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Development and Evaluation of the Possibility of Using Epoxyurethane Mastic in Railway Transport

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Abstract. In scientific work is presented the solution of scientific and practical task – the development and evaluation of the possibility of using difficult combustible epoxyurethane (EU) mastic with increased vibration-damping properties and the necessary physical and mechanical properties for cladding the internal metal surfaces of railway rolling stock bodies. The compositions of the developed mastic based on the mixture of oligester cyclo-cab (OCC) and epoxidian (ED) oligomers with the addition of flame retardant – ammonium polyphosphate (APh), as well as hydrophobic filler methylaerosil AM-1-300 to give the composition thixotropic properties. As a result of dynamic-mechanical and viscoelastic researches, it was found that the mastic composition ED: OCC with the content of fire-retardant additive and thixotropic filler has high damping properties ($tg\delta$ = 0.45-0.47) in highly elastic field, and therefore, from practical point of view, this the material can work both at low (from -60°C) and moderate temperatures (to +60°C). A comprehensive fire hazard assessment showed that the developed mastic belongs to the group of difficult combustible and difficult flammable materials with slow flame spread and moderate smoke generating ability, as well as moderately hazardous in terms of toxicity. The achieved level of characteristics of the difficult combustible vibration damping mastic testifies about the prospects of its further use for facing the internal metal surfaces of bodies of railway rolling stock in order to ensure their fire safety and acoustic comfort.

1 Introduction

The technical condition of passenger cars of rolling stock of Ukraine's railways are characterized by significant wear (about 85%) [1, 2]. Due to the lack of investments aimed at the purchase of new passenger crews, it is important to improve the rolling stock with the least material losses to date.

The 21st century in the railway industry is the high requirements for the technical condition of rolling stocks – a decrease in the mass of a passenger car, an increase in speed and axle load, and an improvement in their technical and economic characteristics.

Modernization of the interior of the body at the car repair companies with the help of the introduction of innovative solutions will allow restoring the integrity of structural elements with the extension of its service life [3]. The use of polymeric composite materials (PCMs) is a traditional solution for improving the condition and creating comfort in the carriage of passengers in railway tracks [4, 5].

The use of such PCMs in trains provides its cost reduction, durability, weight reduction and reduced operating costs. However, the use of PCMs in passenger trains may increase the risk of fire and death of passengers.

It is known that a passenger train car can burn out in a matter of minutes (a safe time for evacuating people from the passenger train car is 5-6 minutes from the moment of ignition) [6]. At the same time, walls and partitions in case of fire contribute to intense burning, and the ceiling burning temperature can reach 1100°C.

Another problem of "worn out" passenger rolling stocks is the increased level of vibration and noise that arise due to the movement of the passenger train car and reduce the comfort of passengers during transportation [7]. Vibration affecting the passenger's body leads to irritability, headaches, impaired attention, an increase in the likelihood of neurosis diseases, etc [8].

Therefore, an urgent scientific and practical task is the creation of difficult combustible mastics with increased vibration damping properties for facing the internal metal surfaces of railway rolling stock bodies (side walls, cabin walls) in order to ensure their fire safety and acoustic comfort.

According to DSTU 4049-2001 "Passenger railroad cars of locomotive traction. Safety Requirements" materials that are used in the design of a passenger carriage and for internal non-mechanical equipment must comply with the following fire safety requirements:

combustibility group – difficult combustible (difficult flammable) materials;

smoke generation coefficient – not more than 500 m^2/kg;

indicator of toxicity of combustion products – low hazard or moderately hazardous

flame spread index – not more than 20.

2 Unresolved Issues

According to DSTU GOST 12549: 2019 Passenger cars of the main railways of 1520 mm gauge. Coloring. Specifications (GOST 12549-2003, IDT)» for priming the surface of the interior of wagons) use compositions based on modified phenol-formaldehyde resin, polyurethane composition, epoxy resin, which ensures the durability, corrosion resistance and water resistance of the paint system. However, the purpose of these thin-layer compositions is to ensure the reliability of already existing coatings. In this regard, it is relevant to ensure integrated fire safety and vibration protection of passenger cars using polymer materials (mastics), with a thickness of 2 to 4 mm.

