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# The mechanism of copper (II) oxide particles dispersion in a solution of polymethyl methacrylate

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An effective method for producing dispersion-filled polymer composite materials with a low agglomerate content is based on *in situ* — technology-the formation of filler particles directly in the polymer matrix during composite synthesis. Of particular interest is the synthesis of dispersed particles from precursors within minireactors, where synthesis conditions differ from ambient conditions. The interaction of such minireactors with the environment can initiate the dispersion of particles synthesized *in situ*. A method is proposed for forming films of dispersion-filled polymer composites from a polymethyl methacrylate solution in toluene with the *in situ* generation of copper(II) oxide particles within minireactors. It is shown that the formation of minireactors in the polymer solution is due to the development of a polymer shell around the charge of the precursor (copper hydroxide) during its thermal decomposition. It was shown that heating these minireactors in a microwave field leads to the formation of water vapor within them, an increase in pressure, and rupture of the shell, thereby dispersing copper (II) oxide particles. The highest dispersion efficiency is achieved in a saturated solution of polymethyl methacrylate in toluene. The results of a study of polymer films obtained from solutions with varying concentrations of dispersed particles are presented.

**Keywords:** polymer solution, minireactors, *in situ* method, dispersed particles, polymer composite material.

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## Introduction

A problem of agglomeration of submicron particles significantly limits the fields of application of dispersion-filled polymer composite materials (DPCM). Agglomeration of filler particles in the DPCM results in degradation of composite characteristics: reduction of strength and a modulus of elasticity [1,2], change of target parameters of functional properties of the materials [3]. It is especially important to note reduction of reproducibility of all the DPCM characteristics [4] even when providing technological modes of production of the composite materials. The studies on agglomeration of filler particles in the polymers, for example [5–7], are analyzed to demonstrate that there are two stages of agglomeration: agglomeration of particles in a powder before they are introduced into the polymer and agglomeration of particles in the polymer during DPCM production. Using various methods of dispersion and modification of a particle surface in the powders makes it possible to just reduce an agglomerate concentration, but it is impossible to completely get rid of the agglomerates [8].

One of the effective methods of reducing a number of agglomerates in DPCM, which excludes an agglomeration process in the initial powder, is based on formation (synthesis) of dispersed particles in a polymer matrix directly during production of the composite (the *in situ* method) [9,10]. It makes it possible to reduce particle agglomeration [11] and to provide their more uniform distribution, for example, cellulose particles in a polymethyl methacrylate solution (PMMA) [12], thereby resulting in reproducibility of the DPCM characteristics. However, usually, it is impossible to fully get rid of the agglomerates using the *in situ* method [10].

In our opinion, formation of dispersed particles in polymer solutions by the *in situ* method makes it possible to use additional dispersion mechanisms, for example, by forming minireactors [13,14]. Hereinafter, similar to the study [13], the minireactors will be defined as spatially confined formations (droplets, micelles, etc.) isolated from the environment, for example, the polymer solution. Conditions of synthesis of the dispersed particles in the reactors differ from the environmental conditions, while interaction of the minireactors with the environment can initiate processes of dispersion of agglomerates of these particles [14].

Formation of the minireactors can be exemplified by a process of producing copper (II) oxide particles from copper hydroxide in the polymer solution [15]. It is known that heating is accompanied by a reaction of decomposition of copper hydroxide with formation of copper (II) oxide particles (CuO) and release of water [10]. It can be assumed [15] that implementation of this process in the polymer solution will result in formation of the polymer film on a water–solution boundary [16,17]. As a result, the polymer solution will have the minireactors formed and they will have a polymer shell and contain the copper (II) oxide particles and water inside (after a full transition of copper hydroxide into oxide). Heating of these minireactors will result in an increase of water vapor pressure and, in the end, rupture of the polymer shell, thereby providing collapse of agglomerates of the CuO particles. Even the minireactors are used, in particular, for synthesis of nanoparticles [13], issues of mechanisms of their formation in the polymer solutions for *in situ* production and dispersion of the dispersed particles still remain open.

The present study is aimed at refining the expected mechanisms of dispersion of copper (II) oxide particles from the minireactors formed in the PMMA solution in toluene and determining the PMMA concentration in the solution, at which the most effective dispersion of the particles is achieved.

