MASTERBATCHES

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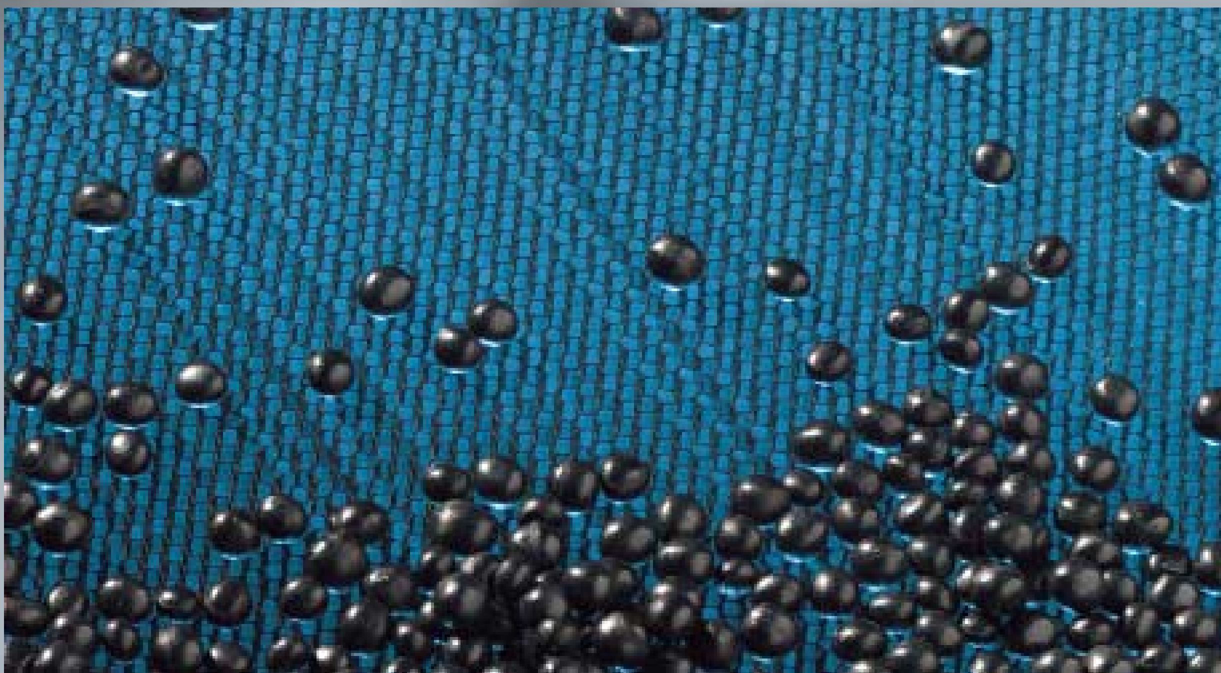
Dispersion and Dilution



Dispersion and Dilution

When using a masterbatch, the following are the two key properties that require careful consideration:

- the **DISPERSION** of pigment (or additives) within the masterbatch;
- the **DILUTION** of the masterbatch into the fabricator's polymer during use.

What is dispersion ?

Dispersion is a measure, at both the microscopic and macroscopic levels, of how a pigment, filler or additive is completely distributed throughout a polymer matrix. **It is probably one of the most fundamentally important properties of any masterbatch.**

What is dilution ?

Dilution is a measure of how well and how easily a masterbatch mixes with a fabricator's polymer. Thus a masterbatch exhibiting good dilution properties will produce processed articles (film, bags, pipe, sheet, etc.) showing excellent distribution of the masterbatch within the fabricator's diluting polymer. Conversely, a poorly diluted masterbatch will produce processed articles showing surface defects (lumps, voids), inconsistent colour and/or opacity, streaking and inferior physical performance.

Why is dispersion important ?

Various problems can arise at the conversion stage if good pigment dispersion in the masterbatch is not achieved by the masterbatch producer. These difficulties include:

- processing problems, such as rapid screen plugging and bubble rupture in film extrusion which will result in lower output and/or higher scrap levels;
- agricultural mulch film will be damaged by reduced protection against sunlight;
- lower impact strength in engineering polymers.

Why is it important to recognise the difference between poor pigment dispersion and incomplete masterbatch dilution ?

