QUALIFYING A MASTERBATCH FOR USE WITH A PRESSURE PIPE RESIN TO MEET PE4710 PIPE REQUIREMENTS

Dr. Taha H. Abdulla and White G. Jee
Sasol Chemicals North America LLC

Abstract

High density polyethylene (HDPE) resins or compounds used in the production of various pressure piping system components (solid wall HDPE pipe, fabricated or injection molded fittings, composite pipe, etc.) are required to meet or exceed certain minimum long-term performance requirements as specified in many application standards. HDPE resin producers, pipe manufacturers, and additive/masterbatch producers must adhere to these standards (including but not limited to ASTM, ISO, PPI, NSF, CSA, etc.) in order to participate in the pressure pipe market. In this paper presentation, we take a deep dive into what it takes to qualify masterbatch/additive producers that will be utilized in the production of the pressure piping system components. While there are official standards, published papers and a host of knowledge in understanding HDPE pipe masterbatches, the intention of this document is to specifically outline the step-by-step process of how one could obtain qualification of a masterbatch with a specific HDPE resin for production of HDPE piping system components. This document will address when one begins the process and the tests one conducts during the qualification period. It will also highlight some potential barriers that could render a masterbatch supplier from meeting qualification requirements.

Introduction

Sasol is an international integrated chemicals and energy company that was established in 1950 in South Africa. One key Strategic Business Unit within the Base Chemicals division include polymers (polyethylene, polypropylene, polyvinyl chloride). Sasol has been a global polymers resin supplier out of South Africa since the 1980s, with a state-of-the-art Polymer Technology Services Center that provides research and development. In an effort to expand a broad portfolio of commodity chemicals globally, Sasol embarked in a joint venture (JV) in North America using the Innovene ™ S technology. The production of high density polyethylene (HDPE) resin commenced in 2017. Sasol decided to expand their product portfolio and participate in the pressure piping system applications market.

HDPE pressure pipe applications require the plastics piping system to meet or exceed certain minimum long-term performance requirements as specified in many application standards. Pipe producers in North America require the use of natural resin that is to be blended with an approved carbon black masterbatch (MB). This is commonly known as the 'Salt and Pepper' process or "machine side blending" process in the pressure pipe manufacturing industry. In order for a resin manufacturer to enter the pressure pipe market, one criteria is to obtain a Plastic Pipe Institute (PPI) independent listing under TR-4[1] using the guidelines provided in PPI TR-3[2].To do that, the Standard Grade shall meet the requirements of the latest revision of "Standard Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials", ASTM D2837[3]. The specific requirements for the Independent Listing for the Standard grade was as follows:

1. E-10 at 73^{o}F, E-6 at 140^{o}F and 140^{o}F Validation (testing performed at 194^{o}F) on lot 1.
2. E-2 at 73^{o}F, E-10 at 140^{o}F, and 140^{o}F Validation (testing performed at 194^{o}F) on lot 2.
3. E2 at 73^{o}F, E-6 at 140^{o}F on lot 3.

Validation is defined as the process of ensuring that for those materials that exhibit a transition from ductile to brittle failure mode, this transition occurs after 100,000 hours at the rated temperature. Based on fulfilling these requirements an Independent Standard Grade listing was granted for a recommended HDB of 1600 psi at 73^{o}F and 1000 psi at 140^{o}F.

While there are many official standards, published papers and a host of knowledge and data about HDPE pipe MB products, this is a paper to provide guidance on how one can utilize a step-by-step process to qualify a black MB for use with a natural pressure pipe resin to meet PE4710 pipe requirements. Please note that this paper is based on Sasol Chemicals North America LLC's approach or experience to qualify black MBs for market entry and this process can vary with each resin supplier's product requirements based upon its background and historical data in the pressure piping industry.

