



Article

Preparation and Characteristics of Fire-Safe Foamed Epoxy Composites Filled with Technogenic Waste from Ceramic Brick Production

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Abstract: As a result of the conducted studies, the method of combining components and foaming parameters for obtaining foam epoxides has been selected. The relationship between the structure of the foam epoxide and the amount of the added blowing agent—ammonium carbonate—has been established. This study explores the development of fire-safe foamed epoxy composites by incorporating technogenic waste from ceramic brick production. The optimized composites demonstrated improved compressive strength, enhanced fire resistance (the LOI—35–44%—and successful UL-94 V-0 testing) through the use of flame retardants (Tris(2-methylphenyl) phosphate and Decabromodiphenyl oxide) and low thermal conductivity (0.030–0.042 W/m K), highlighting their potential as sustainable thermal insulation materials.



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Keywords: foamed epoxy composites; modification; flame retardants; blowing agent; filler; brick dust

1. Introduction

Thermal insulation plays an important role in modern industries, construction, housing, and utilities. It helps solve important issues of life support, organization of technological processes, and energy saving [1,2]. Thermal insulation structures are an integral part of protective elements of industrial equipment, pipelines, and parts of residential, public, and industrial buildings. Reliability, durability, and operational efficiency of buildings, structures, and equipment are significantly increased by thermal insulation materials [3,4].

Epoxy polymer foams have an outstanding set of properties, due to which they have found wide application in packaging, transport, electronics, the aerospace industry, construction, and structural materials [5,6]. Epoxy foams are two-phase materials in which gas bubbles released during the polymerization process are distributed in the volume of the matrix [7]. The nature and structure of the foamed polymer are the sources of its high chemical resistance and shock-absorbing characteristics, low specific gravity, heat, and

electrical insulation properties [8,9]. Epoxy foams are generally rigid and are often used when applications require increased heat resistance, solvent resistance, good adhesion, or more precise controlled foaming than polyurethane foams. Epoxy foams eliminate potential health problems associated with isocyanate sensitivity in the urethane industry, which is often cited as a reason for using foamed epoxy polymers. The advantage of epoxy resins is a wide variety of both resins themselves and crosslinking agents that can be used to obtain a final product with the required properties. Currently, there are studies showing that the modification of the epoxy matrix with dispersed or fibrous fillers, chemicals, and physical effects allows both improving the properties of the epoxy composite as a whole and imparting functional properties to it [10–13].

Xun Fan [14] et al. modified epoxy resin with functionalized multi-walled carbon nanotubes (fMWCNTs) and liquid carboxy-terminated butadiene–acrylonitrile copolymers, and the foaming process was provided by supercritical carbon dioxide (SCCO₂). As a result, a cellular structure with an average particle size of ~400 nm and a cell density of ~10¹² cells/cm^{−3} was obtained. During the formation of the cellular structure, conductive networks are formed due to the fact that fMWCNT particles overcome the percolation threshold, which allows them to achieve an electrical conductivity value of 0.43 S/m when adding 5.0 wt%. At the same time, excellent electromagnetic radiation shielding indicators, an EMI SE of 22.90 dB and a specific EMI SE of 37.54 dB/(g·cm³), were obtained, which is explained by the synergistic effect, as a result of which the transmitted wave is repeatedly reflected from one cell to another, gradually weakening the conductive filler. Waste and crushed fiber-reinforced composites can be used to increase the strength of epoxy foam [5]. The addition of carbon composites with an average fiber length of 59 μm allows for an increase in the compressive strength and elastic modulus to 22.8 MPa and 1060 MPa, respectively. The addition of carbon fibers into epoxy foam in small quantities [15], up to 0.075 wt.% allows, it to increase its shielding properties in the context of microwave radiation, so the reflection coefficient of epoxy foam reaches values below −10 dB in the entire frequency range and values below −20 dB in the range from 2.6 to 18 GHz. In [16], the authors developed a non-standard approach to obtaining epoxy foam modified with silver-coated melamine sponge, microspheres, and MWCNTs. Thus, Yu Xu et al. obtained epoxy foam with an electrical conductivity of 253.4 S·m^{−1}, an electromagnetic interference shielding effectiveness of 68 dB in the frequency range of 0.05–18 GHz, and a thermal conductivity of 0.305 W·mK^{−1}.

It should be noted that in the last few years, there has been a trend to search for new ways of industrial waste disposal, related to environmental initiatives and concern for public health. Branches of industrial production, such as metallurgy, fertilizer production, building materials industry, etc., produce a significant amount of harmful emissions that can potentially be used in the production of epoxy composites. A number of works have shown the efficiency of using slags (blast furnace, ferrochrome, and converter) [17], neutralization sludge [18], marble production waste [19], polypropylene pyrolysis products [20], etc., as fillers for the epoxy matrix. One such waste requiring disposal is dust captured by gas-cleaning filters during the production of ceramic bricks. However, very few publications are available that give information on the effect of brick dust obtained during gas cleaning on the properties of foamed epoxy polymers.

Thermal insulation materials are crucial for energy efficiency in construction and industrial applications, but many suffer from poor mechanical properties, flammability, and environmental concerns. Epoxy foams, with their excellent thermal insulation and chemical resistance, offer a promising alternative. This study addresses two major challenges—enhancing fire safety and incorporating industrial waste—by introducing brick dust and flame retardants into epoxy composites.

2. Materials and Methods

2.1. Materials

Foamed epoxy composites were prepared on the basis of the ED-20 epoxy diene resin and an amine-type hardener, polyethylenepolyamine (PEPA), which were obtained from CHIMEX Limited, St. Petersburg, Russia.

Ammonium carbonate (AC) was used as a chemical blowing agent capable of decomposing at a temperature of 60–80 °C to form NH_3 , CO_2 , and H_2O .

