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Effect of physical properties of samples on the mechanical characteristics of high-density polyethylene (HDPE)

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Abstract. This study aimed an experimental data about the effect of physical properties of samples on the mechanical characteristics of high-density polyethylene (HDPE) used for the manufacture of preinsulated pipes for heating networks. The main waterproofing insulation material in the construction of preinsulated pipelines for heating networks is polyethylene. Outer outer casing of preinsulated pipes made of high density polyethylene (HDPE) are used as a waterproofing insulation of polyurethane foam of steel pipes manufactured in the factory. The sufficient resistances to cracking and mechanical stress, resistance to ultraviolet radiation, high density, high strength, low aging are main properties of HDPE. The study of HDPE of different manufacturers is relevant because of the aging of preinsulated pipelines with HDPE outer casing. In this study are tested physical characteristics of HDPE as a melt flow rate, density at 23°C, water content of PE80, PE100 pipe polyethylene of different world manufacturers. The best samples of HDPE and the effects of various aging factors on the properties of polyethylene are presented.

Keywords: building materials; polyethylene; HDPE; heating networks; material properties

1. Introduction

High Density Polyethylene (HDPE) is defined by EN DIN 8075 as a thermoplastic polymer made from petroleum (EN DIN 8075 2013). HDPE is one of the most popular plastic materials, which used for plastic bottles, cutting boards, milk jugs, piping and more. Polyethylene (PE) belongs to thermoplastic polymers which are composed of long chains forming macromolecules (Kalmagambetova and Bogoyavlenskaya 2020). These chains are formed by polymerization reactions. These chains of PE molecules contain about 50,000 molecules of ethylene. They form a partially crystalline structure, i.e., partly amorphous and partly crystalline (Soltaninezhad *et al.* 2020). Due to this PE has a wide range of physical and mechanical properties. They are low density, good dielectric properties, resistance to radioactive radiation, retention of its properties at negative temperatures, strength, low aging, low water absorption, good weldability (Torres-Huerta *et al.* 2019). The typical properties of HDPE 100 are in the table below (Technical Datasheet Formosa Plastic Corporation 2010).

PE for pipes is classified into grades PE 63, PE 80, PE 100 depending on the level of load. This

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Table 1 Typical properties of HDPE 100

Properties	Units	Test method	Typical value
Melt Index MI 2.16	g/10 min	ASTM D1238	0.05
Melt Index MI 5	g/10 min	ASTM D1238	0.23
Density	g/cm^3	ASTM D1505	0.958
Thermal properties			
Melting point	°C	DSC	129
Brittleness point	°C	ASTM D746	< -70
Mechanical properties			
Tensile strength at yield	Kg/cm^2	ASTM D638	240
Tensile strength at break	Kg/cm^2	ASTM D638	360
Elongation at break	%	ASTM D638	850
Izod Impact Strength	Kg-cm/cm notch	ASTM D256	> 30
Hardness	Shore D	ASTM D2240	64
Thermal stability (200°C)	min	ISO/TR 10837	> 30

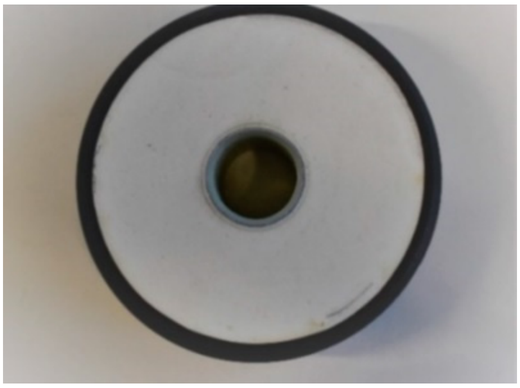


Fig. 1 Cross-section of a PU pre-insulated pipe with outer casing-pipe from HDPE (Doyle *et al*. 2019)

study has considered PE 80 and PE 100 as the most suitable grades for the production of pipes.

