NOVEL LIQUID-DISPERSION TECHNOLOGY FOR MAKING INDUSTRY LEADING HIGHLY FILLED, WELL-DISPERSED MASTERBATCHES

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Abstract

A novel method for making highly filled masterbatches (MBs) has been developed by Interfacial's Development Services Division and now employed commercially by Interfacial. This patent-pending approach has set a new standard for the development and production of the market's highest filled MB materials. The manufacturing method consists of three-steps: pre-coating of mineral or filler, removal of liquid carrier, and finally pelletization. To date, Interfacial has designed, developed and manufactured using this technology MBs consisting of 93+ wt% talc, 93+ wt% calcium carbonate, 90+ wt% wood, 85+ wt% cellulose fiber, and 37+ wt% carbon nanotubes in polyolefin, polyamide, and polyester carriers. Furthermore, it has been shown that these MBs are compatible and disperse extremely well in the main plastic.

Introduction

Industrial manufacturing of polymeric MB relies typically on large-scale and continuous processing methods. Twin-screw extrusion (TSE) and continuous mixers (CM) have been established as two of the most prominent commercial techniques to process MB due to the number of advantages; flexibility, versatility, high throughput, and low maintaince.[1,2] Although successful for processing MB materials, these processing techniques are still limited in terms of creating highly filled, well-dispersed systems that are cost-effective due to basic physics, which include thermodynamic barriers to achieve desired morphologies and viscosity mismatch. There exists a need for a commercial polymer processing technique that can not only manufacture MB with higher filler loadings and enhanced dispersion but also produce these materials at a lower cost. This paper describes a novel method that utilizes a liquid based dispersion system to achieve industry leading highly filled MBs at a cost-competitive price.

Equipment

In this study, a liquid based dispersion technology was utilized to achieve highly filled MBs. Figure 1 shows an image of patent pending liquid dispersion technology.

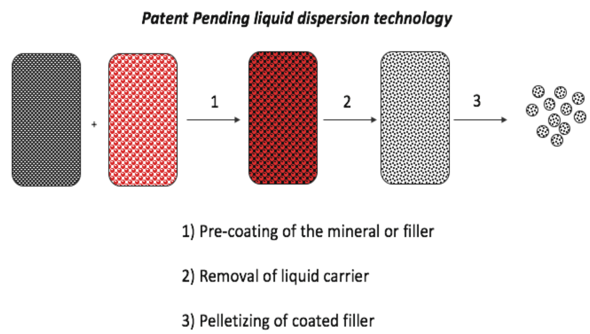

Figure 1. Schematic of liquid-based dispersion technology

From Figure 1, there are three basic steps involved. The first step incorporates pre-coating of the mineral or filler with the liquid dispersion. Following this step, the liquid carrier is then removed and finally, the mixture is pelletized into the final MB pellets. All the MBs were made utilizing this technique. The let-downs were manufactured using a standard 27 mm co-rotating twin-screw extruder in various resins.

Materials

Cellulose fiber is sourced from West Fraser, headquartered in British Columbia. Multi-walled carbon nanotubes (MWCNTs) were purchased from Nanocyl. Polypropylene was purchased from Ineos (H35G-00 PP).

Results and Discussion

Highly filled cellulose fiber masterbatches

Biobased composite materials have been extensively explored as an environmentally friendly, sustainable alternative to materials solely based on petroleum feedstocks.[3] Interest in such materials has increased in recent years due to the need for sustainable products and desire for companies to reduce carbon footprint. Through this dispersion technology highly filled polyolefin and polyvinyl chloride (PVC) biocomposites have been developed to meet the growing sustainability needs and green initiatives of many companies, while meeting the

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performance requirements of engineered applications in the automotive, consumer, and industrial markets. Figure 2 shows SEM images of an 85 wt% filled cellulose/ polyolefin MB.

Figure 2. SEM image of 85 wt% filled cellulose/ polyolefin biocomposite masterbatch

It is evident from Figure 2 that this proprietary manufacturing process yields highly isotropic MBs with good dispersion as well as maintaining an aspect ratio of >10:1. Due to these findings, the cellulose MB allows for excellent processing, coloring, and surface finishing characteristics in the final let-down products. Furthermore, because the MB are made from a high quality, continuous renewable supply of cellulose from West Fraser, the let-down materials show a higher modulus as compared to competing biocomposites on the market. It should also be noted that this novel processing method is cost-effective, which makes them market leading in terms of a price-performance perspective.

Highly filled carbon nanotube masterbatch

Commercially available multi-walled carbon nanotubes are heavily entangled (clusters of > 1 mm in size). Figure 3 shows an image of as-received MWNCTs from Nanocyl.

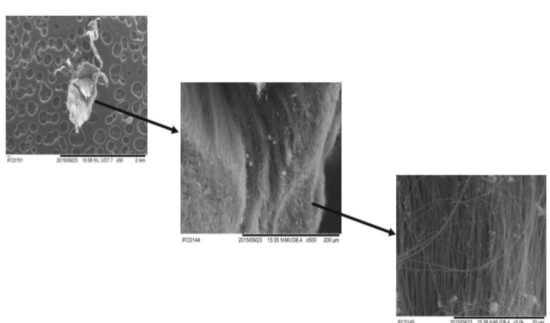

Figure 3. SEM image of commercially available MWCNTs (various size scales)

The key challenge to effectively utilize this material is how to disentangle and disperse using conventional processing methods. Interfacial has developed a commercial liquid based dispersion technology method, now being utilized by Interfacial, to achieve over 37 wt% MWCNT loading with no "bleed-out" effect. Figure 4 shows SEM images of a surface and cross-section area of a MWCNT MB pellet.

