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Influence of doping additive on thermophysical and rheological properties of halogen-free polymer composition for cable insulation and sheaths

Introduction. The demand for halogen-free fire-resistant compositions for the manufacture of fire-retardant wires and cables is constantly growing. Problem. Therefore, the creation and further processing of these materials is an urgent problem. Goal. The aim of the article is to study the effect of the doping additive on the thermophysical and rheological properties of halogen-free compositions for power cables with voltage 1 kV with the determination of both the temperatures of phase and structural transformations of polymer compositions. Methodology. Experiments investigating the phase transformations were carried out with the help device of thermogravimetric analysis and differential scanning calorimetry TGA/DSC 1/1100 SF of METTLER TOLEDO company. Rheological studies of polymeric materials were conducted by using the method of capillary viscosimetry in the device IIRT-AM. Results. The influence of the doping additive on the formation of the supramolecular structure of the filled polymer compositions for cable products was determined, that resulted in the temperature increase of the decomposition beginning by 11 °C and the end of decomposition by 7 °C. Originality. The effect of a doping additive on reducing the effective melt viscosity of a polymer composition from $6 \cdot 10^4$ to $1 \cdot 10^4$ Pa ·s with increasing shear rate has been shown for the first time. The shear rate of the polymer composition containing the doping additive increases from 0.5 to 20 s⁻¹ with increasing shear stress. Practical value. The research results provide an opportunity to reasonably approach the development of effective technological processes for the manufacture of the insulation and sheaths of power cables from halogen-free polymer compositions. References 29, tables 3, figures 8.

Key words: cable production, doping additive, rheological properties, melting temperature, decomposition onset temperature.

Попит на вогнестійкі композиції, що не містять галогенів, для виготовлення пожежобезпечних проводів та кабелів безперервно зростає. Тому розробка цих матеріалів є актуальною проблемою. Метою статті є дослідження впливу легувальної добавки на теплофізичні та реологічні властивості композицій. Теплофізичні властивості визначено з використанням приладу TGA/DSC 1/1100 SF компанії METTLER TOLEDO. Реологічні дослідження полімерних матеріалів проведено методом капілярної віскозиметрії на приладі ИИРТ-АМ. Визначено вплив легувальної добавки на формування надмолекулярної структури наповнених полімерних композицій. Встановлено зниження ефективної в'язкості розплаву полімерної композиції в 6 разів зі зростанням швидкості зсуву в 40 разів при зміненні температури від 150 до 190 °С. Швидкість зсуву полімерної композиції з легувальною добавкою зростає в 40 разів з підвищенням напруження зсуву в 9 разів. Результати досліджень дають можливість обґрунтовано підходити до розроблення ефективних технологічних процесів виготовлення ізоляції та оболонок силових кабелів. Бібл. 29, табл. 3, рис. 8. Ключові слова: кабельна продукція, легувальна добавка, реологічні властивості, температура плавлення,

температура початку розкладу.

Introduction. In the last few decades, the demand for halogen-free fire-resistant compositions for the manufacture of fire-resistant wires and cables is constantly growing. This is largely due to their advantages in reducing smoke and reducing toxic and corrosive gases during combustion compared to traditionally used halogen-containing non-combustible cable materials. Such compositions are preferably materials containing a polymer based on polyolefins and a significant proportion of inorganic flame retardants, in particular aluminum hydroxide $Al(OH)_3$ [1, 2]. Polyolefins are one of the most flammable materials with high heat of combustion, low oxygen index and high heat dissipation, leaving little or no coke residue [3, 4].

In order to pass various tests for compliance with fire safety standards, compositions with content of the appropriate filler at the level of 60-80 % can be used. Of course, in this case there are quite complex problems manufacturability regarding the and mechanical properties of the compositions, which have to be solved by both manufacturers of cable compounds and manufacturers of cable products [5].

Rheological measurements of polymer melts are widely used in processing technologies of polymer compositions for quality control and process optimization. Another interesting field of rheology is to obtain

information about the molecular parameters of polymers and the structure of heterogeneous polymer systems. The publication [6] provides an overview of the influence of molecular weight, molecular weight distribution, the branching various degree of on rheological characteristics. For dispersed polymer systems, such as materials with particles and polymer mixtures, rheological measurements can be used as a simple method of qualitative study of interactions between different phases and changes in geometric structures created by inhomogeneities [7].