Masterbatch Approval Criteria

To ensure a consistent process of selecting a black MB supplier, it was imperative to develop a baseline criteria. The criteria is as follows:

1. Black MB shall be commercially available.

2. Black MB shall be commonly used in the pressure pipe market.
3. Black MB supplier shall be willing to provide a sample and work collaboratively with Sasol during the testing process.
4. Black MB supplier shall be willing to sign a mutual non-disclosure agreement (NDA).
5. Black MB supplier shall be willing to undergo a Quality Audit to validate their manufacturing and quality processes as a reputable long-term supplier in the industry.

It is just as important to work closely with pipe producers and understand their preferred black MB suppliers; this helps in narrowing the black MB suppliers' list to the top few. Based on this criteria, Sasol selected five black MB suppliers (A, B, C, D and E) to work with initially with some of the suppliers supplying more than one black MB product to evaluate. Of those, all A through D fulfilled the criteria, while E did not. Sasol proceeded in obtaining qualification for those black MB suppliers that fulfilled the criteria.

Trials

Upon confirmation and agreement with black MB suppliers, a trial is then coordinated with the pipe producer. Based on desired ratios of black MB to natural resin to achieve pipes with a carbon black content in the range of 2.0 to 2.6% carbon black, a 1" or-2" pipe is manufactured to the pipe specifications based on appropriate ASTM F17[4], Plastic Piping Systems standard the pipe producer is using such as ASTM D2513[5], F714[6], D3035[7], or D2239[8] . A minimum of 100 pipe samples approximately 3 feet in length were collected for each MB sample to be evaluated. The pipe samples were made using the same natural resin lot so that the black MB samples were the only material component variable during the pipe production. The same black MB was run at two different pipe producers to ensure consistency and repeatability. This 'addition' of a black MB could potentially have an effect on the long term service life and thus requires hydrostatic test data to confirm the modified compound's HDB (Hydrostatic Design Basis) or MRS (Minimum Required Strength) is unchanged. At this stage, one must locate a testing facility that could perform hydrostatic testing at the various temperatures to establish equivalency. The HDB equivalence requires the following minimum levels of data (also illustrated in Table 1):

1. E-2 per Part A of TR 3 at 73 °F
2. E-2 per Part A of TR 3 at the highest other listed temperature (140°F)
3. Validation at the highest listed temperature (194°F)

Table 1. Equivalency Testing on One Lot of Pipe for each black MB.

LOT ID	73 °F	140 °F	Validation	MB
I	Complete	Complete	Complete	A
II	Complete	Complete	Complete	B
III	Complete	Complete	Complete	C
IV	Complete	Complete	Complete	D

This process was conducted for each chosen black MB that met the Masterbatch Approval Criteria. It is prudent to note that during the trial, Sasol monitored extrusion conditions to understand the impact of adding this black MB.

Along with the HDB Equivalency testing on the pipe samples, other tests on the sample blend of the natural resin and black MB were conducted to check the ASTM cell classification and other requirements in accordance with D3350[9]. Since the original black product formulation in which an independent listing was granted had a D3350 cell classification of 445574C or 445576C, it was important to verify that the black MB impact (if any) on the D3350 cell classification short-term properties as follows:

1. The carbon black content shall be in the range of 2.0% to 3.0%. Test Method D1603[10] shall be used.
2. For Elongation at Break, all pressure rated materials shall have a minimum extension at break of 400% when tested with 10.1.6 of D3350.
3. Thermal Stability: The material shall contain sufficient antioxidant so that the minimum induction temperature shall be 220 °C
4. The Slow Crack Growth Resistance is to meet the minimum requirement for the appropriate cell classification when tested in accordance with Test Method F1473[11] for the average failure time from two test specimens.

