Improvement of a Gram-scale Mixer for Polymer Blending

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ABSTRACT: A small-scale mixer designed by Bryce Maxwell is commercialized by Custom Scientific Instruments, Inc. under the name of MINI-MAX Molder. It is very useful for the study of polymer blends, especially when the available amount of polymer specimen is limited, for example, less than a few grams. However, it gives essentially shear flow and suffers from poor distribution and dispersion capabilities in comparison with large scale extruder and internal mixers. We propose here an improvement of the mixing and dispersing capabilities of the Maxwell small-scale mixer by the addition of Teflon disk and steel balls together with the mixed materials. When polypropylene and high impact polystyrene were mixed at 70/30 wt. ratio at 180°C without the disk and balls, the high impact polystyrene particle size (D) was 6.27 μm. A finer dispersion (D = 1.44 μm) was achieved by the introduction of one Teflon disk at the center of mixing cup and three steel balls. Furthermore, with increasing the number of steel balls from three to seven, and with using different sizes of balls, much finer dispersions were achieved (D = 0.58 μm and 0.47 μm). This may be caused by: (1) the addition of Teflon disk eliminates the low shear regions in the mixing cavity, and (2) the addition of steel balls induces asymmetric circulation of the materials, some changes in the flow lines going from the center to the border or from the top to the bottom, some reorientation of the materials, and higher shear fields. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 1–5, 2006

Key words: MINI-MAX Molder; blending; mixing; particle size

INTRODUCTION

Blending of dissimilar polymers offers attractive opportunity for the development of new materials with useful combination of properties.[1] The laboratory development of new polymer blends may need expensive materials whose processing requires small-scale mixers. One of these small-scale mixers was designed by Bryce Maxwell, named MINI-MAX Molder®, Custom Scientific Instruments, Inc. [Fig. 1(a)]. It is particularly useful for the analysis of the morphology during processing since it is very easy to open and remove samples from the melt at different times. Moreover, test samples may be molded by injection of the melting materials through a small hole at the bottom of the mixing cup.

However, it was shown that the mixing capability of MINI-MAX Molder is significantly lower than that obtained by a small internal mixer with two blades (e.g., Plastograph®) or an extruder.[2] The poor mixing capability of MINI-MAX Molder seems to be caused by symmetric rotation axis and pure shear. The pure shear is ineffective for the dispersion, especially when

the ratio of melt viscosities of component polymers is above 4 and the less viscous polymer turns to the matrix.[3] Moreover, in comparison with twin screw extruder or Plastograph, MINI-MAX Molder does not induce reorientation of the mixed materials without periodic lifting of the rotor; consequently, it has lower efficiency for the distribution of the dispersed phase.[4] Furthermore, MINI-MAX Molder does not provide a time variation of the shear on a flow line, which has been proved to be helpful for fine dispersion.[5]

To get a better homogenization in MINI-MAX Molder, it is suggested to lift the rotor and push it down periodically during the mixing.[6] However, the reproducibility is not achieved sometimes when different polymers adhere differently to the metal parts.[2]

Our previous study showed that the mixing efficiency of MINI-MAX Molder can be ingeniously improved by the addition of steel balls to the mixer. The improvement may be caused by the changes in the flow lines in the mixer, some reorientation of the material, and the presence of high and of low shear fields.[7]

Following this result, we tried to make further improvement in the mixing capability of MINI-MAX Molder, by adding different numbers and sizes of steel balls and/or by adding a Teflon disk into the mixer. The mixing capability was dramatically improved, for example, by the addition of two small balls, five big balls, and one Teflon disk.
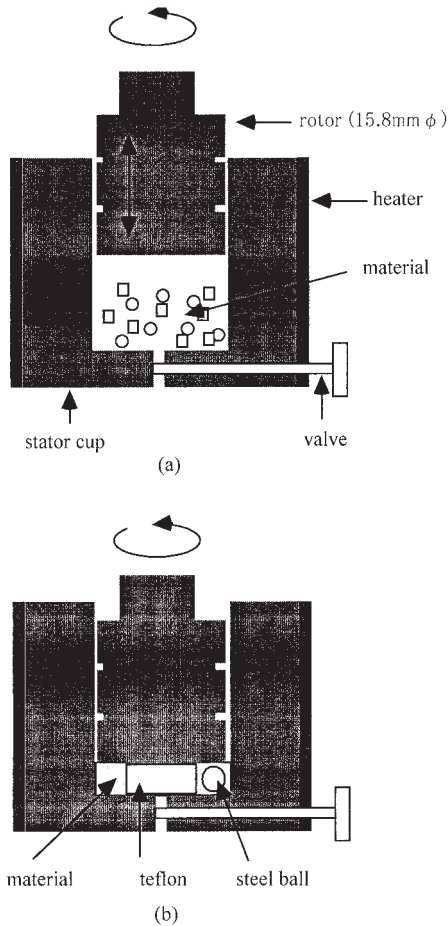

