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FLAME RETARDANT MULTILAYER MICROWAVE ABSORBERS BASED ON POWDERED ACTIVATED CHARCOAL

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Abstract

The article presents the technology for obtaining flame retardant microwave absorbers based on powdered charcoal. These absorbers include three layers. The outer layer of these absorbers is formed based on a mixture of powdered aluminum oxide or titanium dioxide and a flame retardant paint, the intermediate layer is based on a mixture of powdered activated charcoal impregnated with a calcium or magnesium chloride aqueous solution, and a gypsum aqueous solution, and the inner layer is based on aluminum-containing foiled polymer film. The results of an experimental test of the presented technology are provided. They include the results of a study of the process of interaction of an open flame with absorbers manufactured in accordance with the presented technology, the results of a study of the process of interaction of IR radiation with such absorbers, as well as the results of a study of the electromagnetic radiation absorption characteristics in the frequency range 2.0–17.0 GHz of such absorbers. Using the first of the indicated results, it was confirmed that absorbers manufactured in accordance with the presented technology are flame retardant. Using the second of the indicated results, it was confirmed that absorbers manufactured in accordance with the presented technology provide an effective reduction in the energy of IR radiation. Using the third of the indicated results, patterns of changes in the absorption characteristics of electromagnetic radiation in the frequency range 2.0–17.0 GHz of absorbers manufactured in accordance with the presented technology were established, depending on the composition of their outer and intermediate layers.

Keywords: *aluminum oxide, titanium dioxide, microwave absorber, flame retardant.*

Introduction

A number of studies currently being carried out in the field of materials science (in particular, in the field of multifunctional materials) are devoted to the development of flame retardant microwave absorbers. These studies are characterized by practical significance, since microwave absorbers are often used in rooms where increased fire safety requirements are imposed (EMC test chambers, data centers, shielded rooms). It should be noted that to date, about a dozen scientific papers, which present the results of the development and research of flame retardant microwave absorbers have been published [1–8]. The first of these papers dates back to 2021 [1]. Taking into account the indicated features, it should be noted that research devoted to the development of flame retardant microwave absorbers is currently just beginning to develop. It seems promising to direct this research to improve the manufacturability of the technologies for obtaining flame retardant microwave absorbers.

This paper presents the results of the work, the strategic aim of which was the development of research devoted to the flame retardant microwave absorbers. The tactical aim of the work was to substantiate a new technology for obtaining flame retardant microwave absorbers, which, compared to analogues, is characterized by higher manufacturability.

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These results include the following.

1. Description of the new technology developed by the authors for the manufacture of flame retardant microwave absorbers.

2. Description of the results of a study of the process of interaction of an open flame with the absorbers samples manufactured in accordance with the developed technology.

3. Description of the results of a study of the process of interaction of IR radiation with the absorbers samples manufactured in accordance with the developed technology.

4. Description of the results of a study of electromagnetic radiation absorption characteristics in the frequency range 2.0–17.0 GHz of the absorbers samples manufactured in accordance with the developed technology.

Materials and Methods

When developing the technology for obtaining flame retardant microwave absorbers, the results of research presented in [9–11] were taken into account. These papers are devoted to the experimental substantiation of the flame-retardant properties of powdered aluminum oxide and titanium dioxide. In addition, the results of studies presented in the monograph [12] were taken into account. This monograph is devoted to experimental substantiation of the prospects of using powdered activated charcoal for the manufacture of microwave absorbers, characterized by reduced cost compared to microwave absorbers based on graphite, graphene, carbon nanotubes or fibers.

The developed technology includes the following operations.

1. Formation of the outer layer of the microwave absorber.

2.1. Cutting out a fragment from a roll of kraft paper, the overall dimensions and shape of which are determined by the requirements for the overall dimensions and shape of the microwave absorber being manufactured.

2.2. Preparation of a mixture of powdered aluminum oxide or titanium dioxide (30.0 vol. %) and a water-based flame retardant paint (the rest).

