With its ratio of screw of D_a/D_i = 1.55 and specific torque of 18 Nm/cm³, the ZSK Mc[18] provides very good processing conditions for producing masterbatches (figures: Coperion)

Black and Finely Dispersed

Compounding Carbon Black Masterbatches for Polyester Fibers

A carbon black masterbatch used for coloring polyester fibers has to fulfill high standards, especially if the intended results are filmy spinning fibers for very soft and comfy textiles. Taking a Coperion twin-screw extruder as exemplary, it is shown how screw configuration and processing parameters affect the dispersability of carbon black and material degradation in the polyester matrix.

Polyester fibers are utilized in many different applications. In automobile construction, for example, roof linings and interior cladding, seat covers or even complete convertible soft-tops are made from it. In the clothing branch we find functional wear and street clothing, in home textiles, curtains, seat covers and much more. Depending on the application, a number of different types of fibers are available. The differences lie in their fineness, cutting lengths, as well as in the various physical parameters of textiles, such as fiber strength, elongation and hot-air shrinkage.

Polyester fibers made from PET (polyethylene terephthelate) are made by melt spinning, whereby the PET melt compounded in an extruder is subsequently squeezed through spinnerets. The properties desired are obtained by adding functional additives and pigments, generally in the form of masterbatches. These masterbatches are subject to strict requirements, particularly when spinning polyester fibers that are just a few µm thick to produce especially soft and comfy textiles.

High Demands on Carbon Black Masterbatches

Black polyester fibers are generally colored with carbon black dosed as masterbatches when PET is compounded. Only a few types of carbon black that are extremely pure and have very fine particles are suitable for this. An industrially manufactured, hi-tech carbon black is used which is physically and chemically specified and produced under controlled processing conditions: industrial carbon black consists of more than 96 % carbon and contains only small amounts of oxygen, hydrogen, nitrogen and sulfur. Carbon black is available in various types with properties tailored to its area of application.

The production of color batches for fiber applications requires PET with an intrinsic viscosity (IV) of 0.6 to 0.8 dl/g (the IV value is a comparative measure of the average molecular weight). Care must be taken, since water strongly degrades molten PET hydrolytically. At the same time, PET is hygroscopic and can absorb water very quickly up to an equilibrium moisture content of 3,300 ppm.

Twin-Screw with High Dispersing Power

Co-rotating twin-screw extruders are especially suitable for compounding carbon black color batches. Parameters relevant to the compounding process include the screw diameter ratio D_a/D_i »

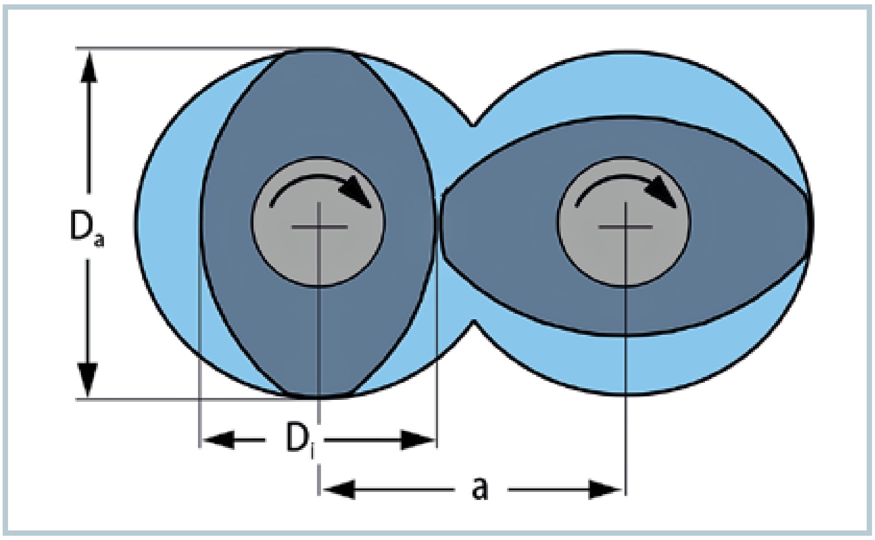

Fig. 1. Schematic presentation of the co-rotating twin-screw with outer (D_a) and inner screw diameters (D_i), as well as centerline distance a

