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NEW CROSSLINKED (PEX) PIPES FROM SUSTAINABLE RESOURCES

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SHORT SUMMARY

This study investigates production of crosslinked polyethylene (PEX) pipes using a resin obtained from a renewable resource via twin-screw extruder technology. PEX pipes were extruded using petroleum-based and renewable high-density polyethylene (HDPE) for comparison. Testing using relevant ASTM standards on the new pipes indicates that the PEX pipes meets or exceeds the vigorous requirements described in the standards such as excessive hot/cold pressure tests and Environmental Stress Crack Resistance (ESCR). The study shows that the renewable resin shows potential as a starting material for PEX pipes.

KEYWORDS

PEX pipes, pipe extrusion, sustainable resources, ASTM Standards

ABSTRACT

In recent years, innovation has progressed at an unprecedented pace where new technologies demand the use of plastics derived from sustainable resources across various industrial manufacturing processes. Though research in bioplastics and sustainable polymers is making great strides, their growth in the plastics industry is still slower than expected. Crosslinked polyethylene (PEX) pipes continue to gain popularity in plumbing and indoor climate applications around the globe. An approach to increasing the sustainability of PEX pipes is to produce them using high-density polyethylene derived from sustainable resources. In this paper, a method for producing a strong and flexible PEX pipe obtained from resin derived from renewable plant resources is described.

The new resin characteristics and pipe properties were compared to resins derived from petroleum resources. The observed difference in crosslink density was found to correlate with the difference in melt flow index between the resins tested.

Production of sustainable PEX pipes was accomplished via twin-screw extruder technology using UV energy as a method for crosslinking. Testing performed on the new

pipes indicates that the pipes produced meet or exceed the requirements set by the vigorous industrial requirements outlined in ASTM standards, such as excessive hot/cold pressure requirements and ESCR tests. Comparison of sustainable PEX pipe performance relative to pipe made with petroleum-derived resources are included in this evaluation.

INTRODUCTION

Rapid growth in plastics production is extraordinary, surpassing most other man-made materials. The vast majority of monomers used to make plastics, such as ethylene and propylene, are derived from fossil hydrocarbons. Product designers and engineers are adding new products constantly as new material technologies evolve around the use of plastics [1]. Plastics produced from sustainable resources are gaining momentum due to the high demand of plastics used in different industrial processes for its exceptional performance.

This paper emerges from the drive for sustainable initiatives taken by the authors in manufacturing of PEX pipes from polyethylene resins derived from renewable resources. PEX pipes continue to gain popularity in plumbing and indoor climate applications due to ease of installation and its ability to withstand aggressive water conditions. An approach to increasing the sustainability of PEX pipes is to produce them using materials derived from sustainable resources that will add value to the new concepts in product development especially for green/energy efficient building concepts. Plastic is generally light-weight and strong. Hence, plastic materials already contribute significantly to energy-efficient building and are an essential part in the construction field. If one can derive plastic products from sustainable resources, it will be an added benefit to use these materials and eliminate the need for depleting petroleum-based resources. Therefore, the authors in this investigation sought out a high-density polyethylene (HDPE) derived from plant resources. This selected material is currently used in several other applications such as blow molding and film applications. In this study, the HDPE from the sustainable resource was crosslinked to produce PEX pipes. Crosslinked pipes produced were tested against the applicable standards to confirm its validity and use in plumbing applications. The pipes were produced using twin-screw extrusion process using UV energy as a way to crosslink the HDPE [2]. Crosslinking using UV energy also introduces a new way of energy savings in PEX production, a more sustainable pathway than the commonly used crosslinking methods. UV crosslinking is considered a “green” and environmentally friendly technology, since no solvents are used in the process and no emission of volatile chemicals takes place. Hence, the resulting PEX pipes have an improved sustainability profile.

Several crosslinking methods are widely used for crosslinking polyethylene pipes. The required chemical crosslinking levels (CCL), as found in ASTM F876, are 65% to 89% depending on different crosslinking methods used in the industry [3]. For example, when a peroxide is employed in the fabrication of PEX-a pipe, the required crosslinking levels are 70% to 89%. For all other crosslinking methods (PEX-b, -c, and –other) a minimum of 65% is required. These requirements are set by industrial governing bodies based on the long-term performance tests conducted on PEX pipes.

Testing performed on the new pipes produced from sustainable HDPE indicates that the pipes meet or exceed the requirements set by the vigorous requirements described in ASTM standards, such as excessive hot/cold pressure requirements and Environmental Stress Crack Resistance (ESCR) tests.

