is that the talc starts out in the most desirable phase for the TPO formulation – the PP phase. The main benefit of the second approach is that material handling would be easier because one would only need to add two components into the hopper of the single-screw extruder of the extrusion sheet line (talc concentrate #2 + PP) whereas the first approach would require the addition of three components (talc concentrate #1 + PP + POE).

Shear Refiner: It was hypothesized at the beginning of this project that talc would be dispersed well in the talc concentrate and that only distributive mixing of the TPO formulation components would be required at the sheet extrusion line. In the event that this hypothesis was not correct, the level of dispersive mixing required from the single-screw extruder would be identified through the use of a shear refiner (**1,2**). A sketch of this instrument is presented in Figure 1. It is essentially a decoupled Maddock-type mixer (**3**) that is fed by a combination of a single-screw extruder and gear pump. This polymer processing assembly allows one to vary the speed of the mixer while holding the output rate of the system constant. Thus, with a shear refiner it is possible to vary the applied shear stress by simply adjusting the rotor speed of the mixer while maintaining a constant output rate.

A significant amount of shear stress is generated during the melting operation of the single-screw extruder that feeds the Maddock-type mixer of the shear refiner. The shear stress developed by the Maddock-type mixer is an additional shear history that can be used to increase the level of dispersive mixing that the material experiences beyond what is generated in the single-screw extruder. If indeed the level of dispersive mixing developed by the single-screw extruder was not sufficient, the level of dispersive mixing could be adjusted by varying the speed of the Maddock-type mixer of the shear refiner.

The shear refiner was operated with a set point temperature of 210°C and the rotor speed was varied from 20 to 180 rpm in 40 rpm steps. Pellets were collected at each rotor speed and the resulting TPO was characterized. Characterization included rheological testing, mechanical testing (performed on injection molded test specimens), and microscopy. As will be described later, these experiments revealed that the level of dispersion mixing was not altered by rotor speed which indicated that the hypothesis regarding the dispersion of the talc in the talc concentrate was indeed adequate for preparing TPO through on-line compounding on a sheet extrusion line.

Sheet Extrusion: Based on the results of the shear refiner experiments, it seemed clear that dispersive mixing would not be an issue regarding the on-line compounding of the TPO formulation. It was not known, however, what degree of distributive mixing would be required to mix properly the blend. Distributive mixing was investigated by blending the TPO formulation on a sheet line using three different screws: a simple single-flighted screw, a screw containing a single-pass mixing blister ring, and a high performance double wave screw. The sheet line was equipped with a 2.5 inch diameter 30:1 L/D vented single-screw extruder and a gear pump, a coat hanger style sheet die, and a three-roll stack. Each of the screws used for these experiments was a two-stage screw but vacuum was not pulled on the vent.

Extruded sheet was fabricated from three different materials. Two of these were the same blend formulations previously described for the shear refiner experiments. The third was a pre-compounded TPO with the same overall composition. This pre-compounded material was prepared on the same FCM described earlier. Sheet was extruded at two different rates: 45 and 91 kg/hr. The extruder temperature profile was maintained at 210/220/230°C and the sheet die temperature was maintained at 230°C. The first roll was maintained at 140°C while the second and third rolls were maintained at 220°C. Ten sheet samples with dimensions 0.5 cm x 61 cm x 91 cm were fabricated for each material at both extrusion rates. The machine direction mechanical properties of these sheets were measured through Izod and instrumented dart impact as well as flexural and tensile testing. Sheet orientation was also quantified through sheet shrinkage measurements.

Thermoforming: The main thermoforming characteristic quantified in these experiments was sheet sag performance. Sheet samples were thermoformed on a ZMD International Model V223 shuttle thermoformer. Each sheet was placed in the clamp frame of the thermoformer, and rigidly clamped on all four sides. Next, the clamped sheet was indexed into the heat station of the thermoformer, where the sheet was heated by absorption of infrared radiation supplied by quartz infrared radiant heaters. As the temperature of the sheet increased, the initially flat sheet began to sag under its own weight.

The amount of sag was limited by the equipment configuration and ultimately on the final part quality. The vertical distance of the sheet sag from the initial position in the clamp frame was measured using an infrared profiling scanner (light curtain) that was positioned to detect sheet sag at the middle of the oven. The time required for the sheet to sag from 57 mm below its initial position to 108 mm from its initial position was recorded. Sag rate was determined by dividing the change in the vertical distance of the sheet by the time required for the change in height. The sheet was removed from the oven when the sag reached approximately 108 mm from its initial position and moved to the form station.