To further clarify, the manner in which materials are identified in the cell classification is illustrated for Class PE445574C as follows in Figure 1 (refer also to Table 1 and 6.2 of D3350)

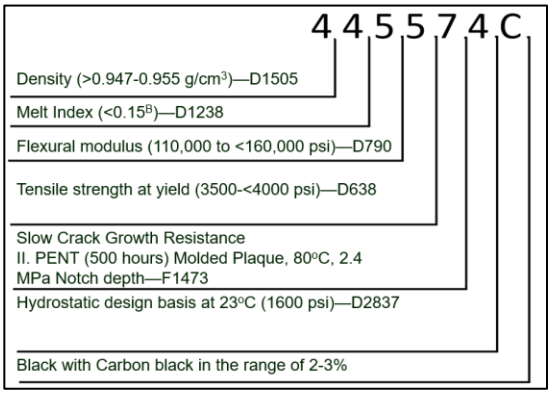

Figure [1] Cell Classification illustration of PE445574C

Barriers:

The steps to qualifying a black MB can be lengthy and rightfully onerous. It is important to realize that the pressure pipe industry entrusts all parties to conduct their due diligence to ensure all the appropriate steps take place to obtain the final qualification. There could be some barriers that can potentially prevent a black MB from being approved to qualify for the pressure pipe industry. Some of these barriers are:

1. Dispersion of black MB may be less than satisfactory which can result in early failure during hydrostatic pressure tests.
    - This can be a result of poor mixing in the production of pipe samples or the presence of carbon black agglomerates in the MB
2. The black MB carrier resin could be of a material that is not compatible to maintain the long-term HDPE pipe resin performance properties necessary for pressure pipe applications.
3. During pipe manufacturing, extrusion settings could result in jeopardizing the integrity of the pressure pipe:
    - This could be due to excessive or lack of cooling
    - Irregular temperature profile resulting in overheating
    - Mixing capability of black MB and natural resin
4. Improper raw material used for black MB
    - Base Resin
    - Carbon Black Type
    - Antioxidant/Stabilization Package

Conclusion

With the assistance of our JV partner and the numerous qualification tests conducted, the Sasol product (natural resin blended with black MB) is listed in PPI TR4 as a PE4710 compound with a recommended HDB of 1600 psi at 23° C and 1000 psi at 60° C. It is also listed in PPI TR-4 as a PE100 compound with a recommended Minimum Required Strength (MRS) of 10 MPa at 20° C. The product is also listed with NSF International as meeting the requirements of ANSI/NSF Standard 14 for potable water and ANSI/NSF Standard 61 for toxicology components in drinking water. It is also listed with NSF International as meeting the material requirements in CSA Standard B137.1. The product is also listed with NSF International as having a CC3 chlorine resistance category as defined in ASTM D3350. To-date, there are four black MB products that are approved with the natural pipe resin. Other black MB products are under evaluation using the criteria as documented in this paper. With the qualification of these black MBs, it allows for the flexibility with customers and potential customers to have a variety of approved suppliers to select from

While the process of ensuring the black MB are qualified can be a lengthy process, it certainly helps to have a single source area that can be used as a reference. The intention of this paper was to be that source.

References

1. Plastic Pipe Institute (PPI). TR-4/2018 HDB, HDS, PDB, SDB, MRS, CRS Listed Materials.
2. Plastic Pipe Institute (PPI). TR-3/2018 HDB, HDS, PDB, SDB, MRS, CRS Policies.
3. ASTM D2873, “Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials or Pressure Design Basis for Thermoplastic Pipe Products”.
4. ASTM F17, “Plastic Piping Systems.”
5. ASTM D2513, “Standard Specification for Polyethylene (PE) Gas Pressure Pipe, Tubing, and Fittings.
6. ASTM F714, “Standard Specification for Polyethylene (PE) Plastic Pipe (DR-PR) Based on Outside Diameter.
7. ASTM D3035, “Standard Specification for Polyethylene (PE) Plastic Pipe (DR-PR) Based on Controlled Outside Diameter.”
8. ASTM D2239, “Standard Specification for Polyethylene (PE) Plastic Pipe (SIDR-PR) Based on Controlled Inside Diameter.
9. ASTM D3350, “Standard Specification for Polyethylene Plastic Pipe and Fittings Materials.
10. ASTM D1603, “Standard Test Method for Carbon Black Content in Olefin Plastics.”
11. ASTM F1473, “Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins.”