



# Wear resistance of epoxy-hard coal composites

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## ABSTRACT

**Purpose:** of this paper is to present results of wear resistance investigations of epoxy-hard coal composites produced by gravity casting

**Design/methodology/approach:** In short introduction wear resistance of polymers is presented, methods used to prepare composites together with short characterization of physical properties of applied fillers are described. Wear resistance of the specimens was determined using Schopper-Schlobach method and APGi device. During the test, the rigidly fixed specimen moves with constant velocity along a rotating cylinder with abrasive paper type corundum P60 on its surface. Wear resistance of specimens was determined after 20 meters distance of abrasion under pressing load of 10N and expressed as the weight loss of the specimen after abrasion.

**Findings:** Addition of the hard coal particles into the epoxy resin matrix caused a decrease of the wear resistance. Wear resistance decreased in a little extend with increasing filler content. Statistical analysis of achieved results showed that the influence of hard coal on wear resistance is significant comparing neat resin and filled resin but is not significant comparing composites with different solid particles content.

**Practical implications:** Tested composite materials can be applied among others in electrical or chemical industry where electrical properties are important but wear resistance is not so important.

**Originality/value:** New types of epoxy-hard coal graded composites were tested. It was demonstrated that by proper gravity casting technology control it is possible to produce graded composites for which hard coal particles do not deteriorate the wear resistance in significant extend.

**Keywords:** Mechanical properties; Wear resistance; Composites; Casting; Hard coal

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## PROPERTIES

### 1. Introduction

One of the most important phenomena, which governs all high performance construction materials, and therefore the polymer materials, is friction. Much of the energy produced on a global scale is used to overcome friction. Under the influence of surface friction materials being in mutual sliding or rolling contact undergo abrasion. The main reason of abrasion is surface

roughness and the presence of solid particles or foreign material at the contact layer. Therefore, reducing these two tribological phenomena (wear and friction) may very well lead to large economic benefits [1-4].

Polymeric materials as the solid bodies are made of macromolecules linked together by intermolecular forces (mainly Van der Waals) or first order chemical bonds in thermosetting resins. Large cohesion forces and thus advantageous characteristics of some polymers enable to apply them as

engineering plastics. One of the main advantages of polymeric materials is that their properties can easily be changed and optimized by different ways of modification. The most widely applied method of modification is addition of fillers and reinforcements. Also tribological properties of polymers can be modified by changes in chemical structure, by mixing different polymers, by plasticizing and by the introduction of additives with special properties [5-8].

Filled and reinforced polymeric materials are being used increasingly in tribological applications. Filled polymers are macroscopically heterogeneous, consisting of filler solid particles homogeneously dispersed in a polymeric matrix. Due to modification possibilities the same materials may be used for sliding elements, where low friction and low wear is required, and for clutches and brakes, where high friction and low wear is required. Operation of fillers depends on their chemical structure, physical structure, degree of fineness, shape of particles, the parameters defining the nature of the interaction in the system polymer-filler. Interaction of polymers with fillers is not limited to phenomena occurring at the interface, but also affects the density of molecules of the matrix around the filler particles. Very important is also volumetric content of filler and not in all cases high content cause wear resistance enhancement. In all cases tribological behaviour of filled polymer is not determined by individual material characteristics but is a function of whole tribological system. Tribological characteristics are determined by microstructure, surface morphology of materials being in contact, the stress and sliding rate conditions and many other parameters of tribosystem [9].

In most cases polymeric materials interact in tribological systems with metals and metallic alloys, especially with steels. There are not so many polymers that are applied in "steel-composite" tribological pairs. The most popular are PA (polyamide), PTFE (polytetrafluoroethylene), PBT (polybutyleneterephthalate), PET (polyethyleneterephthalate) POM (polyacetal), UHMW-PE (ultrahigh molecular weight polyethylene), PI (polyimide), PEEK (polyetheretherketone) and reinforced or filled phenolic resins.

There are many different mechanisms involved in wear of filled polymeric composites. Usually all phenomena are divided into adhesive, abrasive, surface fatigue and tribomechanical mechanisms. For filled polymer composites further classification is also possible: matrix wear (plowing, cutting, microcracking), filler wear (sliding wear), matrix cracking, filler cracking, filler-matrix delaminating (interfacial separation) [9]. Additionally the tribological system is changing during wear process by formation of debris layer between two materials. Depending on the nature and properties, the debris may act as additional abrasive material or as separating and lubricating layer decreasing wear action.

