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FIBER GLASS PLASTIC OBTAINED FROM POLYESTER EPOXY BINDER BY THE CONTACT METHOD AT NORMAL TEMPERATURES

by

L. I. Kravchenko, N. S. Leznov,
and Ya. D. Avrasin



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# EDITED TRANSLATION

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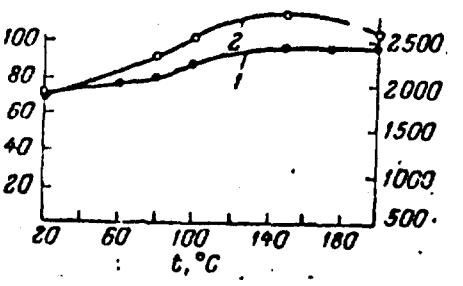
ABSTRACT: Fiberglass plastic ~~(UP-1KhO)~~ containing 60-65% resin was obtained from ~~the~~ polyester epoxy resin ~~11EDSM~~ and benzoyl peroxide-dimethyl aniline - Co linoleate system at normal temperature and ≈0.5 kg/cm$^2$ pressure. The material was compressed at 3 kg/cm$^2$ in vacuo for 24 hr. The plastic obtained was hardened at 20-200C. Increase of the hardening temperature from 20 to 150C increased the yield of insoluble 11EDSM from 70 to 95% and the bending strength from 1860 to 2800 kg/cm$^2$ (Fig. 1). At 150C, the strength of the hardened plastic was highest when hardened for 12 hr. Polymerization of ~~11EDSM~~ with isopropylbenzene hydroperoxide-Co linoleate or benzoyl peroxide-dimethyl aniline systems gave fiberglass plastics with inferior physical properties. The properties of VP-1KhO plastic, affected by the time and temperatures of aging are tabulated. Orig. art. has: 6 tables and 5 figures.

Table 1. Effect of water, fuels, and MS oil on the cold-hardening fiber reinforced plastic [illegible]

| Medium | Weight increase, % | | | |
|---|---|---|---|---|
| | before heat processing | | after heat processing | |
| | 24 hr | 30 days | 24 hr | 30 days |
| A | 0,90<br>0,83—0,97 | 3,2<br>3,0—3,5 | 0,30<br>0,25—0,40 | 1,35<br>1,3—1,45 |
| B | 0,1<br>0,02—0,1 | — | 0,01<br>0,002—0,02 | 0,2<br>0,12—0,25 |
| C | 0,17<br>0,15—0,19 | 0,2<br>0,17—0,21 | 0,1<br>0,07—0,13 | 0,2<br>0,12—0,28 |
| D | 0,65<br>0,64—0,87 | 0,70<br>0,55—0,90 | 0,73<br>0,3—1,0 | 0,97<br>0,85—1,1 |

A = $H_2O$; B = gasoline; C = kerosine; D = MS oil.




1) Yield of insoluble 11EDSM, %; 2) bending strength, kg/cm$^2$. Influence of hardening temperature on yield of insoluble 11EDSM and bending strength of fiberglass plastic.

| | strength, kg/cm² | | | | E | F | G |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | Kg·cm/cm² | Kg/cm² | Kg/c |
| Контрольные ($t_{исп}$=20 °C) | 1125<br>780—1390 | 1310<br>1020—1615 | 1455<br>1075—1620 | 140<br>110—180 | 95<br>70—140 | 65600<br>59600—71600 | 2570 |
| 150 °C—12 ч ($t_{исп}$=20 °C) | 1875<br>1555—2320 | 2240<br>1780—2545 | 1825<br>1315—2325 | 195<br>175—215 | 115<br>85—140 | 89100<br>76600—110800 | 3530 |
| 100 °C—200 ч ($t_{исп}$=100 °C) | 1210<br>965—1420 | 2055<br>1820—2475 | 1105<br>1085—1140 | 85<br>65—110 | 90<br>85—130 | — | — |
| 150 °C—200 ч ($t_{исп}$=150 °C) | 1130<br>995—1295 | 2265<br>1970—2445 | 855<br>810—960 | 65<br>55—85 | 90<br>80—110 | 50600<br>45200—58800 | — |
| 200 °C—200 ч ($t_{исп}$=200 °C) | 980<br>900—1025 | — | 800<br>735—925 | 60<br>50—65 | 85<br>80—100 | 48600<br>37800—66700 | — |

$t_{исп}$ = aging temperature

Table 3. Influence of the duration of aging at different temperatures on the mechanical properties of fiberglass plastic VP-1KhO hardened at normal temperature (60-65% of 11EDSM).

