dispersions of color pigments

In the rubber and plastic industries as well as related fields, most colorants used are classified as "pigments." Pigments are defined as those colorants that are insoluble in the medium in which they are used. This is in contrast to "dyes" that are colorants designed to be soluble in the final substrate. Most color pigments are manufactured to an extremely fine particle size. This ultimate particle size is somewhat irrelevant to dispersion in rubber and similar materials. This is because pigments in everyday life do not exist as individual particles but as aggregates and agglomerates of particles. Good dispersion of color pigments depends on the breaking up of these agglomerates into as small a size as required by product needs, both performance and economics. This **Solutions** *will discuss the various forms of color dispersions available from Akrochem. Each has both attributes (ultimate reduction in particle size; ease of handling) as well as negatives (cost; possibly more difficult handling) that need to be weighed when deciding on a color pigment dispersion.*

rubber-bound colorants

The most popular form of color dispersion in the rubber industry is rubber-bound colorants. These are made by mixing high concentrations of color pigments into a rubber carrier. An internal mixer or a rubber mill is used to generate sufficient shear to reduce the agglomerate size and predisperse the pigment into the rubber. This form allows easy handling; clean, easy weigh-up; no fly-loss of pigments; production equipment and personnel stay significantly cleaner compared to powders. Colored end products are more consistent with fewer "streaks" and "dots" of colorants that are common with powdered pigments (especially organic pigments).

We discussed rubber-bound color masterbatches in a previous **Solutions** issue concerning compounding of colors. You may want to review that issue briefly. We'll want to touch on several ideas presented there. The first is the elastomeric binder for colorants. Most standard line products are bound with either SBR or EPR. The SBR has good compatibility with general-purpose rubbers including NR, SBR, PBD, CR, and NBR. The ability of the SBR binder to crosslink is an advantage at times. In formulations requiring a lot of color, the dilution effect of excessive non-curable polymer like EPR (which has no double bonds to sulfur cure) can have an impact on set properties. Having the ability to cure the binder allows you to either add additional cure to maintain properties or back out some of the primary polymer. In most coloring applications this is not an issue since little color is normally used. However, the presence of double bonds in the SBR binder may also be a disadvantage.

The presence of double bonds in the SBR binder means the shelf life of a masterbatch will be more limited. To the vast majority of customers this minor limitation is never encountered. As long as storage conditions are reasonable (unreasonable would be a shed in Arizona where storage temperatures reach 140°F in the summer), most SBR-bound masterbatches will be viable for years. It is still recommended that inventory be turned over at least once a year. The other problem with a high-diene polymer like

SBR is the effect the double bonds can have on the cure of low unsaturated polymers like EPDM and butyl. The SBR binder will have little effect on an EPDM cure if kept to less than a part per hundred rubber (phr). As the SBR quantity rises above 1 phr, the cure preferentially works on the SBR instead of the EPDM. This can cause the rheology of an EPDM with SBR-bound colorant in it to be scorchy and the state-of-cure to drop due to there not being enough cure left to fully cure the EPDM portion. To solve the issues of both shelf life and diene compatibility, EPR is used as the other primary colorant binder. In the case of peroxide-cured stocks, either SBR or EPR will work.

EPR (ethylene-propylene copolymer with no diene) provides outstanding shelf life as well as good compatibility with saturated or low-diene rubber like EPDM, butyl, CPE, Hypalon, and EVA. Even exotic polymers like Aflas™ and FKM can use EPR as a color binder. Most FKM users unfortunately avoid rubber-bound colors and instead grind powdered pigment colors into FKM on a mill. This is exceedingly messy and often results in color dispersion problems. However, a few FKM users have found that an EPR-bound colorant has minimal effect on the final properties of their compound. This allows a cleaner mixing area, minimum contamination, good dispersion of color, and ease of handling. We recommend that, early in the development of an FKM formula, rubber-bound colors be used so qualification is done properly. Many FKM formulas go through lengthy approval procedures and requalification is out of the question. It is better to do the testing with masterbatches up front than get into a situation where you have to mix messy organic pigments into your rubber. Thick, colored pastes may be a better choice for FKM. Evaluating the color early in the development cycle also gives you an opportunity to evaluate how good the final color pigment dispersion must be in a part. If the part cross-section is small and dispersion is critical, special dispersions may be required. We will look at some ways to improve color dispersion in critical applications later in this **Solutions**.