The next important problem related with wear resistance of polymeric materials is wear testing. All methods are based on appropriate type of tribological loadings such as sliding, rolling, sliding with rolling or oscillating sliding [10]. Less widely for polymeric materials are used stream methods. Almost all wear tests results are given as sample's weight loss or worn material weight. Results are highly method sensitive and because of this for every research results presentation applied method has to be precisely described. Also tests conditions are very important. Temperature highly influences wear properties of polymeric

materials, so it is very important to perform tests in precisely controlled temperature. In many cases temperature chambers are recommended and applied. Also sliding and loading conditions and counter-bodies surface properties must be thoroughly examined and keep constant. In wear tests reports wear parameters also have to be given. Due to complexity of polymeric materials wear processes, test should be adopted as much as possible to conditions during future application [10].

Together with invention of new class of engineering materials, functionally graded materials (FGMs), new possibilities of wear properties modification arose [11]. These materials are especially interesting because wear resistance is usually demanded only in thin outer layer of ready parts and structural elements. Shaping composition and structure gradation it is possible to produce parts with given thickness of wear resistance layer.

Functionally graded materials are an innovative and attractive class of composite materials. If the functional gradient is properly designed, FGMs may show unexpected properties, which differ from those of the single ingredient materials and also from those of the traditional (not graded) composite materials having the same mean composition [12-23].

Polymeric Graded Materials (PGMs) may be constructed entirely from one polymer with changing chemical or physical structure or from mixtures of two or more polymers, or they may be at least two-components composite, composed of a polymer matrix and filler. Gradation of composition and structure and in this way gradation of properties may be achieved due to chemical processes (crosslinking, grafting, co-polymerization) or due to physical processes (sedimentation, diffusion, dissolution, mixing, creating a new phase) or in any other method that changes properties in different extend in different places of a volume of polymeric material. For the purpose of presented research the most important are composites consisting of polymeric matrix and solid filler particles [24-29].

The combination of different matrix and filler (dispersed phase) materials enables to develop composite material with a wide range of functional properties: mechanical, chemical, electrical, magnetic, optical and many others. These materials have many advantages such as ease of processing, high efficiency of manufacturing processes, the resulting low density of composite and other but the main advantage of graded materials and among them polymeric graded materials is combining regions of materials with different properties without definite boundaries. In this way many problems are avoided such as poor adhesion between these regions (e.g. layers), thermal stresses by temperature differences and other [30].

Many technologies have been developed so far for producing graded materials which significantly increased the scope of their application. Polymer graded materials can be prepared using known techniques of polymeric materials processing, among others, compression moulding, extrusion, casting, spraying and coating but also many modern techniques are applied such as selective laser sintering (SLS), in situ polymerization, electron beam radiation, corona discharge and many others. One of the basic requirements to achieve designed properties gradation is elaboration of relations between structure and composition gradation on one side and properties gradation on the other side. The second problem which has to be solved is to find relations between production technologies parameters and gradation

profiles achieved. Because of this the challenge for modern industry is the development of techniques for economically and technically effective production of Polymeric Graded Materials with planned and designed properties gradation.

In this paper an attempt is presented to produce a polymeric graded material by gravity casting method. The main novelty is that a new type of filler was used, namely powdered hard coal. This filler was earlier applied to manufacture polymeric composites [31-34] but without composition and properties gradation. The basic purpose of the elaboration of this material was to change electric properties of epoxy resin, especially to decrease surface resistivity in order to avoid static electricity. But at the same time it was important to preserve wear resistance or at least not to deteriorate it in significant degree. Polymeric graded material was chosen because the task was to decrease electric surface resistivity only on one side of ready material. It is possible to achieve this by coatings applying but in this case adhesion of coating to the rest of material becomes the problem. Applying polymeric graded material enabled to achieve continuous change of electric and wear properties without boundaries problems.

## 2. Experiments

It is well known that inorganic filler particles enhance the mechanical and in many cases tribological properties of polymers. The stiffness, toughness, hardness and wear performance of the composites are extensively determined by the size, shape, volume content, and especially the dispersion homogeneity of the particles. In the present study, various amounts of micro-scale particles of hard coal were introduced into an epoxy polymer matrix as its filler. The influence of these particles on the wear resistance was investigated. At the same time electric surface resistivity was tested and the results will be presented in another paper (in preparation). Aim of wear resistance study was to test if the coal particles introduced into the polymeric matrix significantly alter this property of graded composites. Wear and electric measurements were performed on the same side of graded material samples (Fig. 1).

