

Optimization of Physical and Rheological Properties in Extrusion Moulding of Recycled Combined HDPE-LLDPE Polymer Composite Pipes

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I. INTRODUCTION

The concept of utilizing filler materials to enhance polymer performance in Composite materials has drawn a great deal of research interest. The science and technology of polymer composites has created considerable excitement and expectations in the last five years. In addition to that, researches in this area have been focusing on the second phase embedded in the polymeric matrix that gives physical and chemical properties that cannot be achieved. Researchers have also discovered that incorporating the right amount of fillers into a polymer matrix pose a remarkable strength and flexibility and that industries should be able to integrate the outcome of their researches widely in high performance applications in the field of electrical engineering, aerospace, marine, high speed parts in engines, packaging and sports gadgets. With the extrusion methods of synthesis and tools for characterization, polymer composite science and technology is now experiencing explosive growth. Taking advantage of the need and the properties of the polymer composite material through this research a new enhanced composite is developed through addition of fillers and granules into polymeric matrix to cater for varied applications.

1.1 Classification of Plastics

Plastics are usually classified by their chemical structure of the polymer's backbone and side chains. Some important groups in these classifications are the acrylics, polyesters, silicones, polyurethanes, and halogenated plastics. Plastics can also be classified by the chemical process used in their synthesis, such as condensation, poly addition and cross-linking. There are two types of plastics. (i.e. Thermoplastic and Thermosetting Polymers.)

1.1.1 Thermoplastic Polymers

Thermoplastics are the plastics that do not undergo chemical change in their composition when heated and can be moulded again and again. Examples include polyethylene, polypropylene, polystyrene, polyvinyl chloride (PVC), and polytetrafluoroethylene (PTFE). Common thermoplastics

range from 20,000 to 500,000 amu, while thermosets are assumed to have infinite molecular weight. These chains are made up of many repeating molecular units, known as repeat units, derived from monomer; each polymer chain will have several thousand repeating units.

1.1.2 Thermosetting Polymers

Thermosets can melt and take shape once and after they have solidified, they stay solid. In the thermosetting process, a chemical reaction occurs that is irreversible. The vulcanization of rubber is a thermosetting process. Before heating with sulfur, the polyisoprene is a tacky, slightly runny material, but after vulcanization the product is rigid.

1.2 Recycling of Plastic Materials

Thermoplastics can be remelted and reused, and thermosets plastics can be ground up and used as filler, although the purity of the material tends to degrade with each reuse cycle. There are methods by which plastics can be broken back down to a feedstock state. The greatest challenge to the recycling of plastics is the difficulty of automating the sorting of plastic wastes, making it labor intensive. Typically, workers sort the plastic by looking at the resin identification code, although common containers like soda bottles can be sorted from memory. Typically, the caps for PETE bottles are made from a different kind of plastic which is not recyclable, which presents additional problems to the automated sorting process. Other recyclable materials such as metals are easier to process mechanically. However, new processes of mechanical sorting are being developed to increase capacity and efficiency of plastic recycling. A first success in recycling of plastics is Vinyloop, a recycling process and an approach of the industry to separate PVC from other materials through a process of dissolution, filtration and separation of contaminations. A solvent is used in a closed loop to elute PVC from the waste. This makes it possible to recycle composite structure PVC waste which normally is being incinerated or put in a landfill. Vinyloop-based recycled PVC's primary energy demand is 46 percent lower than conventional by produced PVC. The global warming potential is 39 percent lower. This is why the use of

recycled material leads to a significant better ecological footprint. The common recycling plastic materials are as follows,

- PETE - Polyethyleneterephthalate,
- HDPE - High-Density Polyethylene,
- PVC - Polyvinyl chloride,
- LDPE - Low-Density Polyethylene,
- LLDPE - Linear-Low Density Polyethylene,
- PP - Polypropylene and
- PS - Polystyrene.

1. Objectives and Significance

- To optimize the process temperature and proportion mixing of HDPE and LLDPE.
- To obtain the optimum level of filler material and Resin matrices in terms of weight percentage.