Besides Akrochem's off-the-shelf color masterbatches, we also produce custom-color masterbatches. A rule of thumb: if a formula requires three or more colors (including white and black), a single, blended masterbatch of those colors will provide much more consistent batch-to-batch color results than weighing those items separately. It is simple logic that it is easier to weigh one rubber masterbatch accurately (and because the blend is at a larger concentration than individual colors would be, there is less need to be absolutely precise on a blend weigh-up) than it would be to weigh-up four individual colors. And don't think that four colors are unusual; it is surprisingly commonplace. The more exact a color has to be, the more likely multiple colorants have to be used to get that precise color. In addition to custom color blends, Akrochem makes many rubber-bound color masterbatches to other custom requirements.

While we always recommend you try to find an off-the-shelf colorant that will do the job (off-the-shelf means there are no lead times, product is always available; no minimum purchases are required as with custom masterbatches; costs are better due to the longer production runs of standard products), there are times when a special product is needed. Some examples of special polymers used to bind colors: halobutyls in medical applications; high-ACN nitrile for fuel-resistant applications; low-ACN nitrile for special solvation processes; natural rubber for thread and rubber bands; and silicone for silicone elastomers.

Other custom formulations that can be done for color masterbatches include:

- Various forms are available including pellets, chexs, and slabs. Pellets are very easy to handle and weigh. But they cost a little more and some mill mixers don't like pellets because they "pop" out of the mill nip. Slabs are the standard form. Slabs work well on mills but must be scissor-cut to weigh. Chex (1" x 1" x 1/4" squares) are a nice compromise – easy to handle and weigh, no

additional cost, and perform well on mills. Also, various ways to prevent sticking of the color MB's are available: standard clay dip, calcined clay dusting, calcium carbonate, as well as no parting agent at all. In the latter case, slabs are packed between polyethylene sheets. In addition, color MB's can also be precisely preweighed into batch inclusion bags.

- Super-soft masterbatches – while most of our color MB's are made to be soft for good dispersion properties, occasionally a compound will require a colorant that is extremely soft. Usually this is the case with foamed products, soft rolls, and soft premixed neutral masterbatches that have color added later. It is always better to add color early in the mix cycle of a compound. Shear will be at its highest, leading to better dispersion. Super-soft MB's are for those times where the batch has already been softened and no more mixing shear can be expected.
- The rubber carrier may have a thermoplastic quality to it. The product (usually in a pellet form) is very firm at room temperature to allow automated weigh-up without massing or bridging. Then, at normal processing temperatures, the viscosity drops to an easy-to-mix level.
- Masterbatches can be screened through a 150-mesh (approximately 100 micron) screen to eliminate any large aggregate particles that might remain after the initial dispersion. Screening is recommended for thin-walled, Class A-finish extrusions (primarily automotive) and critical sealing applications.
- Custom-color masterbatches can also be designed for the use of specialized pigment types. FDA pigments are one example. UV-resistant pigments are another. Keep in mind that the more specialized the pigment requirements get, the fewer choices there are for color matching. For example, you may have to compromise on the exact color in order to keep all ingredients acceptable for FDA 177.2600 (178.3297) regulations.
- The **ultimate** color dispersion in rubber is achieved by adding a concentrated colored paste to a rubber binder. As you will find as we discuss paste dispersions, this is the finest particle size to which color pigments can be reasonably reduced. The largest agglomerates of pigment in a paste are about 25 microns, maximum. Most pastes are in the 1-10-micron size. These paste masterbatches (many are NBR-based) are important to roll and printing blanket makers where a soft base stock makes thorough color dispersion difficult and absolutely no pigment particles can be present on the face of the product.

plasticizer paste color dispersions

Rubber-bound color dispersions use an internal mixer or mill to grind the pigment agglomerates down to a usable size. But the nature of this type of equipment only allows the machine clearances to get so close. Thus aggregate breakdown stops at this point. Many products require the maximum pigment size to be smaller. Also, many end-uses of colorants can't use a solid form of dispersion to carry the color into a product. For these applications, concentrated colored pastes are used.

Colored pastes are made by grinding colored pigments into a liquid carrier on a 3-roll ink mill. An ink mill uses very tight clearances to crush the pigment aggregates to a fine dust, which is then dispersed in the accompanying liquid. Typical Hegman dispersion is 7 – the exact meaning of the Hegman scale will be discussed in further detail. In brief, this means there are no particles larger than 10 microns. This kind of size reduction cannot be had with rubber mixing. Even when rubber is screened through a 150-mesh, there may be particles

of close to 100 microns passing through. This may be too large for a critical surface finish. Besides the excellent particle size, the paste allows color to be added to liquid systems like plastisols, solvent-based adhesives, cast urethanes, and LIM silicones as well as some solid systems like PVC compounding or soft rubber mixing.