The novelty and the main purpose of this work was to study and compare the physical and mechanical properties and effect of pipe grades of polyethylene with the subsequent selection of the best raw materials for waterproofing underground pipelines of heat networks in case of its durability and life time.

It is known that preinsulated bonded pipe systems for directly buried hot water networks which made in the factory environment from steel service pipe, polyurethane thermal insulation and outer casing of polyethylene are used with heat operation temperature 90-130°C for district heating (Danielewicz *et al*. 2016, Kayfeci 2014, Turski and Sekret 2018, DIN EN 253-2013 2013, DIN EN 448-2009 2009, Huang *et al*. 2017).

Polyethylene for outer casing of preinsulated pipes of grade PE100 has good technological properties, however, despite the high melt viscosity MFR -0.2-0.7 g/10 min (EN DIN 8075 2013).

The presence of lower molecular weight fraction in the melting plays the role of grease and facilitates the extrusion of the polymer.

The study of the quality of polyethylene of different manufacturers is relevant because of aging of HDPE and life time of underground pipelines.

Therefore, there is necessity to study the raw materials, in particular granulated material for the production of polyethylene pipes, since the properties of polyethylene of different manufacturers are different (Huang *et al*. 2017, Wang *et al*. 2018, Chopra *et al*. 2018).

2. Methods

The most important properties of HDPE for the production of outer casing for preinsulated heat pipes, which are key in the selection of raw materials and influence the quality of the material, are the melt flow index, the density of the polyethylene, and the water content. The Melt Flow Index is important for welding polyethylene (fittings) and can be between 0.24-0.27 g/10 min as a result from experience in extrusion on a specific machine, plastic pipe production line Extruder by Battlefield. The MFR is important for extruding pipes. It must be sufficient to allow the PE mass to exit the extruder within a certain time without scratches, caverns and other inclusions. The density of polyethylene largely determines its further properties, including strength, impact resistance, thermal insulation properties and durability. The water content of polyethylene must be investigated on the basis of polyethylene processing conditions, so that shrinkage of the raw material does not occur when it is heated to the required temperatures.

The test samples were HDPE pellets of PE 80 and PE 100 grades, black and white, from different manufacturers in different countries. The pellets were weighed for each test respectively. The melt flow rate (MFR), water content and density of HDPE were tested. The tests were carried out as follows.

The melt flow rate (MFR) was investigated on the Melt Flow Junior 6942 extrusion plastomer by Ceast.

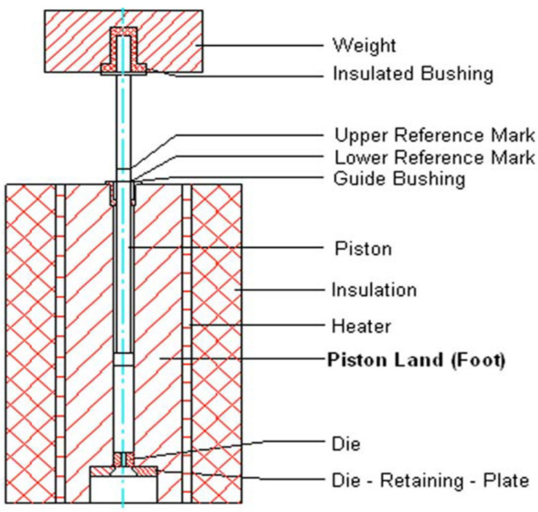


Fig. 2 - General Arrangement of Extrusion Plastometer (ASTM D1238 2013)

Table 2 Normative documents for determining PE properties

Indicator	Normative document	Unit
Melt Index	ASTM D1238	g/10 min
Density	ASTM D1505	g/cm^3
Carbon black content	ISO 6964	%
Water Content	EN 12118	mg/kg