Such traditional polymer mastics based on non-drying sealants as chlorobutyl rubber, polyethylene, polyisobutylene, which are highly combustible substances [9, 10], are known due to the hydrocarbon nature of polymers.

Another group of polymer mastics based on aqueous polyvinyl acetate dispersion [11, 12] used for the inner coatings of railway cars, are characterized by reduced flammability and the stability of technological properties [13, 14]. However, a disadvantage of the known materials is low vibroacoustic indicators (coefficient of mechanical loss of 0.2 at temperature of 20°C).

Difficult combustible mastics [15, 16] based on an epoxy oligomer with targeted additives are also used on the surfaces of machine parts, mechanisms, protective housings, vehicles with the goal of: reducing vibration and noise levels. However, they are characterized by an insufficient level of damping ability (coefficient of mechanical losses of 0.25 at a temperature of 20°C).

Therefore, promising direction is the development of difficult combustible epoxyurethane mastic with increased vibration-damping properties and the necessary physical and mechanical properties for cladding the internal metal surfaces of railway rolling stock bodies.

The aim of this work is the development and evaluation of the possibility of using difficult combustible epoxyurethane mastic with increased vibration-damping properties and the necessary physical and mechanical properties for cladding the internal metal surfaces of railway rolling stock bodies.

To achieve this goal it is necessary to solve the following tasks:

1. To determine the damping ability of the developed materials, to conduct a comparative assessment of the effect of the ratio of oligomers and flame retardants on the viscoelastic and damping properties of the polymer matrix.

2. To conduct a comprehensive assessment of the fire hazard of the developed epoxyurethane mastic by such indicators as the combustibility group, smoke generation coefficient, toxicity index of combustion products, and flame spread index.

3 Main Part

Epoxyurethane (EU) crosslinked polymers were used as the polymer matrix, the preparation of which is based on the interaction of a mixture of cyclocarbonate-containing and epoxy oligomers with amines.

A mixture of oligomers was chosen of oligester cyclo-cab oligomer Laprolat-803 (OCC) and epoxidian oligomer ED-20 (EPOXY). An amine type hardener: diethylenetriamine (DETA) was used to cure the oligomers.

To reduce the flammability of the polymers was used flame retardant additive - ammonium polyphosphate (APh) in an amount of 25 wt.% [17, 18]. The main characteristic of APh for flame retardant is the content of nitrogen and phosphorus, which should be in the range of 14-15% nitrogen and at least 70% phosphorus, respectively. To impart thixotropic properties to the composition was used filler with a hydrophobic surface of methylaerosil AM-1-300 in an amount of 1.5 wt.% [19, 20].

The developed two-component mastic is applied to a metal surface by spraying or with special staple, 2-3 mm thick [21].

As the main method for studying viscoelastic properties, we chose the method of dynamic mechanical spectroscopy, which was implemented on a torsion pendulum-dynamic relaxometer [22]. From the experimental data, the dynamic elastic modulus (G', E'), the mechanical loss tangent *tgδ* and the loss modulus G" were calculated. These indicators are related *tgδ* = G"/G'. The maximum value of *tgδ* corresponds to the mechanical glass transition temperature (T_g).

The study of the viscoelastic properties of polymers was carried out in the ultralow frequency range of 0.7-1 Hz, which minimizes the influence of external mechanical influences on the change in the polymer structure [23] and the temperature range from -100°C to +60°C.

For a comprehensive assessment of the fire safety of the developed mastics, studies were conducted according to DSTU 4049-2001 “Passenger railroad cars of locomotive traction. Safety requirements“.