Paste Viscosity

The viscosity of a colored paste can be important depending on the product to which the paste is added. Ideally a colored paste is similar in viscosity to a liquid end product while a paste going into a solid elastomer should be stiff for ease-of-handling and for economics (higher concentration of color pigment). Specialty additives may be added to a paste to move the viscosity up or down but the main criteria for viscosity is the pigment loading.

Here are a few rules of thumb concerning paste viscosity:

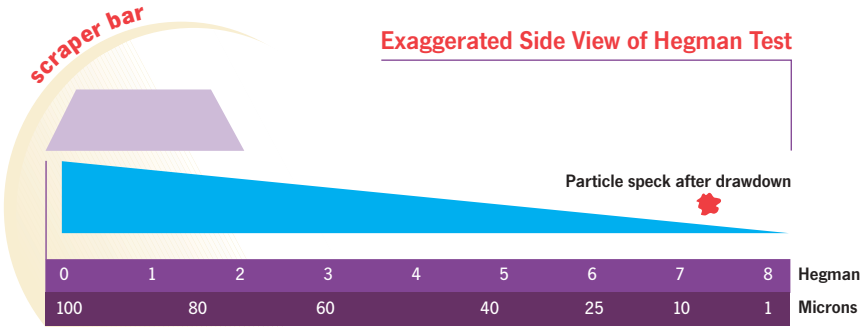
- The thicker pastes are easier to add to rubber compounds on a mill. Roll compounds using phthalate pastes or silicone gums using silicone fluid paste should use a heavy paste to help prevent the paste from squeezing out to the mill guides during mixing and creating a messy mix. Thicker pastes also help stop the rubber from "shredding" and chunking off into the mill pan. If all you have on hand is a thin, pourable paste color, use some easy dispersing filler to dry up the paste before or during addition to the mill (or internal mixer).
- Thicker pastes will reduce settling of pigment during extended storage. For this reason, thin pastes should always be stirred prior to weighing.
- A thick paste can be difficult to disperse in a low-viscosity fluid. A prime example is liquid urethane. A pourable color paste can be added directly to the system. A thick color paste will leave heavy "globs" of paste floating in the thin urethane liquid. Few customers have the high shear capabilities to adequately disperse these globs. Parts end up with dark spots of undispersed color paste. Some customers have in-house equipment that allows them to thin down a heavy paste but most customers would be better advised to request a thinner paste whose viscosity more closely matches the fluid to be colored.

Akrochem will customize your paste viscosity. Because most of our pastes are made-to-order, we can make the paste you need in almost any viscosity. By using certain surfactants, lower viscosity pastes can be made with the same concentration of pigment as a thicker paste. But be aware that if a very thin paste is needed, a lower concentration of color may have to be used. In most cases this is not a major issue since many liquid systems are translucent and, as a result, don't require much color.

Measuring Pigment Maximum Agglomerate Size

After a paste colorant has been made, a sample is taken and run through a "Hegman" test to measure the quality of pigment dispersion in a paste. It is a simple yet very indicative test. It consists of a metal plate with two precisely cut "troughs" running the length of the plate. The paste sample is added to the trough "deep end." The paste is then scraped along the length of each trough using the scraper bar. Each of these troughs gets shallower (see representation below) as it continues. If a pigment particle is too large to fit in the narrowing trough, a dot or streak will appear in the smooth face of the paste. The point on the Hegman scale where this first dot or streak appears

is the Hegman result. A Hegman reading is calculable to a particle size in microns. If no paste surface anomalies appear prior to a Hegman reading of "6," all the pigment particles are less than 25 microns. A Hegman of 7 means the maximum particle size is less than 10 microns. Typically, Akrochem pastes run 7 or better with a guaranteed 6 minimum.



types of liquid carriers for pastes

Akrochem disperses colored pigments in a wide variety of liquid carriers. Following is a brief look at the most common ones. Remember, the liquid carrier does not have to be perfectly compatible with the end product. As long as there is no cure interference or bleed, the paste can usually be used. The typical usage of most pastes is so low that absolute compatibility is not necessary. A good example is DIDP phthalate in EPDM. While DIDP will bleed from EPDM if 20 phr or more is used, the normal amount used for coloring purposes (0.5 to 5.0 phr) is well within compatibility limits.