NOMENCLATURE

CCL- Chemical Crosslinking
ESCR-Environmental Stress Crack Resistance
GPC-Gel Permeation Chromatography
HDPE-High Density Polyethylene.
HT-GPC-High Temperature Gel Permeation Chromatography
OIT-Oxidation Induction Time
SDR-Standard Dimension Ratio
ID-Inner Diameter
OD-Outer Diameter
UV-Ultra Violet

EXPERIMENTAL

Materials and Characterization

The high-density polyethylene resins (HDPE) selected in this study were obtained from leading producers of polyethylene. The resin and respective pipe samples are marked as HDPE-1 (PEX Resin Sample 1), HDPE-2 (PEX Resin Sample 2), and HDPE-SR (Resin from Sustainable Resource). The details of the resins selected along with key properties measured are given in Table 1.

The Melt Flow Index (MFI) was determined using ASTM D 1238 [4]. The measurements were performed at 190 °C using a load weight of 21.6 Kg. Density measurements were performed on extrudate using the ASTM D1505 for resins [5].

Differential Scanning Calorimetry (DSC) measurements were performed using a Mettler Toledo DSC 8823^{e} with a temperature ramp rate of 15 °C/min.

Oxidation Induction Time (OIT) were determined at 200 °C using Mettler Toledo DSC 8823^{e} using method described in ASTM D 3895 [6].

Table 1. HDPE Resin Properties

Properties Evaluated	**HDPE-1**	**HDPE-2**	**HDPE-SR**
Resin Shape	Powder	Powder	Pellets
Polymerization Process	Homo polymer	Copolymer	Copolymer
Density at 23 °C(g/cc)	0.953	0.956	0.952
Melt Flow Index g/min (190 °C/21.6 kg)	2	10	9
Melting Point by DSC (°C)	125	124	121

Pipe performance testing was conducted according the respective ASTM standards. Stabilizer functionality test on pipe samples were performed by keeping six pipe samples continuously for 3000 hours at a hoop stress of ~101 psi or 0.7 MPa at 120 °C as per ASTM F876.

For ESCR, a notch depth of (10% of the pipe wall thickness) was made inside the pipe wall according to the requirements described in the ASTM standard F876. The pipe samples were then filled with the test medium which is 5% Igepal CO-630 (nonylphenoxypoly (ethyeneoxy) ethanol) mixed with 95% of untreated water. Igepal serves as an ESCR accelerating agent. The pipes are then pressurized to 195 psi at 180 ºF (82 ºC). ASTM F876 requires that the pipes hold pressure for a minimum of 100 hours.

Pipe Extrusion

A large 60 mm Twin Screw extruder was used for pipe production. The screw configuration used is a co-rotating model with specially designed elements to achieve proper mixing of the additive package. A pipe die was used to produce 1/2” diameter pipes (15.8 mm x 2.0 mm) with SDR 9 dimensions.

Crosslinking Process

Crosslinking was performed using Ultra Violet (UV) Energy. The achieved crosslinking level depends on the proper selection formulation and process conditions. Since various options of UV devices and lamps are available in the market today, careful selection of UV energy using proper bulbs and the proper selection of raw materials dictates the crosslinking levels achieved in this process.

DISCUSSION

Crosslinking process using UV curing, i.e., photoinduced crosslinking, is a process where a pipe formulation comprising of a combination of a polyolefinic polymer, for example polyethylene, a photoinitiator, a co-agent, and a stabilizer package, is exposed to UV radiation to form a crosslinked polymer. In the case where polyethylene is utilized for extrusion using a pipe die, the final product is a PEX pipe.

Crosslinking in polyethylene follows a complex mechanism. When UV energy is applied to a polymer chain in presence of necessary crosslinking chemicals, a variety of reactions like crosslinking, chain scission, and recombination reactions take place [7-8]. These reactions proceed in a competing way depending on the free radical formation and the chemical kinetics dictated by the reaction environment. Crosslinking level obtained are dictated by the structure of polyethylene resin employed, extrusion temperature, and crosslinking package, in this case the photoinitiator.

Pipe Extrusion Conditions

For the sake of comparing the resins, the extrusion conditions were kept identical as possible while using the resins selected in this study. The resin was fed through a gravimetric feeder to the extruder. The pipe dimensions were controlled with the help of a calibration sleeve housed in a vacuum tank using a nitrogen flow. It is interesting to note that the HDPE-SR produced very smooth and clear pipes using the same conditions applied for extruding other resins used in this study.

The dimensions measured for the pipes produced were in compliance with the ASTM F876 standard requirements. All three pipes produced were subjected to various performance requirements against the testing standards [9]. Results are summarized in Table 2.

Table 2. Summary of testing for pipes HDPE-1, HDPE-2 and HDPE-SR.