## 1. Materials and methods

### 1.1. Preparation of initial components

The solution was produced by using a polymer — PMMA (TU 2216-055-55856863-2009), a solvent — toluene (the c.p. grade as per GOST 5789-78). Toluene is selected to be the solvent due to: a low value of its dielectric loss tangent $(0.01-0.03)$ as compared to water $(\sim 0.17)$, the copper oxide particles $(0.1-0.3)$ and copper hydroxide $(\sim 0.01-0.06)$ at the frequency of 2.45 GHz [18]; the toluene boiling temperature $(110.6\,^\circ\mathrm{C})$ [19] is higher than that of water. It makes it possible to purposefully heat internal volumes of the minireactors, to reduce intensity of evaporation of toluene from the solution, thereby reducing a solution viscosity increase rate during the experiment. A PMMA charge weighing $(13.0 \pm 0.5)$ g was dissolved in $(150 \pm 10)$ mL of toluene when heating to $(50 \pm 1)\,^\circ\mathrm{C}$ and mixing in a heating magnetic mixer (manufactured by Ekros, the ES-6120 model) for $(2.0 \pm 0.3)$ h. Dissolution of PMMA in toluene was followed by producing a viscous transparent solution. In order to achieve the aim of the study, different concentrations $(C)$ of the PMMA solutions were obtained by diluting the produced solution with toluene. Thus, 6 samples of the solutions with the PMMA concentrations from 2 % to 10 % with a step $(2 \pm 0.2)$ % were produced. The concentration of the saturated PMMA solution in toluene was 10 % [20] in the experiments. It should be noted that water is not dissolved in toluene and facilitates displacement of the polymer out of the respective solution [16].

Copper hydroxide $Cu(OH)_2$ was produced by a technique described in the study [10] just before the experiments. Thus, a time of interaction of copper hydroxide with carbon dioxide of atmospheric air was minimized. This interaction results in formation of copper carbonates that have a higher temperature $(290\,^\circ\mathrm{C})$ of decomposition [21,22] with formation of copper (II) oxide than copper hydroxide $(50\,^\circ\mathrm{C})$ [10]. The experiments were done using the copper hydroxide charge weighing $(2.0 \pm 0.2)$ g, which was placed in a cuvette with a specified concentration of the solution.

### 1.2. Experimental technique

During the experiments, the reaction of decomposition of copper hydroxide was initiated by heating in a microwave field using a magnetron microwave oscillator that has adjusted output power within the range 80–800 W and is designed to operate at the 2.45 GHz frequency. The output power was set at $(720 \pm 10)$ W. A resonance chamber of the microwave oscillator included a flat-bottom cuvette made of a heat-resistant polymer insoluble in toluene, with a volume $(50 \pm 5)$ mL of the solution of the specified PMMA concentration $(C)$ and the copper hydroxide charge $(2.0 \pm 0.2)$ g. The solution temperature was controlled during heating by a Bragg grating's fiber-optic sensor [23,24] located at the cuvette bottom at its external side. A temperature measurement error was $\pm 0.1\,^\circ\mathrm{C}$.

The transition of copper hydroxide into oxide was visually determined by changing of a charge color from blue (Fig. 1, *a*) to black (Fig. 1, *b–d*). During synthesis, when heating copper hydroxide, the cuvette bottom formed the minireactors, whose size was gradually decreasing (Fig. 1, *b–d*). The heating time of the samples with the different PMMA concentration in the solution was the same and it was $(600 \pm 5)$ s. Dynamics of variation of the solution temperature can be conventionally divided into three regions (Fig. 2). The regions I and II exhibit gradual reduction of the minireactor sizes (Fig. 1, *b,c*), while the region III exhibits completion of formation of a particle suspension, which is accompanied by a significant increase of its absorbance (Fig. 1, *d*). Temperature oscillations within the region II are due to surfacing of a large number of the minireactors located against the temperature sensor at the cuvette bottom. As long as the minireactor is below the temperature sensor at the cuvette bottom, sensor readings increase, while during surfacing of the minireactor the sensor readings begin to decrease. It indirectly indicates a higher temperature value inside the minireactors as compared to the suspension temperature: the suspension is mainly heated by transfer of heat from the heated minireactors. Within the region II, intensity of suspension heating by impact of the microwave field is additionally increased due to the high dielectric loss tangent of the copper (II) oxide particles.