and the specific torque Md/a^3, calculated as the ratio of the torque transmitted to the screw shafts, and the centerline distance to the third power (**Fig. 1**). The ZSK Mc[18] twin-screw extruder from Coperion GmbH of Stuttgart, Germany, with a screw diameter ratio of D_a/D_i = 1.55 has proven capable of compounding PET-base carbon black masterbatches (**Title figure**). Its specific torque of 18 Nm/cm^3 is tuned to the free screw volume and has high dispersing power.

The processing section is modularly set up from individual housing segments. Therein several processing zones are set with task-specifically configured screw elements: from conveying, plasticizing, dispersing and homogenizing to degassing and pressure build-up on the compounded melt ahead of the die. The tightly intermeshing twin-screw prevents formation of stagnant areas along the entire length of the processing section. The result is continuously high conveying power together with optimum self-wiping action of the screws. The extruder treats the product gently and exhibits very good mixing properties.

Two Different Compounding Processes

The two processes used for compounding PET-based carbon black batch are the Premix method and the Split-feed method.

In the Premix process, all components – matrix polymer, any additives and carbon black – are pre-mixed and fed together into the main intake of the twin-screw extruder (**Fig. 2**). In order to avoid de-mixing effects and the formation of carbon black agglomerates in the intake

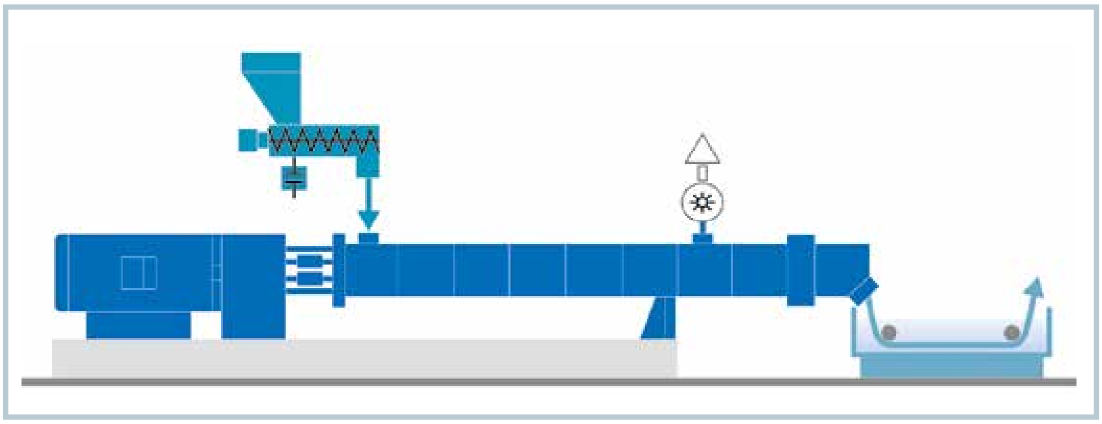

Fig. 2. Setup for processing carbon black masterbatch by the Premix method: the pre-mixture of PET and carbon black powder is dosed volumetrically at the intake area of the compounding extruder