A = tensile; B = compression; C = bending; D = shearing; E = notch toughness; F = modulus of tensile elasticity; G = modulus of shearing elasticity; H = Poisson coefficient.

# FIBER GLASS PLASTIC OBTAINED FROM POLYESTER EPOXY BINDER BY THE CONTACT METHOD AT NORMAL TEMPERATURES

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For the hardening of polyester maleinate resins at normal temperature as oxidizing-reducing systems one used peroxide of methyl ethyl ketone – naphthenate of cobalt, hydro peroxide of isopropyl benzene – naphthenate of cobalt, peroxide of benzoyl – dimethylaniline, and others [1-5].

For the hardening of polyester acrylate resins one uses the system of hydro peroxide of isopropyl benzene – linoleate of cobalt and peroxide of benzoyl – linoleate of cobalt [6].

In the use of these oxidizing-reducing systems for hardening at normal temperature polyester acrylate epoxy binder 11EDSM [7] it has been established that polym · ation is extremely slow. In this connection there were investiga other oxidizing-reducing systems.

## Choice of the Oxidizing-Reducing System for Hardening the Binder 11EDSM

There were tested the following oxidizing-reducing systems: hydro peroxide of isopropyl benzoyl – linoleate of cobalt and peroxide of benzoyl – dimethylaniline. In the case of using the first system the binder 11EDSM is polymerized to the state of gelatinization during 24 hours; the solid polymer, however, is formed after 15 days.

In the making of glass plastic on the basis of this binder with the use of the systems of oxidation peroxide of benzoyl – dimethylaniline on the uncovered surface of the glass plastic there remained a sticky unpolymerized layer of binder, which did not disappear even after heat treatment of the glass plastic, which is explained by the inhibiting action of the oxygen of the air (apparently the interaction of the oxygen with the free radicals formed in the system [8]).

Taking into account that dimethylaniline in its optimal content together with peroxide of benzoyl speeds up the reaction of hardening of the epoxy resin and polyester acrylate, and also the fact that linoleate of cobalt together with peroxide of benzoyl facilitates the formation of films hardenable at normal temperature on the basis of polyester acrylates without surface tack [9] we in the further work tested a three-component initiating system of peroxide of benzoyl – dimethylaniline – linoleate of cobalt.

In Table 1 there is presented the change in the limit of strength in static bending at normal temperature and after aging at 200°C during 200 hours, as depends on the type of oxydizing-reducing systems used for hardening.

All the plates were prepared by rolling up the impregnated layers of fiber glass filler with the aid of a roller under a pressure of about 0.5 kgf/cm$^2$. The thickness of the plate was 10 mm and the content of the resin 60-65%.

From Table 1 it is seen that the system peroxide of benzoyl – dimethylaniline – linoleate of cobalt assures the best strength properties of glass plastic.

For obtaining fiber glass material of uniform structure, and also explaining the inhibiting action of the oxygen of the air in hardening material at normal temperature there was used the method of impregnation of the fiber glass filler under the action of a vacuum and pressure in a closed mold (Fig. 1).

Table 1. Dependence of strength of fiber glass plastic on type of oxidation-reduction system used for hardening the binder 11EDSM at normal temperature.

| Oxidation-Reduction System | Normal temperature | | State of uncovered surface of glass plastic after 5 days | Static bending strength, kgf/cm² | |
|---|---|---|---|---|---|
| | Stage ⌚ | Stage ⌚ | | at 20°C* | 200°C after 200 h aging |
| Hydroperoxide of isopropyl benzene + linoleate of cobalt . . . . | 24 h | 15 days | weak tack | 790<br>700—850 | 600<br>485—650 |
| Peroxide of benzoyl + dimethyl-aniline . . . . . | 45 min. | 2 h | some tack | 1450<br>1100—1900 | 850<br>800—905 |
| Peroxide of benzoyl + dimethyl-aniline + linoleate of cobalt . . . . | 60 min. | 2 h | tack absent | 1445<br>1070—1855 | 800<br>735—925 |

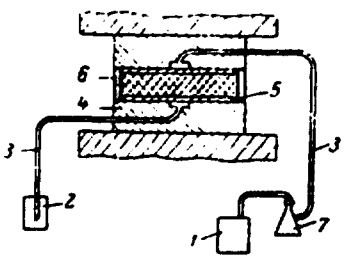


Fig. 1. Sketch showing impregnation under the action of a vacuum and pressure in a closed mold.