Brick dust (BD), which is formed on filters during the production of ceramic bricks and is a waste product of this production, was used as a filler for foamed epoxy composites.

To reduce flammability, fire retardants, such as Tris(2-methylphenyl) phosphate (TCP) with purity $\geq 99\%$ (Aladdin Reagent, Shanghai, China) and Decabromodiphenyl oxide (DBDPO) with purity $\geq 98\%$ (Aladdin Reagent, Shanghai, China), were used. The chemical structure of fire retardants is shown in Figure 1.

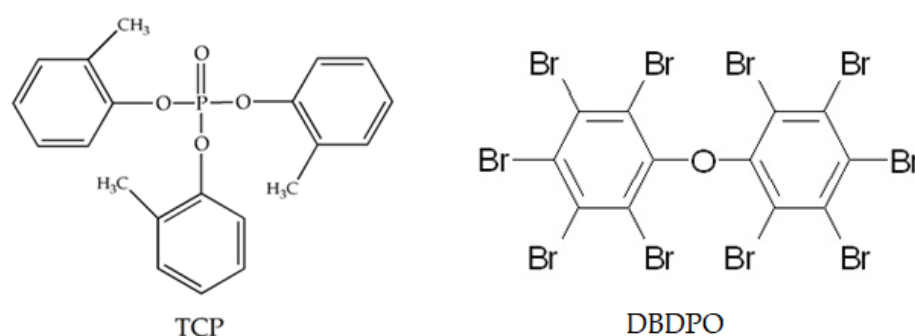


Figure 1. Chemical structure of the TCP and DBDPO.

2.2. Characterization of Filler

X-ray fluorescence analysis was used to study the local X-ray fluorescence elemental microanalysis of brick dust (BD) with the possibility of elemental mapping. The principle of qualitative elemental analysis of the sample is based on the characteristic secondary X-ray radiation—X-ray fluorescence excited by primary X-ray radiation and recorded using special sensors. Quantitative analysis is carried out by comparing the radiation intensity of the elements under study in the sample and standards with a known content of these elements. X-ray fluorescence analysis of the CP was carried out on an X-ray analytical microprobe, aRAM 30- μ microscope (JSC “Scientific Instruments”, Saint Petersburg, Russia).

The analysis of the distribution of BD particles by size in the aqueous suspension was carried out using the laser diffraction method based on the physical principle of electromagnetic wave scattering. To analyze the distribution of the filler particles by size, a laser particle size analyzer Fritsch Analysette-22 Nano Tech (Fritsch, Idar-Oberstein, Germany) was used, allowing measurements to be carried out in the range of 0.01–2000 μm .

The morphology of the filler surface (FS) was studied using the scanning electron microscope TESCAN VEGA3 (Brno, Czech Republic).

2.3. Testing of Foamed Epoxy Composites

The curing kinetics of foamed epoxy composites were determined using the thermometric method. An investigation into the composition curing process involved, creating the relationship $G = f(\tau, T)$, where τ represents the time taken for the process in minutes and T denotes the temperature in degrees Celsius. The analysis was conducted with the objects thermally insulated, allowing for an examination of how heat dissipation influenced both the curing and foaming processes [21].

The structure of the cells of foamed epoxy composites was studied using a research-automated complex based on the Zeiss Axio Imager.A2m microscope (Carl Zeiss Microscopy LLC, New York, NY, USA).

The water absorption of foamed epoxy composites was determined in accordance with [22]. The method consists of measuring the amount of water absorbed by the test sample using hydrostatic weighing of the sample after it has been in distilled water for a specified time at $(23 \pm 2)^\circ\text{C}$ under normal pressure. The water absorption relative to the initial volume (WA_v) was determined using the following formula:

$$WA_v = \frac{m_3 + V_1 \cdot \rho - (m_1 + m_2 + V_c \cdot \rho)}{V_0 \cdot \rho} \cdot 100$$

where m_1 , m_2 , and m_3 are the mass of the original sample, frame, and frame with the sample after testing, respectively, V_0 and V_1 are the volumes of the original and tested sample, ρ is the density of the water, and V_c is the correction for the volume of the water in the cutting of surface cells.

The water absorption relative to the initial total surface (W_s) was determined using the following formula:

$$W_s = \left(\frac{m - m_0}{S \cdot \rho} + \frac{V - V_0}{S} \right) \cdot 10^4$$

where m_0 and m and V_0 and V are the mass and volume of the original sample and sample after testing, respectively, ρ is the density of the water, and S is the full geometric surface of the sample.

The moisture absorption of foamed composites was determined using the following method. The samples were kept for 24 h at a temperature of 23°C . Cubic samples with an edge length of (30 ± 0.3) mm (at least three for each material) were made, without visible defects of the surface film, the dimensions were measured, and the initial mass of the sample was determined. The samples were placed in a desiccator that was pre-filled with water at the bottom. The samples were placed on a grid above the water and kept in a tightly closed desiccator for 24 h with a relative humidity of 98%. After the specified time passed, the samples were weighed again, and moisture absorption was determined using the following formula:

$$B = \frac{m_1 - m_0}{m_0} \cdot 100$$

where m_0 is the mass of the original sample and m_1 is the mass of the sample after exposure to the desiccator, g.

The apparent density of foamed epoxy composites was determined in accordance with [23]. The essence of the method is to determine the mass of the sample per unit of its volume.

Stress was determined at 10% relative compression strain of foamed composites using the WDW-5E universal testing machine (Time Group Inc., Beijing, China) in accordance with [24]. Tests were carried out on samples that were 50 mm in length, 50 mm in width, and 50 mm in height. The permissible deviation for these dimensions is $\pm 1\%$.

