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Eco-Biocompatible Epoxy Composites with Vegetable Fillers

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ABSTRACT

The purpose of this work is to study eco-biocompatible epoxy composites with vegetable fillers, the comparison of their chemical and physical qualities, and the prospect of application in various areas of social life. The plant fillers investigated in the work are cheap and available, which makes them an interesting subject of research with the potential of real applications. During the work, 7 samples of epoxy composites with various vegetable fillers (tea, ground coffee, cocoa, ground apricot pit, crushed fruit tree shavings, flour, and micro cellulose) and 1 sample without filler were tested for comparison according to the following properties: microhardness, resistance to abrasion and mechanical compression, adhesion to metals during tearing and shearing, and resistance of samples to aggressive chemical environments and water.

As a result of our research, we were able to obtain data based on our samples, which confirm the effectiveness, feasibility, and potential of using eco-biocompatible epoxy composites for various fields of medical and industrial production, depending on the vegetable filler added to them. It is established that the introduction of some fillers increases compressive strength (load), microhardness, bending angle and abrasion resistance. Infilling with some vegetable composites, such as cocoa and micro cellulose, enhances the adhesion with metals. It is shown that the introduction of vegetable fillers into the composition of composites improves their resistance to the studied aggressive environments. All samples demonstrated good resistance to water absorption, which is practically equal to the indicator for the unfilled polymer.

Introduction

Over the last time, the relevance of the study of epoxy composites has increased significantly. The components for making these composites are relatively affordable, so many industries have the potential to use them. Eco-biocompatible epoxy composites, depending on the filler and its properties, can be used in the production of membranes for water filters, prostheses, hydrogels, coatings and plastics, paper, and innovative electronic technologies [1]. In this work, the properties of 7 samples of eco-biocompatible composites with the following vegetable fillers were investigated: tea, ground coffee, cocoa, ground apricot pit, crushed fruit tree shavings, flour and micro cellulose. They were compared with a sample of unfilled epoxy composite to understand where composites with certain vegetable fillers can be effectively used in industry and medical production.

The fillers were chosen so that some of them had only microparticles (micro cellulose, flour), some had a micro- and coarse-

(meso-) dispersed phase (tea, coffee, dispersed stone) and one each – nano-sized particles (cocoa) or coarse-sized (wood shavings). All fillers (except laboratory-derived micro cellulose) are available and are often waste products from food or technical industries. The amount of filler is fixed - 33% by mass, and often - the maximum amount to preserve the normal consistency of the epoxy composition. The object of the study: the influence of disperse vegetable fillers on the physical and mechanical properties of epoxy polymer composites. The subject of the research: Epoxy polymers with 33% by mass content of vegetable dispersive fillers. The purpose of the research: to investigate the possibility of creating low-cost plant-polymer composites with the desired properties from available resins and materials using a simple direct filling technology, for the current needs of industry, household, and medicine. Scientific novelty: obtaining new experimental data on the effect of plant particles on the physicochemical properties and resistance to different chemical environments of epoxy composites. Based on these data, a conclusion will be drawn regarding the prospects of using such compositions for industrial, medical, and household needs.

Epoxy composites are very strong materials compared to conventional plastics and wood-paper materials. Also, recent studies have revealed that epoxy composites are biocompatible when they are fully cured, which is achieved in 2-3 days. There are modern studies that confirm the possibility of creating high-quality and biocompatible poly epoxy exo prostheses or other biomaterials [1-5]. Composites made of epoxy resin are valuable because their fundamental properties do not depend either on the place of production or on the time of storage. Epoxy resins are well combined with non-polar and polar liquids and, accordingly, with almost all fillers. They are widely used to produce various coatings, eco-packaging, and restoration. Epoxy-vegetable composites can also be used for the needs of construction, design, decoration, for domestic purposes (creating glues and materials like wood), in service (gluing). At the same time, a wide variety of fillers are used. We will give examples of works published by international scientific teams in recent years. Nanocellulose-epoxy polymers are studied in institutes and are of great interest to scientists, for example works [5-7]. There is considerable interest in epoxies with micro cellulose [7,8] and cellulose fibers. Epoxy composites with pine dispersions. Suhaimi investigated the reinforcement of epoxies with coconut fibers.