The sheet surface temperature on the bottom side of the sheet was measured at the end of the heat cycle using an infrared pyrometer. Once the heated sheet was positioned in the form station, a vacuum box contacted the sheet from below. Vacuum was applied to draw the sheet into the vacuum box and pre-stretch the sheet. A machined aluminum mold was lowered into the top of the pre-stretched sheet and vacuum applied to draw the extended sheet against the mold while the vacuum was simultaneously released from the vacuum box. Details of the mold have been published previously (**4**). The part was formed and allowed to cool and was ultimately removed from the clamp frame.

RESULTS AND DISCUSSION

Shear Refiner: The shear refiner experiments revealed that varying the amount of applied shear stress experienced by the blend in the melt state did not significantly alter properties of the TPO blend. The viscosity curves of TPO prepared from the PP/talc concentrate formulation on the shear refiner at high and low rotor speeds were nearly identical (Figure 2). The curves overlay perfectly at high shear rates and only a slight difference was observed at low shear rates. This slight difference was likely due to polymer degradation when subjected to a high rotor speed in the shear refiner – an effect which would be more pronounced at low shear rate than high shear rate.

The shear refiner experiments also showed that the choice of talc concentrate type influenced the rheological properties of the TPO formulation (Figure 3). The melt flow rates (MFR) of the TPO formulations prepared from the PP/talc concentrate were higher than those prepared from the POE/talc concentrate across the full range of rotor speeds explored. It is believed that this difference was due to the PP carrier resin experiencing a greater degree of degradation during the FCM compounding of this concentrate than that experienced by the POE carrier resin during the FCM compounding of the POE/talc concentrate. For each given TPO recipe, no clear trend in MFR with rotor speed was observed.

Similarly, no clear trends in mechanical properties with rotor speed were observed for each of the two TPO recipes. However, the flex modulus (Figure 4) of TPO samples prepared from PP/talc concentrates was higher than those prepared from POE/talc concentrates – despite the speculation that the PP/talc concentrate experienced more degradation during its preparation than the POE/talc concentrate. It is believed that this difference in flex modulus was due to the location of talc in the resulting TPO samples. In order to maximize stiffness, one would want all of the talc to be dispersed in the PP phase of the TPO with no talc located in the POE phase. The contribution that talc particles would make to the stiffening of the TPO formulation would be significantly diminished if the talc was buried within a soft elastomer phase. For TPO samples prepared from the POE/talc concentrate, all of the talc was located within the elastomer at the beginning of the blending process so it stands to reason that the TPO blends prepared via this route would tend to have more talc embedded in elastomer phases than one would achieve using a PP/talc concentrate.

A similar explanation is used to explain the tensile yield data (Figure 5) collected from the shear refiner TPO samples. Again, no trend with rotor speed was observed but the TPO samples prepared from the POE/talc concentrate exhibit a larger tensile yield than those prepared from the PP/talc concentrate. It is also believed that this difference was caused by a difference in talc distribution across the PP and POE phases of the TPO. Samples prepared using the PP/talc concentrate likely contained a high concentration of talc particles (i.e. defects in the stiff phase) which caused yielding to occur at a lower applied tensile stress than that experienced by samples prepared using the POE/talc concentrate.

No clear trends were observed in the Izod impact data (Figure 6) collected from the shear refiner TPO samples: neither with respect to rotor speed nor talc concentrate type. This behavior could be due to two different competing effects counterbalancing each other. TPO samples based on the PP/talc concentrate yielded at a lower stress (due to an increased population of talc defects in the PP phase) but the elastomer phase particles are better impact modifiers (due to a decreased population of talc defects in the elastomer phase) whereas just the opposite would be the case with TPO samples based on the POE/talc concentrate.

Close inspection of the transmission electron microscopy (TEM) images prepared from the TPO shear refiner samples indicate that the talc particles were more likely to be imbedded in the elastomer phase for the case of samples prepared using the POE/talc concentrate (Figure 7). It was also observed (Figure 8) that rotor speed did not significantly alter TPO morphology. Such a result was no surprise considering that there were no trends observed between rotor speed and mechanical performance for the TPO samples prepared on the shear refiner.