It is important to recognise differences between the two as a different course of corrective action may be needed to remedy the various problems caused. There is generally very little that the masterbatch user can do in the case of inherently poor pigment dispersion in the masterbatch. Generally, this product would have to be scrapped or re-worked into non critical applications. In the case of poor dilution however, there are several possible remedies. All of these hinge on increasing shear in the system.

How can poor dispersion be avoided ?

The responsibilty for good pigment or additive dispersion lies entirely with the masterbatch producer. By the careful selection of pigments, additives, resin and processing aids during the formulation stage and using the appropriate mixing equipment for a particular masterbatch, an experienced and technically competent masterbatch producer is able to provide a masterbatch with the necessary and consistent dispersion quality.

How can poor dilution be avoided ?

The masterbatch producer and end user are jointly responsible for ensuring good dilution. They have to design a total "system" to ensure the dilution process is optimised. "System" refers to the various factors which influence the dilution process, i.e.:

- melt flow index of the masterbatch
- melt flow index of the diluting resin
- types of processing equipment (extruder, injection moulding machine, etc.)
- processing temperatures
- production speeds
- back pressure (screen packs, etc.)

Dilution problems can be easily avoided if masterbatch producers and users pay proper consideration to these factors. However, problems will arise if some of the basic rules are overlooked. For example, an injection moulder will develop problems if he tries to pigment a high flow (melt index 60) polyethylene by adding a masterbatch containing a 40% fine particle size black dispersed in a melt index 1 polyethylene directly into the injection machine. No matter how perfectly the black pigment is dispersed within the masterbatch, the dilution resulting from this extreme example will always be of poor quality.

Concerning pigments and additives, which are more difficult to disperse?

Carbon blacks are generally the most difficult pigments to disperse, although several organic pigments such as phthalocyanines and rubine toners are equally difficult. Synthetic and naturally occurring silicas commonly used as antiblock agents in films are also difficult to disperse. High shear intensive mixing machinery is essential for all these materials to achieve masterbatch with the desired dispersion properties. Other additives such as calcium carbonate, lithopone, most titanium dioxides, barium sulphate, UV stabilisers and anti-static agents are generally easier to disperse than carbon black and the other materials listed above. There is a wide variety of carbon black grades used in masterbatch manufacturing and the degree of difficulty in achieving good dispersion across this range varies enormously. As a rule, the finer the particle size, the more difficult it is to disperse.

The structure level of the carbon black also plays a key role.
Low structure blacks are quickly incorporated into the polymer but because they have a less stiffening effect, the shear achieved is less, making it more difficult to achieve good dispersion.

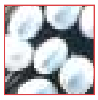
The opposite is true of high structure grades. Of course, if low structure grades are properly dispersed by the masterbatch manufacturer, they offer distinct advantages to the masterbatch user, as they offer better dilution quality due to their lower viscosity characteristics.
Nevertheless, it is possible to achieve equally good dispersion with fine particle grades (20 nanometres) and large particle grades (400 nanometres).

Many masterbatch users misunderstand the term "large particle" and mentally associate this with poorer quality and more agglomerates.

To dispel this notion, it should perhaps be remembered that even the large particle size blacks are still very fine indeed when compared to other pigments and fillers. For example, titanium dioxide and calcium carbonate have particles between 0.2 and 4 microns versus 0.06 microns for the so-called "coarse" grades of carbon blacks.

How is dispersion measured ?

There are actually many different ways to assess dispersion and the technique used often varies according to the type of pigment or additive. Most procedures documented in specifications refer to measurement of carbon black dispersion and most of these are discussed below. Some of these procedures may be appropriate to use with other pigments or additives. Tests carried out are normally described as either microscopic or macroscopic tests.



Microscopic techniques

The most common microscopic test used is referred to as the press-out method, so-called because this involves examination under a microscope of thin hot-pressed compound samples using transmitted light. Most masterbatches contain between 25% and 50% carbon black and hence would be completely opaque even in very thin sections. For this reason, the dispersion quality is assessed using some form of dilution.

Generally, a compound containing 2.5% carbon black (6% addition of a 40% masterbatch to 94% natural resin) is produced. This can be prepared on a laboratory internal mixer, two roll mill, twin screw or even a single screw extruder. Pinhead size pieces of this compound are then taken and hot pressed between two clear glass microscope slides. The resultant "buttons" are then examined under a microscope at say x100 magnification and rated against a series of standard photographs.

Recently, there has been much work by the plastics industry, particularly the pipe specification sector, to introduce more objectivity into the rating procedure and in devising a system which involves an actual count of the number and size of each agglomerate (ISO 18553).