According to Indian patent no. 202041000392, epoxy polymers with various exotic plant fibers (Prosopis Juliflora Bark Fibers, Phoenix Pusilla Leaf Fibers) will be very promising for industrial purposes. A. Musaeva investigated filled epoxy resin with nut powders - hazelnut and walnut. As a result of research, it was proved that with the addition of fillers, the impact toughness, density, and hardness of the composite tend to gradually increase. In some works, it is shown that the introduction of waste from lignin-cellulose production (scoop) into epoxy resin allows obtaining composites with satisfactory properties [6]. The team of O. Daramola investigates the effect of plants and other natural powders and fibers on the properties of polymers - in particular, for biomedical purposes. Such a number and variability of research on the topic of eco-biocompatible epoxy composites with vegetable fillers confirms the interest of scientists in this direction.

Devices and Materials

Epoxy 520 epoxy resin, DETA hardener and the following vegetable fillers were used to manufacture the samples of the studied composites: tea, coffee, cocoa, fruit tree shavings, apricot stone, micro cellulose, flour. The mass of each of the vegetable fillers was 3 g, where 33% was the filler itself and 66% of the mixture was occupied by the DETA hardener. Next, the filler mixture was filled with epoxy resin Epoxy 520 in a ratio of 3:5. One sample of the mixture without filler, respectively, with epoxy resin and hardener in the ratio of 3:5 was also prepared for further comparison with other samples. The following were used for the experiments: electronic caliper, P80 sandpaper, ASTM 2240, Luis Shopper press, ID-1 tearing machine, UMM press, a mixture of propanone and ethyl acetate (1:1), a mixture of 50% hydrogen peroxide, and boiled water.

Figure 1: Micro images (x100) of epoxy cellulose compositions.

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After making suitable mixtures of vegetable fillers and epoxy resin (Figure 1), various molds were used for the curing process. Thus, for the epoxy composites with each filler, the following were produced: cylinders with a surface area of 0.42 cm^2 and a height of 1.2 to 1.5 cm, cubes with a side of 1 cm, regular quadrangular pyramids with a surface area of 1 cm^2, flat hexagons, gluing of steel and tin plates with an area of 2 cm^2, steel cylinders with an area of 5 cm^2, which were glued with appropriate mixtures of composites. Strength characteristics were measured as follows. Compressive strength-taking into account State Standard 4651-2014, on cylinder samples with a diameter of 0.65 cm and a height of 1.1+- 0.1 cm, on a Luis Shopper press. Abrasion – according to the mass of the abraded composite after 40 passes of 20 cm on P80 sandpaper. Abrasion resistance is calculated as an inverse characteristic, taking into the account the relative density of the samples according to the formula I = ρ/Xρpro (ρ/ρpro – the ratio of the density of the composite and H-polymer, X-the mass of the abraded composite, mg).

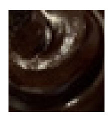
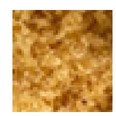
Adhesion at separation was according to State Standard 14760-69, on steel cylinders with an area of 5 cm^2, on the UMM machine. Shear adhesion was measured according to State Standard R 57834-2017, on gluing of steel and rigid plates with an area of 2 cm^2 using an ID-1 tearing machine. Microhardness was measured by indenting an indenter into composite materials in the shape of a flat hexagon using the ASTM 2240 device. Swelling of pyramid and cube samples was classically determined by growth, calculated according to the formula q = (m - m0)/ m0, mass %, where m and m0 are the final and initial masses of the sample.

Experimental Results

Determination of Microhardness of Samples

The hardness of materials is usually measured in the following ranges: macro-, micro-, nano-. Macro hardness is measured by the load on the indenter from 2 N to 30 kN; micro-range - according to the load on the indenter up to 2 N and the penetration depth of the indenter by more than 0.2 μm; the nanorange is measured by the penetration depth of the indenter, which should be less than 0.2 μm. According to the obtained data (Table 1, microhardness), the following can be said: epoxy composites with vegetable fillers naturally reduce microhardness, since the porosity and fiber structure of the fillers disrupts the integrity of the polymer structure. At the same time, cocoa does not cause deterioration of this characteristic, compared with the unfilled sample. Epoxy composite with vegetable filler apricot stone gives a slight improvement in the microhardness of the material by 1%.

