A NOVEL MICRO-BATCH MIXER
THAT SCALES TO A SINGLE SCREW COMPOUNDER

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Abstract

At Antec 2008, a simple, spiral fluted extensional mixer (SFEM) was introduced for the single screw extruder (SSE) [1]. The essence of the SFEM was arranged into the compounding element of a micro-batch mixer. The objective of this work was to compare the mixedness of the SFEM SSE to the batch mixer using several material systems at the macro and microscopic level. The micro batch mixer yielded similar mixedness of particle-polymer and polymer-polymer systems, as when processed using the SSE with SFEM elements even at the 2 micron scale.

Introduction

Micro-batch mixers are useful when only very small quantities of material are available, either because the components are rare or expensive. SSEs are a very common processing tool, and certain SSEs are effective compounders [1, 2]. A micro-batch mixer that enables scale up to a SSE is potentially extremely valuable.

Several micro-batch mixers are known and have been compared in the literature [3]. Common micro-batch mixers include a miniature conical twin screw with a recirculation loop, a cup and rotor mixer, an internal batch mixer with roller blades [4] and miniature dual pistons driving material back and forth [5]. But, the geometry of such micro-batch mixers do not resemble the mixing element geometry of any SSE. This makes commercial scale up from laboratory trials extremely challenging.

The closest production analogue among these is the conical twin. The conical twin is popular for processing and compacting a mixture of dry blend PVC and wood flour but is not otherwise known for its compounding capability. Conical twin screw extruders are naturally compressive and are known to cause agglomeration of nano-particles when used for particulate mixing. One study shows a conical twin severely agglomerated 60 nm calcium carbonate particles in a PVC mixture [6]. However, conical twins can be useful when mixing a filler that has a fibrous, spring-like nature such as wood.

Except for the conical twin, the other micro-batch mixers do not extrude a strand. The molten material must be removed from the mixing chamber and element(s) by using a spatula to scrape off and collect it. The material is exposed to air and oxidation during removal, after which it must be ground prior to molding. However, the industry standard for feedstock is pellet form since a regular geometry melts more efficiently than ground material.

SSEs capable of processing by use of repeated extensional flow fields provide excellent mixing of particulate-polymer and polymer-polymer systems. A new mixing element for SSEs, the Elongator, (hereafter SFEM) produces extensional flow fields and has a forwarding pitch [7]. The SFEM excels in a variety of applications such as venting water from 25% undried wood flour/LDPE [8]; venting undried PMMA [8] processing thermally sensitive materials such as reclaim containing EVOH and, most surprisingly, processing RPVC dry blend at 180 rpm and very high output without degradation or use of a crammer feeder or vacuum hopper [9]. These positive results catalyzed the concept of designing a micro-batch mixer with the SSE SFEM geometry incorporated as the mixing element in the micro-batch mixer with the capability to extrude in order to vacate the molten material.

In this work, four extrusion macroscopic experiments were performed on mixtures that broadly describe different areas of extrusion compounding capability utilizing the SFEM mixing element. These same mixtures were then processed using the novel micro-batch mixer, incorporating similar geometry. A comparison of the resulting mixedness produced by both processing methods is presented.

Additionally, an immiscible polymer blend (IMPB) of polystyrene (PS) and high-density polyethylene (HDPE) in two composition ratios, 20/80 and 30/70 % PS/HDPE, were processed using a 24:1 SSE compounder with two SFEM elements and the novel micro-batch mixer with a similar SFEM geometry. The resultant morphologies produced from the two different processing methods, micro-batch mixer versus SSE SFEM, are compared. The results allow an initial examination into how well the micro-batch mixer scales up to the SSE compounder.

Mixer Descriptions

Superficially, the well known Egan mixer (Fig. 1) or spiral UC mixer, resembles the SFEM (Fig. 2) as both incorporate flutes and spirals. However, the entry flute of the Egan mixer dead ends while the entry flute of the SFEM is open. Thus in the Egan, high pressure is required

to force material over the resistance zone (Fig. 1) while the SFEM pressure can be close to zero (a barrel vent may even be employed). Also, the Egan mixer has an inlet channel, C1, and an outlet channel, C2. The SFEM has channels C1, C2 and C3 and a second pump, separating C2 and C3. Egan mixers are far downstream while SFEM elements start within a few L/D of the feed.