To obtain an effective vibration-absorbing material based on a polymer composition, it is necessary to create such compositions that would possess in the required temperature and frequency range the maximum values of the mechanical loss tangent (*tgδ*) or the mechanical loss modulus G", which are a measure of the dissipated energy, respectively [24, 25]. The maximum values of *tgδ* are observed in the region of transition from the glassy to the highly elastic state.

The results of studies of the dynamic mechanical properties of cross-linked epoxyurethane compositions depending on the ratio of epoxy oligomer and oligester cyclo-cab (EPOXY:OCC) are shown in Fig. 1,2 where temperature dependences of the dynamic shear modulus G '(Figure 1) and mechanical loss tangent *tgδ* (Figure 2).

As can be seen from Fig.1, 2 with an increase in the content of the epoxidian oligomer ED-20 in mixture with oligester of cyclo-cab oligomer from 10:90 to 30:70 wt.% the glass transition temperature T_g of epoxyurethane compositions first decreases by 3°C passing through minimum at content of 20 wt.% ED-20, and then shifted to the region of higher temperatures by 8°C with simultaneous expansion of the peak *tgδ* and decrease in maximum values.

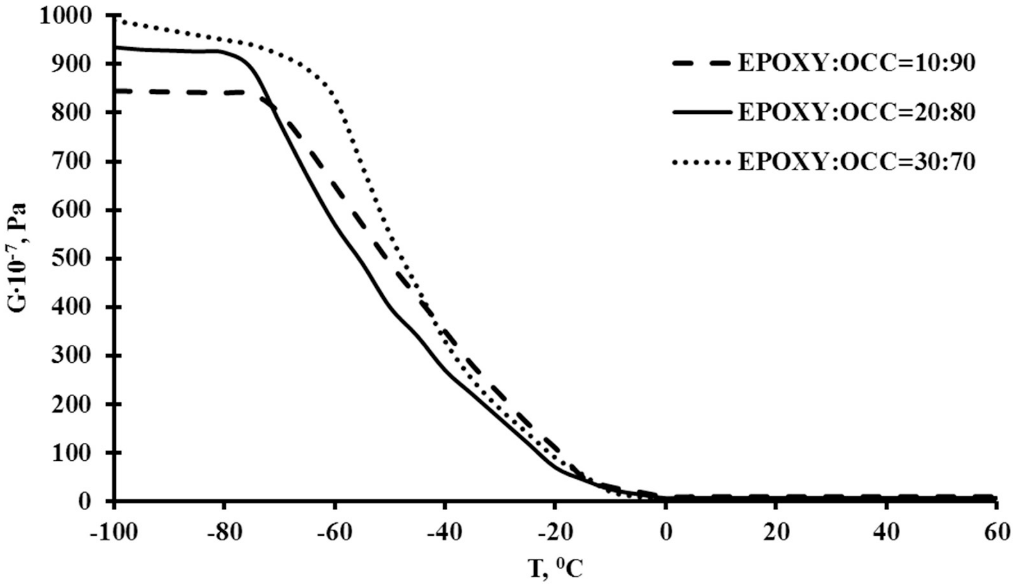

Fig.1. Temperature dependences of the dynamic shear modulus G' of EU compositions on the ratio of oligomers EPOXY:OCC=10:90, 20:80, 30:70 wt.%

