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Technical Paper

Colorants And Their Unique Relationship With Fillers In Cast Polymer Products

Abstract

Pigments and fillers share a unique and important relationship in the coloration of cast polymer products.. The color of cast polymer products can be influenced by many factors during the formulation and manufacturing stages. Raw material types, manufacturing methods, and specific pigment chemistries all contribute to the final color and visual appearance of the final product. This paper explains and investigates some of the capabilities and methods available for troubleshooting and preventing potential color issues associated with thermo-set cast polymer products.

Introduction

Cast polymer products are typically made from a thermosetting polyester resin, catalysts, and common fill-ers such as calcium carbonate or alumina tri-hydrate. Pigments (or colorants) are incorporated to match differ-ent ranges of colors. There are other specialty hybrid fillers that are lower density and have improved final thermal shock properties. Filler selection is normally based on the optimum cost, or in other cases the nature of the performance environment.

Many generally focus on the resin and the pigment system when it comes to final coloration effects. While the color and consistency of color of resin is important, the other building blocks in the formulation have a major color contribution to the finished part.

Popular colors are range from bright whites to light pastel, and other lighter color families. For example, colors such as glacier white, biscuit, and bone are quite common. Many of these formulations contain blends of pigments, with the majority component usually based upon Titanium Dioxide (also abbreviated as TiO_2).

Objective

Since titanium dioxide is present in many formula-tions using fillers previously listed, we set out to under-stand how the pigment formulations could be optimized based on some of the inherent properties of some com-monly available fillers or cast polymers.

The intent was to measure the lightness value (L*) of the dry fillers, and also when the fillers were wetted at high concentrations in a low color (APHA 10 maximum) resin system. Once that data was collected and exam-ined, two fillers (one alumina trihydrate, one calcium carbonate) with lower matrix L* lightness values were combined with titanium dioxide to study the change in lightness with stepwise increases of pigmentation levels.

Experimental

Dry L* Value (Dry Filler Only)

The lightness (or brightness) of a dry filler is normally reported as a dry brightness value. Filler sup-pliers use this tool to report brightness of their fillers, and values in the 88+ range are common. When this test is used for different lots of fillers, the supplier can ensure that lot-to-lot consistency is closely monitored. Other industries such as inks, rubber, paint, coatings, and plas-tics deal with similar quality specifications; this method continues to be the most popular test method for dry fillers.

Dry Test Method

While a supplier may use a special optical cell or apparatus for measuring brightness or lightness, a simple method to judge the dry brightness of the filler is to transfer a small portion into a small polyethylene bag., using approximately the same amount for each sample. Once this is done, a visual comparison can be made by placing the different sample bags side by side. Laboratory testing indicates that the visual judgment and measurement values are very close as long as the plastic bag type and thickness are not varied.

After the polyethylene bags were filled, a computer study was initiated to measure the L* (or the lightness) values.

Matrix L* Value (Filler Wetted with Resin)

One useful technique given here for considera-tion is the measurement of the unpigmented matrix brightness. It is simply a measurement of the filler in the wet matrix state, and a tool to use in conjunction with dry brightness.

The reason for this is that the final color contribu-tion of a filler cannot always be directly determined by dry brightness values. When the filler is wetted with a resin, there are other potential color attributes of the filler, even visually detectable shifts to red/green tints or yellow/blue tints.

Matrix Test Method

For these experiments, we chose a clear un-promoted resin. While it is common knowledge that the promoter, catalyst, and cure schedule will affect the neat and cured color of a cast polymer part, we elected to re-duce the number of variables by using a resin with an APHA color of 10 maximum for testing purposes.

To incorporate the fillers, 1000g (total weight) resin and filler matrix mixtures were charged into a Kitchen-Aid planetary mixer. For calcium carbonate fillers, we chose a filler level of 77.5%, with the balance of the formulation being resin. For aluminum tri-hydrate fill-ers, a filler level of 65% was chosen, with the balance of the formulation being resin.

Wet samples of the matrix were placed in polyethylene bags were filled for visual comparison. Again, a computer study was initiated to measure the L* (light-ness) values

Pigmented Matrix L* Value

Once the dry brightness and resin/filler matrix brightness studies were completed, we chose to make a study of the effect of pigmentation versus changes in brightness and color. The experiments were designed to show the effect of pigment and its ability to change the L* lightness value when combined with the overall color body of the resin/filler matrix.

It is fairly common in our industry to use "ladder" type studies, where a variable, in this case the pigment, will be tested in a step-wise fashion. We know that generally these usage levels of Titanium Dioxide are below 3.0 percent based on the matrix weight, but generally above 0.5 percent. Thus a ladder study was initiated at levels of 0.5 to 3.0 percent, in increments of 0.5 percent.

The pigment used in these studies was a liquid dispersion of Titanium Dioxide (75% active solids). We started with unpigmented matrix mixture and added 0.5% pigment dispersion. After the mixing process was completed, samples were retrieved from the mixing vat and placed in polyethylene bags. Samples were taken when the pigment loading equaled 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 percent.

A computer study was initiated to measure the brightness and overall color properties of the pigmented matrix mixtures. Figure 3 has a listing of the Calcium Carbon-ate fillers. Table 4 has a listing of Alumina Tri-Hydrate fillers, and Table 5 has a listing of the hybrid fillers.

Decoding L*-a*- b* values

- This three-axis system is used by various industries (especially the coatings and plastics segments) to communicate color and/or color differences. (For the testing presented in this paper, the L* axis will be the primary axis for measurements and discus-sion).