It is well known that elongational forces are more effective for dispersive mixing than shear mixing [10] and it is important to understand how the SFEM element used in the SSE and the micro-batch mixer generates elongational flow. For the SSE, the first SFEM is placed within a few L/D of the water cooled feed section of the barrel. As shown in Fig. 2, the flow is split into two channels and each channel feeds an SFEM composed of three channels (C1, C2, C3) and two intermediate pumps (P1 and P2). Material is pushed into C1 by upstream flights. By means of drag flow, P1 pumps material from C1. The combination of pressure flow up the channel and drag flow perpendicular to C1 flow, produces an elongating flow in the approach to P1. This can act to melt, drain melt or mix depending on the state of the material in C1. The material between P1 and barrel experiences shear. The velocity of the material at the P1 surface is near zero and the velocity of the material adhering to the barrel is close to the barrel velocity. If the material is sufficiently soft or melted, it will adhere to both screw and barrel and elongate over C2 (Fig. 3). This is the critical region of high elongation. The material conveys into C3 and is pumped from the SFEM onto a conveying flight and downstream.

The micro-batch mixer geometry, shown in Figure 4, is very similar to the SSE SFEM. It too is composed of a first group, of channels (C1, C2, C3) and pumps (P1, P2) that operate as described above. The first group is contained on one side of a shaft pitched forward to feed and melt material. Exiting this group from C3, material enters a second group with the opposite pitch on the other side of the shaft. Material flows downstream on one side and upstream on the other, as many times as desired. Figure 5 shows a flat view of the rotor.

The micro-batch mixer in this study has a 25 mm diameter rotor 4 L/D long, C1 sufficiently large for pelletized feedstock and a P1, P2 clearance of 1 mm as in the SFEM SSE. A feed port allows material entry through the barrel (Fig. 4) and is equipped with a ram within the port either for encouragement of the input into the channel or to seal the mixture for mixing once entered. There is a die hole under the feed port that is sealed off with a door during mixing.

The micro-batch mixer is easy to operate. It is heated to the desired process temperature. With the door closed, material enters the feed port and C1 either by gravity or with the ram's assistance. Typically, the charge of 12 cc requires 1 to 4 minutes to enter the rotor, which is primarily dependent on rotor speed and melting properties of the material. After mixing, the rotor is stopped and the door opened to expose the die. The rotor is re-started to induce extrusion of a molten strand of about 6 cc that is air cooled. The entire process typically takes 3 to 10 minutes. The same quantity extruded may then be added to the mixer, if multiple batches are desired. A nitrogen blanket or vacuum over the rotor and process are possible.

Experiments and Results

A starve fed 25 mm, 36/1 L/D SSE with three SFEM elements, similar to Fig. 2, with a P1, P2 clearance of 1 mm, was used for the broad extrusion experiments showing macroscopic results. The comparisons include: 1) natural low-density polyethylene (LDPE) pellets (1 MI) with 1% red color at 200 ˚C into film, representing color mixing, 2) a salt/pepper blend of 35 wt % calcium carbonate powder dry-blended with 65 wt % polypropylene (PP) pellets (35 MFI) was blended into extruded strands, representing the high filler loading category of compounding, 3) a salt/pepper blend of 10 % elastomer pellets and LDPE pellets was made into a thermoplastic olefin (TPO) film representing compounding requiring high shear stress, 4) rigid polyvinyl chloride (RPVC) pellets and flexible polyvinylchloride pellets were compounded into semi-flexible strands, representing different viscosity compounding and a thermally challenging mixture. All four material systems were simply dry-blended prior to extrusion.

The same four mixtures were then processed using the micro-batch mixer. The 1% red color concentrate pellets were loaded into the micro-batch mixer and then followed with natural LDPE. The rotor speed was set at 4.3 rpm. Input and mixing totaled 4 minutes and produced a uniform colored strand from end to end. This implies significant axial mixing in less than 18 total revolutions. The strand was similar in color to the extruded film, as shown in Fig 6.

The calcium carbonate/PP blend was processed at 204 ˚C and a rotor speed of 100 rpm. Input time was 3 minutes, and mixing time was 4 minutes. A surprisingly smooth rod resulted from both the batch mixer and SSE—nearly as smooth as a rod of 100 % PP. At 100X magnification, both surfaces appear smooth (Fig 7).

The 10 % elastomer/LDPE blend was processed at 204 ˚C at a rotor speed of 100 rpm. Input time was 2 minutes, and mixing time was 4 minutes. A smooth strand was produced that while still hot was pressed into a film of about 0.25 mm. There were no visible domains in the extruded film or the rod pressed into film from the batch mixer unlike films processed using a SSE with a typical mixing element (Fig 8).