II. MANUFACTURING PROCESS

Pipe Extrusion

Pipe extrusion is defined as a process of forcing the polymer melt through a shaping die (in this case circular). The extrudate from the die is sized, cooled and the formed pipe is pulled to the winder or a cut off device with the aid of haul off device. Prior to this, the plastic material in the form of polymer granules is fed into the hopper, conveyed by a rotating screw through a long cylindrical barrel. This is subjected simultaneously to high temperature and pressure, forcing the melt through the die at a predetermined rate. The principal aspects of a solid wall polyethylene pipe manufacturing facility are presented in fig.1.



Fig. 1 Typical Conventional Extrusion Line

Raw Materials Description

The quality of the starting raw material is closely monitored at the resin manufacturing site. The test methods and codes in this are used to ensure that the resin is of prime quality. The pipe manufacturers need to monitor the important physical and mechanical properties. The recycled raw

materials in the shape of granules is preferred by this industry are shown in fig. 2.

Extrusion Line

The raw materials used to manufacture polyethylene pipe are generally supplied as pellets. The resin is stabilized against thermal oxidation. The resin is supplied in either the natural state which is blended at the extrusion facility to add pectitized color and UV stabilizers or in a pre-colored form. The most common colors are black and yellow. The choice of color will depend upon the intended application and the requirements of the pipe purchaser. Carbon black is the most common pigment used for water, industrial, sewer and above-ground uses shown in fig. 3. Color concentrate is important for protecting the pipe from the effects of ultraviolet radiation that can cause degradation. Black concentrate alone is very effective in absorbing ultra-violet radiation whereas non-black concentrates use a ultra-violet stabilizer to provide protection. The horizontal blue line appeared in the pipe indicates the manufacturers coding design in the extrusion process. These pigments are in different colors and the combinations of the 20% in weight of blue pigment and 80% in weight of dull weight pigments are preferred by this industry. These pigments are extruded at a particular temperature in a separate die on the extruded pipes. These pigments are shown in fig. 4.



Fig. 2 Combined Recycled Granules of HDPE and LLDPE Pipe Materials



Fig. 3 Carbon Black Pigments Fig. 4 Color Line Pigments

Extrusion Principles



Fig. 5 Typical Extrusion Screw

The function of the extruder is to heat, melt, mix, and convey the material to the die, where it is shaped into a pipe. The extruder screw design is critical to the performance of the extruder and the quality of the pipe. The mixing sections of the screw are important for producing a homogeneous mix when extruding natural and concentrate blends. A typical extruder is shown in fig. 5.

There are many different types of screw designs, but they all have in common the features. Each screw is designed specifically for the type of material being extruded. The extruder screw operates on the stick/slip principle. The polymer needs to stick to the barrel so that, as the screw rotates, it forces the material in a forward direction. In the course of doing this, the polymer is subjected to heat, pressure and shear (mechanical heating). The extent to which the material is subjected to these three parameters is the function of the screw speed, the barrel temperature settings and the screw design. The design of the screw is important in the production of high quality pipe. The wrong screw design can degrade the resin by overheating and shearing it, which will reduce the physical properties of the pipe.

Extruders

The single-screw extruder shown in fig. 5 is generally used to produce polyethylene pipe. An extruder is usually described by its bore and barrel length. Pipe extruders typically have an inside diameter of 2 to 6 inches with barrel lengths of 20 to 32 times the bore diameter. The barrel length divided by the inside diameter is referred to as the L/D ratio. An extruder with an L/D ratio of 24:1 or greater will provide adequate residence time to produce a homogeneous mixture. The extruder is used to heat the raw material and then force the resulting melted polymer through the pipe extrusion die. The barrel of the machine has a series of four to six heater bands. The temperature of each band is individually controlled by an instrumented thermocouple. During the manufacturing process, the major portion of the heat supplied to the polymer is provided by the motor. This supply of heat can be further controlled by applying cooling or heating to the various barrel zones on the extruder by a series of air or water cooling systems. This is important since the amount of heat that is absorbed by the polymer should be closely monitored. The temperature of the extruder melted polymer is usually between 390°F and 450°F, and under high pressure (2000 to 4000 psi).

Pipe cooling Operations

The pipes must be cooled sufficiently in the calibrating tank and in the bath to retain enough stability to withstand the stresses. Water bath, water sprays are the two

types used. Smaller diameter pipe is usually immersed in a water bath. Cooling water temperatures are typically in the optimum range of 40°F to 50°F (4°C to 10°C). The total length of the cooling baths must be adequate to cool the pipe below 185°F (85°C) in order to withstand subsequent handling operations. The typical cooling operations are shown in fig. 6a) and 6b).