Extrusion is the main method of polymer processing in the cable industry. Almost all polymer processing operations require an extruder for melting, mixing and forming products [8, 9]. To understand and optimize the extrusion process, it is first necessary to understand the rheological properties [10]. In other words, it is difficult to understand and optimize the polymer processing operation without first having a complete understanding of the thermoreological behavior of the polymer material over a wide range of time. Moreover, using the rheological properties in both shear and longitudinal flows, it is necessary to determine the appropriate equation that can capture the correct rheological response of the material forced through the capillary and slit extrusion heads [8-11].

Some important rheological properties of polyolefins and their mixtures related to extrusion are discussed in [12-17], including: inlet pressure during extrusion, important for determining the expansion of polymer melts; influence of temperature and pressure on rheological properties [18, 19]; wall penetration of polymers [20-25].

However, for dispersed polymer systems, the relationship between structure and rheological characteristics is not clear, additional research methods should be used to assess the contribution of different structural elements [26]. In [27] the influence of the modifier on the thermophysical properties of fire-resistant composite materials was investigated.

Among the requirements for halogen-free cable polymer compositions is the ability to provide high linear extrusion rates. Thus, the study of the effect of alloying additives on the rheological and thermophysical properties of halogen-free polymer compositions is an urgent problem.

The goal of the work is the study of the effect of the alloying additive on the thermophysical and rheological properties of halogen-free polymer compositions for power cables for voltages up to 1 kV with the determination of the temperatures of phase and structural transformations of polymer compositions.

Polymer cable compositions. Halogen-free fireresistant polymer compositions were studied: sample 1 and sample 2. The polymer matrix (sample 3) for polymer compositions is a mixture of polyolefins (linear low density polyethylene; polyolefin elastomer and linear low density polyethylene modified with maleic anhydride). Flame retardant filler is alumina trihydrate. The content of flame retardant in polymer compositions is 60 %. The polymer composition in sample 2 contains an alloying additive in an amount of 2 %.

Paraffinic hydrocarbons were used as an alloying additive. The technical properties of the alloying additive are listed in Table 1.

Table 1

Properties of the alloying additive			
Indicator	Value		
Melt viscosity at temperature 140 °C, Pa \cdot s, 10^3	180-300		
Droplet temperature, °C, not less than	103		
Penetration hardness, %, not more than	5		
Volumetric resistivity at temperature 110 °C and voltage not less than 100 V, $\Omega \cdot cm$, not less than	$1 \cdot 10^{14}$		

The polymer composition is made on the compounding line of the X-Compound Company, Switzerland. The line includes the following equipment: compounder/mixer 120-16 L/D, feed extruder GS 140-6 L/D with granulating head, ingredient dosing system, transport systems of ingredients and finished products, granule cooling system.

Equipment and methods. A series of experiments to study phase and structural transformations, thermal oxidative degradation processes were performed using

thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) TGA/DSC 1/1100 SF instrument of the METTLER TOLEDO Company at heating rate of 10 and 50 deg/min.

Operating temperature range is from room temperature to 1100 °C; heating rate is from 0.01 deg/min to 100 deg/min.

Microbalance: the maximum weight during mounting is 1 g. The resolution of the TGA device is 1 μ g in the entire weighing range.

DSC measurement range: $1 - \pm 350$ mW; resolution -0.04μ W.

Determination of temperatures of phase and structural transformations is carried out according to the characteristic points of the description of physical phenomena on DSC diagrams.

The beginning of the melting or vitrification effect is defined as the point of intersection of the baseline with the diagram before the phase transition. The end of the effects is defined as the point of intersection of the baseline with the diagram after the phase transition and the tangent curve, which is drawn to the point of inflection.

Determination of decomposition temperatures of polymer compositions was performed using DSC diagrams obtained using the instrument TGA/DSC 1/1100 SF (temperature rise rate is 10 deg/min). Decomposition start and end temperatures were defined as the points of intersection of the baseline with the diagram before and after the decomposition of the polymer compositions and the tangent to the diagram drawn to the inflection point. Mass loss is defined as the distance on the ordinate axis between the tangent to the TGA diagram and parallel to the abscissa axis at the end of the process.

The study of the rheological properties of polymeric materials was carried out by capillary viscosimetry on the IIRT-AM instrument.

The properties of polymer compositions were determined at temperature of 150-190 °C and loads from 37.24 to 211.82 N. Using the obtained data, the shear stress, shear rate and effective viscosity were calculated.

