Xplore's MC 40 micro-compounder in continuous compounding mode:
Part-1: Performance of the new screw design in blending immiscible polymers

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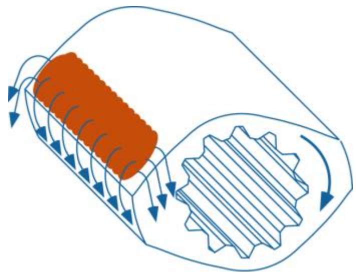
Introduction

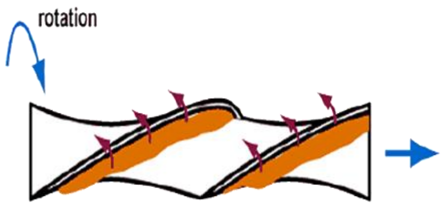
The MC 15 HT, Xplore's flagship micro-compounder with 15 ml capacity, offers many advantages, such as high shear, good dispersion, and operational stability. Over time, Xplore was inspired by customers' needs to process larger amounts of material; as a result, our engineers developed an MC 40, which can process a higher compound volume of 40 mL. This sample amount can then enable the usage in subsequent high-volume applications, experimental or analysis steps.

An MC 40 has a longer overall screw length; therefore, as well as batch processing, it can also potentially enable continuous processing. Proper mixing along the screw's longitudinal axis must be ensured to unlock this potential. In this first demonstration, the effect of the screw geometry on the MC 40 continuous mixing performance has been studied. For this purpose, a new screw geometry was designed. The primary revision incorporates mixing elements to promote dispersion further (Figure 1a).

Dispersive mixing involves particle size reduction of cohesive components, which requires considerably high flow stresses during compounding under both shear and elongational deformation conditions. This way, solid fillers or viscous ingredients can be de-agglomerated, or even immiscible viscous liquid droplets can be broken up. In the existing screw design, shear deformation is dominant, and maximum shear conditions occur over the screw flank due to the small gap. Besides, some portion of the melt can also flow backwards over the screw flights, resulting in elongational mixing. However, this only applies to a minor part of the melt (Figure 1b). In the new screw geometry, to enhance the dispersive mixing further, new mixing elements were incorporated into both mixing screws to promote elongational deformation, which leads to improved dispersive mixing of the melt.



(a)



(b)

Figure 1. (a) Kneading block [1]
(b) Elongational mixing along the screw

In this work, we compared the mixing efficiency of the conventional (regular) screw of the MC40 with the new design (dispersive) under continuous compounding conditions (Fig. 2). We mainly focused on two critical indicators of the mixing, residence time and mixing quality (particle size reduction and homogeneity). One notes that higher mixing quality means that the particles can be dispersed effectively with the correct shear level, which leads to a higher surface area at the interface.
Sufficient residence time is crucial in the compounding process to achieve a high mixing

quality (also without unmelts),i.e., good dispersion and distribution. The second primary revision made in the new screw design is the shortening of the pitch of the screw. In this way, a longer residence time can be achieved compared to the regular screw design of the MC40 compounder.

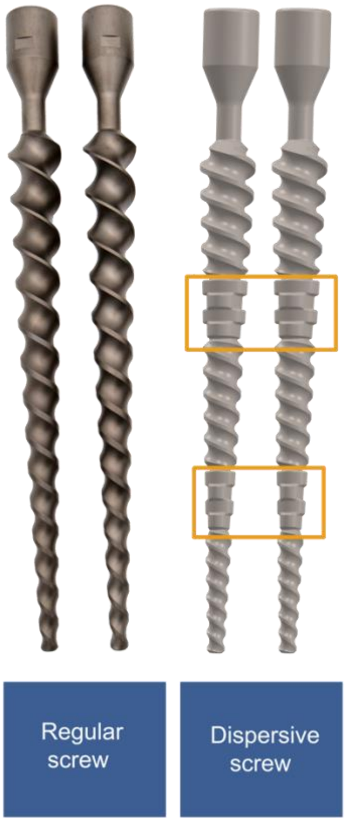


Figure 2. Regular and dispersive (new) screws

In the first part of this study, the residence time of 2 screw designs was compared by processing a colored LDPE incontinuous mode (details are given in the Materials and Testing Section). In the second part, the mixing quality of the two screws was compared. A compatibilized PP/PA6 blend system was chosen as a model system. Due to the difference in the polarities of these two semi-crystalline polymers, they are highly thermodynamically immiscible. Therefore, the mixing quality depends on not only the shear conditions but also the extent of compatibilization reaction where residence time plays an important role.

