Research of fire resistance and physical-mechanical properties of secondary polyethylene

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Abstract. This article provides information on a new type of filler used for secondary polyethylene and improving the fire resistance of secondary polyethylene. The article summarizes the physico-mechanical results of fillers, improves the properties of secondary PE, and creates the scientific basis for the production of composite materials with pre-planned properties.

1 Introduction

Aluminum hydroxide, magnesium hydroxide, polyphosphate, zinc borate, microencapsulated red phosphorus, basalt and other substances are used to increase the fire resistance of composite polymer materials based on polyolefins, including used polyethylene [1, 2].

Below are physical descriptions of some flame retardants used to modify polymer materials (PE, PP, PS, PVC, etc.), including secondary PE, to increase their fire resistance.

Table 1. Hydrofy G 2.5 from Nuova Sima of branded magnesium hydroxide physical descriptions.

No	Physical characteristics	Values	Test method
1	Density, -g/cm^3	2.36	ISO 787/10
2	Apparent density, (until compacted), g/cm^3	0.40	DIN 1060
3	Apparent density, (uncompacted), g/cm^3	0.60	ISO 787/11
4	Decomposition temperature, ^{0}C	350	ТГА/TGA
5	Comparative surface,- m^2/g	70	Flowsorb 2300
6	Moisture, %	0.35	ISO 787/2
7	Residual mass unit (sieve 44 μm), %	0.01	ISO 787/7

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Table 2. Aluminum hydroxide brand Alufy 6 of Nuova Sima physical descriptions.

No	Physical characteristics	Values	Test method
1	Density, -g/cm^3	2.40	ISO 787/10
2	Apparent density, (until compacted), g/cm^3	0.60	DIN 1060
3	Apparent density, (uncompacted), g/cm^3	0.80	ISO 787/11
4	Decomposition temperature, ^{0}C	180	ТГА/TGA
5	Comparative surface,- m^2/g	2.5	Flowsorb 2300
6	Moisture, %	0.02	ISO 787/2
7	Residual mass unit (sieve 44 μm), %	0.1	ISO 787/7

Table 3. Antimony trioxide (Sb_2O_3) produced by Nuova Sima physical descriptions.

No	Physical characteristics	Units of measurement	Norm
1	The mass content of antimony trioxide (Sb_2O_3) is	%	99.60
2	Average particle size		1 Micron
3	Belizna	Conditional size	94.7
4	Mass content of lead (PbO)	%	0.0115
5	Mass content of iron oxide (Fe_2O_3)	%	0.0012
6	Mass content of arsenic (As_2O_3)	%	0.03
7	Ressidue on 325	Mesh	0.010%

Table 4. Zinc/borate ($2ZnO•3B_2O_3•3.5H_2O$) manufactured by Nuova Sima physical descriptions.

No	Names of indicators	Values
1	Belize	98.4
2	Surface moisture content, %	≤ 0.28
3	Particle size, mcm	2.36
4	Mass content of zinc oxide (ZnO),%	37.5
5	Mass content of boron anhydride (B_2O_3), %	47.51
6	Amount of crystallized water, %	13.5
7	Relative mass, g/cm^3	2.67
8	Melting point, ^{0}C	980
9	Dehydration temperature, ^{0}C	320
10	Refractive index	1.58
11	The mass amount of the substance that did not pass through the mesh of the sieve with a size of 45 μm, %	≤ 0.1

2 Materials and methods

It is known that almost all polymers burn at temperatures above 350°C (in air). In order to reduce this flammability, we have chosen I-0760 grade of polyethylene (PE) raw material designed for processing under pressure injection by “Shurtan Gas Chemical Complex” LLC for experimental work. Because many types of polymer products are produced on the basis of this brand, as well as technological waste is generated during production processes. On the basis of technological waste, many secondary composite polyethylene materials are obtained, and during the period of operation of the obtained products, fire situations must be eliminated.

For this purpose, we conducted the following experiments with secondary PE of the selected brand I-0760. First, without adding any flame retardant to secondary PE, we studied its physical-mechanical, rheological, strength, heat and fire resistance properties according to Ts 17642168-04-2013 (Table 5).