Melt Flow Junior extrusion plastometer was used to determine the melt flow index of the HDPE pellets. The essence of the method consists in determining the mass of the material in grams extruded from the device for 10 minutes under given conditions of temperature and pressure. The samples in the form of granules (pellets), ensuring its introduction into the hole of the extrusion chamber were used for testing. The device was heated without sample to temperature of 190°C and kept at this temperature for 15 minutes. Upon reaching the required temperature (190°C), the cylinder was checked for contamination. The piston was taken out, a material sample of 4-5 g was loaded into the extrusion chamber with the help of a funnel. To prevent air from entering the material under test, its loading time should not exceed 1 minute. The device was heated (heating time is 360 seconds). The piston was inserted into the chamber, slightly pressing, the material was manually compacted, and loading of 5 kg was placed on the sleeve. The time interval for cutting extrudate (flow HDPE) - 120 seconds was set on the device. The samples were taken in the form of 5 segments of extrudate, coming out one after the other. Each segment was weighed and the total weight of five segments was calculated. This indicator is an indicator of melt flow and is measured in g/10 min.

Density at 23°C was studied on the Melt Flow Junior 6942 extrusion plastomer (ASTM D1505 2018). Melted samples from the Melt Flow Junior extrusion plastometer in the amount of 3 pieces (l = 4 cm) were subjected to further density determination tests. The glass was filled with water and installed on a stand with weights. After installing the instrument on the scale, the scale is reset. Next, the sample was weighed. A sample of the extrudate was placed under the lid in a glass of water and its bulk weight (V) was recorded. The average value of three samples was taken as the result.

The mass fraction of volatile substances (water content) was determined in an oven (dryer) produced by Binder FED (EN 12118-2013 2013). The dryer was pre-heated to 105 ± 0.2°C. The test glasses were numbered and weighed with an accuracy of ± 0.0002 g. Further, the glasses were filled with the test material (20 ± 1) g, weighed with the same accuracy and dried at temperature (105 ± 0.2)°C for 2 hours. Then the glass with the sample was cooled for 1 h to temperature of (23 ± 0.2)°C and weighed. The average value of three samples was taken as the result. Table 2 shows the regulations for determining the basic properties of PE for pipes.

The study of the microstructure of HDPE of the selected samples of the initial samples by electron scanning microscopy using a JSPM-5200 atomic force microscope produced by JEOL, Japan.

3. Results and discussion

The test samples were HDPE pellets of PE 80 and PE 100 grades, black and white, from different manufacturers in different countries. The pellets were weighed for each test respectively. In Table 3, the samples are numbered 1-17 for ethical reasons.

The samples of HDPE pellets are shown in Fig. 3.

Table 3 Research data on HDPE of different grades and manufacturers

Polyethylene sample number	Color, grade	Melt flow rate, g/10 min	Mass fraction of volatile substances, %	Density at 23°C, g/cm^3	Soot content, %
1	Black, PE 100	0,25	0,02	0,957	2,25
2	Black, PE 100	0,23	-	0,958	2,25
3	Black, PE 100	0,241	-	1,013	2,25
4	Black, PE 100	0,21	-	0,97	2,3
5	Black, PE 100	0,24	-	0,959	2,3
6	Black, PE 100	0,221	0,02	1,02	2,21
7	Black, PE 100	0,227	0,27	1,015	2,25
8	Black, PE 100	0,8	-	-	2,25
9	Black, PE 80	0,138	0,05	0,977	2,25
10	White	0,158	-	1,019	0
11	Black, PE 100	0,43	-	1,017	2,25
12	Black, PE 100	0,43	-	1,01	2,25
13	White	0,259	-	0,988	-
14	Black, PE 100	0,263	-	0,986	2,2
15	Black, PE 80	0,272	-	0,994	-
16	Black, PE 80	0,26	-	1,012	2,2
17	Black, PE 80	0,56	-	1,008	2,2