In order to analyze sizes of the particles and their agglomerates and to produce the DPCM films from the solutions, we have used the solution produced after synthesis of the particles within the cuvette volume. Precipitate that was the undispersed minireactors was left on the cuvette bottom. During the experiments, the polymer concentration in the solution, which varies due to partial evaporation

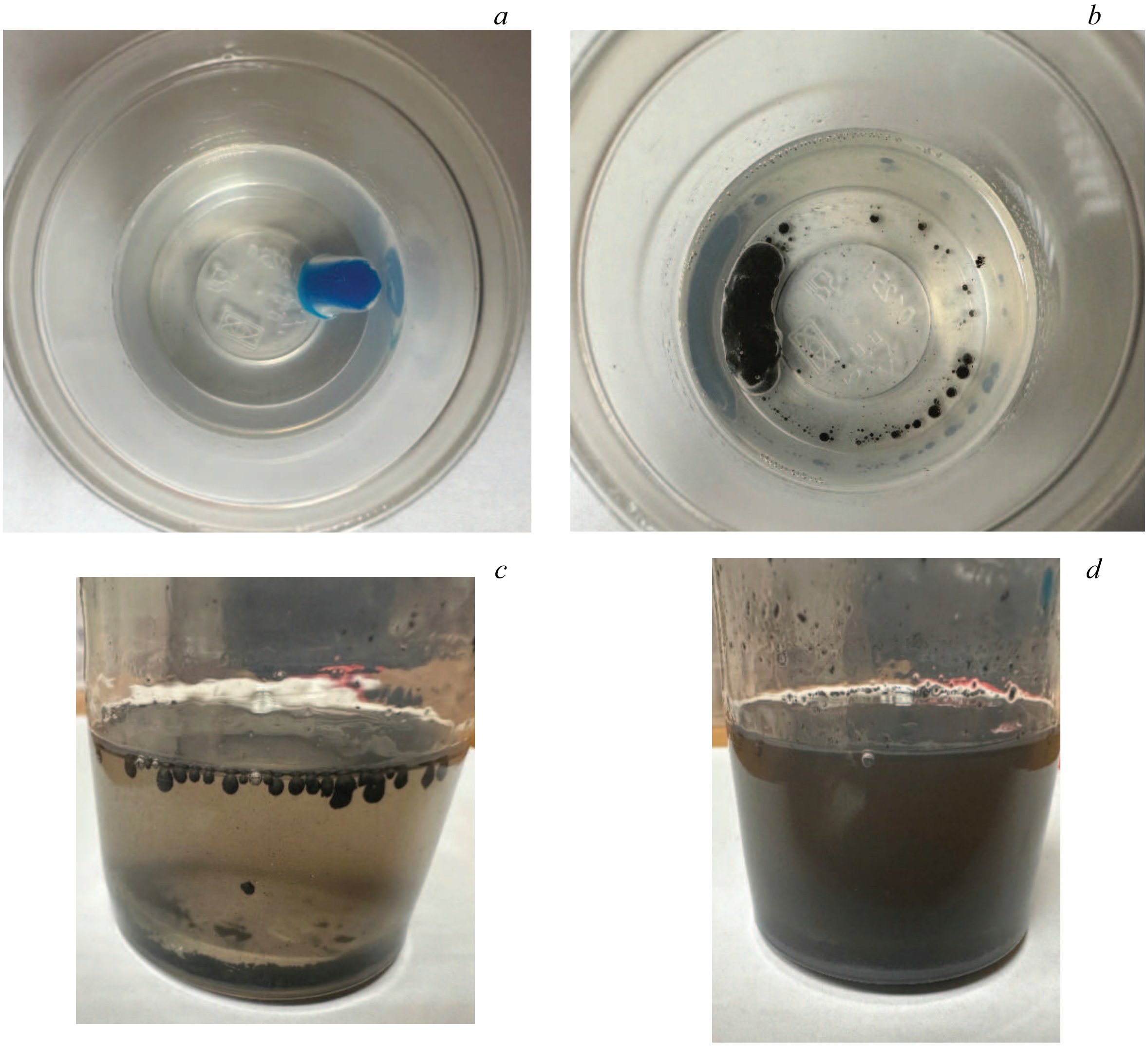


**Figure 1.** Diagram of transformation of copper hydroxide into copper (II) oxide in the minireactors (the saturated polymer solution).