The PVC mixture was fed for 3 minutes and mixed for 3 minutes at 100 rpm. A 6 gram rod was extruded from a 12 gram input. The rod is semi-flexible, as is the rod produced from the SSE and SFEM. A 50/50 mixture was then added to replace the extrudate of 6 grams. This was repeated 5 times without obvious yellowing or discoloration in the rod from the batch mixer.

For the experiments showing microscopic results, an IMPB of PS/HDPE was dry-blended into two composition ratios, 20/80 and 30/70 % PS/HDPE. Each was separately starve-fed into a 24/1 L/D extruder with dual SFEM elements, a P1, P2 clearance of 1 mm, a barrel temperature 204C and processed at 120 rpm. The same mixtures were separately fed into the micro-batch mixer processing at 204 °C Feeding time was 3 minutes, and mixing time was 1, 2, 4 and 6 minutes. The morphology of the PS/HDPE IMPB processed using the micro-batch mixer and SSE was examined using a Zeiss Gemini 982 Field Emission Scanning Electron Microscope (FESEM). The samples were fractured at liquid nitrogen temperatures and gold coated prior to viewing.

FESEM micrographs corresponding to the PS/HDPE IMPB are presented in Figure 9, in order to analyze the resultant morphologies. The samples shown in the micrographs in the left column were processed using the micro-batch mixer and the samples shown in the micrographs in the right column were processed using the SSE fit with two SFEM elements. The 20/80 % PS/HDPE composition is shown at 10 and 2 µm scales in Figures 9A – 9D. The 30/70 % composition is shown at 10 and 2 µm scales in Figures 9E – 9H. In the micrographs, the PS surface appears smooth, while the HDPE surface appears rough. This is due to the brittle fracture behavior of PS and the more ductile fracture behavior of HDPE. For the 20/80 % PS/HDPE composition, the PS minor phase domains appear as discrete particles and range in size from 0.2 – 2 µm, when processed using the micro-batch mixer and the SSE. This is a remarkably small domain size for the PS minor phase especially considering the processing was performed using a SSE and a micro-batch mixer with similar mixing elements. This level of compounding approaches the nano-scale and is usually not exhibited in SSE or small-scale batch processing. The 30/70 % PS/HDPE composition is near the co-continuous region, dependent upon the composition ratio and component viscosities. Thus, the PS domains are no longer only discrete globules. Rather, the PS domains are elongated and are intertwining around the HDPE domains in the same manner when processed using the micro-batch mixer and SSE. The PS domains range in size from 2 – 20+ µm, when processed using the micro-batch mixer and SSE.

Discussion

The micro-batch mixer produced good macroscopic and microscopic results that were similar to SSE results. Both processing methods resulted in a well-mixed red LDPE film, a smooth calcium carbonate filled PP extruded strand, the TPO showing no visible domains, easy and successful processing of vinyl with no burning, and a semi-flexible vinyl rod with multiple batches showing no burning. These experiments represent major mixing scenarios for the extrusion industry, including color mixing, highly filled materials, melt blending, and thermally sensitive processing. As is evident from Figure 9, the resulting morphologies of the PS/HDPE IMPB are very similar when processed using the micro-batch mixer and SSE.

Conclusions

The micro-batch mixer is shown to produce similar mixedness as the SSE for a variety of materials systems because of its similar geometry to the SSE compounder with SFEM elements. The mixing times are fairly short and comparable to extrusion residence times. This micro-batch mixer adds a powerful tool for the polymer researcher because only small quantities of material are necessary, and these experiments successfully scale-up to large-scale extrusion processing equipment. The micro-batch mixer is a vital, simple piece of equipment that provides results quickly and conveniently.