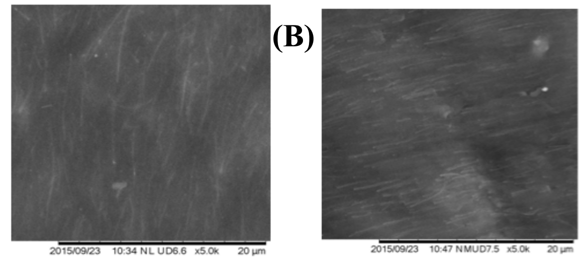

Figure 4. SEM image of a) surface of MWCNT MB pellet, and b) internal cross-section of MWCNT MB

To the best of the authors knowledge, this is the highest loading of competing MWNCTs in the industry (typical MBs are 15 wt% or less). Another important aspect of this dispersion technology is that the MWCNTs can be let-down and dispersed in virgin polyolefins (and other resins) while maintained a high length to diameter ratio of greater than 100, which leads to an extremely low electrical percolation threshold. Figure 5 shows the surface and volume resistivity of several MWCNT MBs processed with virgin PP. It can be seen from Figure 5 that the percolation threshold is reached with 6 wt% (or ~2 wt% MWCNT) of the MWCNT MB.

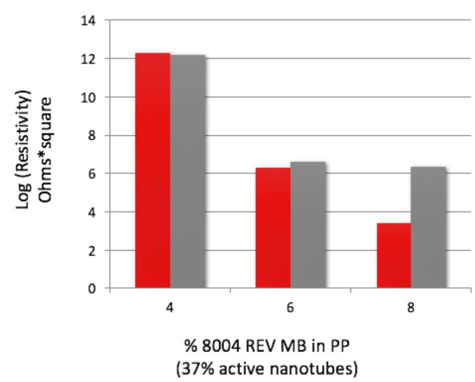

Figure 5. Surface (red) and volume (gray) resistivity of a PP/MWCNT nanocomposite made from let-downs of the MWCNT MB

When compared to carbon black (CB), a commonly used additive for electrical conductivity, this is about 10 times less material needed to achieve the same electrical performance. If one compared the price per amount of material needed to achieve the percolation threshold, the MWCNT MB are approximately the same compared to CB powder. However, due to the fact that one needs 10 times less, other performance properties are significantly improved, including mechanicals and rheology in the end MWCNT polymer product.

Other highly-filled masterbatch systems

In addition to cellulose fiber and MWNCTs, this proprietary process allows for the manufacturing of other highly-filled MB materials. Table 1 gives a list of materials that have been manufactured on a commercial scale with the percent loading.

Table 1. Current MB materials utilized

Carrier	Additive	% loading
Polyolefin	Talc	93
Polyolefin	Calcium Carbonate	93
Polyolefin	Wood	90
Polyolefin	Carbon Nanotube	37
Polyolefin	Halloysite	90
Polyolefin	Glass Microsphere	50
Polyolefin	Wollastonite	90
Polyolefin	HAR Talc	90
Polyolefin	Cellulose Fiber	85
Polyolefin	Lightweight Talc	90
Polyolefin	Boron Nitride	93
Polyamide	HAR Talc	90
Polyamide	Wollastonite	90
Polyvinylchlorid	Cellulose	85
Polyester	Calcium Carbonate	95

Another interesting opportunity with these MB is light-weighting. As shown in Table 1, this liquid based dispersion can yield a MB that has 50+ wt% glass microspheres. Figure 6 illustrates that this MB can be blended with PP to significantly drop the specific gravity (> 10% reduction). The key to achieving such of a reduction is the ability to preserve the spheres during the production of the MB. Ultimately, this demonstrates another advantage of the liquid based dispersion technique.

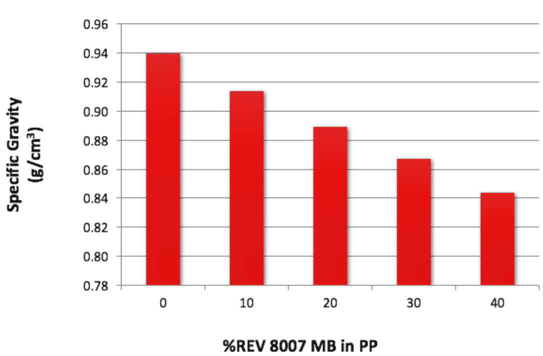

Figure 6. Image of specific gravity as a function of wt% glass microsphere in PP

Other benefits to dispersion technology

In addition to achieving new, industry leading approaches to the development and production of the market's highest filled MBs, there are several major benefits that have been shown through this liquid dispersion technology. They are as follows:

- Excellent dispersion of MB in main resin leading to enhanced properties
- Eliminate shipping of hazardous powders by making highly filled pellets (ease of handling)
- Lower cost MB due to effectiveness of processing method
- Preserve structural integrity of additives and minerals, leading to better end properties as compared to competing products
- Platform technology that can be applied to many other additives and/or fillers
- Low temperature process that eliminates degradation of heat sensitive additives and/or fillers

Conclusion

This paper describes a novel liquid dispersion technology for making industry-leading highly filled, well-dispersed MB. To date, there are over 20 MB and let-down products on the market that have already demonstrated their effectiveness in final products. Further developments will investigate the production of highly filled MB in other carriers that include nylons, polyesters, and other higher temperature engineered resins.

Acknowledgments

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Key Words: Dispersion, multi-walled carbon nanotubes (MWCNT), cellulose, masterbatch