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2016: MACHINERY & MATERIALS REVIEW

COLOUR COMPOUNDS FOR POLYOLEFIN PIPE

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Formulating superior colour compounds for polyolefin pipe

Although smaller in volume than the market for black pipe, a substantial amount of high density polyethylene (HDPE) pipe manufactured in North America is colour-coded – and much of that is for non-pressure applications. When formulating colour masterbatches for these applications – such as conduit – a variety of organic and inorganic additives are used. Unlike carbon black, these additives will not always enhance pipe performance in the field – and can actually detract from it. However, adding certain ingredients to the masterbatch can improve production, and enhance the pipe's performance for both pressure and non-pressure applications.

Let-down ratios

A major difference between black and colour masterbatches is in their let-down ratios. Black concentrates are typically let down in polyolefin resins at relatively high mix ratios between 4% (24:1) and 6.5% (14:1). This is because a greater concentration of carbon black (usually 2-2.5% in the pipe) is necessary to achieve the right amount of UV protection. Let-down ratios for **Joseph Serbaroli** of Ampacet lays down some guidelines to help ensure that polyolefin pipe does not lose performance when adding colour to it

colour masterbatches are lower: for colours such as yellow, blue or lavender, these typically need around 1% (99:1) or 2% (49:1) ratios, for applications such as corrugated duct, smooth wall conduit and gas pipe.

For pressure-rated water or gas pipe, the dispersion of the pigments in the matrix is critical. If small agglomerates become embedded in the wall of a yellow gas pipe, it could lead to a brittle failure or Stage Two mechanical failure – in which a tiny hole forms in the wall. As pressurised gas or water spurts through the pipe wall, the opening will become larger until the pipe loses pressure completely. If a compounder cannot attain optimum dispersion of the pigments in a yellow Extruders must take care when formulating colour for polyolefin pipe These images show poor dispersion of blue pigment (left), as seen in thin film samples, together with a better dispersion of pigment (right)



masterbatch for gas pipe, it is advisable to adjust for a higher let-down ratio - for example, from 2% (49:1) to 4% (24:1) or more.

This is more common in thin-walled pipe, where there is a greater the chance that the agglomerates will affect its ability to hold pressure. Large diameter pipes can be more forgiving, thanks to their thicker walls. In this case, agglomerates or poorly dispersed particles will form voids or 'windows' in the wall. This is almost impossible to circumvent, but they must be minimised in both size and number in order to reduce the chance of pipe failure. Studies confirm that dispersion quality is critical to both UV performance and the plastic's mechanical properties. For pressure-rated yellow PE gas pipe, the let-down ratio should be no less than 2% [49:1] in order to ensure adequate dispersion. For non-pressure applications, such as coloured conduit, a 1% (99:1) mix ratio will suffice.

To measure dispersion quality in the masterbatch pellets, Ampacet uses a micro dispersion scale of 1-5 where 1 is the highest quality dispersion. This is done by converting the masterbatch into a thin polyethylene film of about 1.5 mils (38 microns) thickness, then measuring the agglomerates in the film via a visual inspection. This gives the masterbatch producer confidence that the additives are adequately dispersed for the customer to make a pipe wall that is smooth and solid. Also, it provides a company with a traceable lot number in the event of a pipe failure. The dispersion rating is reported on the certificate of analysis for every lot produced. For critical applications - such as pressure pipe or geomembranes - compounders should keep records of each lot and, if necessary, make that data available to the purchaser or an independent third party.

Weatherability factors

When formulating non-black concentrates for outdoor use, weatherability – a plastic's ability to resist chemical change from elements such as sunlight, temperature, and moisture – is a primary concern. Environmental exposure, whether above ground or below ground, will weaken or damage the polymer chain, making it brittle – or cause the pipe to discolour over time.

Proper pigment selection plays a vital role in extending the service life of polyolefin pipe. Studies have shown that not all pigments act the same when exposed to the elements. Some pigments, depending on their chemical composition and particle density, enhance the properties of plastic – sometimes having a synergistic effect with UV stabilisers to protect resin from damaging UV rays. But the reverse can also apply, with many pigments having a damaging effect on polyethylene, degrading it at an accelerated rate. The topic is complex, requiring an understanding of what happens in two distinctly different scenarios: aboveground and below-ground environments.