2.3. Uniform mechanical application of a layer 3.0±1.0 mm thick of the mixture prepared as a result of operation 2.2 onto a piece of kraft paper opened as a result of operation 2.1.

2.4. Atmospheric drying of the mixture applied as a result of operation 2.3.

2. Formation of the intermediate layer of the microwave absorber.

2.1. Cutting out two identical fragments from a roll of self-adhesive polymer film, the overall dimensions and shape of which are determined by the requirements for the overall dimensions and shape of the microwave absorber being manufactured.

2.2. Preparation of powdered activated charcoal impregnated with a magnesium or calcium chloride aqueous solution.

2.2.1. Preparation of a magnesium or calcium chloride aqueous solution (concentration – 35.0±1.0 wt. %).

2.2.2. Immersion of powdered activated charcoal particles into an empty container.

2.2.3. Pouring the prepared magnesium or calcium chloride aqueous solution into a container in which particles of powdered activated charcoal are immersed.

2.2.4. Mixing a magnesium or calcium chloride aqueous solution and particles of powdered activated charcoal.

2.2.5. Separation from the mixture obtained as a result of operation 1.2.4 of particles of powdered activated charcoal impregnated with a magnesium or calcium chloride aqueous solution.

2.3. Mixing powdered activated charcoal, impregnated with a magnesium or calcium chloride aqueous solution and obtained as a result of operation 2.2, with gypsum and water in a volumetric ratio of 2:3:10.

2.4. Uniform mechanical application of a layer 5.0±1.0 mm thick of the mixture obtained as a result of operation 2.3 onto the surface of the adhesive layer of one of the fragments of self-adhesive polymer film, opened as a result of operation 2.1, so that a field remains along the perimeter of this fragment 10.0±1.0 mm wide, free from this mixture.

2.5. Atmospheric drying of the mixture applied as a result of operation 2.4.

2.6. Placing the second of the fragments of self-adhesive polymer film, opened as a result of operation 2.1, with the adhesive side on top of the layer of the mixture dried as a result of operation 2.5.

3. Fixing the outer layer of the microwave absorber, formed as a result of operation 1, on one of the surfaces of the intermediate layer of the microwave absorber, formed as a result of operation 2, using spray adhesive.

4. Formation of the inner layer of the microwave absorber by cutting off a fragment from a roll of aluminum-containing foiled polymer film, the overall dimensions and shape of which are determined by the requirements for the overall dimensions and shape of the microwave absorber being manufactured.

5. Fixing, using spray adhesive, the opened fragment of aluminum-containing foiled polymer film on the surface of the formed intermediate layer of the microwave absorber on which the outer layer is not fixed.

In accordance with the developed technology, 4 types of microwave absorber samples were manufactured. A sample of each species differed in the composition of the outer and / or intermediate layer. The characteristics of these samples are presented in Table 1. The thickness of the sample of each type was 8.0±1.0 mm.

Table 1. Characteristics of samples of each type

Sample name	Components included in the outer layer of the sample	Components included in the intermediate layer of the sample
Sample of type 1	Aluminum oxide, water-based flame retardant paint	Magnesium chloride aqueous solution, powdered activated charcoal, gypsum, water
Sample of type 2		Calcium chloride aqueous solution, powdered activated charcoal, gypsum, water
Sample of type 3	Titanium dioxide, water-based flame retardant paint	Magnesium chloride aqueous solution, powdered activated charcoal, gypsum, water
Sample of type 4		Calcium chloride aqueous solution, powdered activated charcoal, gypsum, water

The study of the interaction of an open flame with microwave absorbers samples manufactured in accordance with the developed technology was carried out in the following order.

1. Wrap the test sample with a sheet of foil.

2. Making a round hole with a diameter of 140.0 mm in a sheet of foil with which the test sample is wrapped, so that the center of this hole coincides with the center of one of the surfaces of this sample.

