PROCESSING MICA BASED PIGMENT MASTERBATCH

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Abstract

Many effect pigments used in thermoplastics such as those used for pearlescent appearance are comprised of coated mica crystalline platelets. For effective coloration of thermoplastics the mica structure should not be altered. If the mica platelet is reduced in size while being compounded into the plastic it will alter the appearance of the plastic. It is desirable to maintain the mica structure in order to produce the best possible appearance of such pigments. To avoid attrition of the platelet structure exposure to shear during processing should be avoided, particularly when compounding high concentrations of pigment for masterbatch production. Co-rotating twin screw extruders are used for the compounding pigments into thermoplastics. Typical kneading blocks have predominantly been used with this equipment for the compounding of pigments for many decades. The mixing properties of this element include compression and simple shear which causes damage to the mica structure. Avoiding compression through the use of elongational mixing can significantly reduce damage to the crystalline structure of mica and enhance the appearance of mica based pigments. New mixing alternatives are available today that eliminate the shear peaks that are characteristic of typical kneading blocks, and utilize highly effective and efficient elongational mixing for the dispersion and distribution of mica based pigments while preserving the platelet structure.

Compounding of effect pigments

There are four distinct and important steps for the incorporation of effect pigments into a thermoplastic.

- Wetting
- Dispersion
- Distribution
- Stabilizing

The precise implementation of these steps is necessary to protect product quality and ensure an effective and economical product 1.

Wetting

Wetting of pigments prior to dispersion is a very important step, particularly for masterbatch production where high volumes of pigment are used. The importance of pre-wetting the pigment prior to dispersion is often not considered by masterbatch producers. If high loadings of pigments are exposed to elevated shear rates without pre-wetting then particle-to-particle friction will occur. The dry pigment will be compressed so that the particles will abrade against each other often at extreme pressures. This results in attrition of the platelet structure physically damaging the pigment. This also results in very high localized temperatures which can cause oxidation and/or discoloration of sensitive coatings on the mica.
For effective wetting the compression of raw pigment should be avoided. Mixing should be through elongation and laminar means in a non-filled process section. If dry powder becomes constrained prior to wetting the compression of the dry particles can occur resulting in attrition of the mica structure. Elongation in a non-constrained (filled) environment is a very effective and efficient means of mixing. As the melt pool is stretched tensile forces gently pull the pigment particles away from each other. Surface renewal through frequent splitting and recombination of the melt pool is also a highly effective method for low energy mixing and wetting of pigment particles. Special mixing elements are available that accomplish this task during conveying through special features that can include pins and grooves, however retain a fully intermeshing and self-wiping characteristic.

Effective wetting also occurs in the intermeshing zone between the adjacent rotating screws as the melt pool is transferred from one rotating shaft to the adjacent shaft. As the melt pool is transferred from the one screw to the next the melt folds or rolls enhancing surface regeneration. The resin pool is then quickly stretched into the root of the receiving screw creating effective elongation. To increase this mixing effect shorter lead angles for conveying and mixing elements should be used to increase the frequency of folding.

Element are available for effective pre-wetting of pigment through a combination of elongation and surface renewal

To improve wetting capability and reduce energy requirements for such operations it is advised to keep melt temperatures high and melt viscosity low prior to adding the pigment, particularly with high volumes of pigment. The (room temperature) mica will need to be heated which will inherently reduce resin melt temperatures and increase viscosity. If moisture or other volatiles are present the drop in melt temperature will be greater. At a higher viscosity more energy will be required to transport and wet the mica which could cause damage to the pigment.

**Dispersion**

Shear peaks, cause and effects

Most modern twin screw extruders utilize two lobe kneading blocks for energy input to facilitate dispersion. The fully intermeshing and self-wiping kneading block was patented by Rudolf Erdmenger in 1949 and has changed little over the decades. The kneading block (KB) has been the predominant element used for kneading and mixing in a TSE for over 60 years. The appearance and performance of the kneading block has remained largely unchanged since its inception.
A negative attribute of bi-lobe kneading blocks pertains to the existence of shear peaks during the compression of melt as one lobe of a kneading block intermeshes with the lobe of the kneading block on the adjacent shaft. During this occurrence up to 3% of the melt experiences a very high shear environment as it is exposed to extreme compression that forces materials between the two lobes at the tips of the barrel apexes. Material passing through this environment will experience a significant amount of shear energy input and high temperatures from frictional heating. If the solids content within the resin is high, as is experienced in masterbatch production, additional abrading of the mica can occur from particle-to-particle friction.