UV and thermal protection

For above-ground piping systems and conduit, exposure to solar radiation and high temperatures will cause both photo-degradation and thermal degradation, which can vary with season and geographic location. Most pipes need a degree of protection from the elements, due to the lengthy times they are stored at construction sites. When exposed to heat, cold, UV rays and moisture, the molecular chains of the polymer will break over time. Free radicals are produced, which cause the PE or PP to become brittle and lose its durability. This leads to environmental stress cracking (ESC), which accounts for some failures of plastic pipe and fittings.

With the addition of carbon black, two mechanisms work to impart Environmental Stress Crack Resistance (ESCR) and extend the service life of the piping system – protecting against UV degradation and thermal degradation. This is why more than 80 % of polyolefin pipe made in North America is black: no single ingredient provides more cost-efficient protection than carbon black. Many studies explain how carbon black morphology and particle size can mitigate the harmful effects of UV radiation. Most have concluded that a minimum of 2% carbon black in the pipe wall is necessary to protect the polymer over the long term



Solar radiation varies widely according to geographic location, which can cause a problem for pipe manufacturers from the effects of UVA and UVB rays. There is also evidence, from a study performed by Cabot Corporation, that the dispersion of carbon black in the polymer can positively influence the expected life of plastics.

When pigments other than black are used, UV inhibitors, light stabilisers and anti-oxidants must be added to give equivalent outdoor protection. Without UV inhibitors and anti-oxidants, UV radiation will break down the chemical bonds of the polymer. Depending on the geographic location of the piping system, the degradation can accelerate quite rapidly. Coloured HDPE pipe left outdoors for any length of time will be subject to embrittlement, chalking, fading and loss of tensile strength – which ultimately leads to ESC.

UV inhibitors and anti-oxidants

Different colours are used to identify various piping systems and conduit lines Hindered Amine Light Stabilisers (HALS) are by far the best light stabilisers. Their effectiveness in scavenging free radicals also makes them the most cost-effective. For coloured pipe, it is advisable to use the high-performance polymeric HALS, as they are less migratory than most others. This, together with their long-term anti-oxidative effect, makes them the products of choice for the extended outdoor exposures of pipe and conduit. Within certain limits, they are also NSF acceptable for potable water applications.

Heat from the sun also increases the temperature of exposed pipe – making it as much as 60F higher than ambient, depending on colour. This causes thermal degradation. Such extreme temperature fluctuations over time will also affect the polymer bonds. In addition, it should be noted that chemical reaction rates increase exponentially as temperature increases. So, it is important in some cases to add anti-oxidants or heat stabilisers into colour masterbatch formulas to prevent thermal degradation of the pipe or conduit.

Two kinds of anti-oxidants – primary and secondary – are generally used in colour masterbatches as thermal stabilizers. Primary AOs are chemicals that slow the thermo-oxidation of the polymer outdoors over time, and extend the functional lifespan of the plastic; secondary anti-oxidants are used in pipe production to inhibit the thermo-mechanical effect of processing the polymer, which degrades it for as long as it resides at high temperature in the extruder zone.

Secondary anti-oxidants are especially important when a polymer is exposed to two or even three heat cycles – such as when regrind is added to pipe. Without enough secondary AO, the thermo-mechanical degradation of a mixture containing regrind could cause the compound to stick to the extruder. This happens particularly on the inside of the pipe, where more heat is contained in the process, and can result in surface fracture and even tearing on the inside surface. Formulators must take this into consideration, and add enough AO to prevent this type of surface fracture from occurring.

These protective anti-oxidants improve ESCR and protect against Slow Crack Growth (SCG), but they also increase cost. Add colour to the mix, combined with UV inhibitors, and it will be even more expensive per foot of pipe extruded. As a result, the cost of using a colour is generally two to five times that of black concentrates.

Accelerated weather testing

To determine the optimum amounts of UV and AO necessary for a given application, accelerated weathering tests can be performed on pieces of pipe. With the right laboratory equipment, accelerated weathering can be conducted by exposing pipe samples to controlled cycles of intense energy, light, heat and simulated rain spray. To gauge and verify the weather resistance of customers'





Ampacet's uses weatherometers to test samples of polyethylene pipe and film products, Ampacet uses two different kinds of weatherometer at its research facility in Terre Haute, Indiana.