3. Fixing the gas burner on a movable platform located on the frame.

4. Fixing the test sample on a holder located on the same frame as the movable platform on which the gas burner is mounted, so that the hole in the foil sheet with which the test sample is wrapped is located opposite the gas burner nozzle.

5. Adjusting the location of the gas burner, taking into account that the distance from its nozzle to the surface of the test sample was 15.0 mm.

6. Turn on the burner and keep it on for 15.0 minutes or until the test sample ignites.

7. If necessary, registration of the moment in time at which the test sample ignited.

The study of the interaction of IR radiation with microwave absorbers samples manufactured in accordance with the developed technology was carried out in the following order.

1. Fixing the test sample on the holder.
2. The location of an IR radiation source based on halogen lamps opposite one of the surfaces of the test sample at a distance of 50.0 mm (the surface temperature of the specified source was 70.0±2.0 °C).
3. The location of the MobIR M4 thermal imaging camera at a distance of 50.0 mm from the other surface of the test sample (to the surface opposite which the halogen lamp-based IR radiation source is placed).
4. Turn on the IR radiation source based on halogen lamps.
5. Registration of television images of the surface of the screen sample under study for 60.0 minutes at certain points in time (Table 2).
6. Analyze each of the 23 recorded thermal images using the special Guide IrAnalyser software supplied with the MobIR M4 thermal imaging camera. The results of this analysis are the average surface temperature of the sample at each time point at which its television images were recorded, as well as its thermal profiles.
7. Plotting a graph of the dynamics of changes in the average surface temperature of the sample based on the analysis results obtained in step 6.

Table 2. Procedure for recording thermal imaging images of sample surfaces during research

Thermal image number	Time point from the start of research, min
1	0.00
2	0.21
3	0.47
4	0.78
5	1.15
6	1.61
7	2.16
8	2.83
9	3.64
10	4.62
11	5.81
12	7.25
13	9.0
14	11.12
15	13.68
16	16.78
17	20.54
18	25.10
19	30.62
20	37.31
21	45.41
22	55.23
23	60.00

Electromagnetic radiation absorption characteristics in the frequency range 2.0–17.0 GHz of each of the microwave absorber samples manufactured in accordance with the developed technology were obtained in accordance with the following procedure.

1. Measurement of the reflection coefficient of electromagnetic radiation in the frequency range 2.0–17.0 GHz of the sample using a panoramic reflection and transmission coefficient meter SNA 0.01–18 and one horn antenna P6-23M.
2. Measurement of electromagnetic radiation reflection coefficient in the frequency range 2.0–17.0 GHz of the sample using a panoramic reflection and transmission coefficient meter SNA 0.01–18 and two horn antennas P6-23M.

3. Convert each of the measured electromagnetic radiation reflection coefficient in the frequency range 2.0–17.0 GHz of the sample from decibels to relative units using the following formula:

$$R(f) = 10^{\frac{S_{11}(f)}{10}}, \text{ rel. units,} \quad (1)$$

where $R(f)$ – electromagnetic radiation reflection coefficient value of the sample at a certain frequency in the range 2.0–17.0 GHz, expressed in relative units, $S_{11}(f)$ – measured electromagnetic radiation reflection coefficient value of the sample at a certain frequency in the range 2.0–17.0 GHz, expressed in decibels.

4. Convert each of the measured electromagnetic radiation transmission coefficient in the frequency range 2.0–17.0 GHz of the sample from decibels to relative units using the following formula:

$$T(f) = 10^{\frac{S_{21}(f)}{10}}, \text{ rel. units,} \quad (2)$$

where $T(f)$ – electromagnetic radiation transmission coefficient value of the sample at a certain frequency in the range 2.0–17.0 GHz, expressed in relative units, $S_{21}(f)$ – measured electromagnetic radiation transmission coefficient value of the sample at a certain frequency in the range 2.0–17.0 GHz, expressed in decibels.