Figure 1 (a) Scheme of MINI-MAX Molder CS 183MMX; (b) scheme of melt mixing with Teflon disk and steel balls.

Figure 3 SEM pictures of the 70/30 PP/HIPS blend mixed at the normal mixing condition. Sampling was performed at different places: (a) top-border, (b) top-center, (c) bottom-border, (d) bottom-center, and (e) middle (see Fig. 2).

EXPERIMENTAL

Materials used are polypropylene, PP, supplied by Idemitsu Petrochemical Co. (F-200SP, melt flow index (MFI) = 2.0 g/10min.) and high impact polystyrene, HIPS, supplied by Idemitsu (HT50, MFI = 4 g/10min.).

The small mixer used is MINI-MAX Molder CS 183MMX, Custom Scientific Instruments, Inc. It is schematically presented in Figure 1(a). The diameter
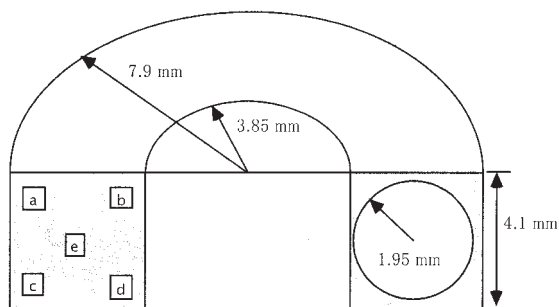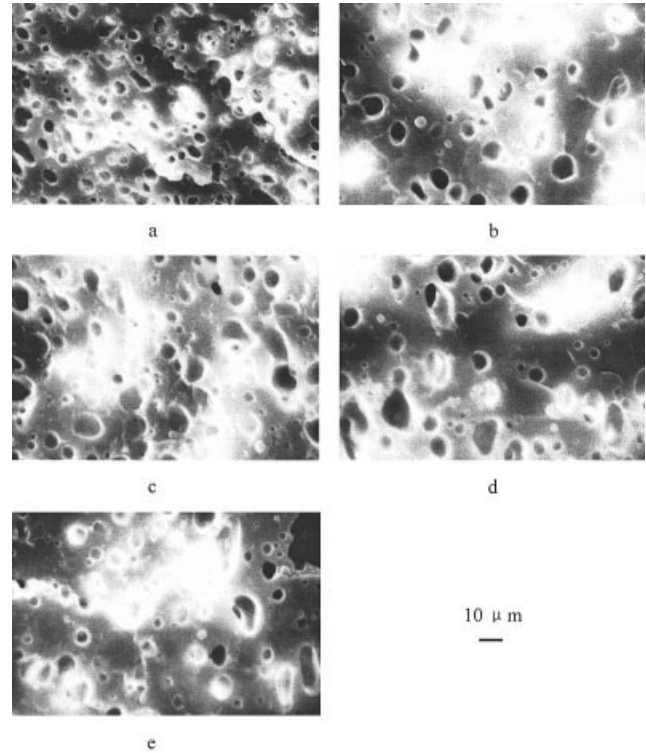

Figure 2 Sampling places (a–e) for SEM observation.

of the mixing cup is 15.8 mm and the rotational speed of the mixer is fixed at 92 rotations per minute. Mixing operations were carried out at four different conditions:

1) Normal mixing: only the polymer specimens were loaded on the mixer.

2) Mixing with Teflon disk: one Teflon disk (e.g., 7.7 mm diameter, 4.1 mm thickness) was placed at the center of stator cup and the polymer specimens were charged [Fig. 1(b)].