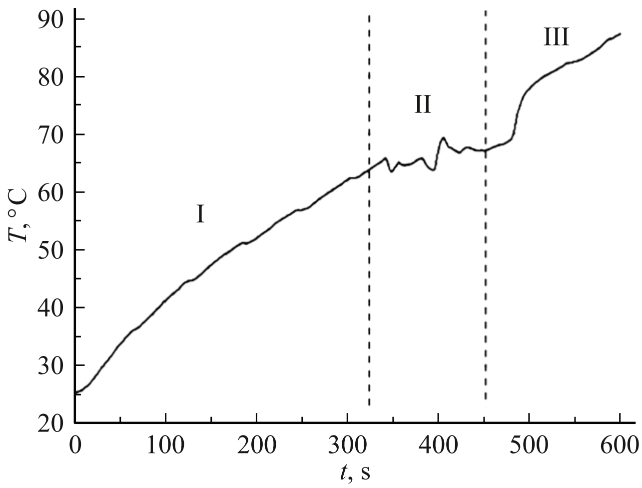


**Figure 2.** Dynamics of variation of the temperature during dispersion of the copper (II) oxide particles from the minireactors in the saturated PMMA solution on toluene.

of the solvent, was maintained. For this purpose, every $(200 \pm 10)$ s of solution heating a sample weight was controlled with accuracy of $10^{-4}$ g. If required, toluene was added up and its temperature corresponded to the solution temperature.

The polymer films were produced from the respective solutions by evaporating the solvent in a vacuum cabinet under pressure of about $(10 \pm 5)$ kPa for $(24 \pm 1)$ h.

### 1.3. Research methods

The sizes of the copper (II) oxide particles and their agglomerates were determined by Transmission Electron Microscopy (TEM) using the microscope Carl Zeiss AURIGA Cross Beam with EDI Inca X-Max 80 mm$^2$. For this purpose, $2\,\mu$L of the solution with the particles was applied to a copper mesh of the 3 mm diameter with a substrate Formvar/Carbon (TedPella). Average sizes of the dispersed particles and their agglomerates were determined using statistical processing of images of at least 500 particles and their agglomerates, respectively, in the ImageJ software by a technique proposed in the study [27].

A volume portion of the copper (II) oxide particles in the samples was determined by dielectric spectroscopy in the films produced from the solutions. Dielectric measurements were performed by means of a measurement system NovocontrolBDS-80 within the frequency range from $10^{-3}$ to $10^4$ Hz. The temperature was changed from 0 °C to 80 °C with a step of 5 °C.The temperature maintenance accuracy was $\pm 0.5$ °C.A probing oscillation amplitude was 3 V; a relative error of measurement of permittivity was 1 %.

A content of the agglomerates of the copper (II) oxide particles in the solutions was comparatively estimated based on analysis of sedimentation curves obtained on a photometer described in the studies [25,26]. The same conditions of sedimentation of the copper (II) oxide particles and their agglomerates, which were synthesized in the PMMA solutions in toluene with the different PMMA concentration, were provided as follows. The particles were sedimented in toluene by adding the respective solution with dispersed particles into it. At the same time, we provided the same initial value of absorbance $D$ and viscosity of the suspension prepared in this way. Viscosity was monitored by means of a Brookfield viscometer BGD 155/4SL (Intelligent Touch-screen Rotary Viscometer), with sampling. The value of viscosity $(0.71 \pm 0.01)\,\mathrm{Pa \cdot s}$ was provided at the temperature $(25 \pm 1)$ °C of the suspensions in order to study sedimentation of the particles and their agglomerates. It made it possible to compare the sedimentation curves without a need of using additional calculation procedures.

Mechanical characteristics (ultimate strain and strength) of the DPCM films produced from the solutions were measured according to GOST 11262-80 in a test machine Shimadzu AG-X 50 kN. The test parameters: a tensile loading rate — 1 mm/min; the environmental temperature — $(23 \pm 2)$ °C.

## 2. Results and their discussions

### 2.1. Sizes of the particles and the agglomerates

Typical microphotos of the TEM images of the dispersed particles and their agglomerates, which are produced from the samples with a 2 % (Fig. 3, *a,c*) and a 10 % (Fig. 3, *b*) PMMA content, are shown in Fig. 3.

All the particles have a shape close to an elliptic one, thereby coinciding with results of the study [10]. Dependences of the average values of the major axes $b$ of the dispersed particles and the average values of the sizes $d$ of the agglomerates (Fig. 3, *c*) on the PMMA concentration in the solution are shown in Fig. 4. The average values of the lengths of the minor axes of the ellipses were actually unchanged for the particles produced in the solutions with the PMMA concentration of up to 8 % and were $a \approx (0.50 \pm 0.03)\,\mu$m. The average value of a was reduced for the particles produced in the solution with the PMMA concentration of 10 % and was $a \approx (0.40 \pm 0.02)\,\mu$m.