To achieve PA6/PP compatibility, maleicanhydride grafted PP (PP-g-MA) was used as a compatibilizer (emulsifier). PP-g-MA preferentially resides at the interface of both polymers and improves interfacial adhesion through the chemical linkage of the anhydride groups with the polyamide end groups (Figure 3) and, at the same time, through sustaining the thermodynamical compatibility with the PP phase. This reduces the interfacial surface tension between two blend components and leads to a better and more stable blend morphology.

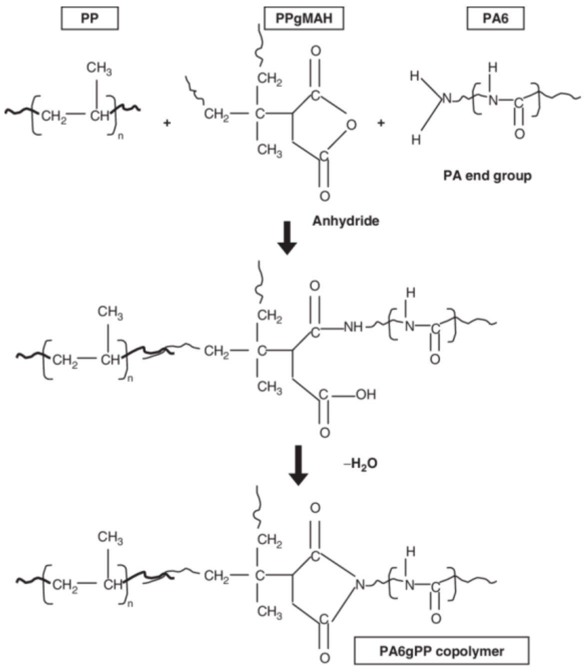


Figure 3. Reaction between PA6, PP, and PP-g-MAH i.e., PA6-graft-PP formation [2]

Materials and Methods

Some important experimental parameters are summarized in Table 1. In the first part, LDPE/LDPE-masterbatch system was used to compare the residence time of regular and dispersive screws in continuous mode. LDPE (MFI 10 g/10min) was fed to MC40 with an Xplore volumetric continuous feeder at a 10 g/min feed rate. The screw speed was set as either 50 or 100 rpm. The barrel temperature was 200°C. The recirculation valve was kept open to allow continuous compounding, which means the polymer was allowed to pass one time over the twin screws, and the extrudate was collected. Once the steady state was obtained, 0.07 gram of colored masterbatch was fed to the micro-compounder from the top at once, and t_i and t_f values were recorded (Figure 4):

- t_i: time at which the color appears
- t_f: time at which the color disappears

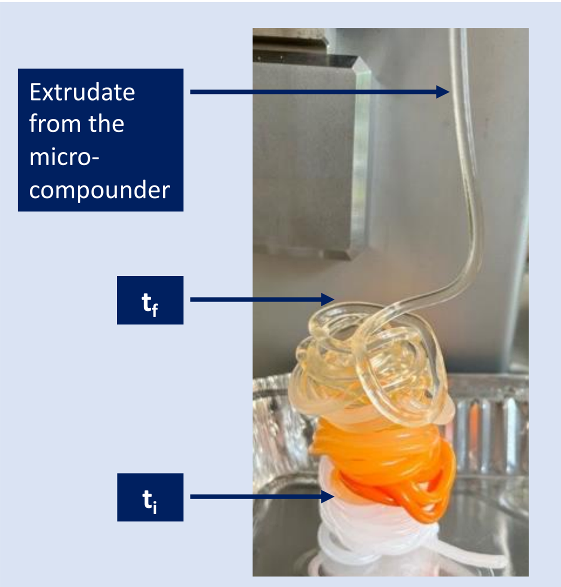


Figure 4. Residence time measurement using a color masterbatch

In the second part, PA6/PP/PP-g-MA blends were prepared under continuous compounding conditions to see the effect of screw geometry on the blend morphology. Before processing, PA6 was dried at 80° C for 16 hours in a vacuum oven to remove the moisture.