References

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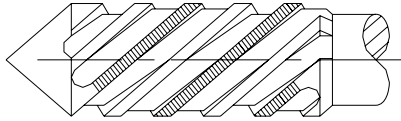
Figure 1. *Schematic of the Egan mixer, or spiral UC mixer, showing the shaded resistance zone and channels. Entrance flute dead ends at the wiping land.*
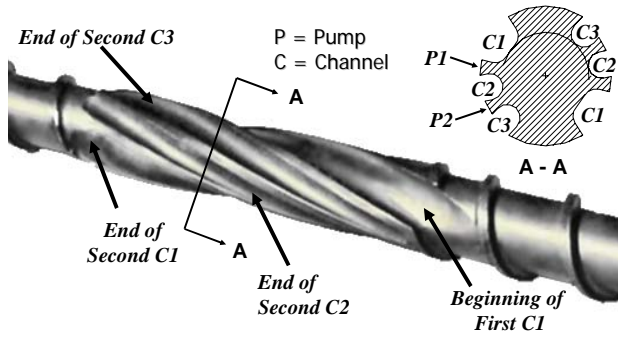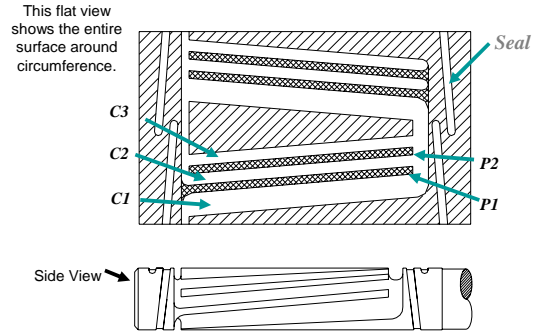

Figure 2. *SFEM element for the SSE. Mixer nomenclature/cross section is the same for SSE and micro-batch mixer.*
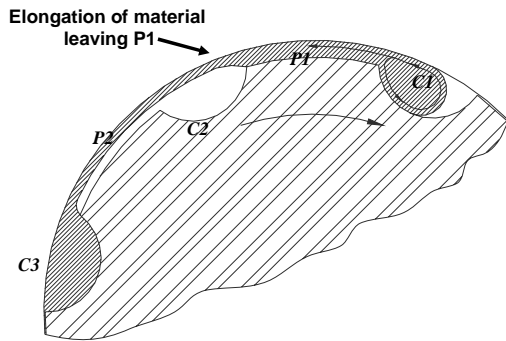
**Figure 3.** *SFEM element showing elongation upon leaving P1*
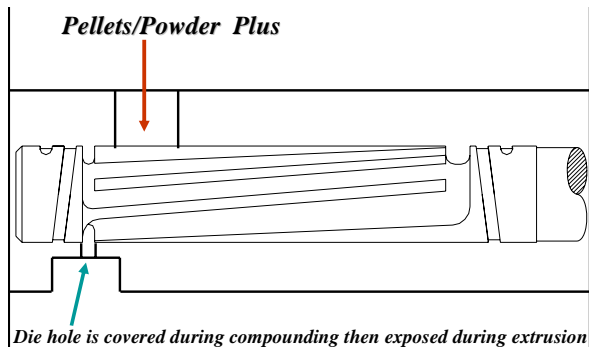
**Figure 4.** *Schematic of the mixing element for the micro-batch mixer*

Figure 5. *Flat vies schematic of the mixing element for the micro-batch mixer*


Figure 6. *1% red color concentrate pellets/LDPE showing the film produced using the SSE and rod produced using the micro-batch mixer, both with the SFEM mixing elements.*

(A)   (B)
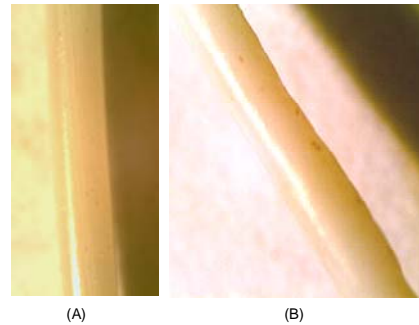
Figure 7. *35 % calcium carbonate/PP blend at 100X magnification processed using the (A) SSE and (B)micro-batch mixer*