DIDP

(di-isodecyl phthalate) plasticizer coloring paste is preferred in NBR rolls and printing blankets, PVC plastisols, and solvent-based adhesives. By not using solvents in the coloring, a non-hazardous paste can be made which reduces shipping, storage and health problems. DIDP is very soluble in almost all common solvents (glycols being one exception). DIDP also maintains FDA status under CFR 21, 177.2600 without the questions that surround DOP (dioctylphthalate). Speaking of FDA materials, if you need an FDA colorant, check with Akrochem Technical Service for an FDA-colored paste or rubber-bound masterbatch.

SILICONE

(dimethyl polysiloxanes) fluid pastes make excellent additives to color silicone gums as well as LIM silicones. They are 100% compatible with silicones and provide the same outstanding heat resistance of a silicone gum. It is extremely difficult to disperse pigment colors into silicone products. Using a thick color paste for mill operations and a thin one for liquid injecting molding allows easy addition of colors to silicones. The paste viscosity can be varied by altering the viscosity of the silicone fluid (100 centistokes to 60,000 centistokes are available) or by changing the pigment loading. FDA silicone fluid is also available.

CARRIERS for URETHANE PRODUCTS

Akrochem makes pastes in three different urethane-compatible fluids: A polyester that will co-cure in polyester urethane systems; a polyether paste that will co-cure in polyether urethane systems; and a low-moisture, dipropylene glycol dibenzoate (DGD) which is non-reactive and acts essentially as a urethane plasticizer or diluent. The polyester urethane can also be co-cured in epoxy coloring while DGD can be used as a non-reactive diluent in epoxies.

Many cast urethane producers prefer the DGD fluid since it can be used with either ester or ether systems. As long as the color levels are not too high, there is seldom a problem with the dilution effect of the plasticizer. However, in a few cases, the curable fluid has resulted in a more consistent product. This is usually formula-dependent, so trials should be run to see which fluid type produces the best parts. Most makers of liquid urethane products also prefer thin pastes to allow easier mixing with the polyol.

MINERAL OILS

Naphthenic, paraffinic, as well as "white" mineral oils have all been used as liquid carriers of colored pastes where a petroleum oil is preferred.

MISCELLANEOUS LIQUID CARRIERS

A wide variety exists of other liquid carriers that might be used to carry color into a product. These are typically very small-volume, niche items. Be sure to evaluate standard line carriers first before using a more "exotic" liquid. Other possible carriers might include:

Epoxidized Soybean Oil (ESO) and Trioctyltrimellitate (TOTM) are higher-performance plasticizers for PVC than the more commonly used DIDP. ESO has good heat stability and resists extraction by solvents or water. TOTM is excellent for high-temperature PVC.

Di (butoxy-ethoxyethyl) formal (better known by the tradename, TP 90B®) is used in extreme low-temperature applications.

Trioctyl phosphate is a phosphorus-bearing plasticizer for use in flame-resistant formulas.

* DRY PASTES *

To overcome one of the major disadvantages of colored pastes (the messy handling), Akrochem has developed a dry "paste" form of colorant. The color pigment has been ground to a paste-quality micron size and then impregnated on a dry filler. Dispersion is outstanding; handling is similar to a de-dusted powder; cost is similar to paste. These products are primarily for ease of rubber addition in critical applications. FKM coloring may find this product useful.

CUSTOM-COLORED PASTES

One of the real advantages to using Akrochem pastes is the possibility of customizing the colored paste.

- First, Akrochem's lab will determine (at no cost) a color match to any color desired by a customer. "Any color" has to be stated with some caveats: an exact match may not be possible if there are other pigment requirements that must be met, like FDA or UV-resistance. Also, if your base compound will not support a certain type of color, an exact match may not be possible. A good example would be metallic-looking pigments. If the base compound is not at least translucent, the metallic glint will be impossible to reproduce.
- We will design the pigment selection in a custom color to special needs such as UV-resistance or FDA uses.

- We'll put this custom color match in the liquid carrier of your choice and design a paste viscosity that fits your needs.
- We'll manufacture this custom color in quantities between 8 and 10,000 lbs.
- Please note that environmentally regulated pigments like cadmiums and lead chromates are not used in our normal production facilities. Nor is any solvent permitted. Akrochem will work with you to find a safer color pigment or a safer, compatible carrier.

other types of pigment dispersions

Akrochem offers other broad lines of pigment dispersions. These subjects are too complex to be included in this **Solutions**. For right now we'll briefly review these product lines.