One is the fluorescent UV accelerated weathering tester (ASTM G 154) often called by its brand name QUV. It does not attempt to reproduce sunlight entirely, but is designed to produce mainly ultraviolet (UV) rays – especially the damaging effects that occur in the 300-400nm spectrum. The other is the Xenon arc test chamber (ASTM G 155) with lamps that provide the closest possible equivalent to natural sunlight, including UV, visible and infrared (IR) light from 295 to 800nm. It also uses heat and water cooling to control temperature, and simulate the effects of moisture with direct water-spray and humidity controls. As a general rule, 12 months of outdoor exposure in Miami can be simulated with Xenon arc weathering in about 2,000 hours or about 83 days.

Preserving buried colours

For below-ground pipe, separate criteria apply. The pigments should have stability underground, because sulphides in the soil can severely discolour the pipe: if lead chromate pigments come into contact with ammonium sulphide, for instance, this forms lead sulphide – which over time can turn a yellow pipe black. To understand the chemical effects on buried pipe, Ampacet's R&D facility in Messancy, Belgium, carried out a study on colours for underground use – testing more than 80 colour masterbatches for resistance to ammonium sulphide, a highly reactive element commonly found in soil. Their analysis found that diarylide pigments are generally resistant to ammonium sulphide and other sulphides in the earth. Some of those that were resistant, when combined with other reactive colours, can have a stabilising effect on the formulation as a whole. This kind of research is critical – because pipelines that are buried for long periods of time must demonstrate recognisable colour so that repair technicians can identify them many years later.

When formulating colours specifically for pressure applications, it makes sense to use as few additives as possible – as any one of them could interfere with dispersion and potentially weaken the pipe wall. In non-pressure applications, such as conduit, dispersion is not as critical. In extreme cases, too much additive masterbatch can cause 'blooming' – in which additives rise to the pipe's surface. If severe, blooming can affect butt fusion – the process used to join sections of pipe together.

For the additives that are essential, these should be easily dispersible in a concentrate. In other words, there should be no agglomerations of pigments or other additives. Some pigments – such as molybdenum orange, copper-based greens and particularly phthalocyanine blue – can accelerate the degradation of a pipe structure over time. This is because their chemistries interfere with the polymer bonds, causing plastic to become brittle. This is termed a 'Stage Three' failure – which can also be caused by high levels of chemicals in the water such as free chlorine or chloramine.

Four other criteria should be considered when formulating colour masterbatches for pipe:

- 1) Only pigments that are light stable for at least two years should be used.
- Only sulphide-resistant pigments should be used for yellow gas pipe.
- 3) Only pigments that are NSF-acceptable should be used for potable water applications
- Only pigments that are stable to 500F should be selected.

Other factors

In contrast to the use of colour throughout the pipe wall, companies that co-extrude a skin layer of yellow for gas pipe – or blue for potable water – will see significant cost reduction. This is because the amount of

Questions to consider before specifying a masterbatch for performance piping systems:

- Will the pipe be used in a pressure or non-pressure application?
- Will the pipe be installed indoors, outdoors above ground or outdoors below ground?
- Does the pipe require ultraviolet protection? If yes, for what length of time?
- Are heavy metal pigments OK? Or are non-heavy metal pigments required?
- Will the pipe need to meet certain ASTM or ISO standards for OIT or other test criteria?

Co-extruded potable water pipes with a skin laver on the outside can be far cheaper than pipe that is coloured all the way

masterbatch used for an outer shell layer is much less than it would be to colour the whole structure. For potable water applications, co-extrusion is advisable as colour pigments can influence the taste of water that passes through it. It could also avoid costly toxicology testing required by NSF for drinking water system components (NSF Standards # 42/53).

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And while most polyolefin pipe in North America is HDPE, some speciality applications use polypropylene (PP). When extruding high-performance pressure pipe from PP, it is not advisable to use masterbatches in PE carriers. While the polarities of the olefins make them compatible as polymers, the melt viscosities and other characteristics can be quite dissimilar. For pressure

Colour coding/designations of PE pipe for US and Canada

Pressure-rated pipe Yellow = Fuel gas Blue = Potable water Lavender = Re-claimed water Green = Geothermal Grey = Slip lining of older pipe

Non-pressure Pipe Orange = Communications conduit Red = Electrical conduit



applications, only masterbatches using a PP carrier should be used in PP pipe.

Summary

Although the factors outlined here play a role in the manufacturing of high quality polyolefin pipe, failures are more commonly caused by poor extrusion - or improper handling and installation of the pipe - than by poor quality materials. It is essential, however, that masterbatch formulators and pipe producers understand and adhere to fundamental material guidelines. Joseph Serbaroli is strategic business manager at Ampacet (www.ampacet.com)



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