Table 1: Physical and mechanical parameters of the studied samples (*- estimated).

	H	Ch	Kv	Ka	MC	A	O	M
Composition, visual								
Adhesion at separation, kgf	40	-	-	-	80	-	-	60
Shear adhesion, kgf	60	-	30	85	60	-	-	40
Abrasion, mg/mm	75/2.5	110/3	110/ 3	93/ 2.5	80/2.1	90/2.6	150 /4	115/3.1
Compression, kgf	320	190	215	220	290	260	30	220
Microhardness, x.F	86	81	80	86	80	87	60	80
Shrinkage, mm	1,5	1	1,1	1,3	1,3	1	-	1
*Maximum bending angle°	80	-	40	40	60	-	-	40

In general, except for the composite with shavings of fruit trees, all our composites show a microhardness like (or slightly lower than) that of the pure polymer. This makes it possible to recommend these fillers as effective cost-saving additives for polymer products that must withstand the load of sharp objects (furniture legs, appliance racks, heels, etc.).

Determination of Abrasion Resistance of Epoxy Composites with Vegetable Fillers

As mentioned earlier, eco-biocompatible epoxy composites can be effectively used in many industries. One of the possible areas of application of epoxy composites is the production of coatings. High-quality coatings are distinguished by their resistance to abrasion. That is why we tested the abrasion resistance of epoxy composites with vegetable fillers and an unfilled sample, and then compared these values. P80 sandpaper was used for the experiment. In the experiment, cylinders of epoxy composites with a surface area of 0.42 cm^2 and a height of 1.2 to 1.5 cm were used. Before the start of the experiment, the masses of the samples and the height of each of them were also pre-measured. After that, 40 passes of 20 cm of each sample were made with sandpaper. At the end of the rubbing passes, a second measurement of the mass and height of the epoxy composites was

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made. Abrasion resistance was calculated according to the formula I = ρ/Xρpro(ρ/ρpro – the ratio of the density of the composite and H-polymer, X – the mass of the abraded composite, mg).

Based on the data we received (Table 1), we can draw the following conclusions. All fillers reduce abrasion resistance, as the mass of the abraded substance increases. This is natural, since soft vegetable fillers are not abrasive. The only filler that almost does not affect this indicator is micro cellulose. Also, when adding the dispersion of apricot bones and cocoa, we have a barely noticeable decrease (the mass of the erased substance and the height of the erased column increase slightly). This is obviously explained by the fact that these fillers are either quite resistant to abrasion (bone, due to dispersion), or have a non-fibrous nanostructure (cocoa).

Evaluation of Adhesion during Detachment and Shearing of Epoxy Composites with Vegetable Fillers with Metals

Adhesion is a molecular connection between the surfaces of heterogeneous bodies brought into contact [18]. To understand whether epoxy composites with vegetable fillers are suitable to produce coatings for industrial or medical products from metals, it is important to measure the adhesion of epoxy composites with them. In this work, the adhesion of the composites during tear-off and shear was measured. To measure the adhesion upon separation, metal cylinders with a surface area of 5 cm^2 were used, on which the composite mixture was previously applied and glued together. According to the obtained data (Table 1), we can draw conclusions: epoxy composite with micro cellulose gives an improvement in adhesion by 87.5%, compared to the unfilled sample, which indicates the feasibility of using epoxy composites with this filler for the manufacture of coatings for metal products; the powder epoxy composite gives an improvement of 50% from the unfilled sample, so it can also be effectively involved in the production of coatings for industrial metal products.

To measure the shear adhesion, steel and tin plates were used, on the edges of which the investigated composites were applied. The area of application of epoxy composites is 2 cm^2. After that, the plates were glued together in pairs. The experiment was carried out on the tearing machine ID-1. According to the results of the experiment: the epoxy composite with cocoa filler gives an improvement in shear adhesion by 42%, compared to the unfilled composite; epoxy composite with micro cellulose has the same bond strength to metal as unfilled composite; epoxy composites, where vegetable fillers are coffee and flour, show a deterioration in adhesion compared to the unfilled sample. From this it can be concluded that epoxy composites with such vegetable fillers as cocoa and micro cellulose can be used to make coatings for metal products, in particular prostheses or industrial metal equipment.