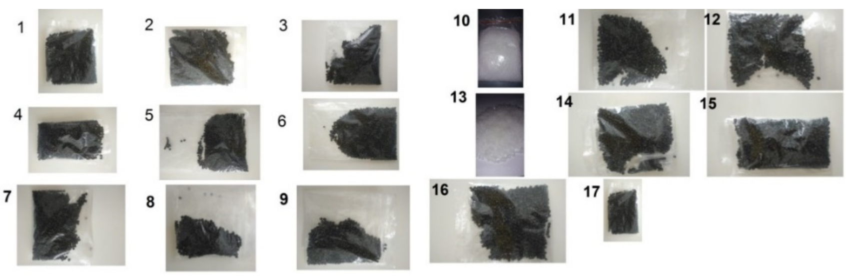


Fig. 3 Samples of HDPE pellets of different grades and manufacturers

A comparative analysis of the melt flow index of polyethylene granulated material of different manufacturers is shown in Fig. 4.

The melt flow rate (melt index) of polyethylene (MFR of polyethylene) characterizes its viscosity, and the higher this indicator, the more fluid and less viscous polyethylene is. Extrusion processes for the production of outer casing pipes require high melt viscosities; therefore, raw materials with low melt flow index are used.

The MFR of polyethylene pipe is in the range of 0.2 ... 0.7 g/10 min and the lower the index, the better for welding of polyethylene. This indicator is important in the manufacture of shaped products made of polyethylene on special welding machines. It should be noted that the polymer melt viscosity substantially depends on the applied load.

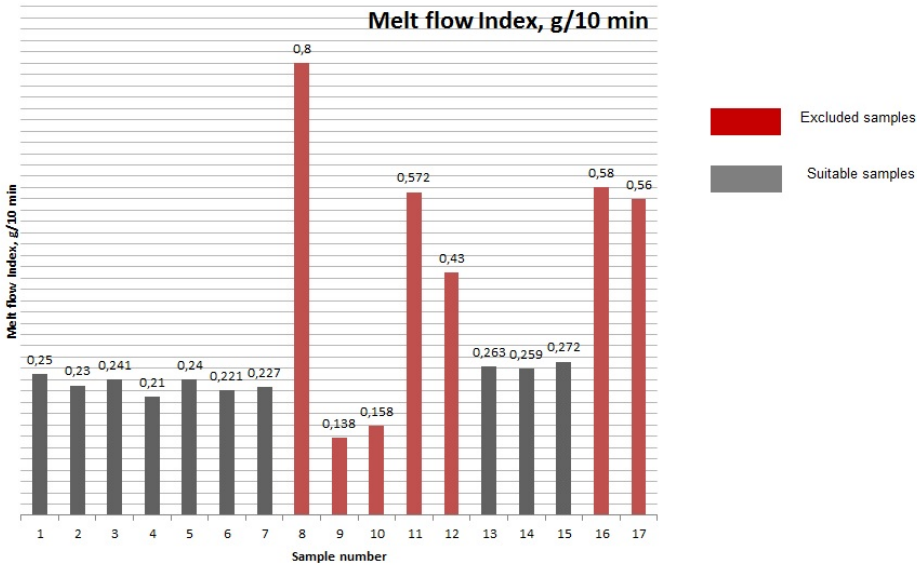


Fig. 4 A comparative analysis of the melt flow rate of the HDPE of different manufacturers

As can be seen from a comparative analysis of the flow index of polyethylene of different manufacturers (Fig. 4), the following samples can be used as raw materials for the production of outer casing pipes: 1 (MFR 0.25); 2 (MFR 0.23); 3 (MFR 0.241); 4 (MFR 0.21); 5 (MFR 0.24); 6 (MFR 0.221); 7 (MFR 0.227); 13 (MFR 0.263); 14 (MFR 0.59); 15 (MFR 0.272); 16 (MFR 0.26).

The study of the density of polyethylene pipe at 23°C determines not only the physical and mechanical properties, but also the possibility of processing on the extrusion line. With increasing density, the strength, rigidity and chemical resistance of the material, including heat resistance, increase. However, impact resistance drops at low temperatures, elongation and permeability to moisture, gases and vapors decrease at break.