3) Mixing with balls: the polymer specimens and steel balls were loaded.

4) Mixing with Teflon disk and balls: the Teflon disk was placed at the center of stator cup and polymer specimens were loaded after the steel ball was put in (Fig. 2).

In all these experiments, the mixing cup was kept filled with the polymer materials. As the size and/or number of the Teflon disk and/or steel ball added were different in every experimental run, the amount of polymer specimens was different for the different conditions as will be shown later.

Height of the sample was determined by the size of the steel ball. For example, in experimental run of B4 in which the 3.3 mm diameter steel balls were used and were set as shown in Figure 2, a 3.5 mm height of the Teflon disk was chosen to maintain clearances
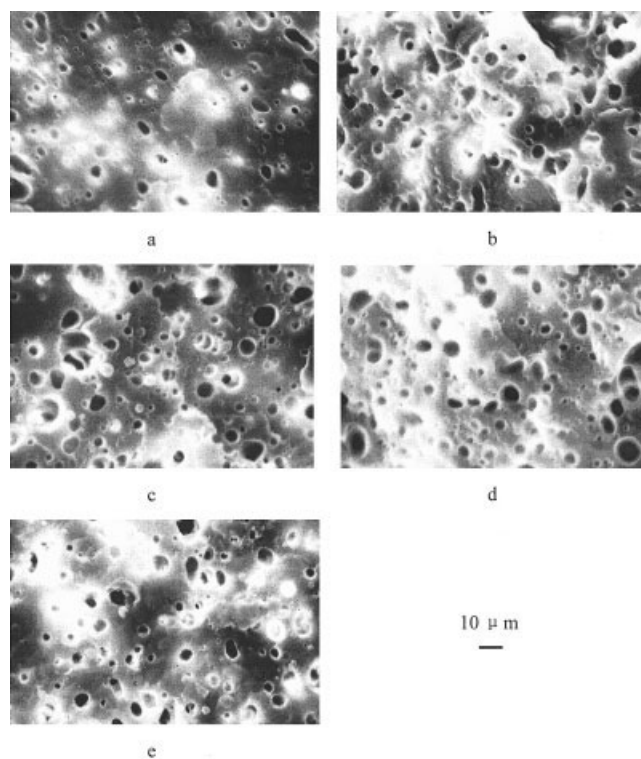
Figure 4 SEM pictures of the 70/30 PP/HIPS blend mixed by placing Teflon disk (8.4 mm diameter/3.5 mm height) and adding three steel balls (3.3 mm diameter). Sampling was performed at different places: (a) top-border, (b) top-center, (c) bottom-border, (d) bottom-center, and (e) middle (see Fig. 2).

between the rotor with the ball and the ball with the bottom of the mixing cup as narrow as possible, here equaling 0.1 mm, respectively. Because the polymer materials filled with the mixing cup, the height of the sample thus equals the height of the Teflon disk, 3.5 mm. Similarly, in experimental run of B8–B11 in which the 3.9 mm diameter steel balls were used, 4.1 mm height of the sample was chosen to maintain the clearances between the rotor with the ball and the ball with the bottom of the mixing cup equaling 0.1 mm.

Pellets of the materials were introduced in the mixer at 180°C. The rotor was pushed down on the material for 5 min before any mixing. This procedure allows the complete melting and temperature equilibration of the materials before mixing. PP and HIPS were melt-mixed at 70/30 (PP/HIPS) wt. ratio.

After mixing for a fixed time, rotation was stopped and the mixed material was immediately quenched. The quenching was performed by putting the whole mixing cup into a water bath. Then, from the quenched cup, a cylindrical specimen was removed. From the quenched specimen, sampling for microscopic observation was carried out at various places as shown in Figure 2.

The morphology was observed by scanning electron microscopy, SEM. The samples for SEM observation were prepared by fracturing in liquid nitrogen and then solvent etching of the dispersed HIPS with toluene. From SEM pictures, image analysis was performed by Macroscope® software. For calculation of the average particle size, ~ 500 particles were used.

RESULTS AND DISCUSSION

Typical results of SEM observation are shown in Figures 3–5. The average particle size was calculated by averaging the results at various sampling places (a–e).