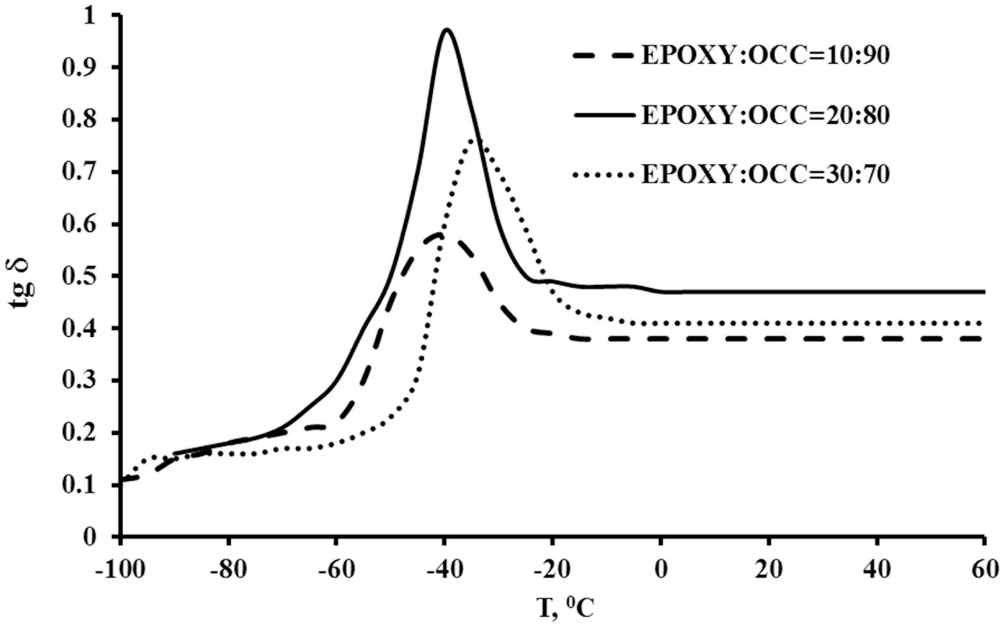

Fig.2. Temperature dependences of the mechanical loss tangent *tgδ* of EU compositions on the ratio of oligomers EPOXY:OCC=10:90, 20:80, 30:70 wt.%

At the same time, for the composition of ED-20:OCC= 20:80 wt.% there is a narrowing of the peak of mechanical losses and an increase in the maximum value of *tgδ* to 0.97. A similar simultaneous change in the indicated characteristics of the peak of mechanical losses indicates an increase in the cooperatively of the process of thawing the molecular mobility of the main segments of the chain polymer network, which is typical for polymers with a more uniform structure. On the other hand, with this ratio in a highly elastic state, the highest level of *tgδ* is characteristic of the composition ED-20:OCC= 20:80 wt.% and is about 0.48-0.50.

The results also show that the change in dynamic properties with increasing ED-20 content is at first glance ambiguous. When the content of 20 wt.% ED-20 in the mixture of oligester cyclo-cab is observed an increase in the peak of the maximum value of *tgδ* and it's narrowing, which indicates the ordering of segmental ability and an increase in structural homogeneity.

However, peak shift *tgδ* and accordingly glass transition temperatures of 3-5°C to lower temperatures, an extension of the glass transition interval, a steeper decrease in the shear modulus in the glassy state, and the appearance of additional peaks *tgδ* of low intensity in the region of (-80) ÷ (-70)°C indicate some loosening of the structure.

With further increase in the concentration of ED-20 to 30 wt.% along with an increase in the absolute values of the elastic modulus in the glassy state (at -100°C), an even sharper decrease is observed with increasing temperature, which indicates further loosening of the structure of the corresponding polymers in the glassy state.

This is due to the fact that with an increase in the ED-20 content, the density of the spatial network and the number of volumetric aromatic structures increase, which reduce the efficiency of intermolecular interaction in the glassy state, as a result of which the local molecular mobility due to the movement of fragments of hydroxyurethane chains will "unfreeze" at more low temperatures.

As can be seen from Fig. 2, the highest values of *tgδ* = 0.48-0.5 in highly elastic state are observed for EU materials based on mixture of ED-20:OCC at a ratio of 20:80 wt.%, so this composition can be used as the basis for creating vibration-absorbing mastics.

We have previously shown [26] that a fire retardant APh was used to effectively reduce the combustibility of mesh compositions in the form of dispersed powder with an average particle size of 50-60 μm in an amount of 25 wt.%. As well as methylaerosil AM-1-300 in the amount of 1.5 wt.% to give the mastic thixotropic properties [27].