Maximum Bending Angle of Samples

After the tear adhesion measurement, the used samples were evaluated for the maximum bending angle relative to each other. The results of the experiment indicate that the unfilled epoxy composite is the most elastic. Vegetable fillers did not improve the elasticity of the material. On the other hand, the deterioration of the elasticity of the material when fillers are added indicates that they can be used to produce products that should have small indicators of this characteristic. Composites with cocoa, coffee and flour show a deterioration of elasticity of 50% compared to the composite without filler, for micro cellulose - 25%.

Resistance of Epoxy Composites to Mechanical Compression

Compressive strength is one of the most important characteristics. Epoxy polymer has high compressive strength, which, as a rule, is difficult to significantly strengthen with filling. It can be expected that some vegetable fillers (due to low stiffness) will increase it. To measure the resistance to mechanical compression of epoxy composites, we used epoxy composites of a cylindrical shape – columns with a height of 1.1+-0.1 cm and a diameter of 0.65 cm. The experiment was carried out on a L. Schopper press machine (Figure 1 No. 5). As a result of the experiment, we obtained the data shown in Table 1. Among the tested samples, micro cellulose and apricot bone proved to be the most resistant to compression. They showed the least deterioration relative to the composite without filler.

From the data in Table 1, we can conclude that most of our fillers, taken in a large amount (33 mass %), do weaken the compressive strength. At the same time, compression diagrams (Figure 2) show some weakening of the modulus during compression (because the straight section of the diagram has a smaller angle of inclination). Of all samples, only micro cellulose gives a slight decrease in compressive load, and when filled with coarse particles (for example, tea powder or shavings), a significant decrease in the indicator is observed.

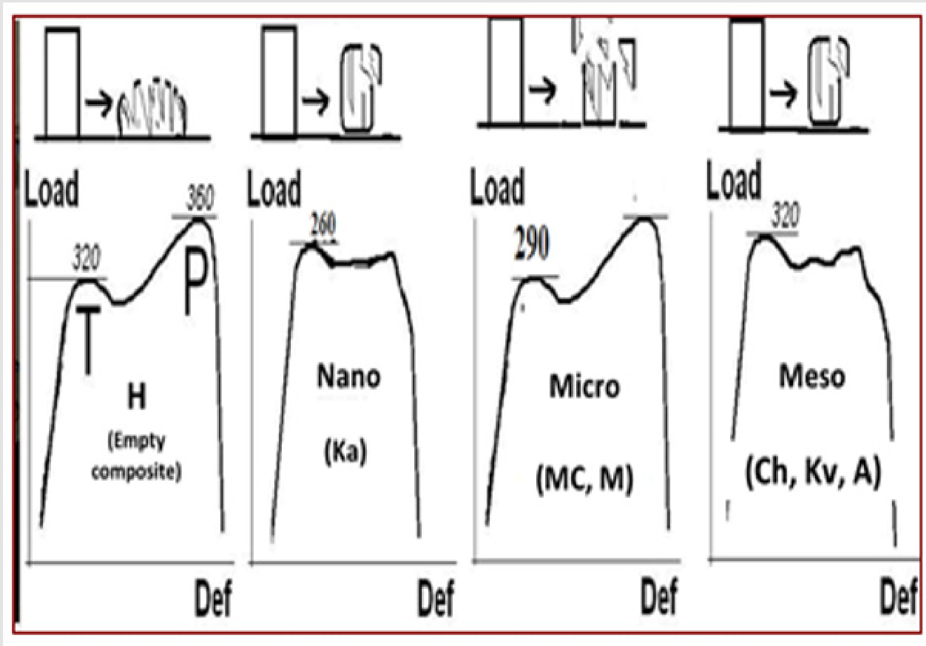

Figure 2: Samples compression diagrams.