5. Calculation of the electromagnetic radiation absorption coefficient values in the frequency range 2.0–17.0 GHz ($A(f)$) of the sample using the following formula:

$$A(f) = 1 - R(f) - T(f), \text{ rel. units.} \quad (3)$$

The panoramic reflection and transmission coefficient meter SNA 0.01–18, used to measure electromagnetic radiation reflection and transmission coefficients in the frequency range 2.0–17.0 GHz of the samples, includes the following devices:

– swing frequency generator;
– measuring signal processing unit;
– blocks of directional couplers.

Results and Their Discussion

Based on the results of a study of the interaction of an open flame with manufactured microwave absorbers samples, it was established that such absorbers are flame retardant (Fig. 1).

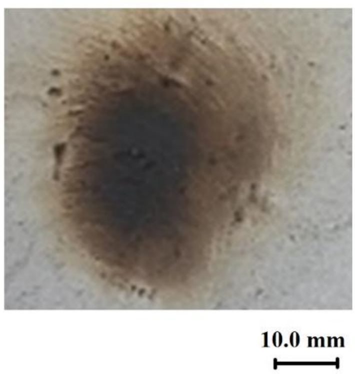

Fig. 1. Photograph of a section of the surface of the sample outer layer after 15 minutes of exposure to a gas burner flame

Based on the results of a study of the process of interaction of IR radiation with microwave absorbers samples manufactured in accordance with the developed technology, it was found that the average temperature (T_{av}) of the reverse surfaces of these samples increases from 22.0±2.0 °C to 28.0±2.0 °C, provided that their front surfaces are exposed to IR radiation, the source surface temperature of which is 70.0±2.0 °C (provided that the air temperature in the room is 21.0±1.0 °C) – Fig. 2.

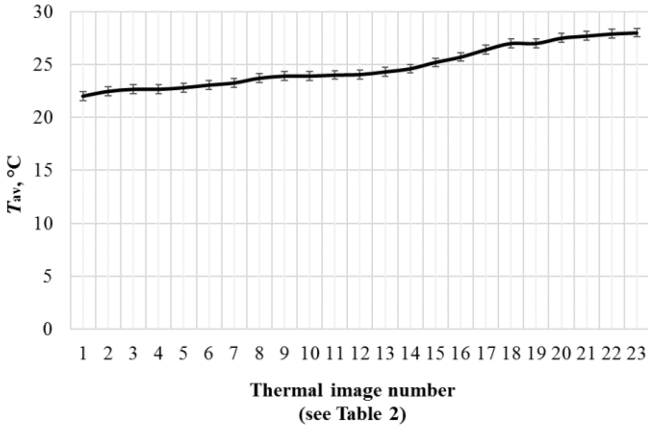

Fig. 2. Change over time of the average temperature of the reverse surfaces of microwave absorbers samples manufactured in accordance with the presented technology

Frequency dependences of electromagnetic radiation absorption coefficient in the range of 2.0–17.0 GHz of the microwave absorbers samples manufactured in accordance with the presented technology are presented in Fig. 3, 4.

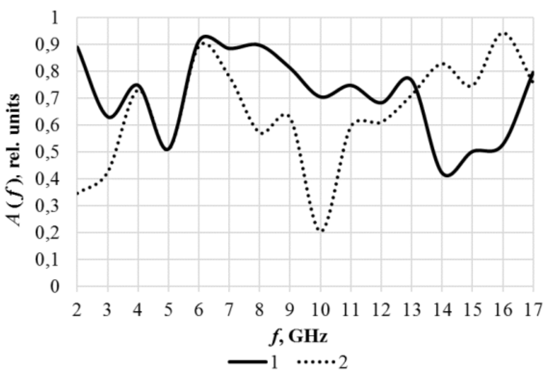

Fig. 3. Frequency dependences of electromagnetic radiation absorption coefficient in the range of 2.0–17.0 GHz of the samples of types 1 (curve 1) and 2 (curve 2)