Therefore, it was of interest to study the effect of dispersed mineral fillers (APh and AM-1-300) on the dynamic mechanical properties of epoxyurethane compositions.

The results of these studies are presented in Fig. 3, 4, which shows that the shape of the curves of changes in the dynamic modulus and the tangent of the angle of mechanical losses from temperature does not differ from similar unfilled compositions.

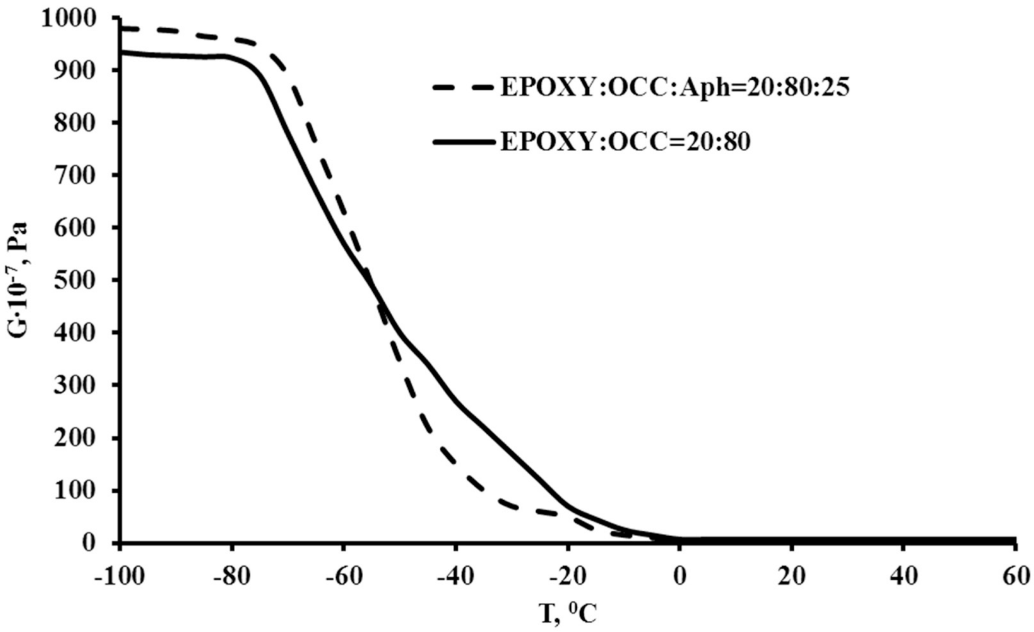

Fig.3. Temperature dependences of the dynamic shear modulus G 'of unfilled epoxyurethane composition (EPOXY:OCC=20:80 wt.%) and filled composition of APh and AM-1-300