Determination of Chemical Stability of Samples in Aggressive Environments

Samples of regular quadrangular pyramids with a surface area of 1 cm^2 were used to study epoxy composites for resistance in aggressive chemical environments. The weight change of the composites was measured using jewelry scales. The experiment was conducted for 8 days. During the experiment, the samples were stored in special little glass bottles.

Figure 3: Appearance of the samples after 1hour (3.a) and 3 days (3.b) exposure in the solvent.

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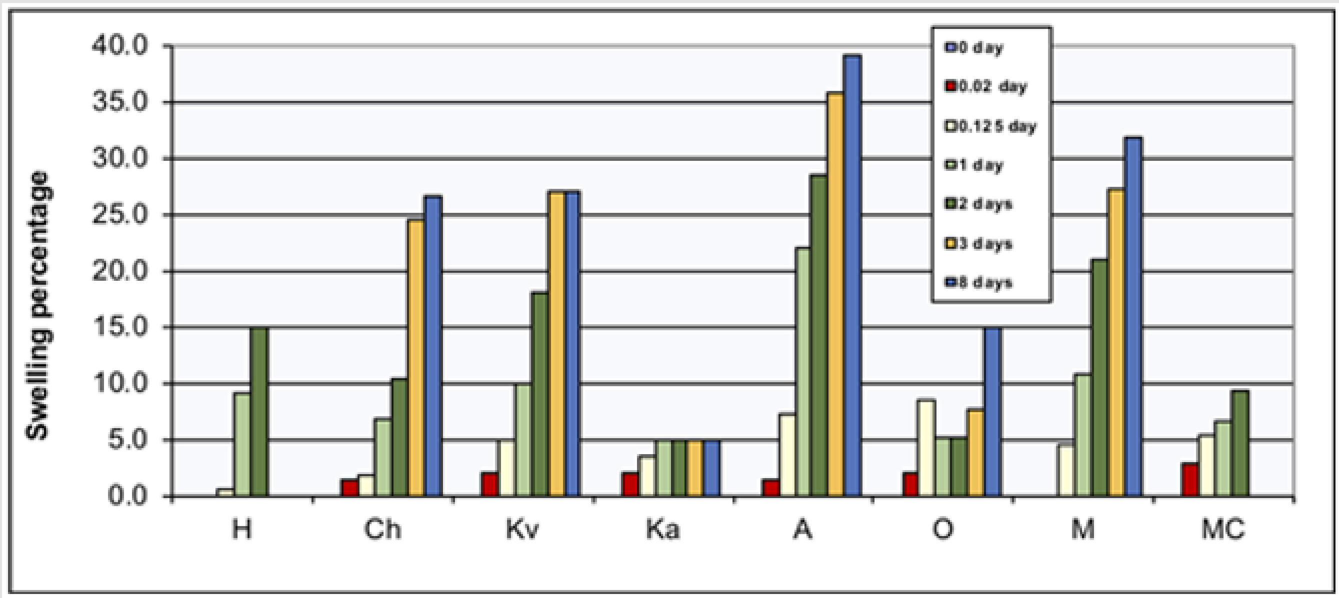

Figure 4: Histogram of swelling of samples in a mixture of acetone: Ethyl acetate (1:1) (aggressive solvent).

Chemical Stability of Samples in a Mixture of Acetone and Ethyl Acetate: According to the results of our experiment, we managed to obtain the data shown in Table 2. In Figure 4, you can also visually follow the dependence of the stability of the epoxy composite on the filler added to it. An unfilled sample quickly swells and disintegrates after 1-2 days of exposure. The sample with 33 mass % micro cellulose behaves in the same way. Note that according to, the introduction of 5 or 10% by mass of micro cellulose gives, on the contrary, a strengthening effect. Therefore, the effect of fillers on the composites resistance to the solvent is highly dependent on the concentration of the filler. Samples with tea, coffee, especially flour and dispersed apricot bone, lighten and swell (Figure 3), but do not disintegrate. The composite with cocoa nanofiller turned out to be even more stable: it neither swells nor decomposes, but only lightens (Figure 3). It is interesting that a very loose sample with flakes shows high resistance to both swelling and disintegration (Figures 3 & 4).

Table 2: Swelling of samples in a mixture of acetone : ethyl acetate (1:1) (aggressive solvent).