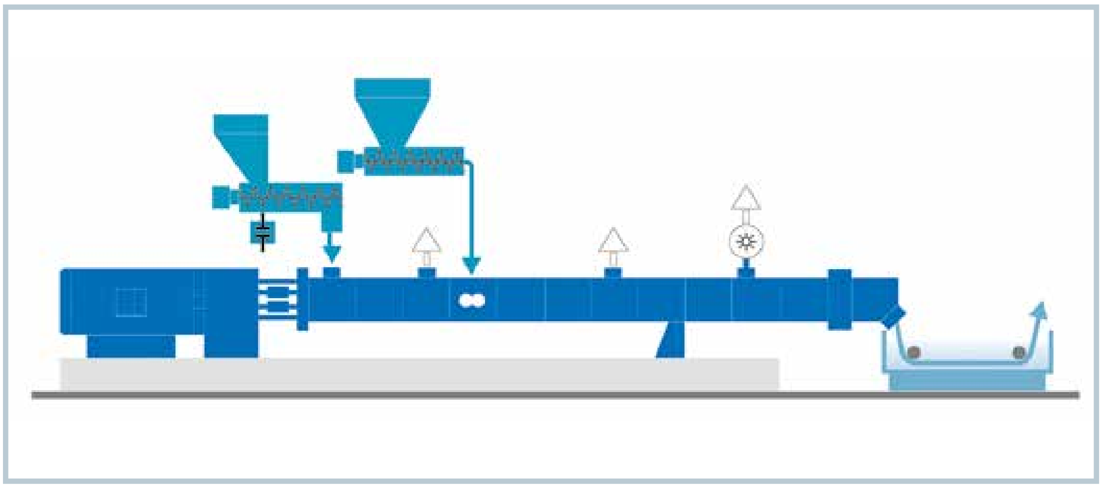

Fig. 3. Setup for processing carbon black masterbatch by the Split-feed method: PET (pellets or flakes) and carbon black powder are gravimetrically dosed into the compounding extruder separately, the carbon black downstream into the already molten PET matrix

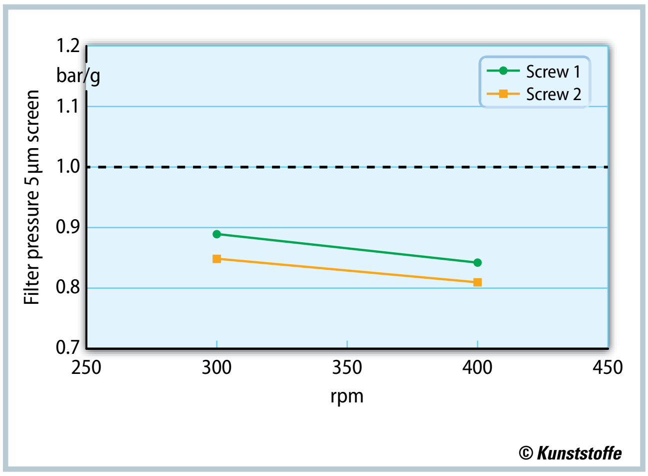

Fig. 4. The influence of screw geometry (screw 2 has more kneading elements than screw 1) and screw speed on dispersion quality

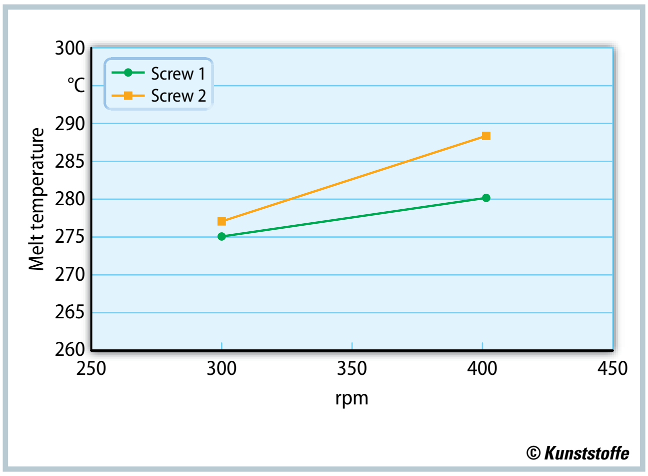

Fig. 5. The influence of screw configuration and speed on melt temperature (explained by the text)

zone, the PET, too, must be present in powder form. Since PET is available on the market only as pellets or as flakes, it has to be pulverized by courtesy of a time-consuming and cost-intensive process. The mixture is made in a container mixer, or also a quick-mixer, and then volumetrically fed to the twin-screw extruder, to get the polymer molten, the carbon black dispersed, and the melt homogenized and degassed. Subsequently, the masterbatch melt is usually shaped into strands and cut up as pellets by a pelletizer.