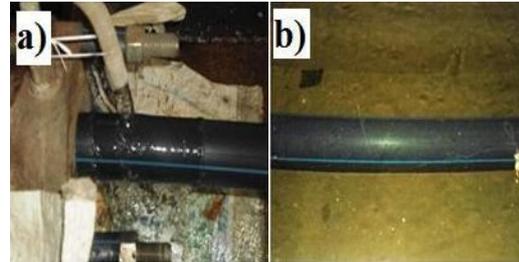


Fig. 6 Cooling operations of HDPE pipes

Traction System

The traction system must provide the necessary force to pull the pipe through the entire cooling operation. It also maintains the proper wall thickness control by providing a constant pulling rate. The rate, at which the pipe is pulled, in combination with the extruder screw speed, determines the wall thickness of the finished pipe. The typical twin traction system is adjustable for different sizes of the pipes and track with proper tensioning arrangement for rubber belt with special grooves are shown in fig. 7. Increasing the puller speed at a constant screw speed reduces the wall thickness, while reducing the puller speed at the same screw speed increases the wall thickness.



Fig. 7 Traction operations of HDPE Pipes

IV. SYNTHESIS OF COMBINED HDPE AND LLDPE

Extrusion techniques can be used to process most thermoplastics and some thermoset plastics. The resins most commonly extruded for tube, hose and pipe products include high and low density polyethylene, polypropylene, polyurethane, polystyrene, fluoropolymers, nylon, polyester, ABS, and flexible and rigid PVC. Extruded plastics often have a higher melt viscosity, which allows the extrudate to retain the shape imparted to it by the die while the extrudate is in the

quenching stages. Incoming raw material evaluations will be performed on all polyethylene resin used for the production of pipe. The incoming raw material evaluations will consist of a density and melt index test on each lot of polyethylene resin for extrusion process.

Combinations of various resins can be used to gain special rheological, physical properties. Many additives can be used during the extrusion process to enhance processing characteristics of the polymer or to alter product properties. Such additives include lubricants, thermal stabilizers, antioxidants, radiopacifying agents, and colorants.

Combinations Of HDPE and LLDPE Material

By identifying the best combination of recycled combined HDPE and LLDPE pipe resin materials by changing various percentages (by weight) of both the resin materials. The rheological properties such as Melt Flow Index (MFI) and physical properties such as Density (ρ), are the major parameters while manufacturing in the HDPE pipes. Changing the various percentages (by weight) in both HDPE and LLDPE recycled granule materials in the extrusion process, the pipe profiles are produced, till it obstructs to extrude in the die. Normally the manufacturers and the customers need the best pipe profiles. These combinations have been done in the HDPE pipe with an outside diameter of 63mm and pressure rating (PN) 12.5 kg/cm² in the industry and the weight of the recycled pipe product are 550 grams/meter. These combinations of recycled HDPE and LLDPE resin materials are extruded in three different temperatures with various percentages weight. The following Table 1 shows the various temperatures and combinations of various percentages (in weight) for both recycled HDPE and LLDPE resin materials could be added in extrusion process. The temperatures are one of the important parameters in extrusion process to melt the resin materials. Decreasing or increasing the temperatures in extrusion process causes changes in chemical, physical properties and the extruded pipe profiles. The three temperatures such as 80°C, 83°C and 86°C are arrived at by industry based on experience and to ensure the best pipe profiles by extrusion process.

Table. 1 Various wt% for HDPE & LLDPE Resins in Extrusion Process at different temperatures

Matl. No	Temp. in °C	HDPE Granl. (wt%)	HDPE Granl. (gms/mtr)	LLDPE Granl. (wt%)	LLDPE Granl. (gms/mtr)
1	80	75	412.5	25	137.5
2		80	440	20	110
3		85	467.5	15	82.5
4	83	75	412.5	25	137.5
5		80	440	20	110
6		85	467.5	15	82.5
7	86	75	412.5	25	137.5
8		80	440	20	110
9		85	467.5	15	82.5

HDPE and LLDPE Resins at 80°C

The temperature at 80°C, in the extrusion process, the combinations of recycled combined HDPE and LLDPE resin materials is not able to flow freely in the extruder barrel, due to the improper density, viscosity and thermal sensitivity of the resin being extruded. The extruder is used to heat the raw material and then force the resulting melted polymer through the pipe extrusion die. The extruder screw operates the resin materials with more stick principles in extrusion process at low temperature of 80°C. The polymer needs to stick to the barrel with the appropriate temperatures.