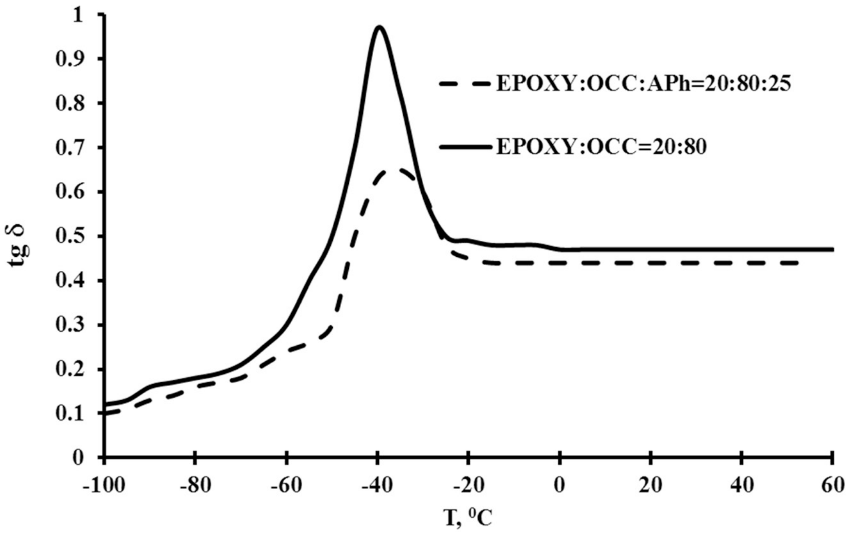

Fig.4. Temperature dependences of the mechanical loss tangent *tgδ* of unfilled EU composition (EPOXY:OCC=20:80 wt.%) and filled EU composition of APh and AM-1-300

Analysis of the curves of changes in the dynamic shear modulus showed that the introduction of APh and AM-1-300 into the composition leads to an increase in the modulus by 20%.

Wherein, there is a slower change in the dynamic module as the temperature rises to the temperatures of the beginning of the process of "devitrification", and the region of "devitrification" itself shifts to different degrees toward higher temperatures, while the temperature interval of the α-transition does not undergo noticeable changes, unlike unfilled compositions.

The temperature dependence of *tgδ* of filled EU compositions also undergoes some changes. It is shown that the introduction of flame retardant additive and thixotropic filler into the EU system leads to slight increase in the glass transition temperature (about 3°C), but helps to reduce the mechanical loss by 33% (from 0.97 to 0.65) compared to unfilled compositions.

As a result of dynamic-mechanical and viscoelastic researches, it was found that the mastic composition ED:OCC with the content of fire-retardant additive and thixotropic filler has high damping properties (*tgδ* = 0.45-0.47) in a highly elastic field, and therefore, from a practical point of view, this the material can work both at low (from -60°C) and moderate temperatures (to +60°C).

For a comprehensive assessment of the fire safety of the developed mastics, studies were conducted according to DSTU 4049-2001 "Passenger railroad cars of locomotive traction. Safety requirements". For a comparative assessment of fire safety the developed epoxyurethane mastics were used analogue based on epoxy resin (Table 1).

From Table 1 it is seen that the developed material is classified difficult combustible by the maximum temperature increment and weight loss. Depending on the time to reach the maximum temperature of the gaseous combustion products of the developed material, they are referred to as difficult flammable materials.

It was found that with the introduction of a flame retardant, the coefficient of smoke formation during combustion decreases (D_m = 485 m^2/kg), which allows us to transfer the developed material to a group with moderate smoke-forming ability. This, obviously, is due to the high adsorption capacity of the resulting coke and the features of the destruction process.

According to the ability of the material to ignite, to generate heat and spread the flame over the surface when exposed to an external heat channel, the developed material refers to the slowly spreading flame.

However, polymeric materials used in modern passenger cars are subject to very stringent requirements not only for combustibility, but also for a number of other characteristics associated with this process. Firstly, polymer materials should not emit toxic, sharp-smelling and irritating airways substances during combustion; Secondly, they should not give thick smoke, which interferes with navigating in a burning room when searching for ways of salvation. Therefore, to obtain difficult combustible polymer mastic used in the confined space of passenger cars, toxicological studies are necessary.

Table 1. Fire safety requirements according to DSTU 4049-2001

Indicators	Epoxy based analog	Developed EU mastic
Combustibility group: - maximum temperature increment Δt_{max}, [°C] - time to reach maximum temperature, s - sample weight loss, m, [%]	combustible medium fire 269 129 92.6	flame retardant difficult ignition 48 300 41.5
Lower heat, ΔH_c, [kJ/kg] according to DSTU ISO 1928: 2006	32060	19780
Smoke factor, D_m, [m^2/kg]	552.6	485.0
Flame propagation index over the sample surfaceaccording to p. 4.19 of GOST 12.1.044-89	22.8 (quickly spread the flame)	8.0 (slowly spread the flame)