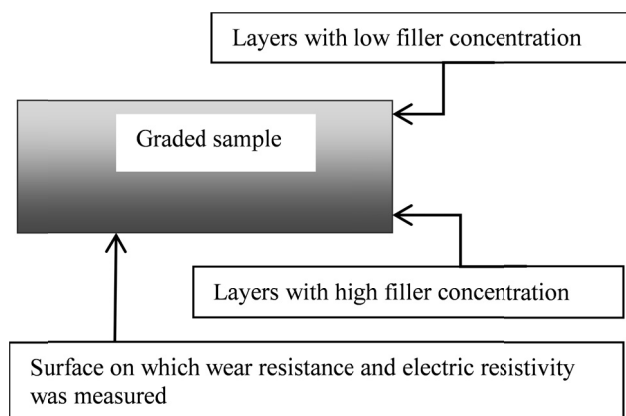


Fig. 1. Measurements position on sample with filler content gradation

### 2.1. Materials for research

Bisphenol A epoxy resin, Epidian 6011 [35], cured at room temperature with hardener ET (modified aliphatic polyamine) [36], both produced by „Organika - Sarzyna” Chemical Plant S.A. (Poland) were used in this study as thermosetting polymeric matrix. Two types of hard coal were used as a filler. The anthracite coal filler mined in Kuzbas of the Kuznetsk Basin (Russia) (designed as “A”) and the second type of hard coal acquired from “Zofiówka” coalmine (designed as “Z”). Basic characteristics of hard coal component materials are shown in Tables 1 and 2.

At this stage of research fillers were applied without particle size fractions separation and in the state in which they were received from the supplier, without any surface modification by surfactants or coupling agents. In the future research fillers surface modification is planned.

Table 1. Basic characteristics of anthracite (A)

Carbon contents [%]	90.1
Ash contents [%]	3.37
Range of particle size* [μm]	d <sub>10%</sub> =7.2 d <sub>50%</sub> =26.5 d <sub>90%</sub> =61.2
Density [g/cm <sup>3</sup> ]	1.37

Table 2. Basic characteristics of hard coal from “Zofiówka” coalmine (Z)

Carbon contents [%]	71.5
Ash contents [%]	16.78
Specific surface [m <sup>2</sup> /g]	0.939
Range of particle size* [μm]	d <sub>10%</sub> =2.92 d <sub>50%</sub> =17.93 d <sub>90%</sub> =64.25
Density [g/cm <sup>3</sup> ]	1.40

\*) Average values of particle size were determined with Mastersizer device of the Malvern company

### 2.2. Samples preparation

The specimens were obtained using gravitational casting method which is one of the technologies relying on gravity and without applying pressure. With this method it is possible to produce one dimensional gradient of component materials content in liquid matrix. The gradient is retained after matrix solidification. Due to sedimentation process, the highest filler content is expected in the lowest specimen layer. The rate of sedimentation depends on polymer and filler densities difference, solid particles size distribution and the viscosity of liquid resins. Increasing resin viscosity during curing process slows down sedimentation and finally stops it. Particles stopped at various positions form gradation of filler concentration. Samples were cured in temperature 23±2°C in 24 hours time. Samples with 0, 15, 20, 26, 30, 36 and 40% vol. filler concentrations were moulded.

The procedure of specimen's preparation is shown schematically in Fig. 2.

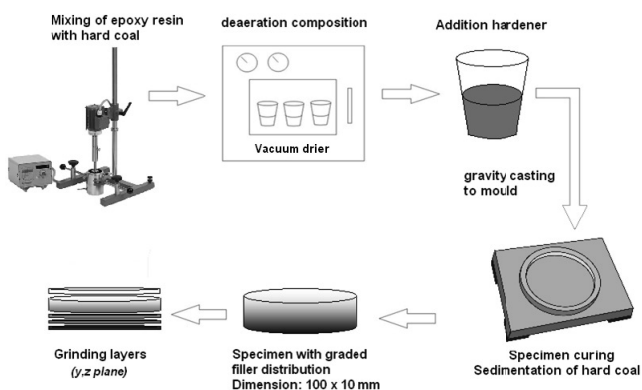


Fig. 2. Scheme of specimen's preparation

Specimens were cast into our own design and construction steel mould. Cavity of the mould has the following dimensions: diameter - 100mm, depth - 12mm. Inner side walls of cavity were made with 3° inclinations to facilitate ready specimen's removal. Additional inner side walls and bottom of cavity were covered with Teflon® fabric. In effect disk shaped specimens with 100 mm diameter and 10 mm thickness were produced in the mould. After demoulding samples were additionally grinded to remove meniscus formed at mould wall. The final thickness was 8,6 mm (Fig. 3).