The binder in the vessel 2 under the pressure of air compressed 3 kgf/cm² and a vacuum created in the system by the pump 1 passing along the flexible hose 3 impregnates the fiber glass filler which is in the device 4 between the metallic linings 5 and the frame 6 and passes into the receiver 7.

After the impregnation of the filler during the course of 45 to 60 minutes the pack was separated from the pump and the vessel with the binder with the aid of two valves and the apparatus was left under the press for 24 hours.

*The tests were continued for 15 hours counting from the moment of preparing the plates.

By this method there were obtained fiber glass plastics with a base of binder 11EDSM at normal temperature and the use of different oxidation-reduction systems (Table 2).

Table 2. Strength of fiber glass plastic hardened at normal temperature as depends on the type of oxidation-reduction system (impregnation under vacuum and pressure in a closed mold).

| Oxidation-Reduction System | State of surface of glass plastic | Static bending strength, kgf/cm² | |
|---|---|---|---|
| | | at 20°C | at 200°C after aging for 200 h |
| Hydro peroxide of isopropyl - benzene - linoleate of cobalt | hard in 10 days | 1100<br>800 - 1100 | 900<br>800 - 1000 |
| Peroxide of benzoyl - dimethyl-aniline | hard in 24 hours | 1950<br>1800 - 2100 | 1250<br>1000 - 1500 |
| Peroxide of benzoyl - dimethyl-aniline - linoleate of cobalt | hard in 24 hours | 1550<br>1100 - 1700 | 1250<br>1000 - 1500 |

A comparison of Table 1 and 2 shows that with the use of impregnation under a vacuum and pressure in a closed mold:

1) $\sigma_i$ of the tested fiber glass plastics is higher than the $\sigma_i$ of material obtained by the usual method of smearing on the binder;

2) the time of the hardening of the fiber glass plastics is shortened;

3) there is eliminated inhibiting action of the oxygen of the air leading to the formation of persistent stickiness on the exposed surfaces of the glass plastic.

Consequently, in the formation of the closed mold one can use the oxidation-reduction system peroxide of benzoyl – dimethylaniline, which assures the greatest strength of the fiber glass plastic both at 20°C and at 200°C after aging at this temperature for 200 hours and also substantially improve the conditions of the work.

## Influence of Heat Treatment on the Strength Properties of Fiber Glass Plastic

Fiber glass plastic was obtained by the described method of contact molding with the starter peroxide of benzoyl and the accelerator dimethylaniline and linoleate of cobalt. The content of resin in the glass plastic amounted to 60%.

For improving the heat resistance and strength of the material obtained after hardening at room temperature it was supplementarily heated at different temperatures up to 200°C.

In Fig. 2 there is shown the influence of the temperature in heating the fiber glass plastic on the yield of insoluble polymer and the static bending strength. For determining this there were used specimens of the size 55 × 15 × 2.5 mm, which somewhat improved the absolute value for the static bending strength as compared with that index of the standard specimens of the dimensions 10 × 15 × 120 mm.

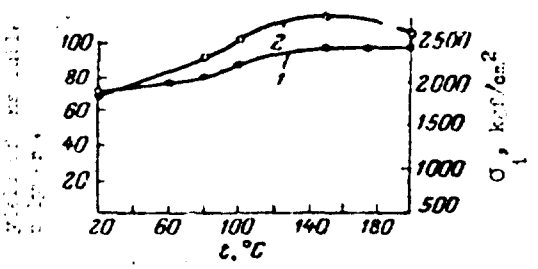


Fig. 2. Influence of the temperature of the heat treatment of the fiber glass plastic obtained from the binder 11EDSM on the yield of insoluble polymer (1) and the static bending strength (2) (heat treatment for 12 hours, thickness of specimens 2.5 mm.

From Fig. 2 it follows that the yield of insoluble polymer of the binder 11EDSM with the rise in the temperature from 20 to 150°C increases from 70 to 95% and $\sigma_i$ correspondingly increases from 1860 to 2800 kgf/cm$^2$.