Shear stress is determined by [28]

$$\tau = (P - P_{in}) \frac{r}{2 \cdot L \cdot \pi \cdot R^2}, \qquad (1)$$

where τ is the shear stress, Pa; *P* is the force required to ensure the flow through the capillary, N; *P*_{in} is the input losses, N; *R* is the radius of the cylinder, cm; *r* is the radius of the capillary, cm; *L* is the length of the capillary, cm.

The shear rate is determined by [28]

$$\dot{\gamma} = \frac{4 \cdot Q}{\pi \cdot r^3},\tag{2}$$

where $\dot{\gamma}$ is the shear rate, s⁻¹; *Q* is the material consumption, cm³/s ($Q = \pi \cdot R^2 \cdot h$, where *h* is the stationary immersion speed of the piston, cm/s; *R* is the radius of the cylinder, cm); *r* is the radius of the capillary, cm.

The effective viscosity is determined by [28]

$$\eta = \tau / \dot{\gamma} , \qquad (3)$$

where η is the effective viscosity, Pa·s.

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Graphs of shear stress versus shear rate and effective viscosity versus shear rate and shear stress are plotted.

In order to estimate the energy required for the transition of the system to the so-called transition state, i.e. when the destruction and the establishment of connections are balanced, the activation energy is calculated.

The activation energy of a viscous flow is determined by [28]

$$E_{act} = \frac{R \cdot T_1 \cdot T_2 \cdot \ln(MFR_2 / MFR_1)}{T_2 - T_1}, \qquad (4)$$

where *T* is the measurement temperature, K; MFR_1 and MFR_2 are the melt flow rates for T_1 and T_2 , g/10 min; *R* is the universal gas constant (8.314 J/(mol·K)).

A series of experiments to determine the electrical strength was performed using an instrument type AII-70, electrical resistivity – an instrument type KISI-1.

Statistical analysis was performed by the method of disperse analysis (ANOVA) using the model of asymptotic regression according to the integrated Levenberg-Marquardt algorithm with a significance level of α 0.05.

Results of investigations. For the extrusion process of halogen-free polymer compositions, it is important for cable products to investigate their thermophysical characteristics such as: temperatures of phase and structural transformations, decomposition start temperatures. The latter are also important for the operation of cable products at elevated temperatures and short-circuit temperatures. For power cables up to 1 kV with halogen-free insulation, the short-circuit temperature is 170 °C (duration not more than 5 s).

Figures 1, 2 show DSC diagrams of a polymer matrix and a halogen-free polymer composition without and with an alloying additive content.



Temperatures of the beginning and end of decomposition (Fig. 2) vary depending on the composition: for the polymer matrix (curve 3) in the range from 229 °C to 254 °C, for the polymer composition (curve 1) – from 258 °C to 275 °C, and for the polymer composition with an alloying additive (curve 2) – from 269 °C to 282 °C.

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Fig. 2. DSC diagram to determine the beginning and end temperature of the decomposition

Figures 3, 4 show TGA diagrams of a halogen-free polymer matrix and polymer composition without and containing an alloying additive.



Mass loss in the range from 200 to 450 °C

Table 2 shows the mass loss data determined from the TGA diagram (Fig. 3) for the polymer matrix and polymer composition that do not contain halogens without and with the content of the alloying additive.

Based on the data in Table 2 it is shown that for samples 1, 2 at mass loss from 5 % to 20 %, the temperature is lower (from 298 °C to 376 °C) than for sample 3 (from 334 °C to 383 °C). When the temperature reaches 275 °C (Fig. 3), there is a sharp change in mass loss (curves 1, 2). This is due to the process of dehydration of alumina trihydrate with heat absorption and water release. Sample 3 loses 50 % of the mass at temperature of 426 °C.

Table 2

Mass loss, %	Temperature, °C		
	Sample 1	Sample 2	Sample 3
5	298,1	301,8	334,6
10	314,0	319,0	356,6
20	351,9	376,2	383,9
30	412,3	435,0	400,5
50	_	_	426,1

Mass loss

Analysis of the TGA diagram (Fig. 4) shows that the polymer matrix loses 100 % of its mass at temperature of 525 °C. For samples 1, 2 already at temperature of 560 °C mass loss does not change, the residue is 38 % of the total mass of the polymer composition, which corresponds to the loss of chemically bound water and coincides with the theoretical calculations presented in [29].



Analysis of DSC and TGA diagrams (Fig. 1-4) shows that polymer compositions should be used for the manufacture of cable products in which the maximum long-term temperature of the conductive core does not exceed 90 °C, and the maximum temperature at short circuit is 170 °C. At the same time, the optimal temperature range of extrusion of polymer compositions was established. Rheological studies were performed at temperatures of 150, 170 and 190 °C.