The samples with the 10 %-PMMA content in the solution (the saturated solution) did not contain agglomerates of the particles. It can be assumed that better dispersion of the particles in this solution is related to an increased thickness of the polymer shell of the minireactors as compared to the solutions with the lower concentration $C$. It seems to contribute to an increase of pressure inside the minireactors, at which they are ruptured. At the same time, there is also an increased value of kinetic energy of the particles, which results in explosive expansion of the particles in the viscous medium and, as a result, dispersion of the agglomerates of the copper (II) oxide particles. Growth of pressure in the reactors is indirectly confirmed by reduction of the average size of the dispersed particles (Fig. 4, *a*). The study [28] demonstrates a dependence of the sizes of the CuO particles on pressure when they are produced from hydroxide by a traditional method of thermal decomposition.

The process of formation of the agglomerates of the CuO particles can be affected by by-products that are formed in the PMMA solution in toluene during the experiment, for example, oxidation of toluene in presence of a catalyst (the (II) copper oxide particles). Moreover, the by-products can affect only agglomeration of the particles, since the very particles are formed inside the minireactors in an aqueous medium isolated from the solution by the polymer shell (Fig. 1). Since the copper (II) oxide particles have aggregative stability within the pH range from 6.8 to 11.98 [29], the experiments included monitoring of the pH value using a pH-meter (the Hanna InstrumentsHI98130 version). The pH value before the experiment was 7.1, and it was 7 at the end of the experiment. Thus, it can be assumed that the by-products that can be formed during the experiment do not significantly affect the process of agglomeration.

Statistical analysis of the content of the agglomerates in the samples by electron microscopy results can have low reliability due to insufficient sampling [30]. Therefore, additional qualitative studies of agglomeration of the particles directly in the solutions with the concentrations $C = 8\,\%$ and 10 %were performed by a method of analysis of the their sedimentation curves [26]. The samples are selected for analysis in this way due to a significant difference of the content of the agglomerates in them by the results of the TEM studies (Fig. 4, *b*). Absorbance $(D)$ was determined by a technique described in the study [26]. The curves of variation of absorbance are shown in Fig. 5.

The sedimentation time constant for the curve *1* (Fig. 5) is about 25 000 s, while for the curve *2* it is about 5500 s. The lower value of the sedimentation time constant for the particles produced in the 8 % PMMA solution confirms that it contains agglomerates, thereby coinciding with the results of analysis of microscope studies (Fig. 4, *b*). A drop of the curve *2* to almost zero indicates a slight portion of low-dispersed particles that provide significantly slower variation of absorbance, which is typical for the curve *1*.