On the basis of the data from Fig. 2 as the optimal there was selected the temperature of 150°C for the heat treatment.

It has been established that with the increase in the time of the heat treatment the yield of insoluble polymer and the static bending strength of the material improve (Fig. 3).

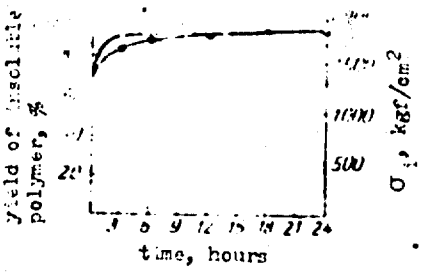


Fig. 3. Influence of duration of heat treatment at 150°C of the fiber glass plastic obtained from the binder 11EDSM on the yield of insoluble polymer (1) and the static bending strength (2).

The influence of the duration of the heating at 150°C on $\sigma_i$ of the fiber glass plastic of "cold" hardening (thickness of the specimen 10 mm, content of resin 61%) is shown below:

| Duration of heat treatment, hrs. | Static bending strength, kgf/cm² |
|---|---|
| — | $\frac{1455}{1075—1620}$ |
| 3 | $\frac{1675}{1210—1780}$ |
| 6 | $\frac{1780}{1510—1915}$ |
| 12 | $\frac{1825}{1315—2315}$ |
| 24 | $\frac{1880}{1400—2380}$ |

As is seen, heat treatment during 12 hours is inexpedient since from this the yield of the polymer and the technological system is considerably drawn out with an insignificant increase in the strength.

We tested the heat stability as to the loss of weight after heating at 100, 150, and 200°C up to 200 hours (Figs. 4a and 4b) and also the change under these circumstances in the static bending strength (Fig. 5).

From Figs. 4a and 4b it follows that the amount of volatile substances given off at 150°C during 200 hours amounts altogether to 3.2%, and at 200°C during the same time to 3.85%.

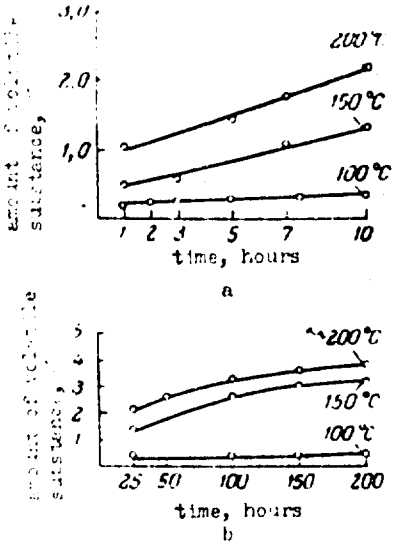


Fig. 4. Influence of temperature and duration of heat treatment on the loss in weight in the fiber glass plastic obtained from the binder 11 EDSM. Time of heat treatment: a — from 1 to 10 hours; b — from 25 to 200 hours.

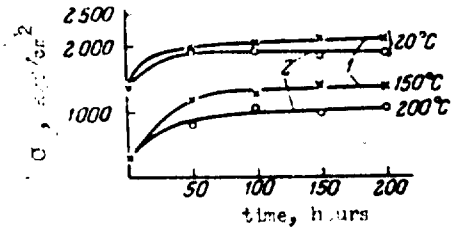


Fig. 5. Influence of duration of heat treatment on the static bending strength $\sigma_1$ of the fiber glass plastic of cold hardening when heat at 150°C (1) and 200°C (2).

From Fig. 5 it follows that $\sigma_1$ both at normal temperature and at 150 and 200°C increases considerably during the first 50 hours of aging which is explained by the completion of the polymerization of the binder in the glass plastic. However, with subsequent aging up to 200 hours there is not noted any lowering of the strength of the material. From what has been explained it follows that fiber glass plastic obtained from the binder 11EDSM of "cold" hardening is heat resistant and after heating at 150°C during 12 hours and can work for a long time up to 200°C.

## Basic Physico-Mechanical and Dielectrical Properties of Fiber Glass Plastic

In Table 3 there are to be found the basic mechanical properties of fiber glass plastic of the brand VP-1KhO, hardened at normal temperature, when it is at the temperatures of 100, 150, and 200°C, after aging at these temperatures for 200 hours.

