Investigation of the Extrusion Process Influence on the Thermophysical, Rheological Properties and Operational Characteristics of Insulating Polymers

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Abstract. In this work, we carried out complex experimental studies of the thermophysical and rheological properties of the initial and secondary (after extrusion) polymer compositions based on low, medium and high density polyethylene, intended for the cable insulation production. On the basis of the obtained experimental data, a comparative analysis of the numerical study results of the flow processes in the extruder metering zone with a constant technological regime was carried out. Changes in the graphics of maximum and average temperature values along the length of the channel are received.

1. Introduction

At present, pure polymers such as polyethylene are rarely used in industry. They are replaced by polymer compositions with various fillers, which make it possible to give the finished product necessary performance characteristics. At the same time, depending on the composition, their thermophysical properties may differ significantly. Among the known methods of processing such materials, extrusion is the most frequently used method, which allows to continuously obtain products of a sufficiently large length [1].

In the modern cable industry during extrusion, along with the well-known problems caused by the occurrence of local overheating, there are also difficulties with utilization of the polymer materials that remain in the transition from one brand-size cable to another [2]. In this regard, it is important to study the extrusion process influence on the thermophysical, rheological properties and operational characteristics of polymer materials and to evaluate the possibility of their reuse in cable production.

2. Experiments

At the first stage, a study was conducted of the polymer materials thermal stability range on Discovery TGA manufactured by the TA Instruments [3]. Determination of the thermal stability range was carried out for polymer compositions based on low density (PE 153-10K) and high density polyethylene (PE 273-81K) of domestic production, and medium density polyethylene (Borealis ME 6052). The maximum heating temperature in experiment was 600 °C, at a heating rate of 10 °C/min. The results of the study are shown in the figure 1 (a).

The next step of the thermal parameter research was determination of the polymer compositions specific heat capacity (Figure 1,b) [4]. The study was conducted on the differential scanning calorimeter DSC Q2000 at a heating rate of 20 °C /min, in temperature range (50–200) °C.
Figure 1. Weight loss curves (a) and specific heat capacity temperature dependence (b) of the original polymer compositions: ● – Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K; and secondary (after processing): ● – Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K.

Analysis of the curves in Figure 1,a showed that the main mass loss of all studied materials occurs at a temperature (400-500 °C). However, it is worth to note that the decomposition of the low molecular weight portion of each sample, which is approximately 5%, begins when the temperature value reaching 300 °C. When samples are heated to temperatures above 400 °C, decomposition of the remaining high molecular weight structure begins. That part of the material that did not burn at a temperature above 600 °C consists of mineral fillers, which are less in domestic polymer compositions (1%) than in foreign material Borealis ME 6052 (3%). It is also worth to note that the processing process does not affect on the thermal decomposition beginning temperature.

From the graph of specific heat capacity temperature dependence (Figure 1,b) it is clear that the phase transition to the molten state in case of low density polyethylene composition occurs at lower temperatures than the other two materials. The magnitude of the maximum value, as well as the curve shape, depends on the polymer material brand and the fillers included in its base. In the case of materials after processing, an increase in the maximum value of specific heat capacity is noted. Herewith in the melt state, all polymer compositions, both the original materials and the secondary materials, have similar average heat capacity values and low temperature dependence.

At the next stage of experimental studies, temperature dependences of thermal conductivity (Figure 2,a) and density (Figure 2,b) were obtained. The first parameter was determined on the FOX 50 analyzer, which is based on the stationary method of heat flux measuring [5]. To determine the temperature dependence of density, a horizontal type dilatometer DIL 802 was used. [6].

Figure 2. Temperature dependence of thermal conductivity (a) and density (b) the original polymer compositions: ● – Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K; and secondary (after processing): ● – Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K.

The difference in the thermal conductivity values of the materials in solid state is more significant
(the maximum difference is 20%). In the melt state, the curve slope is changing, and with increasing temperature the difference in values decreases to 12%. It is also worth to note that the composition based on high density polyethylene in the entire temperature range has the highest value of thermal conductivity. A comparison of the obtained data allows us to conclude that in the case of medium and high density polyethylenes the processing process has a significant effect on the investigated parameters, in contrast to low density polyethylene.

Analyzing the curves of Figure 2,b, it can be noted that all materials in the solid state have a small temperature dependence of density (the value is less than 0.5%). During transition to the molten state, the slope of the curve increases, and the dependence becomes more pronounced (from 120 °C to 240 °C the density value decreases by 1.5%). It is also worth to note that all the studied polymer compositions in the liquid state have a linear temperature dependence of density. At the same time, the extrusion process practically does not affect on the value of studied parameter.

Analysis of the polymer materials rheological properties was carried out on a Discovery HR-2 rotary type rheometer manufactured by TA Instruments [7].

On Figure 3 the dependence of effective viscosity on the shear rate for the studied polymer compositions at a temperature of 200 °C are presented.

![Graph showing the dependence of effective viscosity on shear rate](image)

**Figure 3.** The dependence of the effective viscosity on the shear rate of the original polymer compositions at T = 200 °C: ● – Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K; and secondary (after processing): ● –Borealis ME 6052; ♦ – PE 153 - 10K; ■ – PE 273 - 81K.

The Figure 3 shows that the extrusion process in the case of low density polyethylene (153-10K) has less influence on rheological parameters, in contrast to other two polymer compositions. The maximum difference is observed in the region of low shear rates.