Figure 6 shows the average particle size by SEM as a function of mixing time under the normal mixing condition. One can see a very rapid decrease in the particle size at the early stage of mixing and then no further decrease after 4 min. Similar time variations were observed under different operation conditions. Hereafter, we discuss the results by fixing the mixing time at 5 min.

In Table I, the results by the four different conditions—(B1) normal mixing, (B2) mixing with Teflon disk, (B3) mixing with three steel balls, and (B4) mixing with Teflon disk and three steel balls—are summarized. Note that the height of sample was kept
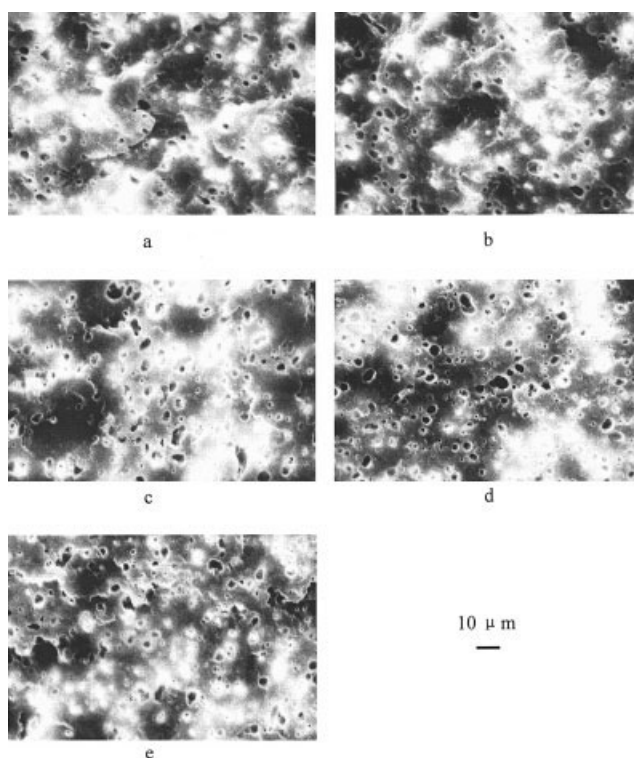

Figure 5 SEM pictures of the 70/30 PP/HIPS blend mixed by placing Teflon disk (7.7 mm diameter/4.1 mm height) and adding five big steel balls (3.9 mm diameter) and two small steel balls (3.3 mm diameter). Sampling was performed at different places: (a) top-border, (b) top-center, (c) bottom-border, (d) bottom-center, and (e) middle (see Fig. 2).
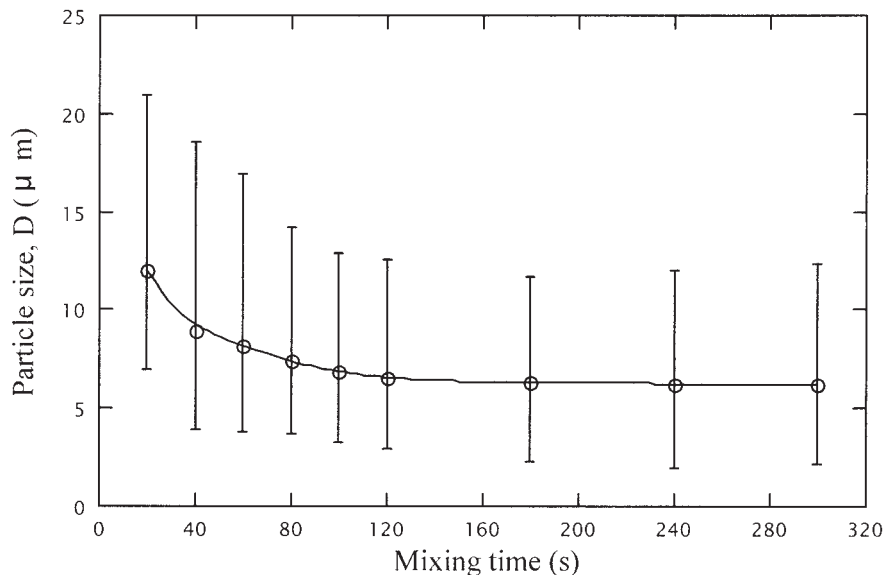
Figure 6   Average particle size by SEM as a function of mixing time at the normal mixing condition.

constant (3.5 mm) by using Teflon disk of 8.4 mm diameter and 3.5 mm height. The normal mixing condition (B1) yields the particle size of 6.27 μm. By adding the Teflon disk or steel balls (B2 and B3), the particle size became smaller. With the addition of both Teflon disk and steel balls (B4), the particle size became even smaller. These may be caused by eliminating the low shear regions in the mixing cavity by the addition of Teflon disk, and inducing an asymmetric circulation of the materials by the addition of steel balls.