These shear peaks cannot be avoided, and exert a non-homogeneous transmission of energy into the polymer. Some of the polymer/mica will be exposed to extreme pressure and shear while other pools of polymer/mica may pass through the kneading blocks and experience little energy input. This can have many negative effects, such as a significant spike in melt temperatures within the intermeshing zone, excessive mean melt temperatures from inefficient energy transmission, degradation of shear sensitive materials, degradation of fibre reinforcements or friable fillers, and consumption of energy that could be better used for other functions.
Shear rate for bi-lobe profile at 400 rpm, the very high shear peak represents the small pool of melt passing between the intermeshing KBs.

Controlling energy

In recent years melting and mixing elements have been developed that significantly reduce or eliminate shear peaks and still maintain a fully intermeshing and self-wiping characteristic. These elements can be designed for task specific functions such as dispersion, kneading, distribution, etc. Since shear peaks are reduced/eliminated the energy input into the polymer is much more controlled, improving both efficiency and effectiveness. Different element geometries facilitate a controlled energy input depending on the needs of the task being performed. Simple shear for the reduction of cohesive aggregates and agglomerates, planar shear for high energy dispersion, low energy elongation for materials that are relatively easy to disperse, etc.

Mixing elements are available that eliminate shear peaks

In the design of the mixing elements the intensity of energy input and reduction/elimination of shear peaks are controlled through the use of multiple lobes (>2) as seen in the cross sectional geometry. As the lobe count increases the melt becomes more constrained which increases energy input. Axial geometry is varied to enable specific forms of energy input. These attributes enable the precise control of energy input and form (planar shear, elongation, etc). To reduce the attrition of mica pigment low energy dispersive mixing should be utilized. Mixing through elongation is the most efficient form of mixing when response is compared to energy input. Low energy elongation in and unconstrained environment (unfilled, no pressure) was described above for wetting. When elongation is utilized in a low pressure constrained environment (no shear peaks) very effective dispersion of pigments can occur. Energy transfer for dispersion can be controlled through the amount of constraint within the
mixing elements. Eliminating shear peaks and improving mixing efficiency prevents pigment degradation.

Cross sectional geometry manages energy input, axial geometry manages the mode of mixing

**Distribution and stabilization**

Distributive mixing should take place to ensure a homogeneous distribution of effects pigment within the resin matrix. Uniformity of pigment dispersion is necessary to prevent any anomalies in the appearance of the molded parts. Many of the newer generation mixing elements described above have inherent distributive mixing characteristics as well as dispersion, eliminating the need for a separate distributive mixing function within the extruder process section.

If a separate distributive mixing function is required there are many effective distributive mixing elements available. Some are comprised of blade like structures or “gears” projecting off a cylindrical core. Distributive mixing characteristics of such elements are highly effective, however at higher screw speeds it is possible to damage mica pigment through impingement as it passes between the blades. Most of the effective distribution elements do not have fully intermeshing and self-wiping characteristics, which can allow resin to accumulate in the un-wiped section of the elements and degrade. Char formation can occur in such regions and can re-enter the resin pool after degrading creating a color body, or black speck, within the resin. This is particularly true with heat sensitive plastics. Since the use of screen filters is usually not recommended for mica pigments (large platelet structure of mica, high and damaging extruder screw energy input to overcome pressure drop) the contamination cannot be removed from the plastic. The presence of color bodies will result in a higher reject rate of the final molded part.

Typical distributive mixing elements
Distributive mixing elements are available that exhibit fully intermeshing and self-wiping characteristics. Some of the elements are designed to facilitate distributive mixing characteristics while generating pressure. It is possible to use these particular elements in the metering (discharge) process section just prior to the die. Another beneficial attribute of these elements is to facilitate melt homogenization prior to discharge from the extruder. This can improve strand stability if a stranding system is used, or dimensional accuracy if direct shape or form extrusion is to occur.

Elements are available to enable distributive mixing in the metering section

Stabilization of the pigment is important for the production of coatings however is not an issue with thermoplastics. The pigment distribution is frozen during stranding or die face pelletization.