PA6/PP/PP-g-MA blends were melt compounded in Xplore MC40 micro-compounder in continuous mode at 100 rpm screw speed and 230°C barrel temperature having 30%/70%/2% weight ratio, respectively, by using an Xplore continuous double feeder. The continuous flow was interrupted with an IM12 Xplore Injection molding machine to obtain tensile test specimens. The injection and holding air pressure was 8 bar, and the cycle time was 15 sec. To compare the mixing quality of the continuous new screw design with batch processing, samples were also prepared with regular screws under batch process conditions. In this route, the valve was kept closed, and the blend was sheared for 5 minutes and then transferred to IM12.

The mechanical performance of the blends was investigated by tensile test. Additionally, a scanning electron microscope (SEM) accomplished the examination of the blends' morphology.

Table 1. Parameters of the study

Details of the Study	Screw Type	Screw Speed (rpm)	Compounding Type
1st part: Residence Time **System:** PE/masterbatch	Regular	50	Continuous
	Regular	100	Continuous
	Dispersive	50	Continuous
	Dispersive	100	Continuous
2nd part: Mixing quality **System:** PP/PA6/PP-g-MAH	Regular	100	Continuous
	Regular	100	Batch (5 minutes recirculation)
	Dispersive	100	Continuous

Results and Discussion

The results of the residence time study are summarized in Table 2. Both t_i and t_f values are higher for dispersive screws than regular screws, indicating longer residence time for a given screw speed.

One can expect that the longer residence time would lead to better dispersion. For both screws, increasing the screw speed (and eventually increasing the shear rate) decreased both t_i and t_f values, meaning that a shorter residence time is achieved with a higher screw speed.

Table 2. Results of residence time study (1st part)

Screw Type	Screw Speed (rpm)	t_i (min)	t_f (min)
Regular	50	0:35	5:30
	100	0:30	5:00
Dispersive	50	1:20	6:15
	100	1:05	5:50

In the second part, we compared the mixing quality of regular and dispersive screws. Figure 5 shows the SEM images of the PA6/PP/PP-g-MA blends. PP is the matrix, and PA6 is the dispersed phase, which is obviously seen as a particle. The average dispersed particle size of PA6 was decreased from 2.05 microns to 0.45 microns when the dispersive screws were used instead of regular screws. The dispersive screws provided better uniformity and homogeneity of PA phase dispersion in the PP matrix with a reduction in the size of the domains thanks to better mixing ability and longer residence time. Since the blending of the PP/PA6 system in the presence of PP-g-MA is a reactive process, the longer residence time of the new screws compared to the regular ones could promote the grafting reaction of maleic anhydride to the chain ends of PA6 by allowing the polymer chains to find time for the reaction.

Using the regular mixing screws under batch process conditions is usually expected to promote the dispersion of the blend components in the most effective way. However, the particle size was observed as 1.12 microns, almost more than double the value (0.45 microns) achieved with dispersive screws. However, it must be remembered that the compounding conditions, either in continuous or batch mode, are not optimized for PA6/PP blends.

Tensile test results given in Figure 6 show that the elongation of the blends is in the order: Dispersive continuous mode > Regular batch mode > Regular continuous mode

This result correlates well with the average particle size measurements in SEM images. Dispersive screw in continuous mode yields the smallest particle size and the highest elongation. In other words, smaller particle size prevents premature failure. Basically, as aligned with the interface phenomena, it can be concluded that the smaller the particle size, the higher the elongation due to the larger interfacial surface area.