Sheet Extrusion: Unlike what was found regarding Izod impact of injection molded samples prepared from the shear refiner TPO samples, the type of talc concentrate used to prepare extruded sheet TPO samples did exhibit a slight effect on instrumented dart impact performance (Figure 9). Sheet samples prepared using the POE/talc concentrate exhibited a slightly lower total energy to break than those prepared using PP/talc concentrate and those prepared from pre-compounded TPO. The insensitivity of impact performance to talc concentrate type exhibited by the shear refiner samples could be a result of the extra mixing history associated with the injection molding of the shear refiner test specimens. The instrumented dart impact test specimens prepared from the extruded sheets did not undergo any addition mixing – test specimens were simply cut from the extruded sheets. It should also be noted that no trend in instrumented dart impact performance was observed with respect to screw design or extrusion rate.

A significant influence of talc concentrate type on TPO sheet modulus was observed (Figure 10). Similar to what was seen for the injection molded shear refiner TPO samples, a lower modulus was exhibited by the TPO sheet prepared using the POE/talc concentrate whereas that prepared using the PP/talc concentrate was similar to that prepared from pre-compounded TPO. The root cause of this behavior is again believed to be a result of the relative distribution of talc in the PP and elastomer phases of the TPO samples. As was the case with instrumented dart impact, no influence of screw design or extrusion rate on the modulus of TPO extruded sheet was observed.

An influence of extrusion rate on ultimate tensile strength was observed (Figure 11). TPO sheet fabricated using a fast extrusion rate exhibited lower tensile strength than that fabricated using a slow extrusion rate. It is believed that this behavior was due to a difference in orientation developed in the sheet as a function of extrusion rate. It was observed (based on shrinkage measurements) that sheet extruded at a high extrusion rate possessed a lower amount of orientation (17.6% shrinkage) than sheet fabricated at a slow extrusion rate (22.3% shrinkage). All things being equal, a faster extrusion rate typically causes an increase in sheet orientation. The opposite effect exhibited by these sheets is thought to be due to a difference is extrudate temperature caused by viscous heating of the extrudate. It should also be noted that the tensile strength of the extruded TPO sheet prepared using the POE/talc concentrate was slightly lower than that of sheet prepared using the PP/talc concentrate and sheet prepared from pre-compounded TPO. No influence of screw design on tensile strength was observed.

An influence of talc concentrate type on yield behavior of extruded TPO sheet was observed. Elongation at yield (Figure 12) of TPO sheet prepared using POE/talc concentrate was significantly greater than that of TPO sheet prepared using PP/talc concentrate and sheet prepared from pre-compounded TPO but yield strength showed the opposite trend (note: for these materials the yield strength was the ultimate tensile strength). Both sets of behaviors are believed to be due to differences in talc distribution across the TPO polypropylene and elastomer phases. It should be noted that the yield strength behavior dependence on talc concentrate type was the opposite of what was observed from the injection molded shear refiner TPO samples. It is believed that this difference may be influenced by sheet orientation as it can also been seen that yield strength of extruded TPO sheet reduced with increased extrusion rate (Figure 11). No influence of screw design yield behavior was observed.

Thermoforming: Since no influence of screw design on mechanical performance was observed, it was decided to only perform a thermoforming analysis of TPO sheet extruded with the simple single-flighted screw design. The results of the sag behavior of the TPO sheets in the thermoforming oven are presented in Figure 13. Sheet sag rate is observed to be influenced by the extrusion rate used during the fabrication of the sheet. For pre-compounded TPO sheet, sag rate of the sheet fabricated at a fast rate is faster than that fabricated at a slow rate. The opposite trend is observed for the on-line compounded TPO sheets. For a given extrusion rate, the on-line compounded TPO sheet prepared from the POE/talc concentrate sags at a faster rate than that prepared from the PP/talc concentrate. This difference is believed to be due to differences in talc localization between these two different types of TPO sheet. Sheet surface temperatures were monitored during the sag measurements and it was observed that sheet temperature was consistent for all the samples.