Thermal conductivity and thermal resistance of foamed epoxy composites were determined using the ITP-MG4 “100” instrument (STROYPRIBOR. Chelyabinsk, Russia) in accordance with [25].

The thermal stability of the foamed epoxy composites was determined by the thermogravimetric method using the MOM Q-1500D derivatograph (Budapest, Hungary).

The LOI was tested using the JF-3 oxygen index device (Jiangning, China), following the ASTM D 2863-2008 standard.

A CZF-5 vertical burning tester (Jiangning, China) was used to perform vertical burning tests according to the UL-94 standard.

3. Results

When selecting the foaming mode for a composition containing fifteen parts by the mass of the PEPA hardener and two parts by the weight of the ammonium carbonate blowing agent per 100 parts by the weight of the ED-20 resin, several methods for obtaining foamed materials were used, as shown in Figure 2.

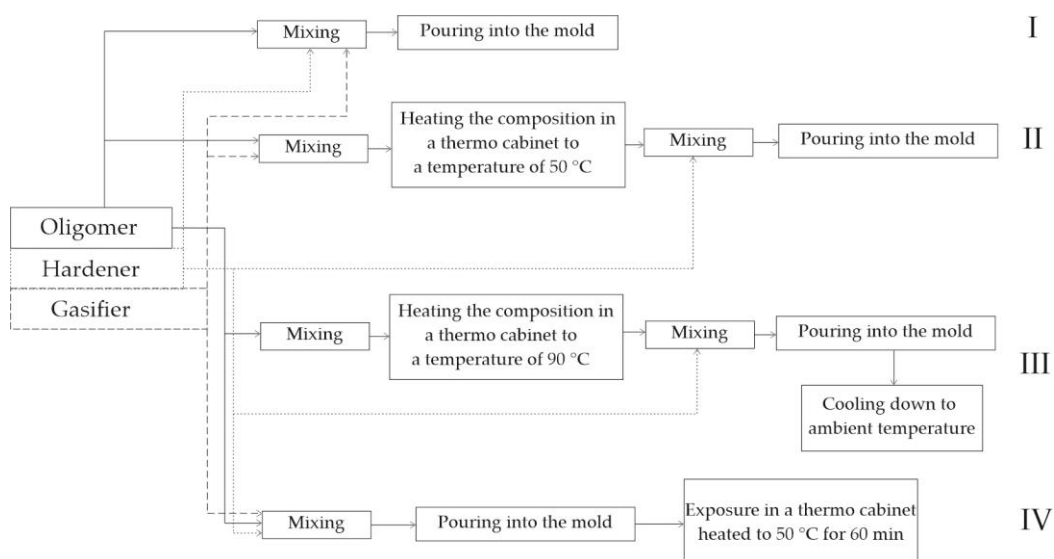


Figure 2. Methods for obtaining foamed epoxy composites.

The first method consisted of mixing all components (epoxy oligomer, blowing agent, and hardener) and placing them in a restriction mold without preliminary heating. Analysis of the curing kinetics of this composition, as shown in Figure 3, showed that no structure formation process was observed for about 60 min, and then a rapid increase in the temperature accompanying the curing process was noted. During the curing process, a temperature of 108 °C was reached due to the process's own exothermicity, thus ensuring the possibility of decomposition of the blowing agent.

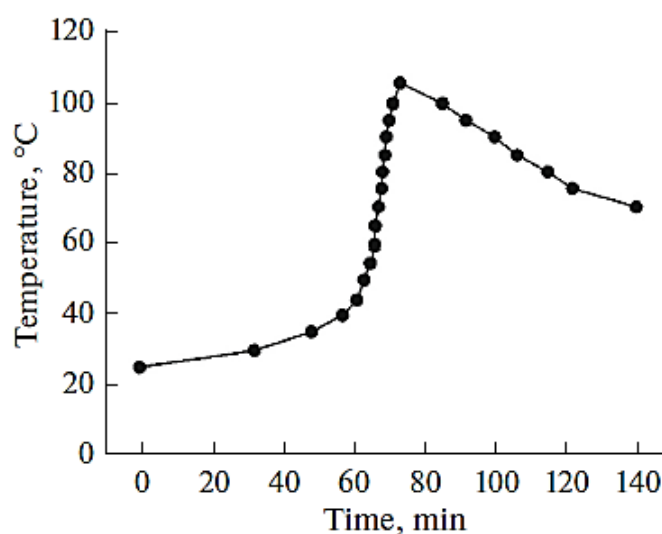


Figure 3. Curing kinetics of the foamed epoxy polymer obtained according to method 1.

In this case, the foaming process takes place in a partially cured system, as a result of which the epoxy foam is obtained with a high apparent density ($>700 \text{ kg/m}^3$).

According to the second method, when combining the components, the composition was poured into the mold, which was placed in the heating cabinet for 60 min. The composition was heated to a temperature of $50 \pm 5 \text{ }^\circ\text{C}$. Then, the mold was removed from the heating cabinet, and foaming and curing took place without additional heating. In this case, a predominantly closed-pore cell structure was formed, as shown in Figure 4a.