From Table 3 it is seen that:

1. After heating at 150°C during the selected optimal time, 12 hours, one notices an improvement in the strength of the material as a result of the supplementary polymerization of the binder. With the continuation of the heat treatment at the indicated temperature up to 200 hours the strength characteristics (E, $\sigma_i$, $\tau_{sk}$*) are lowered. An exception is constituted by the static bending strength ($\sigma_s$), which in the case of tests at normal temperature during 12 hours increases almost to the double and does not change after 200 hours of aging. This, apparently, is explained by the formation of a sufficiently stable and heat-resistant polymer.

2. With the increase in the temperature of the heating up to 200°C with the same time of aging of 200 hours, $\sigma_s$, E, $\sigma_i$, $\tau_{sk}$ show a tendency to become lower, apparently, because of the partial destruction of the polymer, its contraction, and the development of inner stresses.

The basic thermo physical properties of fiber glass plastic VP-1KhO before and after the heat treatment at 150°C for 12 hours are shown in Table 4, and the data about the action on it of water, fuels, and oils in Table 5.

In Table 6 there are presented the dielectric properties at the frequencies of 50 and $10^6$ Hz of fiber glass material, hardened at room temperature without heat treatment and heated for 12 hours at 150°C.

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*$\tau_{sk}$ is the shear strength.

Table 3. Mechanical properties of fiber glass plastic VF-1Km[illegible] hardened at normal temperature as depends on the time of aging at different temperatures (content of resin 60-65%).

| Duration and temperature aging | Ultimate strength, kgf/cm² | | | | Impact strength, kgf·cm/cm² | Modulus of elasticity in tension, kgf/cm² | Modulus of elasticity in shear, kgf/cm² | Poisson's ratio |
|---|---|---|---|---|---|---|---|---|
| | tensile | compression (in thickness of rib), kgf/cm² | bending (in thickness of rib), kgf/cm² | shear | | | | |
| Control ($t_{test}$ = 20°C | 1125<br>780–1390 | 1330<br>1020–1615 | 1155<br>1075–1620 | 110<br>110–180 | 95<br>70–110 | 65000<br>59400–71000 | 25700 | 0.276 |
| 150°C—12 h ($t_{test}$ = 20°C | 1875<br>1535–2920 | 2210<br>1780–2545 | 1825<br>1315–2025 | 195<br>175–215 | 115<br>85–140 | 80100<br>76000–110800 | 15300 | 0.261 |
| 100°C—200 h ($t_{test}$ = 100°C | 1210<br>965–1420 | 2055<br>1820–2475 | 1105<br>1085–1140 | 85<br>65–110 | 90<br>85–100 | — | — | |
| 150°C—200 h ($t_{test}$ = 25[illegible]°C) | 1130<br>995–1295 | 2265<br>1970–2415 | 855<br>810–960 | 65<br>55–85 | 90<br>80–110 | 50000<br>45200–58800 | — | — |
| 200°C—200 h ($t_{test}$ = 200°C) | 980<br>900–1025 | — | 800<br>735–925 | 60<br>50–65 | 85<br>80–100 | 48400<br>37800–66700 | — | — |

Note: 1. The data presented are the average results of 10 tests. 2. $\sigma_{shear} = \frac{810}{730-830}$ kgf/cm² after 12 hours of heat treatment at 150°C = $\frac{1025}{960-1095}$. 3. $\sigma_{bear} = \frac{2815}{2595-2965}$ kgf/cm² after 12 hours of heat treatment at 150°C = $\frac{3750}{3460-3040}$ kgf/cm². 4. Tests for bearing strength were conducted on S. D. Tkachev's apparatus at 20°C.

Table 4. Basic thermo physical properties of glass plastic VP-1KhO.

| Indices | before heat treatment | after heat treatment |
|---|---|---|
| Density, g/cm$^3$ . . . . . | 1,41<br>1,31—1,47 | 1,4<br>1,3—1,48 |
| Heat resistance per Martens scale, °C . . . . . | 65<br>59—71 | 240<br>238—240 |
| Heat conductivity factor $\lambda$ in interval 20-200°C, kcal/m·h·°C . . . . . | 0,27—0,38 | 0,36—0,43 |
| Linear extension factor $\alpha$ at 20-200°C, 1/°C . . . | 32,0—12,0·10$^{-6}$ (in interval 20—150 °C) | 25,0·10$^{-6}$ |
| Temperature conductivity factor a at 20-200°C, m$^2$/h . . . . . | 7,1—6,4·10$^{-4}$ | 8,1—6,1·10$^{-4}$ |
| Heat capacity c at 20-200°C kcal/kg·°C . . . . | 0,26—0,41 | 0,30—0,48 |