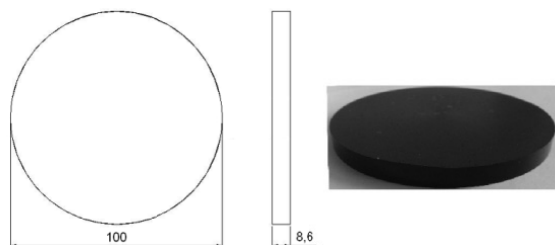


Fig. 3. Shape of the specimen

Exemplary microscopic image of achieved gradation of hard coal particles concentration across sample thickness is shown in Fig. 4 a,b.

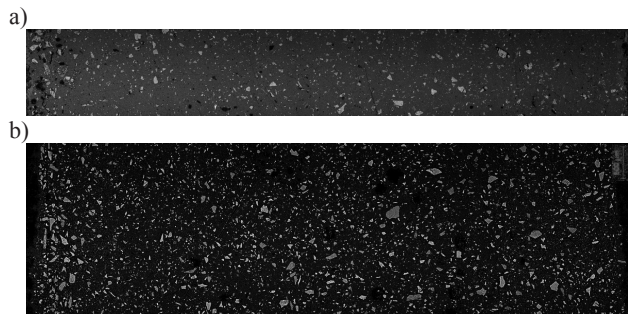


Fig. 4. Microscopic image of exemplary cross section of the sample (a) Epidian 6011 + 15% vol. of hard coal from "Zofiówka" coalmine, (b) Epidian 6011 + 20% vol. of anthracite

### 2.3. Measurements methodology

Wear tests were performed using Schopper-Schlobach method. In the device dedicated to this method a sheet of sand paper with gradation 60 was wound around a cylinder of the diameter 150mm. The cylinder was rotating with approximately 40rpm angular velocity. The sample had a form of a disk with the diameter 16 mm (cross-sectional area approximately 2cm<sup>2</sup>) and the thickness of at least 6 mm. Samples were pressed against the sandpaper with 10N load. During test the sample was additionally turned around its axis to retain flatness of sliding surface of the sample during all sliding path (Fig. 5).

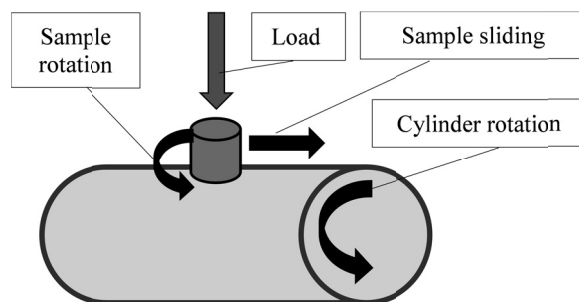


Fig. 5. Tribological system used in wear resistance test

The driving screw moved the specimen holder at a constant velocity along the cylinder so that the specimen was sliding all the time on fresh sandpaper. Specimen was automatically raised up above the sandpaper after passing sliding distance of 40 m or 20 m (depending on the obtained results).

Samples prepared using filled and unfilled epoxy resin are presented in Fig. 6.



Fig. 6. Specimens for wear resistance

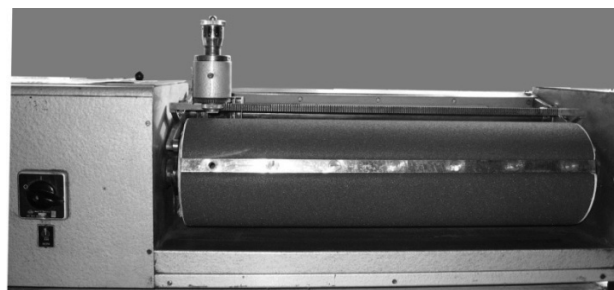


Fig. 7. APGi apparatus for wear resistance test



Abrasion resistance tests were performed applying wear apparatus APGi type (Fig. 7). Schopper-Schlobach testing procedure was accepted according to ISO4649 requirements.

Wear resistance was calculated as the volume loss of the abraded sample (in cubic centimetres,  $\text{cm}^3$ ), according to the formula(1):

$$\Delta V = \frac{(m_1 - m_2)}{\rho} \quad (1)$$

where:

$m_1$  - mass of the sample before abrasion[g],  
 $m_2$  - mass of the sample after wear test[g],  
 $\rho$  - specific weight (density) [ $\text{g}/\text{cm}^3$ ].