For large part thermoforming applications, a slow sheet sag rate is desirable as a means to maintain uniform thickness across the formed part. Sheet sag of on-line compounded TPO sheet prepared from the PP/talc concentrate nearly matches that of the pre-compounded TPO sheet for the case of a fast sheet extrusion rate. Likewise, these TPO sheets exhibited less thinning in the corners during the forming operation as compared to the on-line compounded TPO sheet prepared with the POE/talc concentrate which is also as a desirable characteristic.

CONCLUSIONS

It was possible to match the mechanical performance of pre-compounded TPO formulations through the use of on-line compounding. A simple single-flighted general purpose screw works as well as a high performance dual wave screw for on-line compounding. The use of a PP-based talc concentrate is preferred over that of a POE-based talc concentrate as the former resulted in sheet with improved tensile modulus. The sag rate of on-line compounded TPO extruded sheet can also match that of pre-compounded TPO extruded sheet when a PP/talc concentrate is used. Based on this work, it has been shown that it should be possible to reduce the manufacturing cost of TPO extruded sheet designed for large part thermoforming applications by eliminating a separate compounding step and compounding the formulation directly on a sheet extrusion line through the use of a highly filled talc concentrate.

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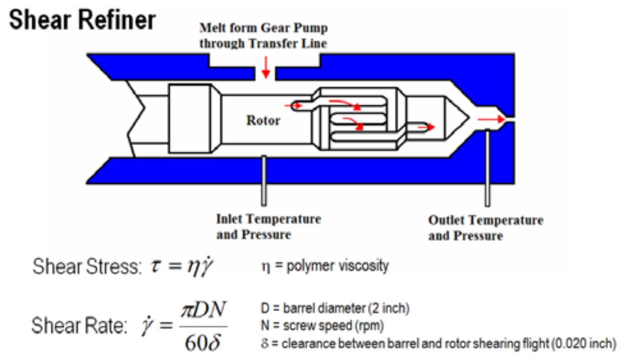

Figure 1: Sketch of Shear Refiner.

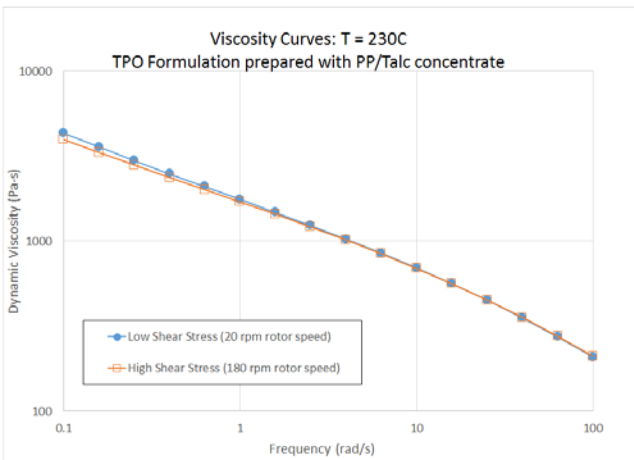

Figure 2: Shear Refiner Mixed TPO: Viscosity Curves.

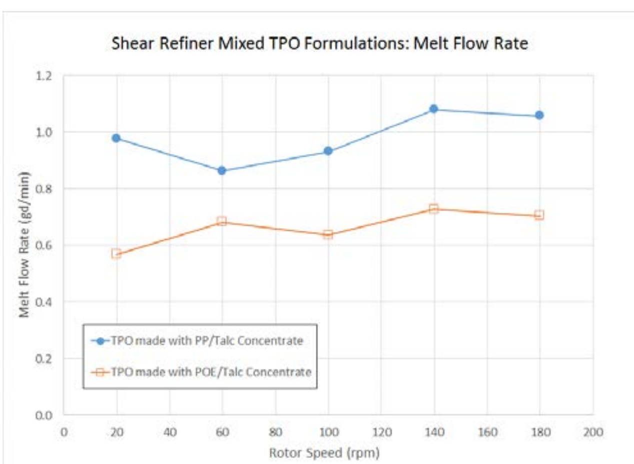

Figure 3: Shear Refiner Mixed TPO: Melt Flow Rate.