Figures 5, 6 present the dependencies of the effective viscosity on the shear rate and the effective viscosity on the shear stress.



Fig. 5. Dependence of effective viscosity at different temperatures on the shear rate of polymer compositions

In Fig. 5 for the polymer composition of sample 2 there is a more significant decrease in the effective viscosity over the entire temperature range with increasing shear rate compared to the polymer composition of sample 1.

In Fig. 6 there is a decrease in effective viscosity with increasing shear stress for sample 1 from $8 \cdot 10^4$ to

 $1.5 \cdot 10^4$ Pa·s, for sample 2 from $6.1 \cdot 10^4$ to $1 \cdot 10^4$ Pa·s. The alloying additive provides efficient distribution of the flame retardant filler in the polymer matrix, resulting in the formation of an ordered supramolecular structure.



Fig. 6. Dependence of effective viscosity on shear stress of polymer compositions at different temperatures

Figure 7 presents graphical dependencies of shear rate on shear stress (flow curves) for polymer compositions of samples 1 and 2, respectively. The nature of the curves indicates that higher shear stress values are required to achieve higher shear rate values during the flow of polymer compositions. For the polymer composition of sample 2, the flow curves (4, 5, 6) at fixed values of the shear stress are shifted toward higher values of the shear rate compared to the polymer composition of sample 1 (curves 1, 2, 3).



Fig. 7. Dependence of shear stress on the shear rate of polymer compositions at different temperatures

Figure 8 shows the graphical dependencies of the activation energy of the viscous flow on the load for the polymer compositions of sample 1 and sample 2.

The activation energy of a viscous flow determines the energy barriers that are overcome in the elementary act of flow and determines the effect of temperature on the effective viscosity: the higher the activation energy, the greater the effect of temperature on the effective viscosity.

For the polymer compositions of sample 1 and sample 2, the activation energy decreases with increasing load, and the activation energy of the polymer composition of sample 1 becomes smaller compared to the polymer composition of sample 2.



The electrophysical properties of polymer compositions of sample 1, sample 2, and sample 3 have been studied. The electrical resistivity is determined by the presence of free charges (electrons and ions) and their mobility. Electrical strength is the electric field strength at which a breakdown occurs.

The results of the study are presented in Table 3.

Table 3

Flectrophysical	indicators	of polymer	compositions
Electrophysical	mulcators	or porymer	compositions

Indicator	Sample 1	Sample 2	Sample 3
Volumetric resistivity, $\Omega \cdot cm$	1,15·10 ¹⁵	$1,32 \cdot 10^{15}$	$1 \cdot 10^{16}$
Electrical strength, kV/mm	45,0	48,5	23,5

It is advisable to compare fire-hazardous polymer compositions that do not contain halogens: sample 1 and sample 2.

From data of Table 3 it can be seen that with the introduction of the alloying additive, the volumetric resistivity increases from $1.15 \cdot 10^{15}$ to $1.32 \cdot 10^{15} \Omega \cdot cm$, the electrical strength increases from 45 to 48.5 kV/mm.

Conclusions.

1. The influence of alloying additive on the formation of supramolecular structure of filled polymer compositions for cable products is determined, due to which the temperature of the beginning of decomposition by 11 $^{\circ}$ C and of the end of decomposition by 7 $^{\circ}$ C increases.

2. The expediency of using fire-retardant compositions for the manufacture of cable products, in which the maximum long-term temperature of the conductive core does not exceed 90 °C and the maximum temperature in the event of a short circuit is 170 °C, is shown.

3. For the first timethe effect of an alloying additive on reducing the effective melt viscosity of a polymer composition from $6 \cdot 10^4$ to $1 \cdot 10^4$ Pa·s with increasing shear rate is shown. The shear rate of the polymer composition containing the alloying additive increases from 0.5 to 20 s⁻¹ with increasing shear stress.

4. For the first time the influence of an alloying additive on the electrophysical properties of fire-

hazardous halogen-free polymer compositions has been studied. With the introduction of the alloying additive, the electrical resistance increases from $1.15 \cdot 10^{15}$ to $1.32 \cdot 10^{15}$ $\Omega \cdot cm$, and the electrical strength increases from 45 to 48.5 kV/mm.

5. The results of research provide an opportunity to reasonably approach the development of effective technological processes for the manufacture of insulation, sheaths of power cables from halogen-free polymer compositions.

Conflict of interest. The authors of the article state that there is no conflict of interest.

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