Even though the Premix process is expensive, it is still used frequently worldwide. One reason for this is the fact that the personnel operating the system require little or no training, and even semi-skilled workers can be employed.

By contrast, the Split-feed process (**Fig. 3**) offers economic advantages, since it eliminates time and cost intensive grinding, as well as the production of the pre-mixture from PET powder and carbon black. Here PET in any form – be it pellets, flakes or powder – is fed into the first housing of the twin-screw extruder and melted down. Downstream the carbon black is added separately directly into the polymer melt using a twin-screw side feeder (ZS-B). There the pigment is gently worked in and dispersed. If PET pellets and carbon black powder were dosed together in the extruder intake, this would tend to compact the carbon black. Given the especially high radial forces occurring here, the still insufficiently wetted carbon black can form agglomerates that subsequently could not be completely broken up.

For the separate feeding of the two product streams – the PET pellets and carbon black powder – a gravimetric dosing scale is required by the Split-feed process at each stream. Notably better trained personnel are required for orderly operation than to perform volumetric dosing by the Premix method.

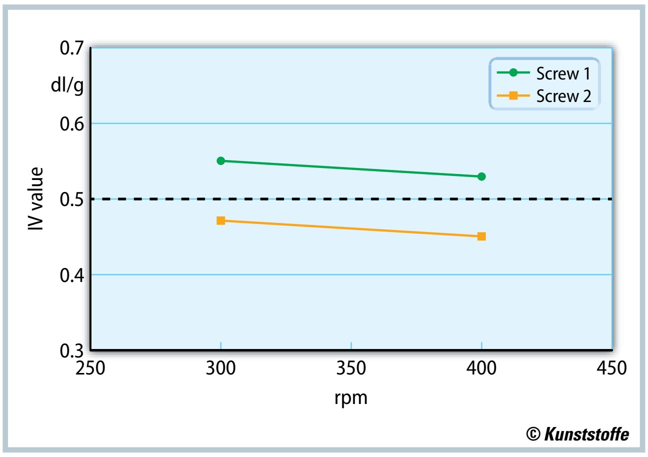

Fig. 6. The influence of screw configuration and speed on IV degradation of PET matrix (IV initial value 0.62 dl/g)

Carbon Black Masterbatch by the Split-Feed Method

Below the results of trials on a ZSK 45 Mc[18] (screw diameter 45 mm) where PET-based carbon black masterbatch was produced by using the Split-feed method are presented (for the comparison the Premix method was investigated using the same type of extruder). The masterbatch contained 30 wt.-% carbon black; the initial IV-value of the PET was 0.62 dl/g. For the substrate, PET pellets have been dosed into the main intake of the twin-screw extruder, the carbon black directly into the polymer melt. Variables were the screw configuration and screw speed.

The goal was to produce a masterbatch with very high dispersability for coloring so-called fine denier fibers. A filter pressure value test according to EN 13900-5 (**cf. box**) was used to determine dispersability. For fine-fiber applications, the screens used usually have 10 µm mesh. In these tests, the quality requirement was tightened further, and an especially fine 5 µm screen was selected. Here, too, filter pressure value ought to lie below 1 bar/g.

Figure 4 presents the influence of screw configuration and screw speed on the carbon black dispersion achieved. Screw 2 has more kneading elements than screw 1 and thus achieves higher material shear. The result is a somewhat improved dispersion of the carbon black (lower filter pressure value) compared »

Filter Pressure Value Test

Standard EN 13900-5 describes a standard procedure for acquiring filter pressure value as a quality criterion for masterbatches. It is performed using a test mixture made from masterbatch and base polymer. The test setup used for it consists of a single screw extruder, a melt pump and a filter test tool (breaker plate with inlaid filter package). The material is melted by the extruder, homogenized and fed to the melt pump that forces the melt through the filter at a constant defined volume flow. The filter mesh varies with the specified quality (fineness of dispersion).