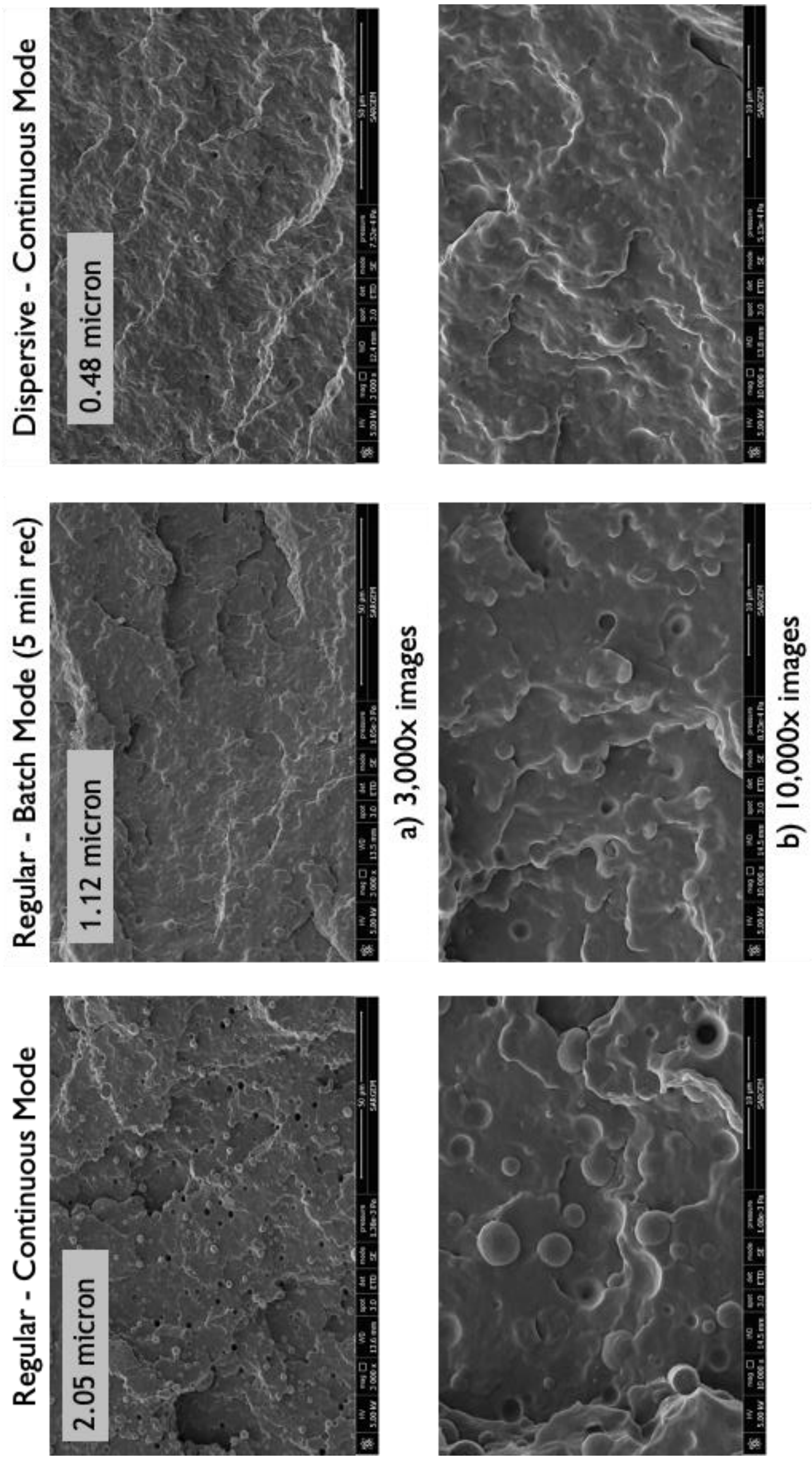


Figure 5. SEM images of the PA6/PP/PP-g-MA blends a) 3,000x magnification and b) 10,000x magnification