All wear tests were performed at temperature  $23 \pm 2^\circ\text{C}$ .

The measurements of the density of the materials of tested specimens were performed applying Mohr scale method by weighing them in air and water on the analytical balance Adventurer ProAV264CMOHAUS. For every material the density was calculated as a mean value of three consecutive determinations (1).

Five samples were tested for every composite. Altogether 35 measurements were performed.

### 3. Results and their analysis

Addition of hard coal particles into epoxy resin only slightly changed abrasion resistance of this polymer. For all filled compounds wear resistance decreased a little in comparison with unfilled resin. Graphic interpretation of relationships between filler volume content and volume loss based on average values of five samples are presented in Figs. 8 and 9. Comparison of the abrasion resistance of composites filled in different degree with two types of filler is presented in Fig. 10.

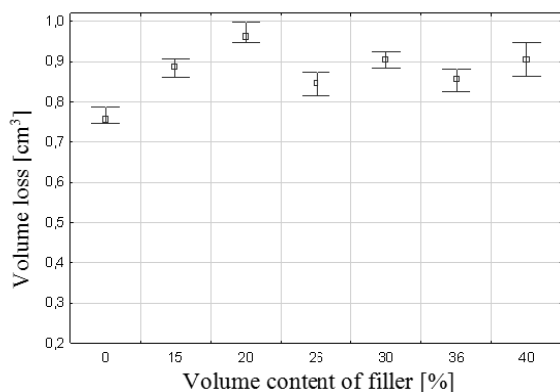


Fig. 8. The dependence of sample volume loss on the content of anthracite (A)

Comparing all filled composites, the minimum value of the volume loss was achieved for compounds with 26% volume fraction of hard coal (Z) (Fig. 6). Also in the case of composites filled with anthracite the smallest loss of volume was recorded for

the composite with 26% volume fraction of filler. Observe relationships can be explained taking into account filler action during abrasion. Two different phenomena determine abrasion resistance in the case of hard particles filled polymers. Hard particles increase total hardness of polymer composite and in this way increase wear resistance of the composite. On the other hand solid particles, depending on adhesion level between matrix and filler, may be more or less easily removed from the matrix increasing surface roughness and in this way decreasing wear resistance. Depending on which mechanism is predominant, wear resistance increase or decrease is observed. In the case of tested materials the later phenomena seems to be predominant. Coal particles, being foreign inclusions in the structure of epoxy matrix weakened the cohesion forces and could cause micro-cracks. It is also possible that filler particles contributed in the formation of relatively large void sand bubbles in the material volume of the composites what strongly influenced abrasion resistance results.

Best balance between positive and negative mechanisms was achieved for 26% vol. fraction of both coal types. To enhance adhesion between solid particles and polymeric matrix fillers surface modification is planned in future research.

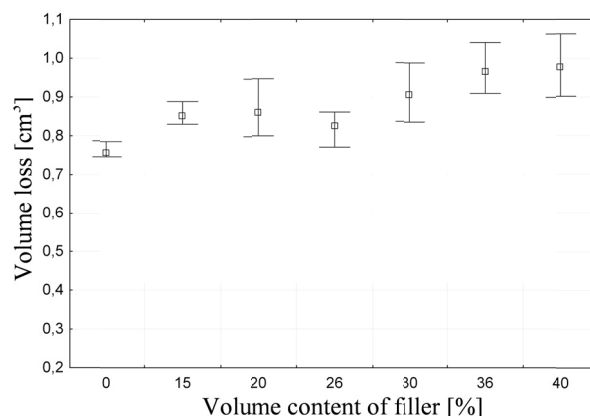


Fig. 9. The dependence of specimen volume loss on the content of hard coal (Z)

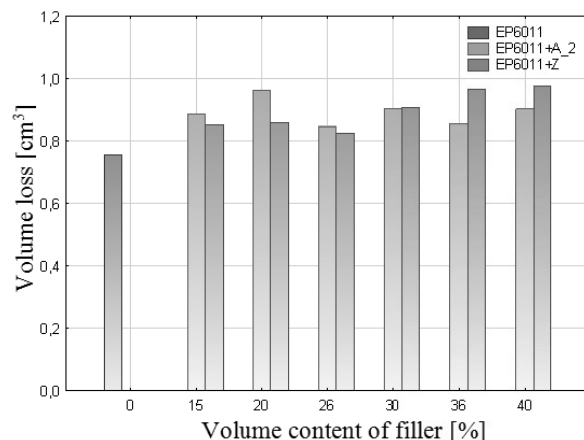


Fig. 10. Comparison of the abrasion resistance of the two types of composites with different content of filler