Table 5. Physico-mechanical, rheological, strength and thermal properties of secondary PE.

No	Names of indicators	Inspection method	Research conditions	Unit of measurement	Nominal values
	Rheological studies				
1	Viscosity index	D 1238-2013	190^0C. 2.16 kg	g/ 10 min	7.0±0.5
2	Viscosity index	D 1238-2013	190^0C. 5 kg	g/ 10 min	20 ±1
	Physical studies				
3	Density	D 792-2020	23^0C	g/sm^3	0.960±0.005
4	Introduction to Molding; 3.2 mm	D 955-21	23^0C	%	1.8∼2.4
	Mechanical studies				
5	Tensile yield strength	D 638-2014	50 mm/ min	MPa	27.0+ 1.0
6	Relative elongation at break	D 638-2014	50 mm/ min	%	1200
7	Modulus of elasticity in bending	D 790-2017	2 mm/ min	MPa	1280± 10
8	Shore hardness	D 2240-2017	23^0C	1sec	66±1.0
	Impact strength studies				
9	Impact viscosity of cut samples according to izod	D 256-2010	23^0C	KJ/m^2	26±1.5
			-30^0C	KJ/m^2	20.7±1.0
	Thermal studies				
10	Vika softening temperature	D 1525-2017	10N/120^0C/h	^{0}C	129.0 ±1.0
11	Bending temperature under load	D 648-2016	0.45 MPa	^{0}C	72.0±1.0

Table 6. Physico-mechanical, rheological, strength, heat and fire resistance properties of secondary PE modified with 30% polyphosphatamine.

No	Names of indicators	Inspection method	Research conditions	Unit of measurement	Nominal values	Compliance with requirements
	Rheological studies					
1	Viscosity index	D 1238-2013	190^0C; 2.16 kg	g/ 10 min	8.0±0.5	Conforms
2	Viscosity index	D 1238-2013	190^0C, 5 kg	g/ 10 min	22 ±1	Conforms
	Physical studies					
3	Density	D 792-2020	23^0C	g/cm^3	0.970±0.005	Conforms
4	Introduction to Molding; 3.2 mm	D 955-21	23^0C	%	2.0 ∼ 2.7	Conforms
	Mechanical studies					
5	Tensile yield strength	D 638-2014	50 mm/ min	MPa	25.0+ 1.0	Conforms
6	Elongation at break	D 638-2014	50 mm/ min	%	1400	Conforms
7	Modulus of elasticity in bending	D 790-2017	2 mm/ min	MPa	1300± 10	Conforms
8	Shore hardness	D 2240-2017	23^0C	1сек	61±1.0	Conforms
	Impact strength studies					
9	Impact viscosity of cut samples according to Izod	D 256-2010	23^0C	KJ/m^2	28±1.5	Conforms
			-30^0C	KJ/m^2	22.7±1.0	Conforms
	Thermal studies					
10	Vika softening temperature	D1525-2017	10N/120^0C/h	^{0}C	135.0 ±1.0	Conforms
11	Bending temperature under load	D 648-2016	0.45 MPa	^{0}C	76.0±1.0	Conforms
	Fire resistance studies					
12	Flammability	UL 94	23^0C	Класс	V-1	Conforms
13	the maximum temperature of fire resistance under the influence of heated wires	ГОСТ 27483	23^0C	^{0}C	650	Conforms

In order to decline the flammability of composite materials obtained on the basis of secondary PE of brand I-0760, polyphosphatamine used as a flame retardant was used and its amount was taken up to 10%, 20%, 30% and 40%. According to the experimental results of these options, the best flammability performance indicators were obtained when polyphosphatamine was taken in the amount of 30%.

Now, we determine the physico-mechanical, rheological, strength, heat and fire resistance properties of modified secondary PE with the addition of 30% polyphosphatamine, which was found to be the optimal option, according to Ts 17642168-04-2013 (Table 6).