A simple representation of this system comprised of the three axes L*, a*, and b*.

- L*: (Light-Dark) Higher L* values would be attrib-uted to a lighter color, and lower numbers would be attributed to a darker color.
- a* : (Red-Green) Higher a* values indicate that the matrix color is more red (may appear "pink") ; lower a* values indicates that the matrix color is more green.
- b* : (Yellow-Blue) Higher values for b* indicate that the matrix color is more yellow, lower values for b* indicates that the matrix color is more blue.

Figure 1 illustrates the L* a* b* coordinate system.

Results and Discussion

The dry L* lightness measurements for alumina tri-hydrate ranged between 99.2 (filler A40) and 99.8 (filler A50) when measured in the polyethylene bags. The dif-ference between the highest and lowest alumina tri-hydrate dry values was 0.6 units.

The matrix L* lightness measurements for alumina tri-hydrate ranged from 90.8 (filler A20) and 95.9 for (filler A50). The difference between the highest and lowest alumina tri-hydrate matrix values was 5.1 units

Figure 2 illustrates the differences in dry versus matrix measurements for the alumina tri-hydrate fillers.

The dry L* lightness measurements for calcium carbonate ranged between 96.7 (filler C35) and 98.0 (filler C25) when measured in the polyethylene bags. The total difference between the highest and lowest cal-cium carbonate dry values was 1.3 units.

The matrix L* lightness measurements for calcium car-bonate ranged from 86.9 (filler C35) and 91.7 (filler A50). The difference between the highest and lowest calcium carbonate matrix values was 2.8 units

Figure 3 illustrates the differences in dry versus matrix measurements for several calcium carbonate fillers.

The pigmented alumina tri-hydrate matrix exhibited a leveling effect with respect to L* lightness when the pig-mentation level approached 1.5 percent based on the ma-trix weight. Addition levels above 2.0 percent did not raise the L* lightness value significantly, which leveled near a L* lightness value of 98.0.

The case for pigmented calcium carbonate matrix was somewhat different. The L* lightness value continued to increase slightly, even after the 1.5 percent level. In fact, the change from 1.5 percent to 3.0 percent raised the L* lightness value over 1.6 units. At 3.0 percent, the L* lightness value was 95.6 units.

Conclusion

It has been observed through laboratory and field experi-ences that dry measurements, while very useful as a quality control monitoring tool, cannot always predict the final matrix mixture properties when considering the multitude of raw material types, including resins, cata-lysts and pigments.

It is indicated from the experimental data that when the filler is wetted with resin, the resulting matrix mixture color is not directly linked to the dry brightness value.

All of the common filler types tested in the dry state were very close in dry L* lightness values. Color differ-ences could be seen when the matrix mixtures were measured.

In general, the calcium carbonate tended to have lower matrix L* lightness values when compared to the alu-mina trihydrate fillers. The change in L* lightness val-ues tended to be unique for the filler type and grade.

The pigmented alumina tri-hydrate matrix exhibited a leveling effect when the pigmentation level approached 1.5 percent based on the matrix weight. The case for pig-mented calcium carbonate matrix was somewhat dif-ferent. The L* lightness value continued to increase slightly when tested above the 1.5 percent level.

Tests such as these offer potential tool when working with white and pastel colors where the pigment level can potentially be adjusted due to the inherent properties of the filler in the matrix.

Acknowledgments

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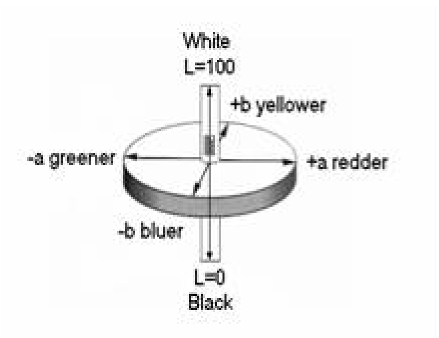
Figure -1



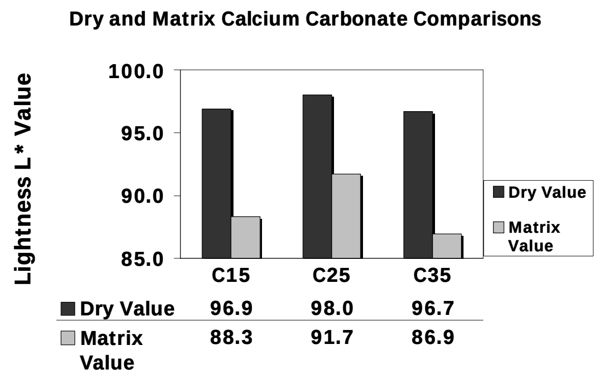


Figure – 2

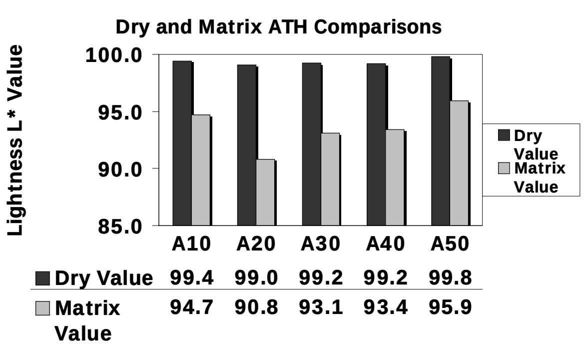


Figure – 4