It can be seen from Figure 3 that electromagnetic radiation absorption coefficient values in the frequency ranges 2.0–4.0 GHz and 7.0–13.0 GHz of the sample of type 1 are higher by 0.05–0.55 rel. units of the sample of type 2. Electromagnetic radiation absorption coefficient values in the frequency range 13.0–17.0 GHz of the first of these samples are lower by 0.05–0.4 rel. units than of the second one. Electromagnetic radiation absorption coefficient values in the frequency range 4.0–7.0 GHz of such samples are comparable. This is due to the following features (Fig. 5):

1) electromagnetic radiation reflection coefficient values in the frequency ranges 2.0–4.0 GHz and 7.0–13.0 GHz of the sample of type 1 are lower by 0.05–0.6 rel. units than of the sample of type 2;

2) electromagnetic radiation reflection coefficient values in the frequency range 13.0–17.0 GHz of the sample of type 1 are higher by 0.05–0.4 rel. units than of the sample of type 2;

3) electromagnetic radiation reflection coefficient values in the frequency range 4.0–7.0 GHz of the above samples are comparable.

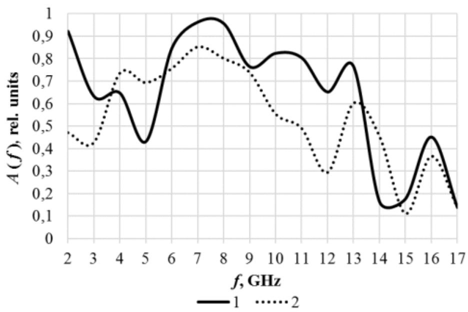

Fig. 4. Frequency dependences of electromagnetic radiation absorption coefficient in the range of 2.0–17.0 GHz of the samples of types 3 (curve 1) and 4 (curve 2)

It can be seen from Figure 4 that electromagnetic radiation absorption coefficient values in the frequency ranges 2.0–3.5 GHz, 5.7–13.0 GHz and 14.5–17.0 GHz of the sample of type 3 are higher by 0.05–0.35 rel. units than that of the sample of type 4. Electromagnetic radiation absorption coefficient values in the frequency ranges 3.5–5.7 GHz, 13.0–14.5 GHz of the first of these samples are lower by 0.01–0.28 rel. units than of the second one. This is due to the following features (Fig. 6):

1) electromagnetic radiation reflection coefficient values in the frequency ranges 2.0–3.5 GHz, 5.7–13.0 GHz and 14.5–17.0 GHz of the sample of type 3 are lower by 0.05–0.35 rel. units than of the sample of type 4;

2) electromagnetic radiation reflection coefficient values in the frequency range 13.0–17.0 GHz of the sample of type 3 are higher by 0.05–0.35 rel. units than that for sample of type 4.

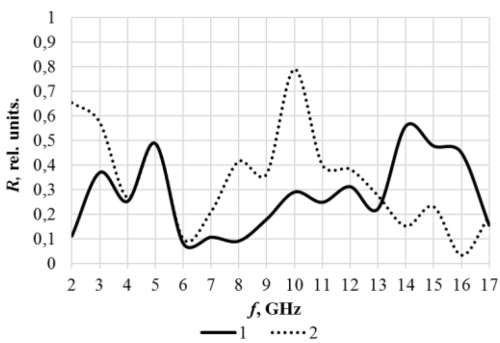

Fig. 5. Frequency dependences of electromagnetic radiation reflection coefficient in the range of 2.0–17.0 GHz of the samples of types 1 (curve 1) and 2 (curve 2)