Decreasing the temperature in the extruder barrel there is not enough sufficient to provide to flow for resin materials by the extruder screw. Due to the improper molecular interaction between these both resin materials in extrusion process, there is a change in properties such as density and melt flow rates, so that, as the screw rotates, it forces the material in a forward direction with the bad pipe profiles. At 80°C the polymer materials of the recycled both HDPE and LLDPE resins are providing the bad physical changes in the extruded pipe profiles with these combinations such as, HDPE and LLDPE in 75, 80, 85% and 25, 20, 15% (by weight) respectively. These pipe profiles are shown in fig. 8a), 8b) and 8c).

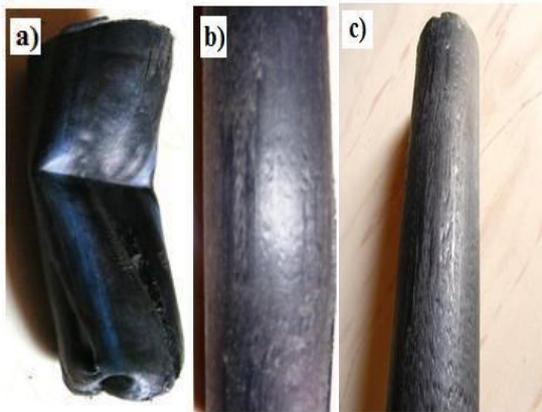


Fig. 8 Bad pipe profiles Extruded at 80°C

HDPE and LLDPE Resins at 83°C

Maintaining the temperature at 83°C, in the extrusion process the combinations of recycled combined HDPE and LLDPE resin materials is to flow freely in the extruder barrel. By increasing the temperature from 80°C to 83°C, the polymer materials of the recycled combined HDPE and LLDPE resins are providing the better extruded pipe profiles with these combinations such as, HDPE and LLDPE at 80 and 20% (by weight), due to the density, viscosity and thermal sensitivity of the resin materials. In HDPE and LLDPE at 75, 85% and 25, 15% (by weight) of these resin materials are not providing the sufficient pipe profiles in extrusion process. The perfect molecular bonding and the melt flow rate of HDPE and LLDPE at 80 and 20% (by weight) combinations of the resin materials are achieving the better pipe profiles at 83°C. The materials melt flow rate is mainly depends on molecular weight, additives, and other ingredients, the melt flow index value allows for direct comparison of materials to each other, so that better pipe profiles can be achieved by providing the proper temperature at 83°C of recycled HDPE and LLDPE granules in extrusion process. These pipe profiles are shown in fig. 9a), 9b) and 9c).

HDPE and LLDPE Resins at 86°C

By increasing the temperatures from 83°C to 86°C in the extrusion process that extrudes the HDPE and LLDPE resins with less viscous and more thermally stable. Temperature in 86°C the combinations of various percentages in weight additions of recycled combined HDPE and LLDPE resins are flow freely in extruder die. Increasing the temperature in melting zone at the heating chamber by the temperature control unit, melting temperature of these resin materials are very high. The melting temperature also mainly depends on molecular weight,



Fig. 9a) and c) shows the bad pipe profiles extruded at 83°C and the fig. b) shows the extruded a better pipe profile.

These extrusion process has been done in the combinations such as, HDPE and LLDPE at 75, 80, 85% and 25, 20, 15% (by weight) respectively. The bad pipe profiles can be extruded with variations of thickness, inner and outer surfaces have been identified in these pipe profiles by the melting temperature at 86°C in the extrusion process. These pipe profiles at various combinations of HDPE and LLDPE resin materials are shown in fig. 10a), 10b) and 10c).



Fig. 10 Bad pipe profiles Extruded at 86°C

additives, and other ingredients, the melt flow index value of its own. The melt flow rate allows for direct comparison of materials to each other.