(A)   (B)
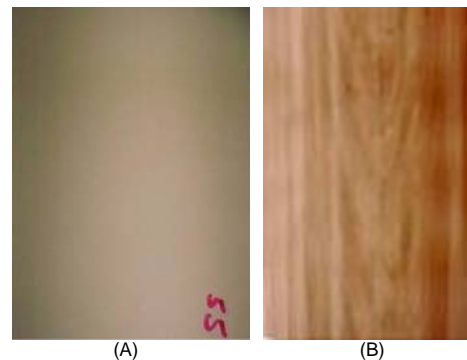
Figure 8. *10 % Elastomer/LDPE blend processed using the (A) SSE with SFEM mixing elements and (B) SSE with a typical processing mixing element*
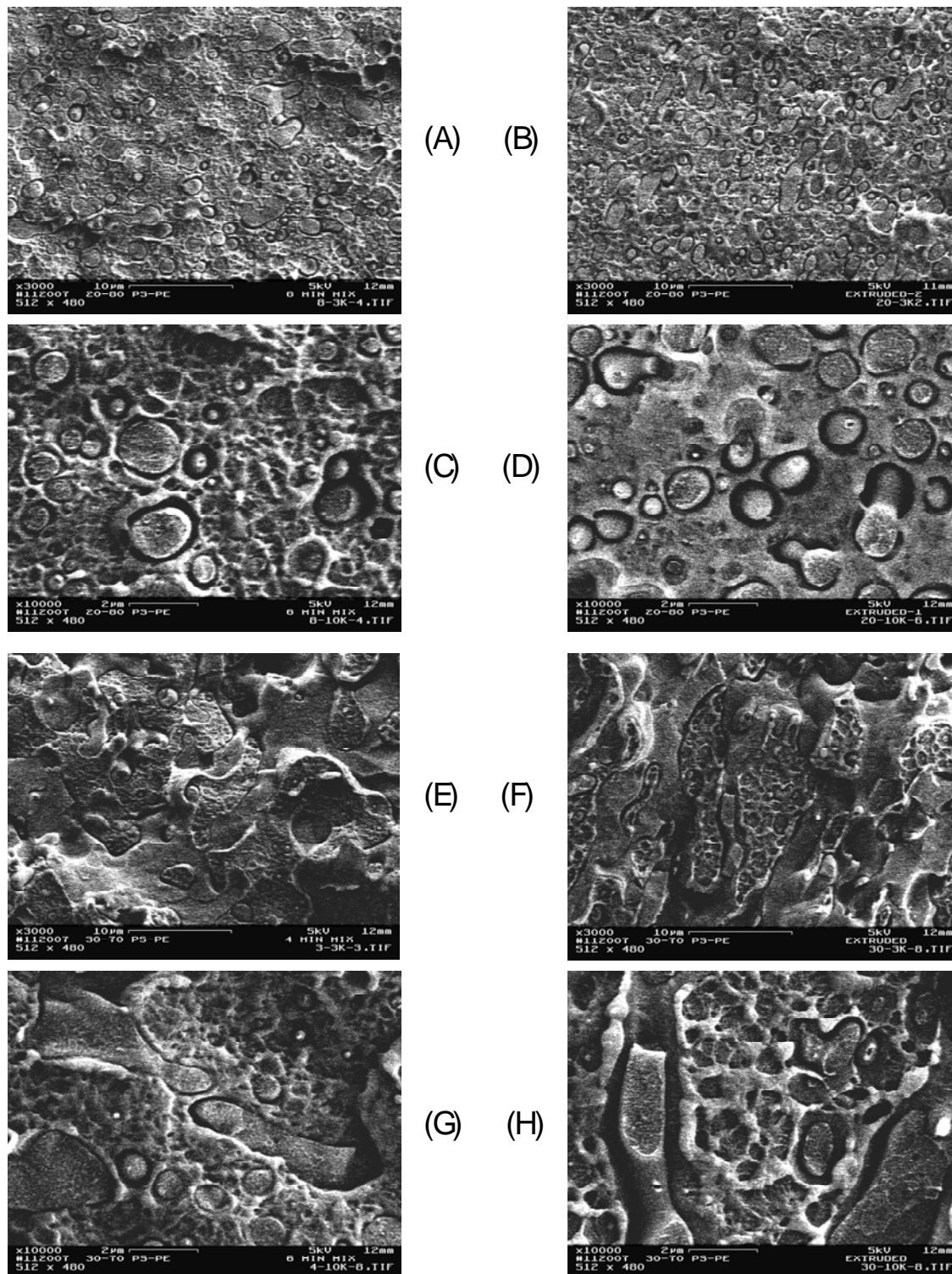
Figure 9. *SEM Micrographs of a PS/HDPE IMPB prepared using a micro-batch mixer and extruder fit with two SFEM elements. Micrographs labeled (A) – (D) correspond to a 20/80 % PS/HDPE composition ratio, micrographs (E) – (H) correspond to a 30/70 % composition ratio. The samples in micrographs labeled (A), (C), (E), and (G) were prepared using the batch mixer, while the samples in micrographs labeled (B), (D), (F), and (H) were prepared using the extruder.*