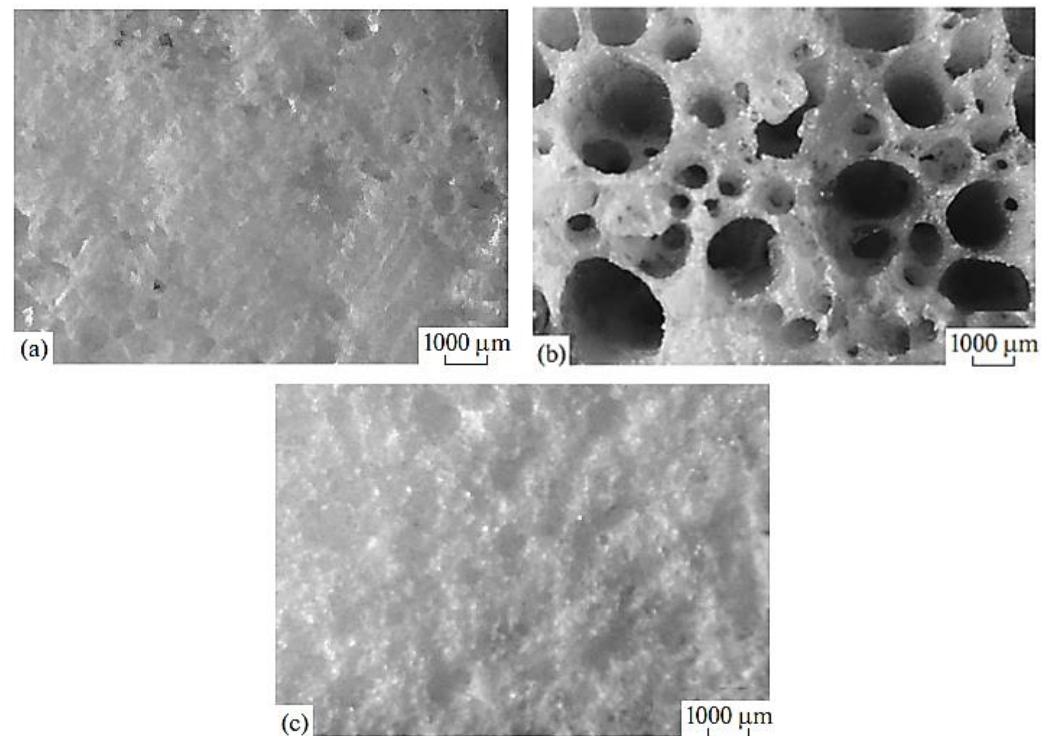


Figure 4. Structure of cells of foamed epoxy polymers obtained using methods (a) 2, (b) 3, and (c) 4.

During the curing process of a resin, the increase in viscosity—caused by the curing reaction—happens at a faster pace than the breakdown of the blowing agent. If the decomposition of the blowing agent is slower than the increase in viscosity, the gas molecules are less likely to escape or merge into larger bubbles before the resin fully cures, which ensures the production of a predominantly closed cell structure [26].

According to the third method, the composition (epoxy oligomer and blowing agent) was poured into the mold and heated in the heating cabinet to a temperature of $90 \pm 5 \text{ }^\circ\text{C}$. In this case, the blowing agent decomposed, and foaming occurred. On reaching a temperature of $90 \text{ }^\circ\text{C}$, the mold was removed from the heating cabinet, the hardener was added, and subsequent curing occurred without additional heating of the composition. In this case, a predominantly open-pore cell structure was formed, as shown in Figure 4b, since gases, being removed from the cell walls during curing, destroyed them. In an open-pore foam, the pores are larger and the cell walls are thinner, providing pathways for air and moisture. This structure allows for greater flexibility, although the density and strength are lower [27,28].

The fourth method consisted of combining all components of the composition, pouring them into the mold, and placing them in the heating cabinet heated to a temperature of $100 \pm 5 \text{ }^\circ\text{C}$ for 60 min. The foaming and curing processes occurred simultaneously. In this case, a predominantly closed-cell structure of cells was formed, as shown in Figure 4c. Like using the second molding method, heating occurred at such a rate that an increase

in the viscosity of the curing resin somewhat exceeded the decomposition of the blowing agent, which ensured the production of a predominantly closed cell structure. This means that the individual cells within the polymer matrix are encapsulated, creating discrete compartments that do not interconnect with each other. The cells are spherical or elliptical in shape and are evenly distributed throughout the polymer. The closed pores provide good thermal and acoustic insulation properties, as well as enhanced moisture resistance [29–31].

The analysis of the apparent density of the obtained compositions showed (Table 1) that depending on the method of carrying out the process, foamed polymers with different apparent densities are obtained—from light ($<200 \text{ kg/m}^3$) to heavy ($>700 \text{ kg/m}^3$).

Table 1. Dependence of apparent density on the molding conditions of epoxy foam.

Methods for Obtaining the Foam Epoxy Polymer	Apparent Density, kg/m^3	Coefficient of Variation, %
1	777	2.3
2	539	3.5
3	287	5.5
4	170	1.2

The conducted studies have shown that obtaining foamed epoxy composites using method 4 ensures not only obtaining more gas-filled composites but also more uniform foaming; the coefficient of variation in determining the apparent density was only 1.2%, which is a very good indicator of the uniformity and stability of the foaming process, as shown in Table 1.

Due to the simplicity of combining components, the ability to obtain foamed polymers with uniform properties, as shown in Table 1, and the ability to carry out foaming and curing processes simultaneously, method 4 was chosen for further research.

Since the amount of the blowing agent also affects the structure formation, its content in the composition was changed from 1 to 5 parts by mass, as shown in Table 2. The processes of the gas formation with an increase in the AC content proceed more intensively, the content of the polymer phase decreases, and the apparent density decreases from 255 to 61 kg/m^3 . Adjusting the content of the blowing agent in the foamed epoxy composite allows for precise control over various characteristics of the material, including its apparent density and porosity. As the blowing agent content increases, the formation of pores within the composite material rises, leading to enhanced water and moisture absorption, as seen in Table 2. This relationship is critical for optimizing the performance of the composite for specific applications, where properties such as weight, thermal insulation, mechanical strength, and moisture resistance are essential [32,33].

Table 2. Characteristics of foamed epoxy polymers.