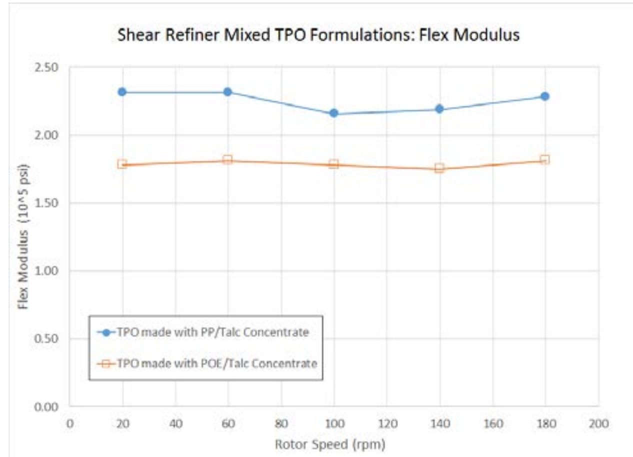

Figure 4: Shear Refiner Mixed TPO: Flex Modulus.

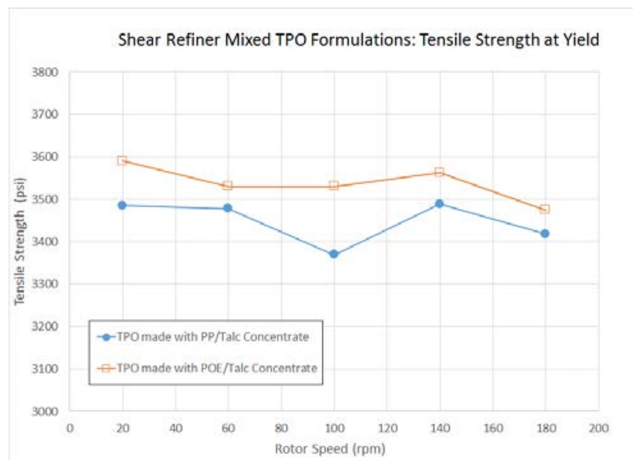

Figure 5: Shear Refiner Mixed TPO: Tensile Strength.

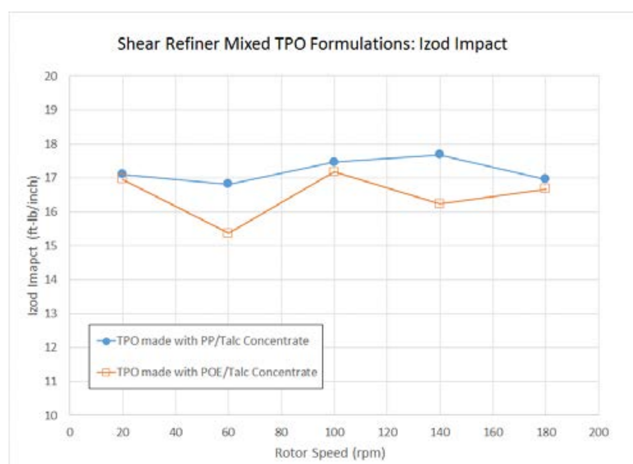

Figure 6: Shear Refiner Mixed TPO: Izod Impact.

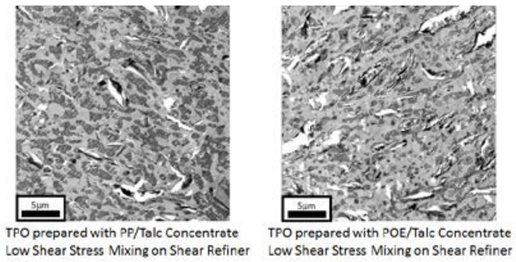

Figure 7: TEM of Shear Refiner Mixed TPO: comparison of two talc concentrates.

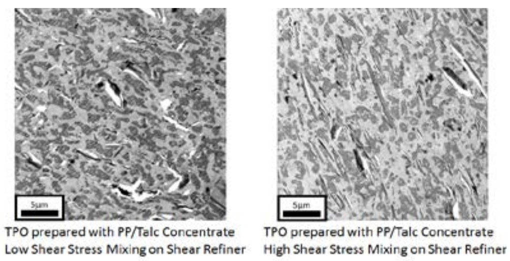

Figure 8: TEM of Shear Refiner Mixed TPO: influence of applied shear stress.