SEM pictures of the blend prepared under the normal mixing showed that the particle sizes were significantly different from positions (a) to (e) (see Fig. 3). By contrast, in the blend prepared by the addition of Teflon disk and three steel balls, no significant difference in particle size was seen between the positions (a) to (e) (see Fig. 4). Thus, the addition of Teflon disk and three steel balls seems to render a homogenous shear field in the mixer.

As summarized in Table II, when the number of steel balls was increased from three to seven, the

average particle size decreased. The addition of too many (nine) balls resulted in poor dispersion. The results may imply that by increasing steel balls up to an appropriate number, the higher shear fields are attained around the balls; however, when more balls are added, the balls started to touch with each other and rotate less freely, resulting in lower shear fields.

By choosing the size of Teflon disk and that of steel balls, one can change the clearance between the wall of mixing cup, the surface of ball, and the surface of Teflon disk. The clearance for the set in Table II was 0.2 mm. If one chooses a Teflon disk of 7.7 mm diameter and bigger steel balls of 3.9 mm diameter, the clearance can be reduced to 0.075 mm. The smaller clearance could generate the higher shear rates and render the finer dispersion.

As expected, it was realized as shown in Table III; that is, compared with the results in Table II, the average particle sizes in Table III are much smaller.

Another interesting choice of mixing condition was to use small and big steel balls. As shown in Table IV,

TABLE I
Mixing Conditions and Average Particle Size (D) After Mixing at 180°C for 5 min

Sample code	Material weight (g)	Number of Teflon disk[a]	Number of steel balls[b]	Particle size, D (μm)
B1	0.70	0	0	6.27
B2	0.49	1	0	3.07
B3	0.54	0	3	2.91
B4	0.34	1	3	1.44

[a] 8.4 mm diameter/3.5 mm height.
[b] 3.3 mm diameter.

TABLE II
Mixing Conditions and Average Particle Size After Mixing at 180°C for 5 min by Setting Teflon Disk[a] and Adding Small Steel Balls; Number of Balls Was Varied

Sample code	Material weight (g)	Number of steel balls[b]	Particle size, D (μm)
B4	0.34	3	1.44
B5	0.30	5	1.20
B6	0.27	7	0.83
B7	0.23	9	0.96

[a] 8.4 mm diameter/ 3.5 mm height.
[b] 3.3 mm diameter.

TABLE III
Mixing Conditions and Average Particle Size (D) After Mixing at 180°C for 5 min by Placing Teflon Disk[a] and Adding Big Steel Balls; Number of Balls Was Varied

Sample code	Material weight (g)	Number of steel balls[b]	Particle size, D (μm)
B8	0.45	3	1.41
B9	0.40	5	1.06
B10	0.35	7	0.58
B11	0.30	9	0.66

[a] 7.7 mm diameter/ 4.1 mm height.
[b] 3.9 mm diameter.

TABLE IV
Mixing Conditions and Average Particle Size (D) After Mixing at 180°C for 5 min by Placing Teflon Disk[a] and Adding Small and Big Steel Balls

Sample code	Material weight (g)	Number of big balls[b]	Number of small balls[c]	Particle size, D (μm)
B11	0.45	7	0	0.58
B12	0.40	5	2	0.47
B13	0.35	3	4	0.55
B14	0.30	1	6	0.65

[a] 7.7 mm diameter/ 4.1 mm height.
[b] 3.9 mm diameter. [c] 3.3 mm diameter.

by combining small (3.3 mm diameter) and big (3.9 mm diameter) steel balls, finer dispersion was achieved. Furthermore, note that there is an optimum number fraction of small and big balls (2 small/5 big balls) for the fine dispersion and SEM pictures shown in Figure 5.

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