3 Results

As can be seen from Tables 1 and 2, the physical-mechanical, rheological, strength and thermal properties of secondary PE of the I-0760 brand without adding fire retardant (polyphosphatamine) physical-mechanical, rheological, strength of the secondary PE of the I-0760 brand with 30% flame retardant (polyphosphatamine) added and thermal properties are quite different.

It was found that the burning rate of composite materials obtained on the basis of secondary PE modified with 30% polyphosphatamine increases as the amount of polyphosphatamine decreases.

The slowing down of the burning speed of the composite materials obtained on the basis of secondary PE modified with 30% polyphosphatamine compared to the pure polymer, the polyphosphatamine contained in the polymer prevents the participation of air oxygen, which actively participates in the combustion process of the composite, and the formation of a certain flame-resistant layer consisting of polyphosphatamine on the surface of the composite, the material can lose mass and slow down the rate of destruction of the polymer.

At the same time, the rheological properties of secondary PE modified with 30% polyphosphatamine were also studied. The obtained results showed that as the concentration of flame retardants among polymer macromolecules increases, accordingly, the fluidity of the obtained composites decreases.

It was assumed that the flame retardant should be located between the polymer macromolecules and reduce the fluidity of the composite as its amount increases, because the flame retardant does not undergo any physical and chemical effects at the secondary PE processing temperature (ie 200-2200C) and the size of the flame retardant particles in the polymer is 2 Considering that it is -3 mm, it was expected that the polymer would serve as a mechanical barrier to the movement of macromolecules during the liquefaction of macromolecules. However, the effect of the amount of flame retardant on polymer macromolecules is more, it was proved by the obtained results.

Due to the high heat and fire resistance of these composites, while maintaining the initial physical and mechanical properties of the finished parts produced on the basis of these composites, it not only allows to save material mass due to the reduction of material thickness, but also expands the scope of use and operation of such composites.

Determination of softening temperatures of secondary PE modified with polyphosphatamine.

The softening temperatures of secondary PE modified with polyphosphatamine were investigated in a Vicat / HDT brand laboratory device according to GOST 15088-2014 (ISO 306:2004).

4 Discussion

During the conducted experiments, 3 samples were prepared and tested in the process of studying the softening temperatures of secondary PE modified with polyphosphatamine. The thickness of the samples was taken according to the standard: sample 1 was 3.54 mm, sample 2 was 3.63 mm, and sample 3 was 4.0 mm. The experiment started at 24.5 ℃ at room temperature. The heating speed of the device is 500 S/h. The time taken for the needle to sink 1 mm under the load (10 N) placed on the sample 1 was 112.68 minutes, and the temperature at this time was 118.4 ℃ [16].

Experiment 1. Determination of softening temperatures of modified secondary PE.

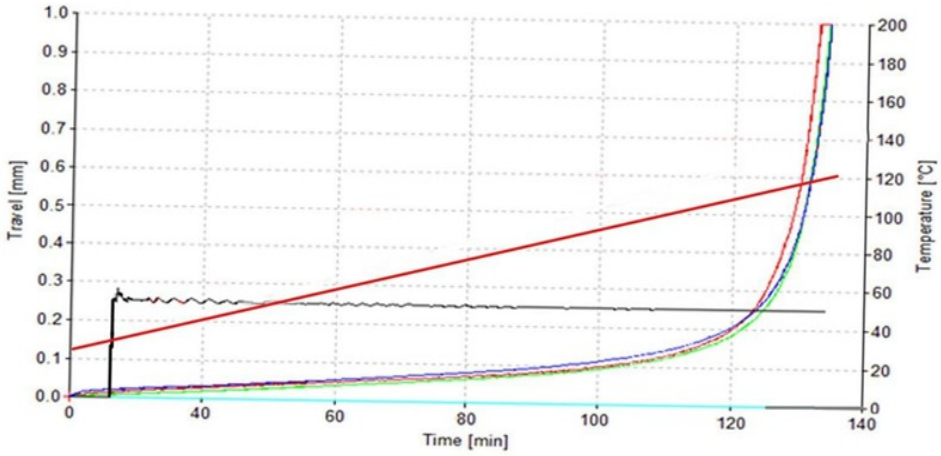

Fig. 1. Secondary PE modified with 30% polyphosphatamine softening temperatures.