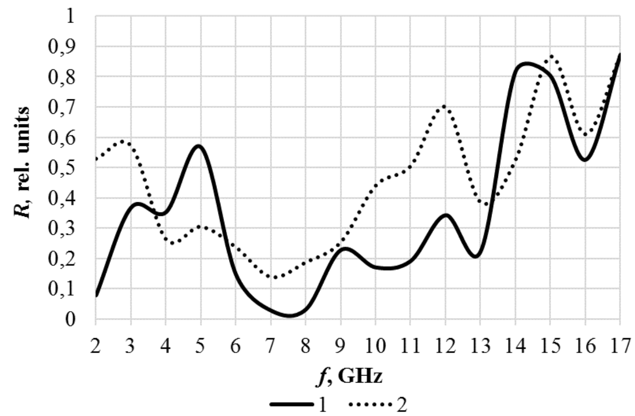

Fig. 6. Frequency dependences of electromagnetic radiation reflection coefficient in the range of 2.0–17.0 GHz of the samples of types 3 (curve 1) and 4 (curve 2)

Parameters of effective absorption band of electromagnetic radiation of the studied samples, as well as of microwave absorbers, which are their analogues (flame retardant microwave absorbers, characterized by a flat surface), are presented in Table 3. When determining the range and width of the effective absorption band of electromagnetic radiation, it was taken into account that this band corresponds to the frequency range in which the value of electromagnetic radiation absorption coefficient of the absorber is equal to or exceeds 0.5 rel. units [13].

Table 3. Parameters of effective absorption band of electromagnetic radiation of the studied samples and their analogues

Sample name	Range of effective absorption band of electromagnetic radiation, GHz	Width of effective absorption band of electromagnetic radiation, GHz
Sample of type 1	2.0–13.5	11.5
Sample of type 2	3.2–9.2 10.8–17.0	6.0 6.2
Sample of type 3	2.0–4.5 5.2–13.5	2.5 8.3
Sample of type 4	3.2–11.0 12.5–14.0	7.8 1.5
Microwave absorber with hydrophobic and flame-retardant functions based on $NiCo_2O4@MnO_2$ nanosheets array and 3D porous expanded graphite hybrids [1]	13.4–18.0	4.6
Microwave absorbers based on fire-resistant iron-based phosphates/phosphorus-doped carbon composites derived from phytic acid-treated metal organic frameworks [2]	12.24–18.0	5.76
Flexible electrothermal microwave absorbers based on Ni-carbon microtube / polytetrafluoroethylene [6]	4.0–8.0	4.0
Microwave absorber based on multifunctional foamed ceramics prepared by the sintering method [7]	8.0–18.0	10.0
Microwave absorber based on aramid nanofiber, polypyrrole and nickel porous aerogel [8]	8.0–16.42	8.42

It can be seen from Table 3 that the widest effective absorption band of electromagnetic radiation of the absorbers manufactured in accordance with the presented technology using powdered aluminum oxide and magnesium chloride aqueous solution. The width of the effective absorption band of electromagnetic radiation of such absorbers exceeds by 1.5–7.5 GHz the width of the effective absorption band of their analogues due to the fact that such absorbers are characterized by a multilayer structure.

Conclusion

It can be concluded that the multifunctionality of microwave absorbers manufactured in accordance with the presented technology has been experimentally confirmed. Such conclusion was made on the base of the following experimental results obtained in course of the conducted research.

The absorbers provide an effective reduction of the microwave radiation energy. The width of their effective absorption band reaches 11.5 GHz. Their electromagnetic radiation absorption coefficient values in the effective absorption band change from 0.5 till 0.95 rel. units.

The absorbers provide an effective reduction of the IR radiation energy. It was found that the average temperature of their reverse surfaces increases from 22.0±2.0 °C to 28.0±2.0 °C, provided that their front surfaces are exposed to IR radiation, the source surface temperature of which is 70.0±2.0 °C (provided that the air temperature in the room is 21.0±1.0 °C).

The absorbers are flame retardant ones. They are not destructed in course of interaction with the open flame.

These absorbers seem promising for use in solving problems related to ensuring the protection of electronic devices from the effects of microwave and thermal interference. It is most advisable to manufacture panels based on these absorbers for finishing the walls of rooms in which electronic devices are located.

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