	H	Ch	Kv	Ka	A	O	M	MC
0 min.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
25 min.	0.0	1.4	2.1	2.0	1.5	2.1	0.0	2.9
3 hours	0.6	1.8	5.1	3.5	7.2	8.5	4.5	5.3
1 day	9.2	6.8	9.9	5.0	22.0	5.1	10.8	6.7
2 days	14.9	10.3	18.1	5.0	28.4	5.1	20.9	9.3
3 days	Decomposed	24.6	26.9	5.0	35.8	7.7	27.3	Decomposed
8 days	-	26.7	26.9	5.0	39.1	15.0	31.8	-

Note: the number of times the mass of each sample increased over time is indicated in the cells.

Based on the results of this experiment, the following conclusions can be drawn Composites with all vegetable fillers, except micro cellulose, gave an improvement in stability. The composite with micro cellulose and without filler was destroyed in one day – on the 3rd day of the experiment. The most resistant to such an aggressive chemical environment as a mixture of propanone and ethyl acetate (1:1) turned out to be the composite with the addition of cocoa as a filler.

Chemical Stability of Samples in 50% Hydrogen Peroxide Solution: Data obtained during the experiment of testing composites for resistance to hydrogen peroxide are shown in Table 3 Also in Figure 6, you can see how samples of composites with different plant fillers changed their weight according to the time spent in an aggressive environment. According to these data, the following can be said: the most resistant to such an aggressive environment as hydrogen

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peroxide was the epoxy composite with shavings of fruit trees, which gave a two-fold improvement; a composite with tea as a filler also gave a 12% improvement. Composites with apricot bone and micro cellulose did not give a noticeable improvement. Composites with flour, coffee and cocoa turned out to be the most unstable to hydrogen peroxide relative to the unfilled sample.

Table 3: Swelling of samples in a 50% solution of hydrogen peroxide H_2O_2 (concentrated hydrogen peroxide, an aggressive oxidizer).

	H	Ch	Kv	Ka	A	O	M	MC
0 min.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
25 min.	2.4	3.0	5.2	0.0	2.6	15.4	3.6	2.6
2.5 hours	2.4	5.7	10.6	28.7	8.1	31.3	9.5	10.1
1 day	15.1	17.0	31.9	51.6	23.5	39.0	31.0	26.5
2 days	22.3	27.2	48.9	64.3	33.3	39.0	46.2	38.1
3 days	29.5	33.2	58.0	72.0	41.6	32.1	59.3	46.3
8 days	62.0	54.3	90.5	93.6	72.8	32.1	95.7	71.6

Note: The number of times the mass of each sample increased over time is indicated in the cells.