Extruder selection

The shear signature of a co-rotating extruder can vary significantly between suppliers and models. Shear characteristics are influenced by the geometry of the extruder and the dimensional tolerances that exist within the extruder.

The root depth of the extruder screw can determine how much of the melt pool is exposed to the barrel wall of an extruder during compounding operations. Root depth is expressed as a ratio of the outer diameter of the screw as compared to the inner diameter, expressed as Do/Di. A deeper root depth, or larger Do/Di ratio, draws more of the resin into the screw root and away from the shear forces that occur where the melt contacts the barrel wall. Shear is created where the melt within the rotating screw contacts the stationary barrel wall.

A deeper flight depth exposes less of the melt to the high shear that exists where the rotating screw meets the barrel wall.
Perhaps more damaging to effect pigment is the shearing that can occur when plastic containing the pigment “leaks” back into the trailing screw channel. This occurs when the screw-to-barrel or screw-to-screw dimensions are large enough to allow leakage of the polymer. For example the OEM tolerances for screw to barrel clearance can range from 0.15mm to 2mm or more depending on the supplier and model of the extruder. Effect pigment passing through these high shear regions can suffer from attrition. It is possible to have tolerances so small that leakage will only occur under high pressures.

Dimensional tolerances within the process section differs between extruder manufacturers and models

Other considerations

Generally the throughput in the production of masterbatches with mica pigments is less than expected because these pigments have a (bulk) density similar to inorganic materials such as chalk or talc. This can reduce the feeding capability of side feeders and limit the feed rate of the pigment. This will result in a lower production line rate and loss of productivity. It is generally not desirable to use two side feeders since some of the pigment will see more work and damage than pigment introduced through the second side feeder, and a longer process section will be required. Residence times within an extruder increase as throughput decreases. Running an extruder with a low fill factor will increase the time that the effects pigment remains in the extruder, and the amount of damage that can occur to it. Densified, or agglomerated, mica is available to improve handling and feeding, however also at higher costs. These products may have other processing issues related to their agglomerate form.

Transport elements are now available for side feeders that significantly increase throughput capabilities while reducing attrition that can occur with standard conveying elements when raw pigment is trapped between the trailing flank of one conveying element and the leading flank of the adjacent element. These high capacity elements possess a negative angle on the leading flank which captures large volumes of powders and forces them forward. Standard elements have a positive angle that can push low density powders up and away from the screw through impingement and centrifugal force. These high capacity elements have demonstrated a 3X to 5X increase in feeder capacity with low bulk density powders, even higher with powder lubricants.
Conveying elements are available for side feeders to significantly increase throughput.

**Conclusion**

Effect pigments are increasingly being used in thermoplastics to produce products with brilliant and aesthetically pleasing appearance. Because of the sensitivity of the mica structure damage occurs to the pigment when compounding them into plastics, particularly during masterbatch production where high volumes of free powders are introduced into the extruder. Pre-wetting of the pigment is particularly important prior to dispersion. Effective wetting is achieved through the use of proper mixing attributes, including elongation, laminar, and surface renewal, and by maintaining a low melt viscosity. For dispersive mixing the use of standard two lobe kneading blocks should be avoided due to their characteristic shear peaks and non-homogeneity of mixing energy input. Excessive energy input and damage to mica pigment cannot be avoided. Product quality can be improved through the utilization of fully intermeshing multi-lobe mixing elements that significantly reduce or eliminate shear peaks and possess highly efficient task-specific mixing functions. Pigment distribution, or homogeneity, is important to ensure that there is a uniform surface appearance in molded parts. Many of the newer mixing elements also possess excellent dispersive mixing characteristics so that a separate distribution process section is not required. If additional distributive mixing is needed then elements with fully intermeshing and self-wiping characteristics should be used to prevent stagnation and degradation of the resin. This can cause color body formation (black specks) that will result in a higher reject rate of the final molded part.

Extruders possess different shear signatures depending on make and model. Selecting an extruder with a lower shear signature can reduce pigment damage.

Low bulk density can reduce the capacity of side feeders used to introduce the pigment to the polymer melt. This can cause the extruder to be operated below design capacity. Side feeder elements are available where the leading flank has a negative angle (vs. positive on standard element) that forces high volumes of powders into the side feeder, thus eliminating line rate restrictions.

Utilizing the technologies detailed above can improve product quality and manufacturing productivity.

**References**

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