Regardless of the assessment method, it must be understood that the let-down technique used cannot possibly coincide with every set of process conditions/resin systems used by every masterbatch user. Hence, good dilution cannot necessarily be guaranteed. By its very nature, the press-out technique applies some "work" to the compound and this conceivably could have a minor influence on the dispersion rating. For this reason, there are a few purists who prefer to assess the microscopic dispersion using a microtome section technique. Microtoming has the added advantage that larger sections (eg the full cross section of a pipe) can be examined. The main disadvantage of this method is that it is very time-consuming and also requires a great deal of expertise in the actual sample preparation. For this reason, it is not very commonly used.

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Macroscopic techniques

The so-called macroscopic methods for assessing dispersion used by masterbatch producers are designed to duplicate as closely as possible certain application processes. They include:

Film Extrusion: blowing a film then counting the number of agglomerates in a specified area.

Profile Extrusion: extruding a pipe or a tape and then counting the number of surface "pips".

Screen Extrusion: extruding a quantity of masterbatch through a screen pack (60 mesh to 325 mesh) purging clear resin, then counting any agglomerates retained on the screen (a modified version of this test is the DELTA P test which uses a pressure transducer to monitor the increase in pressure as the screen becomes blocked).

Tinting Strength: tinting strength methods are still commonly used for assessing dispersion. This involves adding a given quantity of carbon black masterbatch to a standard white compound in a mixer or injection machine then checking the resulting grey tint, either visually or instrumentally, against standard references.

Masterbatches of poor quality can be produced due to:
- inadequate dispersion of good quality carbon black can occur because of poor mixing capability or poor black/polymer combinations;
- the use of poor quality carbon black which may have had high grit content, high ash content and possibly high moisture content;
- the use of low quality polymer with high gel content and possible polymer degradation;
- the use of very coarse fillers/extenders;
- contamination of an extraneous nature which inadvertently enters the system, e.g. paper from paper bags, wood splinters from pallets, metals from various sources.

Experience has shown that a high proportion of so-called dispersion problems occurring with masterbatches have, in fact, been the result of incorrect choice of masterbatches for given applications, in other words, **a masterbatch application mismatch.** This mismatch can arise due to a reluctance on the part of the customer to disclose specific process related information. Many situations have been observed where, because of incorrect masterbatch selection, the masterbatch user is using more masterbatch than is needed, is using a more expensive grade than necessary, or is compromising product performance.

In conclusion, it should be stressed that the more masterbatch users involve masterbatch suppliers in optimising dispersion and dilution of pigments and additives for their specific problems, the fewer problems they will experience.

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Cabot (headquarters)
Interleuvenlaan 5
B - 3001 Leuven
BELGIUM
Tel.: (32) 016.39.24.00
Fax: (32) 016.39.24.44

Cabot Plastics Belgium S.A. (plant & sales office)
Rue Prevochamps 78
B - 4860 Pepinster
BELGIUM
Tel.: (32) 087.39.27.11
Fax: (32) 087.46.98.69

Cabot Plastics Deutschland (sales office)
Tel.: (0800) 182 40 86
Fax: (0800) 182 40 87

Cabot Plastics Ltd (plant and sales office)
Gate Street
Dukinfield SK16 4RU
ENGLAND
Tel.: (44) 161.934 4500
Fax: (44) 161.934 4501

Cabot Italiana S.p.A. (plant)
Plastics Division
Zona Industriale 4
I - 38055 Grigno (TN)
ITALIA
Tel.: (39) 0461. 775 211
Fax: (39) 0461.765.146

Cabot Latin America Division (sales office)
Av. Joao Castaldi 88
04517-900 Sao Paulo, SP
BRAZIL
Tel.: (55) 11.5091 8300
Fax: (55) 11.5542 6037

Cabot Plastics Hong Kong Ltd (plant & sales office)
18 Dai Kwai Street
Tai Po Industrial Estate/Tai Po (NT)
HONG KONG SAR
PRC
Tel.: (852) - 2666-2666
Fax: (852) - 2666-0807

Cabot Shanghai Liaison Office
Rm 606-617, Shanghai Central Plaza
381 Huai Hai Zhong Road
200 020 Shanghai
CHINA
Tel.: (86) 21-538 20 534
Fax: (86) 21-538 27 782

http://www.cabot-corp.com/plastics

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