Table 5. Action of water, fuels, and oils in fiber glass plastic VP-1KhO produced by cold hardening.

| Medium | Increase in weight, % | | | |
|---|---|---|---|---|
| | before heat treatment | | after heat treatment | |
| | 24 hrs. | 30 days | 24 hrs. | 30 days |
| Water | 0,90 | 3,2 | 0,30 | 1,35 |
| Gasoline | 0,83—0,97<br>0,1 | 3,0—3,5<br>— | 0,25—0,40<br>0,01 | 1,3—1,45<br>0,2 |
| Kerosine | 0,02—0,1<br>0,17 | 0,2 | 0,002—0,02<br>0,1 | 0,12—0,25<br>0,2 |
| Oil MS | 0,15—0,19<br>0,65 | 0,17—0,21<br>0,70 | 0,07—0,13<br>0,73 | 0,12—0,28<br>0,97 |
| | 0,54—0,87 | 0,55—0,90 | 0,3—1,0 | 0,85—1,1 |

Table 6. Dielectric properties of glass plastic VF-1Kh at different frequencies*.

| Characteristics of glass plastics | Normal conditions | Φ = 95 ± 3% during 24 h | Temperature of experiment, °C | | | |
|---|---|---|---|---|---|---|
| | | | 20 | [illegible] | 150 | 200 |
| $tg\delta$ at 50 Hz: with heat treatment | 0.020 | does not congeal on existing equipment ** | 0.020 | 0.05–0.07 | does not congeal on existing equipment | |
| after heating 12 h at 150°C | 0.020 | same | 0.020 | 0.021 | 0.011 | 0.311 |
| $tg\delta$ at $10^6$ Hz: without heat treatment | 0.006 | " | — | — | — | — |
| after heating 12 h at 150°C | 0.007 | 0.025 | — | — | — | — |
| ε at 50 Hz: without heat treatment | 4.4 / 4.3–4.6 | does not congeal on existing equipment | 5.0–5.1 | 5.8–5.9 | does not congeal on existing equipment | |
| after heating 12 h at 150°C | 4.2 / 3.9–4.4 | same | 5.1 | 5.4 | 5.8 | 9.0 |
| ε at $10^6$ Hz: without heat treatment | 4.8 / 4.6–4.9 | " | — | — | — | — |
| after heating 12 h at 150°C | 4.7 / 4.3–5.0 | 4.7 | — | — | — | — |
| $\rho_v$ Ω·cm without heat treatment | $7.6–8.6 \cdot 10^{13}$ | $1.6–2.0 \cdot 0.10^{6}$ | $7.6–8.6 \cdot 10^{13}$ | $2.3–4.0 \cdot 10^{9}$ | $8.6–11 \cdot 10^{7}$ | does not congeal on existing equipment |
| after heating 12 h at 150°C | $6.1–8.4 \cdot 10^{13}$ | $5.3 \cdot 10^{11}$ | $6.1 \cdot 10^{13}$ | $7.7 \cdot 10^{12}$ | $7.2 \cdot 10^{9}$ | $1.4 \cdot 10^{9}$. |

*$\rho_s$ of glass plastic before and after heat treatment – $1.2 \cdot 10^{14}$ Ω; disrupvoltage E before heat treatment – 23 kV/mm; after heat treatment more than 23 kV/mm: tgδ and ε congealed on the apparatus "MDP."

**$\rho_v < 10^6$ Ω·cm.

The data of Table 6 reveal the improvement in the dielectric properties of the material, which is explained by the increase in the degree of hardening of the binder and rise in the water resistance of the material (Tables 5 and 6).

The data presented on the physico-mechanical and dielectric properties of the fiber glass plastic VP-1KhO show that this material having been hardened at normal temperature (without heat treatment) can be used for light-load-bearing articles which perform functions at normal temperature and under normal moisture and after supplementary heat treatment (for 12 hours at 150°C) is suitable for articles bearing greater loads used for general and electro technical purposes, which function up to 200 hours at temperatures going as high as 200°C. The use of the material during a longer time, apparently, is quite possible, but requires further experimental checking.

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