Compositions, Parts by Mass, and Molding According to Method 4	γ , kg/m^3	φ_p	φ_g	WA_v , %	W_s , cm^3/m^2	B, %	σ_{com} , MPa
100 ED-20 + 1 AC	255	0.21	0.79	2.1	62	0.5	2.0 ± 0.100
100 ED-20 + 2 AC	170	0.14	0.86	4.9	137	0.7	1.0 ± 0.050
100 ED-20 + 3 AC	100	0.08	0.92	5.0	156	0.80	0.8 ± 0.040
100 ED-20 + 5 AC	61	0.05	0.95	5.2	188	0.84	0.6 ± 0.030

Note: γ —apparent density; φ_p —polymer phase content; φ_g —gas phase content; WA_v —water absorption relative to the initial volume; W_s —water absorption relative to the initial total surface; B—moisture absorption; σ_{com} —10% compression stress.

A clear correlation was noted between the apparent density and compressive stress: an increase in the apparent density results in an increase in the material resistance to compression, as shown in Table 2.

In further studies, brick dust (BD), being a waste product of the production of ceramic bricks and being formed on filters during this production, was used as a filler for foamed epoxy composites.

The results of X-ray fluorescence analysis showed that the BD mainly consists of oxides of silicon, aluminum, iron, magnesium, sodium, potassium and titanium, calcium carbonate, Figure 5.

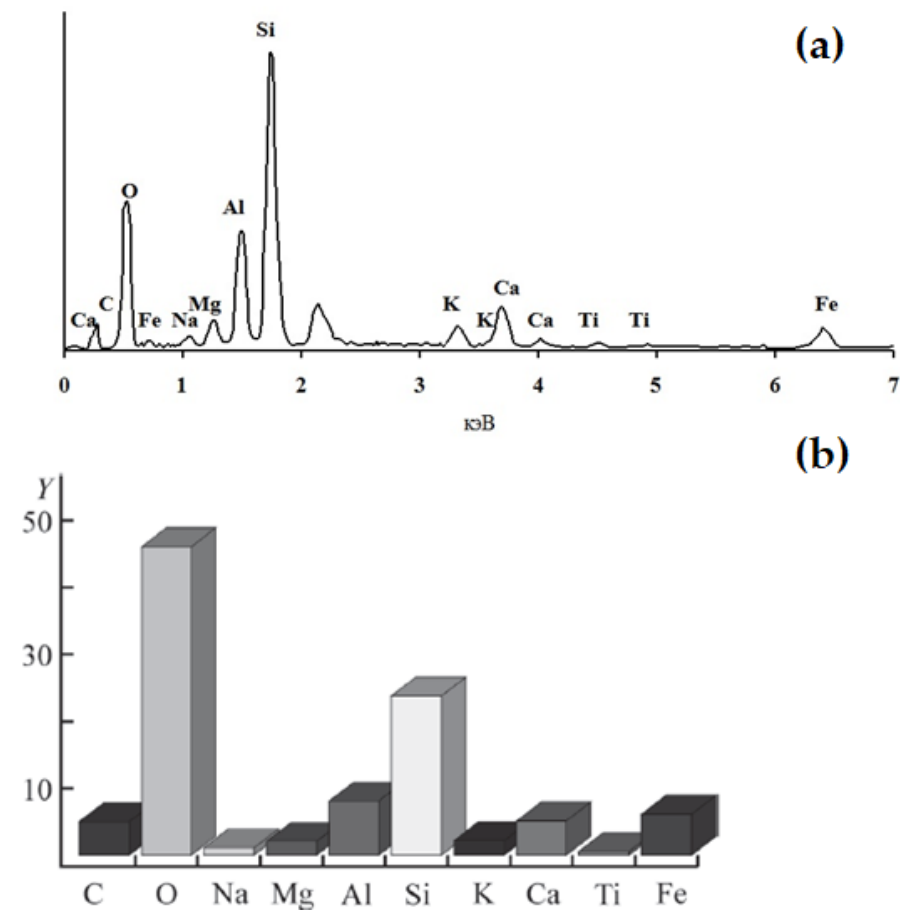


Figure 5. X-ray fluorescence analysis data: (a)—X-ray fluorescence spectrum of brick dust; (b)—quantitative results of X-ray fluorescence analysis of brick dust; Y—mass fraction of components (%).

The fractional composition of brick dust (BD) demonstrates a monomodal distribution of particles in the size range from 0.1 to 50 μm . The average particle size fluctuates within 3–6 μm , which is clearly visualized in Figure 6.

Additional SEM data prove this information. Figure 7 shows that BD particles have a lamellar structure and irregular shape, which can significantly affect their behavior and interaction in various applications.

The optimal content of brick dust (BD) in the epoxy composition, which has the best effect on the properties of epoxy composites, was selected by us earlier [34], and it is 50 parts by mass.

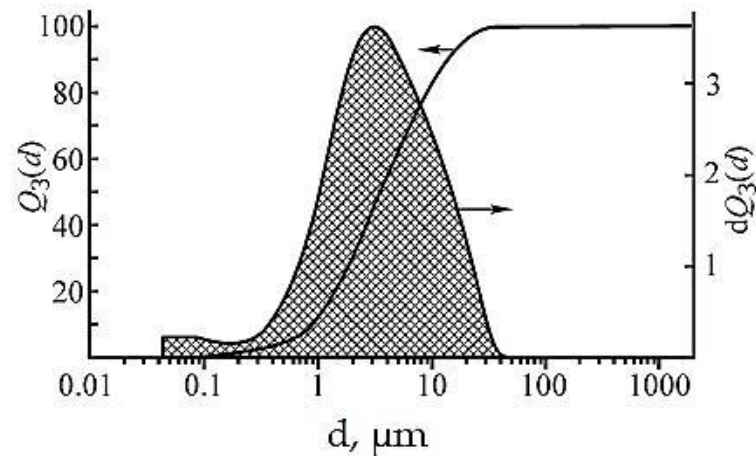


Figure 6. Fractional composition of brick dust.