Pipe Property/Tests	HDPE-1	HDPE-2	HDPE-SR
Pipe Color and Appearance	Clear smooth pipe	Clear smooth pipe	Clear smooth pipe
OIT at 200 ºC-ASTM D3895 (min)	105 ± 5	98 ± 5	98 ± 5
Pipe Dimensions-ASTM F876 (OD range 15.78-15.98 mm, Wall Thickness range 1.78-2.03 mm)	OD = 15.88 Wall =1.93	OD = 15.85 Wall =1.95	OD = 15.85 Wall =1.95
Pipe Density-ASTM F876 at 23 °C (gm/cc) (Minimum Required 0.926)	0.935	0.937	0.940
Gel Content or Chemical Crosslinking-ASTM F876 (%) (Requirement 65-89%)	>70	>65	>65
Burst pressure at 82 ºC or 180 ºF-ASTM F876 (psi) (Minimum Required 215 psi)	298	295	296
1000 Hour Sustained Pressure Testing-ASTM F876/F877 (180ºF, 190 psi)*	>1000	>1000	>1000
Excessive Temperature-ASTM F877 (pipe should be able to hold 48 hours at 210 ºF/99 ºC at 150 psi)	Pass	Pass	Pass
Sustained pressure test-hot bent tube with 90 degree angle shall meet the pressure value of 195 psi at 180 ºF/82 ºC for 1000 hours-ASTM F877	Pass	Pass	Pass
ESCR-ASTM F876, 195 psi at 180 ºF (82 ºC) (Minimum Required 100 hours)*	>100	>100	>100
Stabilizer Functionality Testing (Hoop Stress 101 psi(0.7 MPa) at 120 °C) (Minimum Required 3000 hours)*	>3000	>3000	>3000

*Tests stopped when minimum requirements reached

The performance recorded for HDPE-SR pipes are comparable to other pipes tested. It is very important to note that the tests were stopped once the minimum requirements hours are reached. Hence, the test hours listed in Table 2 are not failure times.

A method for testing the stability of a pipe in the presence of an oxidizing agent is the Oxidation Induction Time (OIT) test. The amount of time required to the induction of polymer degradation is measured in minutes. In this study, the stabilizers used were kept the same for all the three pipe formulations and the OIT values obtained are recorded in Table 2. It should be noted that a higher OIT values are an indication of the amount and the quality of the stabilizer present in the system and not a direct measurement for long-term performance of pipes in actual field application. From the test data, no significant difference between HDPE-SR pipe and other pipes were noticed in terms of performance comparison in this evaluation.

ASTM F876 requires that the PEX formulations must meet the stabilizer functionality test. Stabilizer functionality testing is a measure of resistance to oxidative degradation and provides an indication of the long-term performance of pipes, in relation to oxidative degradation, e.g., by chlorine in potable water. It should be noted that the outside medium is hot air and inside of the pipe is filled with water. This test can be used to demonstrate the specific PEX pipes ability to withstand long-term temperature and sustained pressure conditions. The data obtained for HDPE-SR pipe are given in Table 2 along with other pipe sample tested. It is important to note that the HDPE-SR samples met the requirements for the stabilizer functionality test, which is an indication that the pipes obtained from sustainable grade resins will be able to withstand the real application temperature and pressure encountered in the field.

ESCR test is another of way to measure the ability of PEX pipes to withstand the actual application conditions (environmental stress, higher temperature, pressure, etc.) in the field. In this method, cracking of stressed pipe samples were tested in the presence of surface-active wetting agents. The HDPE-SR pipes produced in this study met the minimum requirement of ESCR per ASTM F876.

Gel content or Chemical Crosslinking Level (CCL) obtained for the pipe sample are given in Table 2. The standard requirement for CCL is 65-89%. A slightly lower CCL obtained for HDPE-2/HDPE-SR pipes could be due the higher melt flow index of these resins. Other factors found to affect the crosslinking are density of the resin, melt flow index, presence of commoner, and the amount of unsaturation present in the polymer chains. Billovits and coworkers while using E-beam crosslinking observed that a lower density and a lower melt flow index of the resin generally favors higher crosslinking levels [10]. While the amount of unsaturation or vinyl content were not quantified for the resins in this evaluation, the difference in resin density does not show a big impact on crosslink levels. Given the application, it is expected that the HDPE-SR resin should have lower unsaturation content compared to other two resins used in this study. It is interesting to note that, the lower melt flow index resin gave slightly higher levels of crosslinking when compared to higher melt flow index resins. This is in agreement with the literature data using E-beam method for crosslinking polyethylene in the above citation. Evaluation of different factors affecting crosslinking levels are in progress and will be topic of discussion in future plastic pipe symposiums.

Test data obtained for the HDPE-SR indicates that the properties are comparable to the petroleum-based resins. Further performance evaluation and testing of sustainable grade resins will be a topic for future plastic pipe meetings.

CONCLUSIONS

The results of this study show that the sustainable grade HDPE resin produced good quality pipes when compared to other similar commercially available resins in the market. The extrusion conditions used were identical for the production of renewable resin pipes. The observed difference in crosslinking attributed to the higher melt flow index of the renewable resin investigated. Performance testing indicates that the renewable resin pipes are comparable in properties and meets the requirements described in the standards.

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