V. EXPERIMENTAL TESTING

In the selection of a polymer for a specific use, a clear understanding of physical and rheological properties is essential to obtain the best performance. Many test methods are available to predict the physical and rheological performance of a polymer under certain conditions. Here the two experimental test methods are predicted in the better

combinations of recycled HDPE and LLDPE composite materials.

Melt Index Measurement as per ASTM Standard D-1238

The melt flow rate measures the viscosity of the polyethylene resin in its molten state and determines the ease of flow of plastic materials. It is a parameter related to the average molecular weight of the resin chains of polymer extruded through a standard size orifice under specified conditions of pressure and temperature in a ten-minute period of time.

$$\text{Flow rate} = (426 \times L \times d)/t$$

(OR)

$$\text{Volume rate} = 426 \times L/t$$

Where,

- L = Length of calibrated piston travel in cm,
- D = Density of resin at test temperature in g/cm^3 , and
- T = Time of piston travel for length in sec.

A true rheological characterization can be carried out by means of rheometers. Melt flow rate is inversely proportional to (shear) viscosity. The melt index test results for recycled combined HDPE and LLDPE polymer composites were shown in Table 2. These MFI test results were plotted in fig. 12.

Density Measurement as per ASTM Standard D-792

The density measurement of the polymer materials are under the physical tests. The density of polyethylene is a measure of the proportion of crystals within its mass. Crystals, a result of the layering and close packing of polyethylene molecules, are denser than the tangled, disordered arrangement of molecules in the amorphous regions. Density is a measure of the “compactness” of matter within a substance and is defined by the equation

The standard metric units in use for mass and volume respectively are grams and milliliters or cubic centimeters. Thus, density has the unit grams/milliliter (g/ml) or grams/cubic centimeters (g/cc). Densities of the films were measured at 23°C with a density gradient column filled with aqueous methanol solutions. The density is measured with a Mettler ME-40290 installed on a Mettler AE 240 scale. The density test results for the recycled combined HDPE and LLDPE polymer composites at different temperatures are shown in Table 3. These density test results were plotted in fig. 13 .

$$\text{Density g/cc, } \rho_1 = (A/B) \times \rho_s$$

Where,

- ρ_1 = Density of the Sample,
- ρ_s = Density of methanol (0.86 g/cm^3 at 24°C),
- A = Weight of the sample in air, and
- B = weight of the sample in methanol.



Fig. 11 Displacement method test for measuring the Density

VI. RESULT AND DISCUSSIONS

Melt Flow Rate Tests Of Thermoplastics by Extrusion Method

In the lower melt flow rate, the length of molecules and molecular weight are as greater. Due to the greater molecular weights the difficulty in extruding the resin through the standard orifice. Thus the results of resins of greater viscosity as measured by a lower melt flow rate. The test is conducted with pressure delivered by a standard load caused by a 2.16 kg weight at a temperature of 374°F (190°C), the resulting melt flow rate is termed the melt index (MI) are shown in table 2.

From the Melt flow index measurement table 2 shows, it was observed that the Melt flow index (MFI) values of recycled combined HDPE and LLDPE polymer composites are higher in different percentages (by weight) at different temperatures such as 80 , 83 and 86°C are compare to the material (i.e. HDPE and LLDPE at 80 and 20% (by weight) at 83°C).

A polymer containing a broad range of chain lengths and have a broad molecular weight distribution (MWD). Resins with this type of distribution have good Environmental Stress Crack Resistance (ESCR), good impact resistance and good processability. MWD is dependent upon the type of process used to manufacture the particular polyethylene resin. For polymers of the same density and average molecular weight, their melt flow rates are relatively independent of MWD. Therefore, resins that have the same density and melt index (MI) can have very different molecular weight distributions.

Table. 2 MFI test results for Recycled combined HDPE-LLDPE composites

Matl. No	Temp. in °C	Recycled Polymer materials in wt%		MFI in g/10 min
		HDPE	LLDPE	
1	80	75	25	0.50
2		80	20	0.48
3		85	15	0.50
4	83	75	25	0.49
5		80	20	0.30
6		85	15	0.48
7	86	75	25	0.50
8		80	20	0.49
9		85	15	0.48

A polymer containing a broad range of chain lengths and have a broad molecular weight distribution (MWD). Resins with this type of distribution have good Environmental Stress Crack Resistance (ESCR), good impact resistance and good processability. MWD is dependent upon the type of process used to manufacture the particular polyethylene resin. For polymers of the same density and average molecular weight, their melt flow rates are relatively independent of MWD. Therefore, resins that have the same density and melt index (MI) can have very different molecular weight distributions.