Explanation. The red straight line in the figure is the temperature of the device, and the green, blue and light reddish colors are the temperatures of three identical samples.

As can be seen in Figure 1, the softening temperature of secondary PE modified with 30% polyphosphatamine is 110.2 ℃.

Determination of liquefaction and recrystallization temperatures of secondary PE modified with 30% polyphosphatamine.

The liquefaction and recrystallization temperatures of the studied modified secondary PE waste were measured in a Differential Scanning Calorimeter (DSC) according to the D 3417-99 method.

Experiment 2. Determination of liquefaction and recrystallization temperatures of secondary PE modified with 30% polyphosphatamine.

As can be seen in Figure 2, the melting point of secondary PE modified with 30% polyphosphatamine is 130^0C, and the recrystallization temperature is 122^0C.

According to the experimental results presented in Table 2, it can be concluded that the addition of flame retardant to the secondary PE modified with 30% polyphosphatamine causes a decrease in the liquefaction and recrystallization temperatures. This is technologically appropriate [11].

Processing of modified secondary PE.

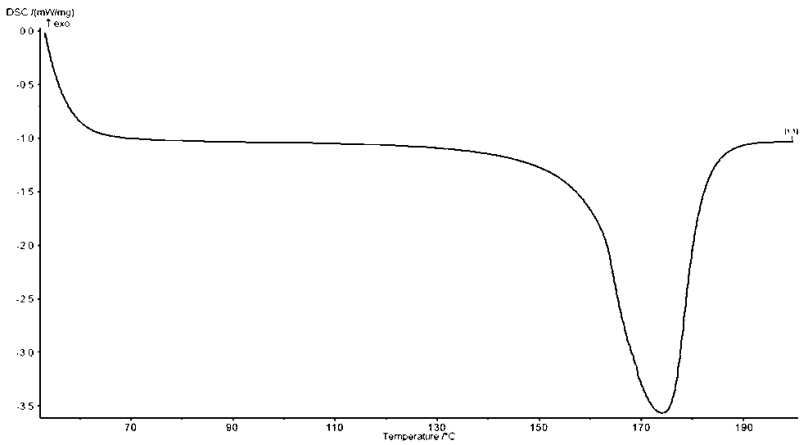

Fig. 2. Liquefaction and recrystallization temperatures of secondary PE modified with 30% polyphosphatamine.

5 Conclusion

Recycled secondary PE modified with 30% polyphosphatamine was used in the process line for the production of pipes with a diameter of 32 mm. The temperatures in the material cylinder and head zones of the extruder were not significantly changed. In order for the quality of the new polymer composite material obtained on the basis of modified secondary PE to be good, several requirements are imposed on the extruder, the extruder must be equipped with a blower (fan) to release volatile gases. In most cases, the screw is inserted directly into the dosing zone to further improve the polymer solution (mixing at the maximum level), which has become a homogeneous liquid due to the rotational movement of the screw inside the cylinder. Volatile substances are transferred to the vent with the polymer. The length of the extrusion zone is usually 2-5D. The depth of the channel in the mixing zone exceeds 0.4D for large diameter mixers and 0.3D for small ones. Air retention is a problem for the extrusion process [16]. This is caused by the infiltration of air with particles of raw materials inside the hopper. Under normal conditions, the pressure of the solid particles of the material in the loading zone forces the air out of the solid phase. In some cases, however, air cannot return to the loading zone and remains in the polymer until it breaks down in conventional extruders. If air bubbles exit the extruder, they can be exposed to the much lower pressure of ambient air and cause the small bubbles to break off with the compressed air. But even with such large breaks, the composite coming out of the extruder is usually defective due to the air bubbles in it [11-15].

During the mixing of the liquid in the cylinder, part of the mechanical energy is converted into heat energy. The heat dissipation increases with the increase in the number of revolutions of the screw. As the value of this released heat increases, there is no need for external heating at times. The performance of the extruder is greatly influenced by the shape and size of the granule. If the granule has a large size, air may remain in the liquid. This leads to the formation of a bubble (vzdutia) in the received item.

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