The purpose of toxicological studies is to determine the toxicity index H_{CL50}, which is characterized as the ratio of the amount of material to unit volume of confined space, the combustion products of which cause the death of 50% of experimental animals. The exposure was 30±0.5 min. In addition, the H_{bCO} value is very important, showing the carboxyhemoglobin content in the blood of dead experimental animals immediately after exposure to the combustion chamber. According to this indicator, the primary biochemical mechanisms of the toxic effect of combustion products are determined. It is believed that the toxic effect of combustion products is mainly due to the action of carbon monoxide, when H_{bCO} values in animals' blood are 50% or more in accordance with p. 4.20.4.4 of GOST 12.1.044-89. However, the authors of [28, 29] believe that only at concentrations of H_{bCO} less than 60%, a toxic effect incompatible with life is due to the presence in the gas mixture of both leading (carbon monoxide and carbon dioxide) and minor components (in particular for polyurethane foams the presence of hydrogen cyanide in the mixture), as well as their combined effect.

The toxicity indicators of the combustion products of the epoxy based analog and developed by EU mastic presented in the Table 2.

From the Table 2 shows that the smallest H_{CL50} value is observed in smoldering mode at 450 °C and is 55.6 for the developed mastic. The data also show that after seeding mice with gaseous mixtures obtained by burning samples of epoxy based analog and developed by EU mastic causing the death of 50% of animals, the H_{bCO} content in the blood of dead animals was 58.4-62.8%. Therefore, for all studied samples of polymers, regardless of their chemical nature and the presence of flame retardants, the leading toxic effect of combustion products is the action of carbon oxides. In accordance with the classification in accordance with p. 2.16.2 of GOST 12.1.044-89, the investigated materials are classified as moderately hazardous.

Table 2. The toxicity indicators of the combustion products of the epoxy based analog and developed by EU mastic

The toxicity index of combustion products at different temperatures		Composition	
		Epoxy based analog	Developed by EU mastic
H_{CL50}, [g/m]	450 [°C]	65.5±6.3	55.6±7.6
H_{bCO}, [%]		58.4±2.6	59.2±2.9
H_{CL50}, [g/m^3]	750 [°C]	86.1±9.9	88.5±10.3
H_{bCO}, [%]		61.6±3.1	62.8±3.1

4 Conclusion

In scientific work is presented the solution of scientific and practical task - the development and evaluation of the possibility of using difficult combustible epoxyurethane mastic with increased vibration-damping properties and the necessary physical and mechanical properties for cladding the internal metal surfaces of railway rolling stock bodies.

The influence of the ratio of oligomers and flame retardant additives on the viscoelastic and damping properties of the polymer matrix is investigated As a result of scientific work. It was determined that the compositions with epoxidian oligomer in an amount of 20 wt.% characterized by the best indicators of damping ability ($tg\delta = 0.97$)

It is shown that the introduction of flame retardant additive and thixotropic filler into the EU system leads to slight increase in the glass transition temperature (about 3 °C), but helps to reduce the mechanical loss by 33% (from 0.97 to 0.65) compared to unfilled compositions. As a result of dynamic-mechanical and viscoelastic researches, it was found that the mastic composition ED:OCC with the content of fire-retardant additive and thixotropic filler has high damping properties ($tg\delta$ = 0.45-0.47) in wide temperature range (from -60°C to +60°C).

A comprehensive fire hazard assessment of the developed epoxyurethane mastic was carried out by such indicators as the combustibility group, smoke generation coefficient, toxicity index of combustion products, and flame spread index It has been established that the developed mastic belongs to the group of difficult combustible materials, with slow flame propagation, moderate smoke generating ability and are moderately hazardous in terms of toxicity.

The achieved level of characteristics of the difficult combustible vibration damping mastic testifies about the prospects of its further use for facing the internal metal surfaces of bodies of railway rolling stock in order to ensure their fire safety and acoustic comfort.

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