Table 3.  
Bartlett's test of homogeneity of variances for specimens filled with hard coal

Compound	Statistic $\chi^2$	Critical value of statistic $\chi_{\alpha}^2$	Degrees of freedom n	Level of significance $\alpha$
EP6011+A	2.150	9.488	4	0.05
EP6011+Z	7.277			

Table 4.  
Test analysis of variances (single classification) for many averages of volume losses for specimens filled with hard coal

Compound	Source of variation	Sum of squares	Degrees of freedom	variance	Statistic $F$	The critical value $F_{\alpha=0,05}$
EP6011+A	between populations	0.12	6	0.0204	4.12	2.45
	in side groups	0.14	28	0.0049		
EP6011+Z	between populations	0.19	6	0.0309	3.54	
	in side groups	0.24	28	0.0087		

With the increase of the filler content increases lightly the value of the volume loss of the specimens. It is more pronounced for composites filled with hard coal (Z). Fig. 7 shows that low levels of filling - less than 26% vol. - the wear resistance of the composites with hard coal (Z) particles is better than those with anthracite (A). For higher filler concentrations (larger than 26% vol.) specimen with anthracite exhibited higher wear resistance.

Wear resistance differences between samples with different levels of filler content and samples filled with hard coal and anthracite seems to be very small and not significant. No clear dependences are visible analysing achieved results. In order to verify if the measured values differ statistically significantly appropriate statistical test were carried out. At the beginning performed was Bartlett's test of variances homogeneity. For this test null hypothesis was assumed that variances of results of volume losses are homogenous (Table 3). Next the test of variances for multiple average values was performed. The null hypothesis for this test was assumed that average values of volume loss did not differ significantly. Results of the test of significance are presented in tabular form (Table 4).

In the Bartlett's test of homogeneity of variances deciding is  $\chi^2$  statistic. Calculated value of  $\chi^2$  statistic is lower than the predetermined critical value of this statistic ( $\chi_{\alpha}^2$ ). This means that null hypothesis cannot be rejected and at significance level  $\alpha=0.05$  verified was hypothesis assuming homogeneity of the variances of abrasion resistance of specimens containing different amounts of filler. This enables to perform the next statistical test comparing averages values of volume losses after wear resistance test. Results of this test are presented in Table 4.

In this test deciding was the F-statistic. Calculated value of the F-statistic is higher than the critical value (given in Table 4), so the null hypothesis  $H_0$  of equality of average values in the study populations should be rejected at significance level  $\alpha=0.05$ . Rejecting null hypothesis is proving a significant influence of various coal contents on the value of volume loss in wear resistance test. It is worth to mention that calculated values of F-statistics are very close to critical value and numerical results of wear test differ not very substantially. Because of this the analysis of variances for many averages of volume losses was repeated at new significance level  $\alpha=0.1$ .

After analysis of the average values of weight losses for composites with filler sat a significance level of 0.1 ( $1 - \alpha=0.90$   $\alpha \Rightarrow 0.1$ ) the null hypothesis ( $H_0$ ) of equality of volume losses in wear resistance test was accepted. Number of schools not only noted the great differences between the numerical values.

The numerical results of wear resistance measurements and the results of the statistical tests to indicate that coal particles introduced into epoxy resin does not improve, but do not substantially worsen studied in this experiment abrasion resistance of composites.

This allows concluding that the main purpose of the research was achieved. Epoxy - hard coal graded composites were developed with abrasion resistance only minutely lower than abrasion resistance of unfilled epoxy resin.

In the future research it is planned to test the influence of surfactants and coupling agents on wear and other properties of graded composites. Application of coal filler with different particle size distributions are also intended. A continuation of research on polymer gradient composites filled with anthracite and hard coal is planned. Research is also planned with compounds filled with particles of specific size fractions.

#### 4. Conclusions

The obtained results allow to formulate the following substantial conclusions:

- Wear resistance tests have shown that introduction of coal particles to epoxy resin resulted in a slight, insignificant decrease in abrasion resistance compared to the cured epoxy resin without fillers.
- Observed was slight decrease in abrasion resistance with increasing filler content.
- For smaller filler content - less than 26% by volume - samples with hard coal (Z) are characterized by better wear resistance, while for larger filler contents - higher than 26% by volume - composites with anthracite exhibit higher wear resistance.

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