Since the investigated polymer compositions are used as electrical cables insulation, their main operational parameters are the specific volume resistance $R$, dielectric constant $\varepsilon$ and dielectric loss tangent $\tan\delta$ [8,9].

Experimental determination of the dielectric constant and dielectric loss tangent was carried out using the MEP-SSA high-voltage AC bridge.

Table 1 presents the experimental data of the measured dielectrical parameters for the studied compositions before and after processing.

| Material | Measurements | $R$, TOhm·cm | $\tan\delta$, $1\cdot10^{-4}$ | $\varepsilon$ |
|----------|--------------|--------------|-----------------|-------------|
| 153-10K  | before processing | >24000       | 6.0             | 2.24        |
|          | after processing | >24000       | 6.0             | 2.17        |
| ME 6052  | before processing | 16890        | 5.3             | 2.41        |
|          | after processing | 20438        | 4.3             | 2.26        |
| 273-81K  | before processing | >24000       | 2.6             | 1.54        |
|          | after processing | >24000       | 2.0             | 2.21        |

Analyzing the obtained data, we can note that the processing process does not have a significant...
effect on the electrical properties of polymer compositions. All parameters are within acceptable limits, in accordance with the regulatory documentation for these materials.

In addition to the dielectric properties, the cables and wires polymer insulation must be resistant to mechanical stress, and at the same time provide the product necessary flexibility. For these purposes tensile tests are carried out.

An experimental study of the materials mechanical properties was carried out on a Walter Bai LFM-50-T tensile testing machine. For testing were used samples in the form of "blades" with a length of 115mm. The resulting stress-strain curves are shown below (Figure 4).

![Figure 4](image_url)

**Figure 4.** Experimental dependence of stress on strain of the original polymer:

- ● – Borealis ME 6052;
-♦ – PE 153 - 10K;
- ■ – PE 273 - 81K;

and secondary (after processing):

- ● – Borealis ME 6052;
-♦ – PE 153 - 10K;
- ■ – PE 273 - 81K.

A comparative analysis of the investigated materials mechanical properties suggests that the processing process has almost no effect on the polymers strength characteristics. The greatest difference in the maximum stress value was 4% for the polymer composition of the brand 273-81K.

3. **Mathematical modeling**

An experimental study of the extruded material properties showed that the processing process for a number of polymer compositions leads to a significant change in the thermal characteristics, especially thermal conductivity. In this regard, it is important to evaluate the behavior of the material during reprocessing and determine the rational mode of the extrusion.

To study the processes of flow and heat transfer in the metering zone of an extruder with an internal cylinder diameter of 160 mm, a screw channel model unrolled onto a plane was used. In the construction of spatial mathematical models of flow in the extruder metering zone following assumptions were made: the process is stationary and steady at constant mass flow, the polymer melt is considered to be a purely viscous, incompressible liquid, mass forces are equal to zero [11]. A more detailed mathematical formulation of the problem and the extruder channel geometrical parameters are described in the article [12].

Modeling was performed for the following technological processing mode: the body temperature was set at 200 °C; the temperature of the screw is 210°C; the temperature of the input material is 225°C; screw rotation speed was 60 rpm. This mode was used both for original polymer and for the material after first processing cycle.

Table 2 presents the values of temperature, viscosity, shear rate and pressure drop for all materials studied during both cycles primary and reprocessing.
Analyzing the obtained results, it can be noted that despite the unchanged technological mode parameters during the materials processing, the values of temperature, viscosity and pressure, that are realized in the extruder metering zone, differ significantly. Whereas the values of the shear rate in the channel remain unchanged. The greatest decrease in polymer temperature at the exit from the dosing zone was observed when reprocessing polymer Borealis ME 6052. The value of the maximum temperature at the channel exit decreases by 16 °C, and the average – by 10 °C.

4. Conclusions
As a result of experimental studies, it was found that the processing process does not lead to a significant change in the materials operational characteristics. It can be concluded that polyethylene-based polymer compositions can be reused in the manufacture of cables and wires electrical insulation. However, there are a number of important conditions, without which it is impossible to obtain high-quality finished product: All material should be collected in clean metal containers, excluding the possibility of various impurities, and the second – is ensuring the correct extrusion technological mode, without exceeding the temperature of thermal destruction.

Obtained in the course of numerical studies values of temperature, shear rate, viscosity and pressure drop allow us to conclude, that it is possible to re-process the investigated polymer compositions at an unchanged technological mode. At the same time, for polymer brand ME 6052, a decrease in the heaters temperature in extruder metering zone is allowed.

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Table 2. Changes in pressure, maximum and average temperatures, shear rates and viscosities for various polymer compositions

| Material | Processing cycle | T, [°C] | γ, [s⁻¹] | η, [Pa·s⁻¹] | ΔP, [MPa] |
|----------|-----------------|---------|--------|----------|---------|
|          | Max. | Ave. | Ave. | Ave. | Ave. |     |
| 153-10K  | first | 253.0 | 231.6 | 134.3 | 759.5 | 40.1 |
|          | second | 249.3 | 229.5 | 134.5 | 684.9 | 35.3 |
| ME 6052  | first | 273.4 | 243.5 | 135.2 | 1038 | 68.0 |
|          | second | 257.3 | 233.6 | 136.0 | 841.9 | 53.8 |
| 273-81K  | first | 309.0 | 263.1 | 135.2 | 1890 | 112.7 |
|          | second | 312.5 | 263.6 | 135.2 | 1938 | 116.6 |