As follows from a comparative analysis of the density of polyethylene granulated material, all the studied grades of polyethylene showed satisfactory results and can be used as raw materials for the production of outer casing pipes.

It is known that soot is the best ultraviolet absorber; therefore, materials for the production of outer casing pipes, pressure pipes for gas and water supply are produced mainly in black. The standards clearly state that the soot content for grades should be 2.0–2,5 wt.%. The crawling of soot can not only lead to a decrease in resistance to UV radiation - large soot agglomerates can work as local stress concentrators, which increase the likelihood of material destruction during the operation of pipes. Hence the requirement for the material for pressure pipes - stuff dyeing at the stage of the production of grade assortment - at the powder compounding (Sadr-Al-Sadati and Ghazizadeh 2019, He *et al*. 2018, Deveci and Fang 2017).

Recently, undyed PE 100 grades, mostly by manufacturers, have appeared on the market, the use of which for the production of pipes is not allowed.

The study of polyethylene granuled material of different manufacturers in terms of soot content showed that samples 10 and 13 are not suitable for the production of outer casing pipes because of white color.

Carbon black (soot) is an amorphous carbon, which differs in grades in the range of particles size, determining its coloring ability, has excellent weather and chemical resistance. Functional groups formed on the surface of carbon black, depending on the technology of production, affect its properties.

In the work (Zanasi *et al*. 2009), it is proved that an increase in the stability of polyethylene is achieved at a dosage of carbon black of 2.0–2.5% a further increase in concentration does not lead to an increase in the inhibitory effect.

The stability of the polymer in atmospheric conditions increases with an increase in the degree of dispersion of carbon black and a decrease in the size of its particles. So, channel black with smaller particles, in contrast to furnace black, has a higher protective effect. The effect of black is not limited to the absorption of sunlight. The studies have shown that soot also serves as an antioxidant. For example, the addition of carbon black reduces the destruction during the flaring of PE (Kimelblat *et al*. 2015, Bazhanova and Erofeev 2012). Soot slows down the rate of oxygen absorption during the oxidation of polyethylene, i.e., it acts similarly to typical antioxidants, increasing the induction period.

The effect of carbon black on the polyethylene aging has been studied in considerable details. Soot is a much more efficient light stabilizer than other light-absorbing pigments, and is quite competitive with some aromatic UV absorbers or oxygen traps. It has been proven that the effectiveness of soot as a stabilizer for thermoplastics directly depends on the content, particle size and degree of dispersion, i.e., the degree of fragmentation of the secondary aggregates of soot particles.

Soot affects the inhibitory activity of other antioxidants. For PE, it has been proven that as a result of adsorption of the volatilization of low molecular weight inhibitors is reduced on soot particles. This inhibitor retention effect also depends on the size of soot particles and is appeared on channel black (particle size less than 300 Å) more than on furnace black (particle size more than 400 Å). Thus, soot grades with a particle size of less than 300 Å are most preferred for concentrates for the manufacture of black plastic products. With the right choice of antioxidant, it is even possible to get a synergistic effect in thermal oxidation of a polymer colored with soot.

The creation of active oxygen-containing groups on the soot surface increases its inhibitory ability. To do this, soot is specially heated in the presence of oxygen, sulfur, selenium. It is known (Bazhanova and Erofeev 2012) that the addition of only 0.2–0.5% of a mixture of channel black (particle size less than 20 Å) and furnace black (particle size more than 250 Å) to polyethylene obtained on Ziegler – Natta catalysts significantly improves its light resistance (Prishchepa *et al*. 2015, Nezbedová *et al*. 2017).

At the production of polyethylene pipes, an undesirable effect is the formation of internal or surface micro- and macropores. The reason for this phenomenon may be the increased content of volatile (low molecular oligomeric) fractions and water in the feedstock (pipe grade polyethylene). Traditionally, polyolefins are considered to be practically inert to water. The presence of surface sorption moisture in PE is not in doubt. However, experimental data show that water is contained not only in the surface layers of PE, but also inside the granules. Not all the samples were taken for the test, but only a selection.