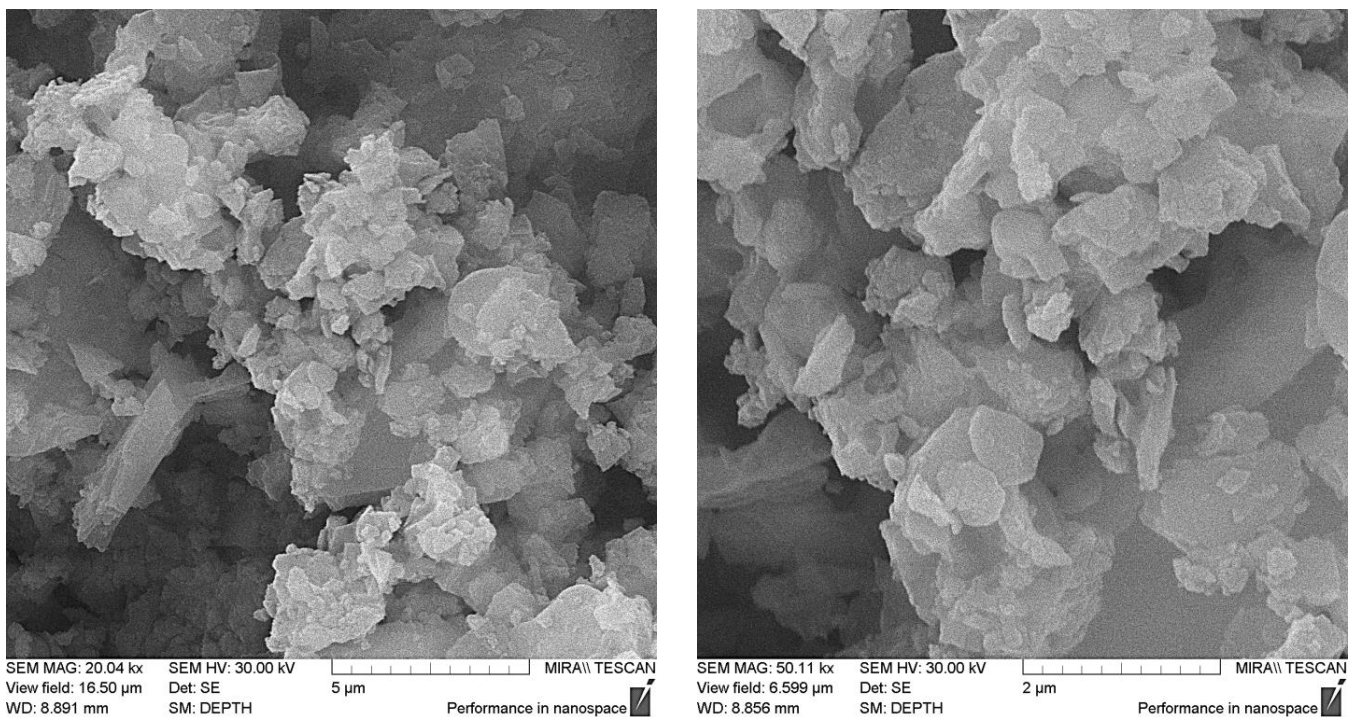


Figure 7. SEM data of BD.

The addition of brick dust (BD) into the epoxy composition ensures the production of foamed epoxy composites with increased resistance to compression. The values of 10% compression strength for foamed epoxy composites when adding 50 parts by the mass of BD increase from 1.0, 0.8, and 0.6 MPa to 1.8, 1.4, and 1.1 MPa with the foaming agent content of 2, 3, and 5 parts by mass, respectively. Thus, it has been established that the addition of technogenic waste from the production of ceramic bricks into the epoxy composition provides an increase in the compressive strength of composites by 80–85%, while the apparent density increases insignificantly, as shown in Table 3. Research findings [35,36] indicate that at high filler concentrations (50 parts by weight), the enhancement in strength can be attributed to the interaction between adjacent particles of filler within the epoxy composite. This interaction results in the formation of linkages between individual macromolecular chains and the filler particles. This connectivity forms bridges among the chains of individual macromolecules situated between the filler particles, thereby restricting the range of possible conformations and the mobility of the macromolecules' segments.

This phenomenon ultimately leads to an increase in both the elastic modulus and the overall strength of the composite material.

Table 3. Properties of foamed epoxy polymers.

Compositions, Parts by Mass, and Molding According to Method 4	γ , kg/m ³	φ_p	φ_g	WA _v , %	W _s , cm ³ /m ²	B, %	σ_{com} , MPa
100 ED-20 + 2 AC	170	0.14	0.86	4.9	137	0.70	1.0 ± 0.050
100 ED-20 + 3 AC	100	0.08	0.92	5.0	156	0.80	0.8 ± 0.040
100 ED-20 + 5 AC	61	0.05	0.95	5.2	188	0.84	0.6 ± 0.030
100 ED-20 + 2 AC + 50 Brick Dust	195	0.16	0.84	2.6	138	0.51	1.8 ± 0.080
100 ED-20 + 3 AC + 50 Brick Dust	162	0.13	0.87	8.4	402	1.47	1.4 ± 0.070
100 ED-20 + 5 AC + 50 Brick Dust	120	0.10	0.90	9.9	459	2.22	1.1 ± 0.060
100 ED-20 + 40 TCP + 3 AC + 50 Brick Dust + 5 DBDPO	132	0.11	0.89	7.1	385	2.56	1.2 ± 0.065
100 ED-20 + 40 TCP + 3 AC + 50 Brick Dust + 10 DBDPO	118	0.09	0.91	6.3	338	3.25	1.0 ± 0.052
100 ED-20 + 40 TCP + 3 AC + 50 Brick Dust + 15 DBDPO	100	0.08	0.92	5.1	315	3.61	0.9 ± 0.035

Note: γ —apparent density; φ_p —polymer phase content; φ_g —gas phase content; WA_v—water absorption relative to the initial volume; W_s—water absorption relative to the initial total surface; B—moisture absorption; σ_{com} —10% compression stress.