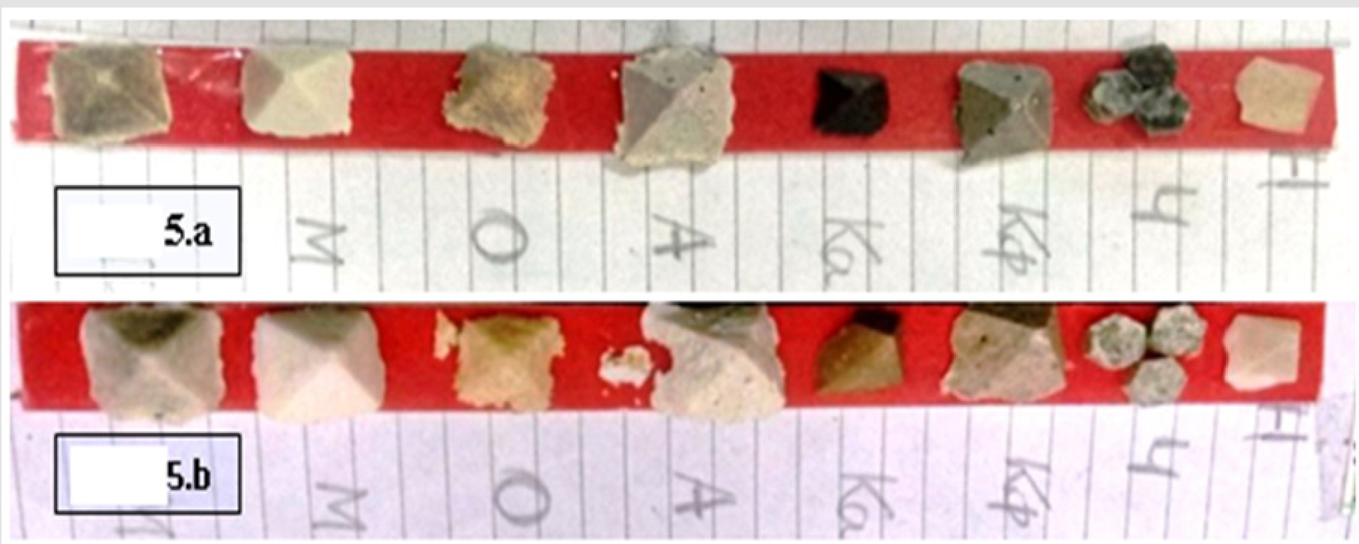

Figure 5: Appearance of the samples after 1hour (5.a) and 3 days (5.b) exposure in an oxidant (50% H_2O_2).

Visually, the samples immediately begin to lighten or even turn white, bubbles appear in the liquid (an oxidation process is underway). As can be seen from Figure 5, the cocoa sample later turned from black to light brown. The sample with bone dispersion immediately changed color from orange to gray (Figure 5a- 1 hour), and later - white, and began to partially disintegrate (Figure 5b). The samples with micro cellulose and tea also lightened and swelled greatly.

According to the data of Table 3, 33 mass % of filling in almost all cases increases the swelling process. The exception is only for the most coarsely dispersed fillers - tea and shavings, due to their resistance to oxidation. Also, the sample with cocoa nanofiller withstands the oxidizer well (Figure 5a) due to the antioxidant properties of cocoa.

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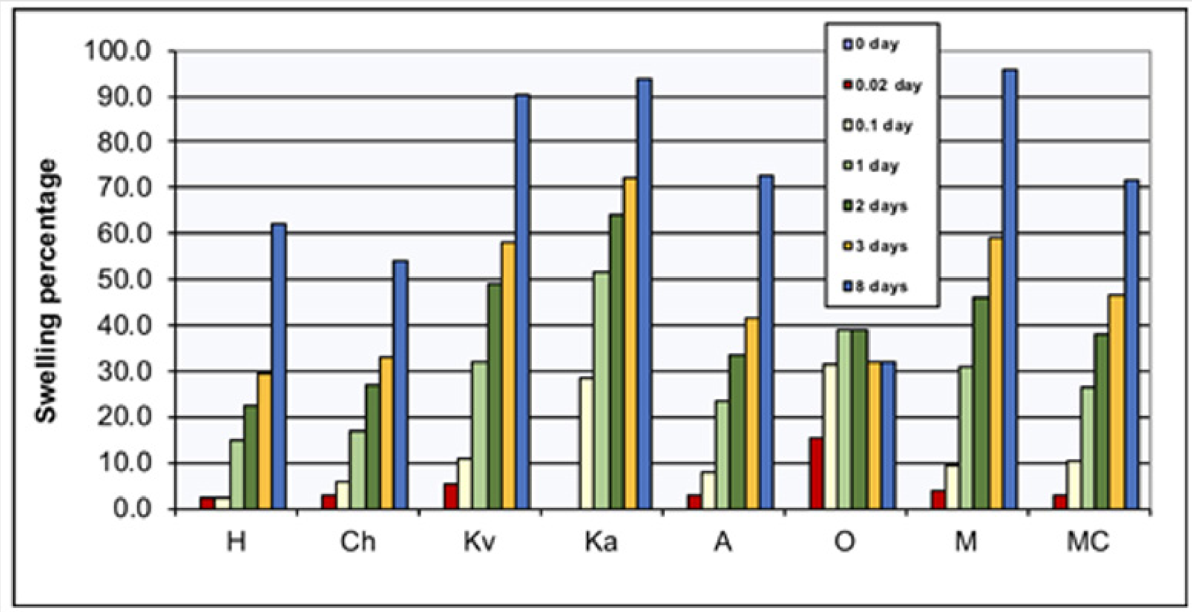

Figure 6: Histogram of swelling of samples in a 50% solution of hydrogen peroxide H2O2 (concentrated hydrogen peroxide, an aggressive oxidizer).