For measurement, the base polymer is put in motion and the rising pressure recorded at the filter. Then the test shifts to the test mixture with a defined pigment content. Agglomerates clogging the filter (and other impurities) cause the pressure on the filter to rise. Maximum pressure is reached as soon as the entire test mixture (the added masterbatch) has passed through the filter. The resulting pressure differential based on the mass of the added pigment is the filter pressure value (bar/g): the smaller this value is, the better (finer) the additive distribution in the masterbatch. For example, the filter pressure value at a screen mesh of 10 µm should not be higher than 1 bar/g for a PET-base carbon black masterbatch used for coloring fine fibers.

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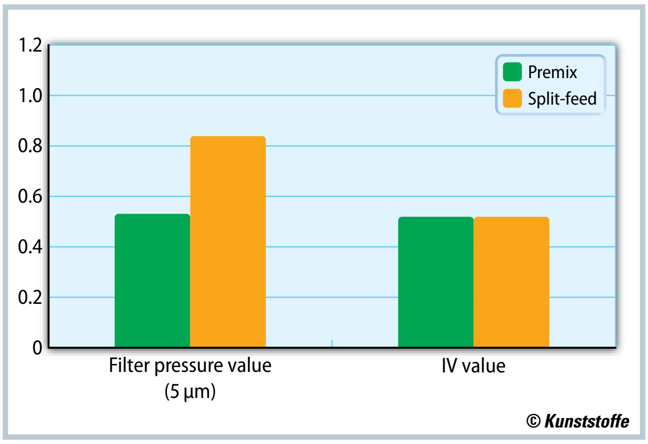

Fig. 7. Comparison of carbon black batch properties according to the production method

with screw 1. The same result comes from increased screw speed: given the same throughput, shear energy input is proportional to screw speed, so that higher rpm's result in improved dispersion in both configurations. Both screw configurations fall below the defined filter pressure value of 1 bar/g limit value.

Increased shear, however, leads not only to improved dispersion quality. The additional energy input simultaneously dissipates in the material in the form of heat, thereby raising melt temperature (**Fig. 5**). The temperature increase is particularly striking at screw 2 with its higher number of shear elements and at simultaneously increased screw speed.

Due to PET's sensitivity to temperature and shear (IV loss due to material degradation), the energy input during compounding can have a negative effect on the properties of the substrate polymer and thus on the entire masterbatch. For applications of carbon black masterbatch to color polyester fibers, the IV value ought to lie as far above 0.5 dl/g as possible. As shown in **Figure 6**, this is the case for screw 1 even at higher rpm's; the IV value for screw 2 lies below the limit value at both screw speeds.

Thus it follows, on the one hand, that the shear force input by the screw has to be high enough to break up pigment agglomerates and disperse the carbon black particles well. On the other hand, shear must not be too high, in order not to cause excessive damage to the polymer. The results (**Figs. 4 and 6**) clearly show that screw 1 supplies the best result at a speed of 400 min^{-1}; by contrast, the higher shear of screw 2 hardly contributes to any improvement of dispersion (**Fig. 4**), but mainly to increased damage to the material (**Fig. 6**).

Comparative Results

For the exemplary comparison of Split-feed and Premix process, a pre-mixture was made consisting of PET powder and 30 % carbon black and processed at the same throughput as in screw configuration 1 at 400 min^{-1}. The results show that the IV values for the Premix method lie at a level similar to those achieved by the Split-feed method (**Fig. 7**). Alone the dispersive power is somewhat better (lower filter pressure value) when processing the pre-mixture of PET powder and carbon black. Contributing to this were the comparatively long dispersion distance and increased mechanical load on the carbon black in the melting zone when the components were added as a powdery pre-mixture at the extruder intake. Filter pressure value, however, falls clearly below the 1 bar/g limit by the Split-feed method, as well.

The tests using the ZSK Mc[18] for the production of carbon black colorant batch have shown that good results can be achieved using both the Premix, as well as the Split-feed method. This applies both to dispersion quality and to the IV value of the masterbatch. The Split-feed method requires somewhat more expertise to operate the equipment; however, it offers the great advantage that it makes automation simpler to implement. ■