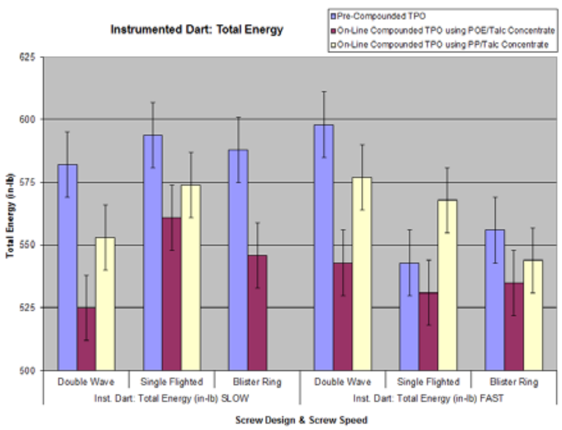

Figure 9: TPO Sheet – Instrumented Dart Impact.

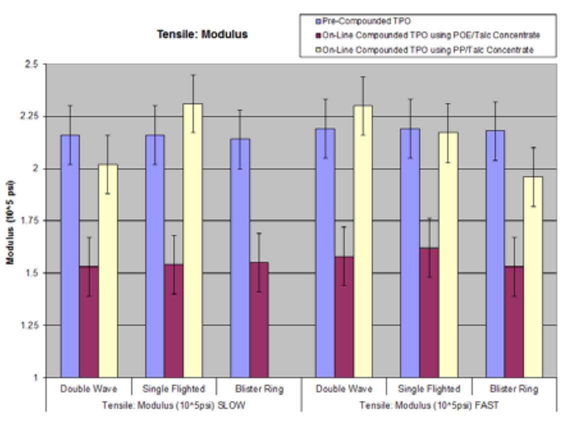

Figure 10: TPO Sheet – Tensile Modulus

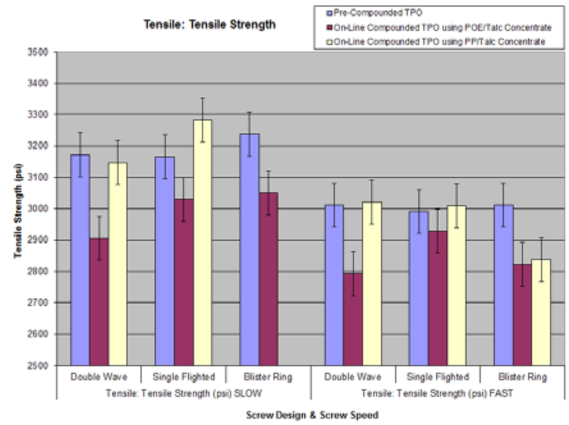

Figure 11: TPO Sheet – Tensile Strength.

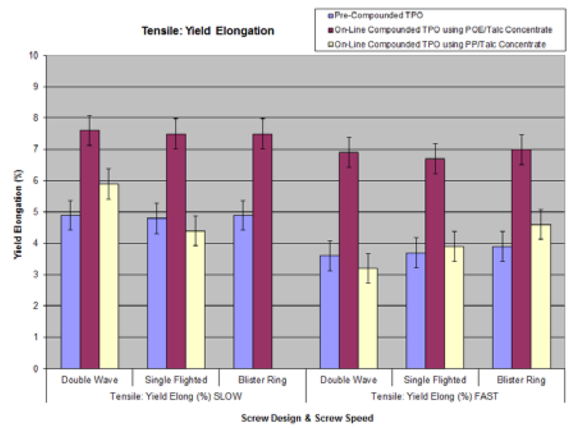

Figure 12: TPO Sheet – Tensile Elongation at Yield.

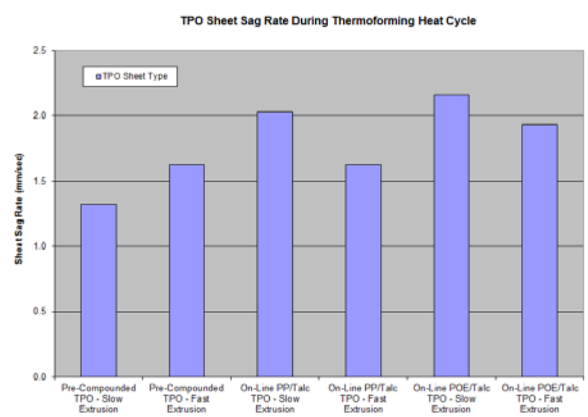

Figure 14: TPO Sheet Sag Rate