Water Resistance of Epoxy Composites with Vegetable Fillers

Important in determining the purpose of epoxy composites with vegetable fillers in various industries is their resistance to water. In our experiment, we checked how the mass of epoxy composites changes depending on the time they were in the liquid. The water in cups with composites was changed every 7-12 hours. The initial water temperature at each change was 100°C. The experiment was conducted for 9 days. The data we obtained are shown in Table 4.

Table 4: Swelling of epoxy composite samples in water.

	H, mg	Ch, mg	Kv, mg	Ka, mg	A, mg	O, mg	M, mg	MC, mg
0 min.	1,00	0,99	1,03	1,09	1,03	0,81	0,92	1,03
1 hour	1,00	1,03	1,03	1,12	1,04	0,86	0,95	1,05
16.8 hours	1,00	1,03	1,05	1,12	1,05	0,88	0,99	1,07
26.3 hours	1,01	1,03	1,06	1,13	1,06	0,88	1,01	1,07
2 days	1,01	1,04	1,06	1,13	1,06	0,89	1,01	1,09
4 days	1,02	1,04	1,07	1,14	1,06	0,89	1,02	1,10
5 days	1,02	1,04	1,07	1,14	1,07	0,89	1,03	1,10
6 days	1,02	1,04	1,08	1,15	1,08	0,89	1,06	1,11
7 days	1,02	1,04	1,08	1,15	1,08	0,89	1,06	1,13
9 days	1,03	1,04	1,08	1,16	1,08	0,89	1,08	1,13

It can be expected that after filling with a significant mass (in particular, 33 mass %) of plant powders, the water resistance of epoxy composites is sharply weakened (due to an increase in porosity and a decrease in the integrity of the structure) – as in the case of 50% H_2O_2. However, this did not happen during our experiment. It can be seen from Table 4 that all samples demonstrate good resistance to water absorption, which is practically equal to the indicator for the unfilled polymer. This is a very promising result, as it allows the use of cheaper (by a third, by adding 33% vegetable fillers) epoxy materials for waterproof structures, products, and coatings.

Conclusion

A. The analysis of the literature during our work showed the relevance and perspective of studying epoxy polymer composites with the use of vegetable fillers, which can improve the physical and mechanical characteristics of the composites.

B. Epoxy compositions were made with tea, ground coffee, cocoa, ground apricot pit, crushed fruit tree shavings, flour, and micro cellulose.

C. The introduction of vegetable fillers in a significant amount (33% of the mass of the mixture) makes it possible to obtain high-quality composites that harden into a composite of black (tea, cocoa, coffee), brown (bone fillers, micro cellulose) or white (flour) color.

D. Physical and mechanical parameters of composite samples were measured: microhardness, resistance to abrasion, resistance to mechanical compression, adhesion to metals during shear and separation. It is established that the introduction of some fillers increases compressive strength (load), microhardness, bending angle and abrasion resistance. Infilling with some vegetable composites, such as cocoa and micro cellulose, enhances the adhesion (as shown in the shear and pull-off adhesion experiment) and significantly improves the shrinkage resistance of the composites.

E. It is shown that the introduction of vegetable fillers into the composition of composites improves their resistance to the studied aggressive environments. They are particularly resistant to acetone and ether (for example, propanone - ethyl acetate) solvents: in them, epoxy composites degrade rather slowly, and often swell less (compared to unfilled polymer). Unlike a polymer without a filler (which decomposes after 1-3 days of exposure in the environment), most epoxy composites can maintain integrity for a very long time. In a strong oxidizing environment (for example, 50% H_2O_2), filled composites swell as a rule more strongly than an unfilled sample (the exception is filling with a dispersion of tea and shavings of fruit trees).

F. Retention of resistance to water absorption after filling with almost all investigated vegetable fillers (except coffee, cocoa, flour) is very important. This allows you to use them as cheap epoxy materials for creating waterproof structures, parts, and coatings.

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