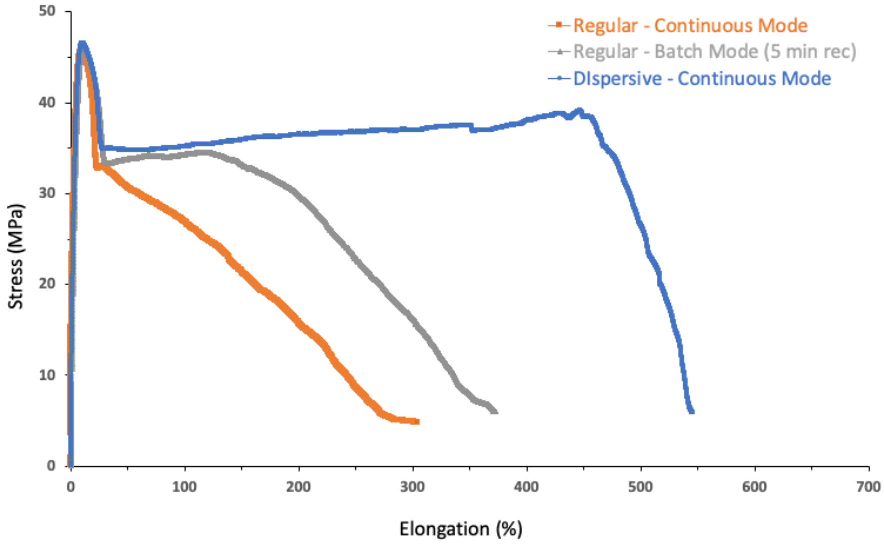


Figure 6. Tensile test results

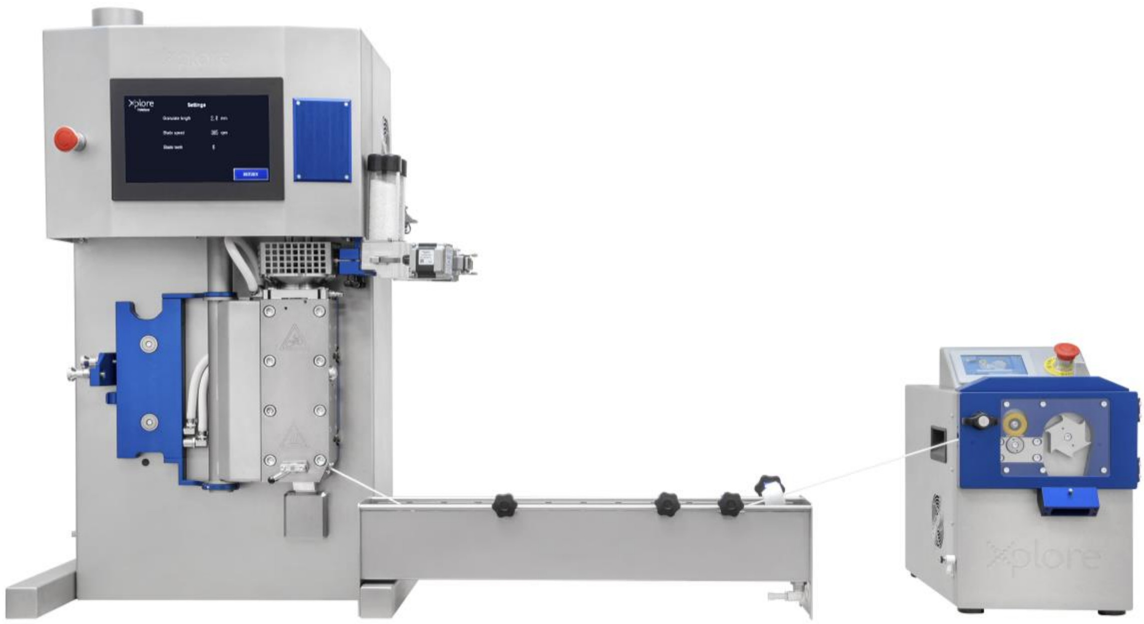


Figure 7. Xplore MC40 micro-compounder demonstrated in continuous compounding mode combination with Xplore water bath and Pro-pelletizer

Conclusion

There are two critical parameters in the continuous compounding of polymers to achieve good dispersion and distribution. One is having a proper shear and elongational deformation during the compounding. The other one is the enough residence time under those deformations. A good screw design for a given compounding system can sustain these two essential factors. In this work, we demonstrated that besides well-established batch compounding in Xplore MC40, with the new screw design, it is capable of being operated in continuous mode as well for preparing small batches (app. 250 g – 3,0 kg per hour, depending on the bulk density of the polymer granules, flakes and powder) of polymer compounds. In addition, in combination with the Xplore water bath and pro-pelletizer, it is now possible to make a continuous pelletizing process using an MC 40, as shown in Figure 7.

For further information, contact our experts at Xplore Instruments BV.

References

1 - Martin, C. Twin Screw Extruders as Continuous Mixers for Thermal Processing: a Technical and Historical Perspective. AAPS PharmSciTech 17, 3–19 (2016).

2 - Wahit M.U., Hassan A., Rahmat A.R., Lim J.W. and Mohd Ishak Z.A., Effect of Organoclay and Ethylene-Octene Copolymer Inclusion on the Morphology and Mechanical Properties of Polyamide/Polypropylene Blends, Journal of Reinforced Plastics and Composites, 25, 933 (2006).