The average molecular weight, as measured by the MI, does not identify the range of chain lengths within the molecules. Polyethylene polymers of the same MI and the same density may have very different properties if the molecular weight distributions (MWD) are different. A polymer with a narrow MWD will crystallize more rapidly and with greater uniformity, resulting in less warpage and greater fidelity to the intended geometry. A polymer with broad MWD may have better stress crack resistance, impact resistance and ease of processing.

At ten-minute period of time in extrusion process the materials are achieving the greater the viscosity and the lower the melt index value. A lower MI (higher average molecular weight) is predictive of greater tensile strength, toughness, greater stress crack resistance, and the greater energy required at any extrusion temperature to extrude polyethylene resins.

It was observed that the recycled combined HDPE and LLDPE polymer composites having the lower Melt Index at 83°C with the combinations of 80 and 20% (by weight) respectively.

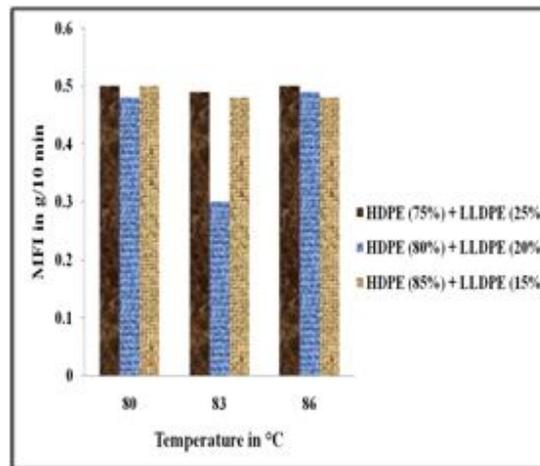


Fig. 12 MFI tests result for recycled combined HDPE-LLDPE by wt%

Density Test of PMC by Displacement Method

Table 3 Density test results for Recycled combined HDPE-LLDPE composites

Matl. No	Temp. in °C	Recycled Polymer materials in wt%		Density at 23 °C in g/cm ³
		HDPE	LLDPE	
1	80	75	0.90	0.50
2		80	0.89	0.48
3		85	0.85	0.50
4	83	75	0.90	0.49
5		80	0.95	0.30
6		85	0.92	0.48
7	86	75	0.89	0.50
8		80	0.89	0.49
9		85	0.90	0.48

From the density measurement table 3, it was observed that the density of recycled combined HDPE and LLDPE polymer composites was different at different temperatures such as 80°C, 83°C and 86°C in the different combination.

It is observed that recycled combined HDPE and LLDPE polymer composites will have higher density at 83°C with the combinations of 80 and 20% (by weight) due to the higher molecular interaction between these both resin materials.

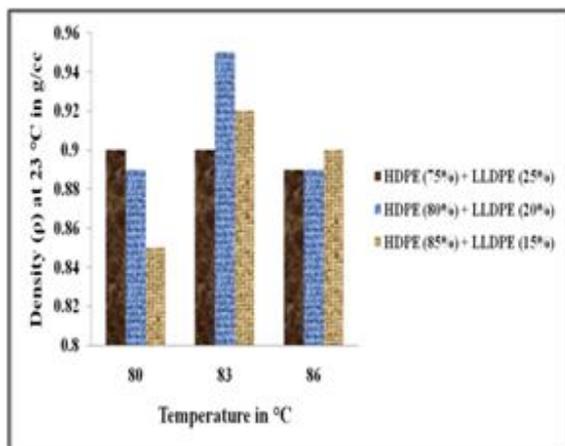


Fig. 13 Density tests result for recycled combined HDPE-LLDPE by wt%

VII. CONCLUSIONS

From the Melt flow index measurement (Table. 2), it was observed that the lower Melt Flow Index (higher average molecular weight) were optimized at 83°C (Extrusion temperature).

By the density measurement (Table 3), it was predicted that recycled combined HDPE and LLDPE at 80 and 20% (by weight) were optimized, due to the higher density of 0.95 g/cc.

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