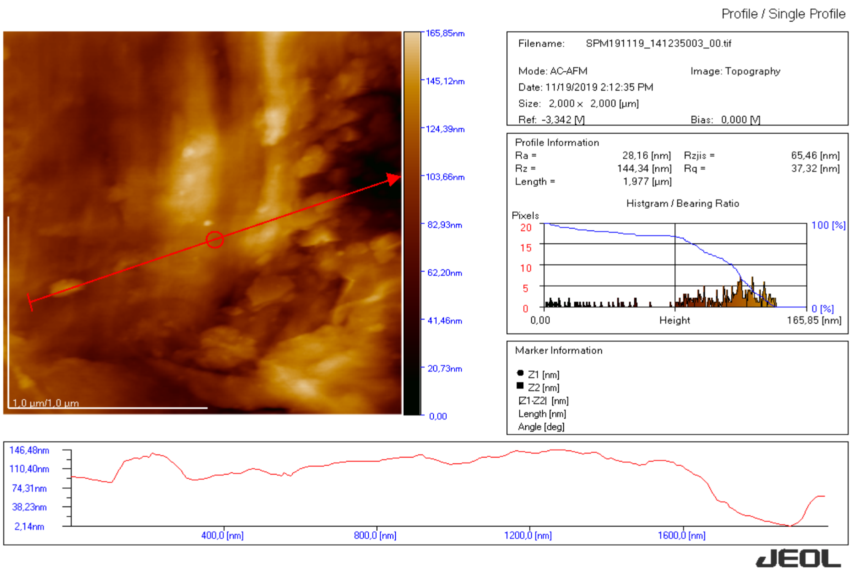
In terms of the content of volatile substances, only some grades of granulated material were investigated: 1 (0.02%); 6 (0.02%); 7 (0.27%); 9 (0.05%) because of low water content (EN 12118-2013 2013).

In order to confirm the correct choice of polyethylene pellets for the production of casing pipes, samples of polyethylene pipes were produced in a factory in Kazakhstan on an industrial extruder. A polyethylene sample was cut out and its surface evaluated using the microscope described in the

"Methods" section.

The polyethylene casing pipe is produced in the factory on a pipe extrusion line. The extruder consists of the following sections: loading material - pellets into the dosing device by mass; its melting at a temperature of around 200°C; the shaping of the tubes-shells in various diameters from 90 mm to 1425 mm; the multi-stage water cooling of the PE pipes. As can be seen, polyethylene is subjected to heating and cooling. The plastic PE mass transforms into a solid substance. Fig. 5 shows a micrograph of the surface of polyethylene.

The microphotographs showed the results of the transformation of substances (liquid phase to solid phase). Two phases - crystalline and amorphous - can be seen in the picture). This gives the pipe polyethylene its basic properties - strength and stability - and at the same time a clearer picture of what happens after extrusion and how it can be used.



(a)

(b)

Fig. 5 Microphotographs of the surface of polyethylene samples: (a) topography of the surface of polyethylene; (b) the phase of polyethylene

4. Conclusions

From the conducted research it should be assumed the effect of physical properties of samples on the mechanical characteristics of high-density polyethylene (HDPE) for outer casing of preinsulated pipes for district heating networks. Polyethylene for the production of polyethylene outer casing of different manufacturers is different. The melt flow index of polyethylene of different manufacturers was different and only a few of the raw material samples showed the right results 0,2-0,7 g/10 min. As follows from a test results on the density of polyethylene granulated material, all the studied grades of polyethylene showed satisfactory results and can be used as raw materials. The study of polyethylene granulated material of different manufacturers in terms of soot content showed that few samples are not suitable for the production of outer casing pipes made from high-density polyethylene. It became clearer that only quality-tested polyethylene samples may be used for pipe extrusion.

The effect of HDPE properties on the durability or aging of the finished product as a preinsulated pipe for heating networks has been assessed.

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