Aqueous Color Dispersions

Akrochem offers a full line of water-based colors for latex products like gloves and balloons, water-based coatings and adhesives, water-based "gels," or virtually any water-based system that needs color. A wide variety of pigments are available ranging from bright organics to durable inorganics. Unusual pigment dispersions include an FDA-certified carbon black [<0.5ppm PAH (polyaromatic hydrocarbons) and <5ppb benzo (a) pyrene], fluorescent colors, and a recent experimental metallic pigment in water. Pigments are dispersed in water using a broadly compatible, glycol-type system or a glycol-and-surfactant-free acrylic resin system designed primarily for the inks and coatings markets.

Plastic Color Dispersions

Plastic is an ideal material for color dispersions. A colorless plastic or thermoplastic rubber (Kraton®, Santoprene®) can be manufactured in large quantities and have various colors blended-in moments before injection. The translucent nature of plastic means bright, vibrant colors can be made with small amounts of colorant. Fluorescent, metallic, and phosphorescent ("glow-in-the-dark") colors are all relatively easy to do in plastic compounds. But dispersion of the pigment is critical. Tiny maldispersions of color are quickly evident in clear plastic. This is where Akrochem's **Akroplast**™ plastic color dispersions are effective.

Single-pigment dispersions and custom-matched plastic color concentrates can be made for virtually any thermoplastic resin. There are numerous pieces of equipment that are being used based on the amount of shear needed, temperature required and volume. A single or twin-screw extruder handles most common forms of pigment dispersion. The use of this type of continuous mixer allows the operator to adjust the amount of shear needed to disperse the pigments by changing many variables: some being the temperature, length of duration, speed and screw configuration. Because the applications vary greatly, the extruder gives you the ability to make various forms from a strand (spaghetti-type) pellet or use an underwater pelletizer to get a sphere. Other types of equipment are the continuous mixer, banbury and open two-roll mill. The continuous mixer uses a combination of banbury and extrusion technology.

Akrochem offers a complete line of pigment dispersions in PVC, EVA ethylene vinyl acetate, PE polyethylene and PP polypropylene using all forms of the aforementioned dispersive equipment. With additional downstream equipment we can offer physical forms such as a spherical pellet, cube or powdered material classified to your size specification. A full line of pigments from

transparent organics to low-cost inorganics can be used. Based on your application, we can formulate a concentrate that will meet NSF, FDA, UV-resistance and weathering requirements.

Recent advancements in the use of TPE, TPO, TPR, TPV, etc. have prompted more work in pigment dispersions for these resin systems. Although there is some discussion on the exact carrier resin to use for the pigment dispersions, Akrochem has developed color concentrates in a variety of general-purpose resins for use in the growing thermoplastic vulcanizate market.

The binder for the plastic colorant must be compatible enough with the plastic to permit good dispersions without molding problems. The melt-flow of the binder also must be similar to the base plastic. The applications for plastics are virtually limitless, so there are many, many options of color and color-type from which to choose. These facts all add up to the need oftentimes for a custom pigment/plastic blender that can help you choose the proper binder as well as the best colorant for your specific application. Rather than having to use a one-type-fits-all colorant, we can custom-design your product.

The pigment choice for a plastic colorant can also be complicated. Color, cost, and serviceability have to be considered. Let Akrochem technical service guide you through the process, especially if you are relatively new to manufacturing colored plastic parts in general or are preparing to mold a particular colored part for the first time.

summary

Type of Dispersion	Typical Binders	Where Dispersion is Used
Rubber	SBR, EPR standard NBR, EVA, HIIR, silicone available (no FKM-bound versions)	Any dry rubber compounding, especially mill mixing. Softened, screened versions needed for some applications.
Plasticizer Paste	DIDP, urethane compatibles, silicone fluid, mineral oils	Liquid systems like 2-part urethanes and silicones, solvent-based adhesives, plastisols, coatings, very soft rubber compounds.
Water Paste	Water with glycol/surfactants; water with acrylic resin	Water-based adhesives and coatings, latex gloves, balloons, condoms. Any water-based system.
Plastic	PVC, PE, PP, EVA, SBS, TPV	Plastic and thermoplastic vulcanizates

For most of the early years of the rubber industry, black was beautiful. Like Model T Fords, you could have any color as long as it was black. But today, bright vibrant colors are commonplace in many products. Even tires are being made in various colors. To achieve the best color possible, the carrier and dispersion of the colorant can be critical. To help you make an informed decision, please contact your Akrochem Technical Sales Representative or the Akrochem Technical Services Dept.

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