Moreover, it has been established that the addition of BD into the epoxy composition leads to an increase in the water and moisture absorption index of foamed epoxy composites, which is apparently due to the fact that BD itself is hydrophilic and absorbs moisture well.

The addition of the foaming agent (ammonium carbonate) into the epoxy composition slightly increases the heat resistance of the epoxy composite. The onset temperature of destruction T_{5%} increases from 256 to 260 °C, and the thermal stability of the composites also slightly increases, as shown in Figure 8, while an increase in the temperature is noted at T_{10%}–T_{70%}, where T_{5%}, T_{10%}, and T_{70%} are the temperatures at mass losses of 5, 10, and 70%, respectively, as shown in Table 4.

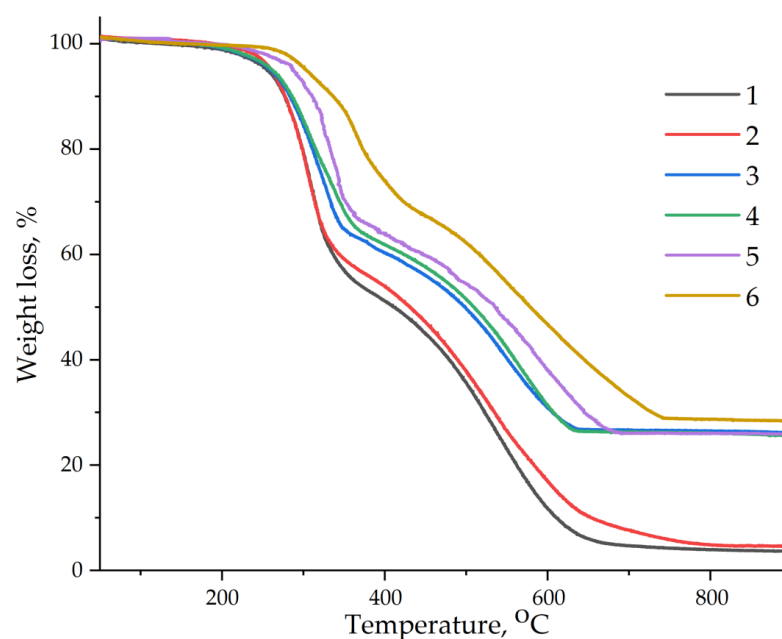


Figure 8. Data of thermogravimetric analysis of samples: 1—100 ED-20 + 15 PEPA; 2—100 ED-20 + 3 AC + 15PEPA; 3—100 ED-20 + 3 AC + 50 brick dust + 15 PEPA; 4—100 ED-20 + 3 AC + 50 brick dust + 40 TCP + 5 DBDPO + 15 PEPA; 5—100 ED-20 + 3 AC + 50 brick dust + 40 TCP + 10 DBDPO + 15 PEPA; 6—100 ED-20 + 3 AC + 50 brick dust + 40 TCP + 15 DBDPO + 15 PEPA.

Table 4. Results of thermogravimetric analysis of foamed epoxy polymers.

Samples	T _{5%} , °C	T _{10%} , °C	T _{20%} , °C	T _{30%} , °C	T _{40%} , °C	T _{50%} , °C	T _{60%} , °C	T _{70%} , °C	Residues at 800 °C, wt%
100 ED-20 + 15 PEPA	256	277	298	314	337	412	480	522	3.94
100 ED-20 + 3 AC + 15PEPA	260	280	300	315	345	432	490	534	4.88
100 ED-20 + 3 AC + 50 Brick Dust + 15 PEPA	262	285	312	334	410	500	552	608	26.50
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 5 DBDPO + 15 PEPA	270	288	316	346	430	510	563	611	26.08
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 10 DBDPO + 15 PEPA	290	311	334	354	452	537	591	648	25.96
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 15 DBDPO + 15 PEPA	306	339	374	428	520	582	646	730	28.69

The addition of brick dust (BD) into the epoxy composition increases heat resistance as well, with the initial destruction temperature T_{5%} increasing to 266 °C. T_{10%}–T_{80%} also increases, which proves an increase in the heat stability of filled epoxy foams, as shown in Table 4.

However, the obtained compositions support combustion in air, as shown in Table 5; therefore, compounds that reduce the flammability of the epoxy polymer were added to the composition of TCP and DBDPO [37,38].

Table 5. The concrete results of LOI and UL-94 tests of the foamed epoxy composites.

Samples	LOI, %	UL-94			
		t ₁	t ₂	Dripping	Rating
100 ED-20 + 15 PEPA	19	>30	-	Yes	No
100 ED-20 + 3 AC + 15PEPA	21	>30	-	Yes	No
100 ED-20 + 3 AC + 50 Brick Dust + 15 PEPA	25	>30	-	No	No
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 5 DBDPO + 15 PEPA	35	3.1	1.5	No	V-0
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 10 DBDPO + 15 PEPA	41	2.8	2.5	No	V-0
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 15 DBDPO + 15 PEPA	44	0.5	0.3	No	V-0

Note: t₁ and t₂ represent an average self-extinguishing time after the first and second ignition, and both are based on the results of five samples.

The selection of DBDPO is explained by the presence of a combustion inhibitor in its composition, bromine, which acts in the gas phase during combustion, diluting flammable gases without reaching their maximum concentration, which ensures self-extinguishing of the sample.

The analysis of the apparent density shows that DBDPO is most likely to act partially as a blowing agent since when it is added, the apparent density decreases from 162 to 100–132 kg/m³, depending on the content (5, 10, 15 parts by mass) of DBDPO in the epoxy composition, as shown in Table 3.

The addition of TCP and DBDPO into the epoxy composition provides an increase in the thermal stability of the material, as shown in Figure 8. The initial destruction temperature increases to 270, 290, and 306 °C, with a DBDPO content of 5, 10, and 15 parts by mass, respectively. Moreover, the addition of TCP and DBDPO provides a significant increase in the thermal stability of the composites in the temperature range of 250–800 °C, as shown in Table 4. The results indicate that the carbon residue at 800 °C for composites containing only BD and BD together with the combustion retardants TCP and DBDPO increases from 3.94 wt.% to 25.9–28.69 wt.%, which is associated with an excellent catalytic ability of both BD and TCP [37] and DBDPO to charring. The increased carbon residue

enhances the thermal stability of the composites by forming a protective char layer, which limits oxygen diffusion and reduces the flammability of the foamed epoxy polymer [39–41].

The fire resistance of foamed epoxy composites was assessed using LOI and UL-94 tests; the results of which are shown in Table 5. The pristine epoxy foamed polymer showed a relatively low LOI value of 21% and did not meet the criteria for the UL-94 rating, and once ignited, it burned vigorously with heavy dripping, which is considered a significant fire hazard and indicates its high flammability. With the addition of brick dust (BD), the LOI value increased to 25%, but when ignited, the sample burned for more than 30 sec and did not meet the criteria for achieving the UL-94 rating as well, while it is worth noting that there was no dripping during the combustion of the sample. The addition of fire retardants TCP and DBDPO into the epoxy composition led to an increase in the LOI value to 35–44% and successful UL-94 V-0 testing, as shown in Table 5. A significant increase in the fire resistance of foamed epoxy composites proved the effectiveness of TCP and DBDPO as excellent fire retardants.

One of the most valuable properties of foam plastics ensuring their wide application in modern technology is low thermal conductivity. The thermal conductivity of foam plastics depends on the thermal conductivity of the polymer matrix and gas, the method of production, and convection. According to the thermal conductivity, materials and products are divided into the following classes: A, low thermal conductivity (<0.06 W/mK); B, medium thermal conductivity (0.06 – 0.10 W/mK); and C, high thermal conductivity (>0.10 W/mK).

Since the developed foamed epoxy composites are intended to be used as thermal insulation, their thermal conductivity and thermal resistance were determined, and the results of these studies are presented in Table 6.

Table 6. Thermal conductivity of foamed epoxy composites.

Samples	Thermal Conductivity Coefficient, W/m K	Thermal Resistance, m ² K/W
100 ED-20 + 3 AC + 50 Brick Dust + 15 PEPA	0.042 ± 0.0021	0.364 ± 0.015
100 ED-20 + 5 AC + 50 Brick Dust + 15 PEPA	0.040 ± 0.0020	0.392 ± 0.016
100 ED-20 + 3 AC + 50 Brick Dust + 40 TCP + 15 DBDPO + 15 PEPA	0.030 ± 0.0015	0.430 ± 0.022

The conducted studies have shown that the developed foamed epoxy composites have low values of the thermal conductivity coefficient (0.030 – 0.042 W/m K), which makes them classified as class A materials (low thermal conductivity).

The foamed epoxy composites we have obtained are slightly heavier than the foamed PU composites, but the epoxy composites are characterized by greater strength and environmental friendliness. In addition, we have solved a very important problem of reducing the flammability of foamed epoxy composites. A comparison of the developed foamed composites with analogs, as shown in Table 7, showed their competitiveness since they have comparable or superior thermal insulation and strength characteristics but surpass most analogs in fire resistance.

Table 7. Comparison of developed foamed epoxy composites with analogs.

Composition	Compressive Strength, MPa	LOI, %	UL-94 Rating	Thermal Conductivity Coefficient, W/m·K
100 ED-20 + 3 AC + 50 brick dust + 40 TCP + 15 DBDPO + 15 PEPA	0.90	44	V-0	0.030
Analogues				
Bamboo-fiber-modified PU [42]	0.63	-	-	0.065
Sawdust-filled PU [43]	0.19	-	-	0.027
RPUF/PVA-ATP ₇ [44]	0.34	36.5	V-0	0.020
RPUF/12%PDEP [45]	0.27	22.9	NR	0.027
Polysilazane (7.50 wt%) epoxy [46]	1.25	NR	NR	0.059
AgNS (20.0 wt%)-modified epoxy [47]	0.38	NR	NR	0.058
TDKPF-modified phenolic [48]	0.24	41.7	V-0	0.036
PSNCFR-modified phenolic [49]	0.33	38.6	V-0	-
Melamine-urea-formaldehyde/nano-Al ₂ O ₃ [50]	0.88	31	V-0	0.067

4. Conclusions

Various methods of adding a blowing agent have been studied, and an efficient method of combining components in obtaining foamed epoxy polymers has been selected. As a result of the studies, the relationship between the structure of foamed polymers and the methods and parameters of foaming has been proven. The effect of the amount of the blowing agent on the structure and properties of foamed polymers has been established. The structure and chemical composition of technogenic waste from ceramic brick production were studied. This study demonstrated that incorporating brick dust and flame retardants (TCP and DBDPO) into foamed epoxy composites significantly improves their compressive strength, fire resistance, and thermal insulation properties. These findings support the use of industrial waste as a sustainable filler for developing efficient, fire-safe insulation materials. Future research could focus on scaling production and testing long-term performance in practical applications.

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