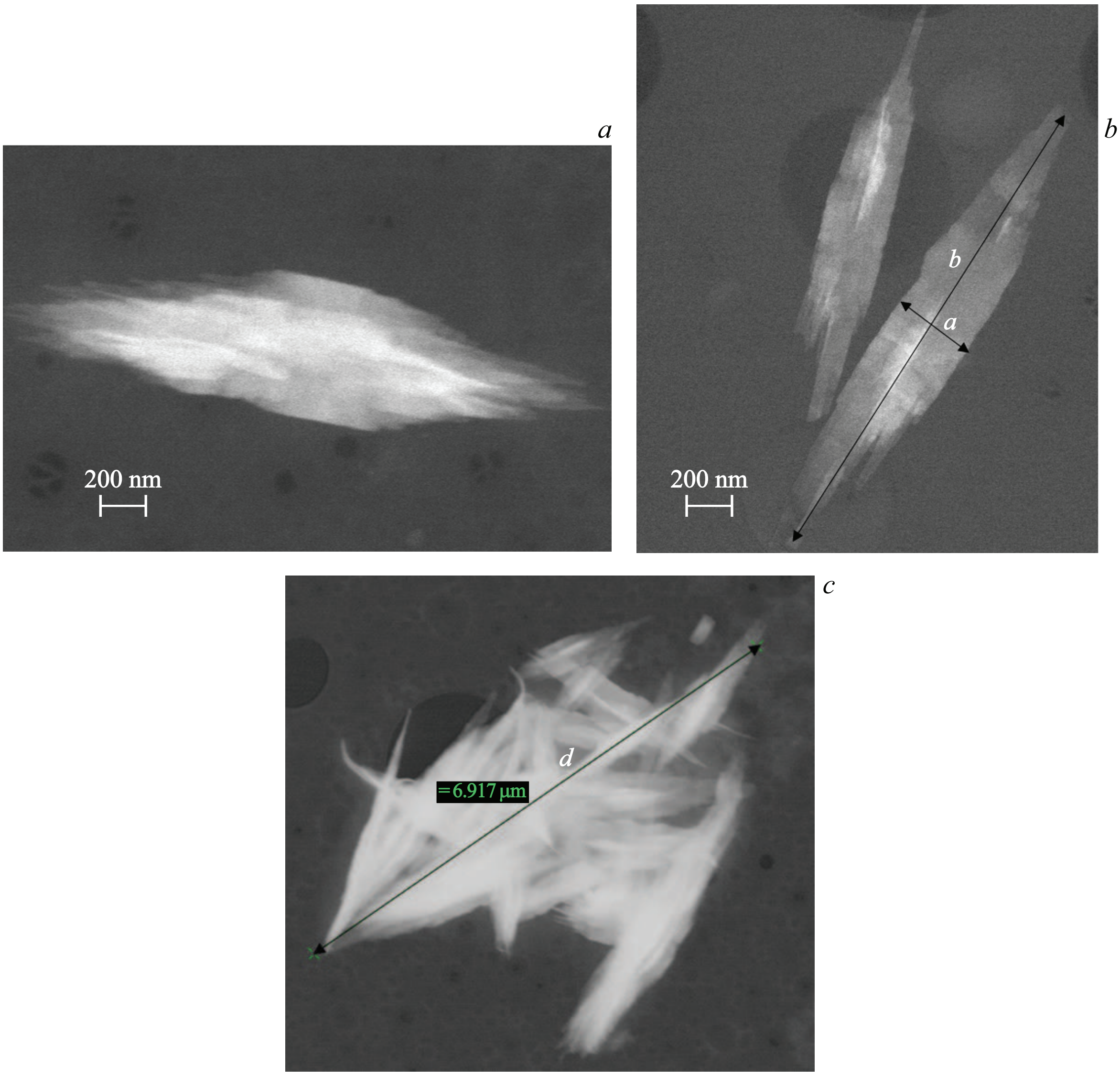


**Figure 3.** Typical TEM microphotos: *a,b* — the dispersed copper (II) oxide particles; *c* — the agglomerates of the copper (II) oxide particles.

In the experimental studies, a rate of formation of the dispersed particles in the sample solutions significantly depended on the concentration of the solution polymer. In order to determine influence of the solution concentration on efficiency of dispersion of the particles from the minireactors, we have produced the films from the volume of the solutions that contained a dispersed phase and determined a volume portion ($\phi$) of the particles in them. The volume portion $\phi$ was determined by dielectric spectroscopy using the Bruggeman formula [31,32]:

$$\frac{\varepsilon_f - \varepsilon_{\text{eff}}}{\varepsilon_{\text{eff}}^{1/3}} = \frac{(1-\phi)(\varepsilon_f - \varepsilon_m)}{\varepsilon_m^{1/3}},$$

where $\varepsilon_f$, $\varepsilon_{\text{eff}}$, $\varepsilon_m$ are permittivities of a polymer composition, a filler and a polymer matrix at the zero frequency, respectively; $\phi$ is a volume content of the filler. The values of permittivity of the polymer films at the zero frequency were determined by approximating dielectric spectra with a function that consisted of superposition of the functions of Cole–Cole and Jonscher [33]:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\Delta\varepsilon_1}{1+(i\omega\tau_1)^{\alpha_1}} + \frac{\Delta\varepsilon_2}{1+(i\omega\tau_2)^{\alpha_2}} + \frac{B}{\omega^n}, \quad (1)$$

where $\Delta\varepsilon = \varepsilon_s - \varepsilon_\infty$ is an amplitude of a relaxation process; $\omega$ is a cyclic frequency; $\tau$ — a typical relaxation time; $\alpha$ is a parameter of symmetrical broadening of the spectrum; $\varepsilon_\infty$ is a limit value of permittivity at the high frequencies; $\varepsilon_s$ — static permittivity (permittivity at the frequency $\omega = 0$); $B$ is a coefficient of the Jonscher function; $n$ is a power exponent of the Jonscher function. The Jonscher function takes into account influences of processes that occur beyond a boundary of a frequency window of measurements. The indices 1 and 2 designate numbering of relaxation processes:

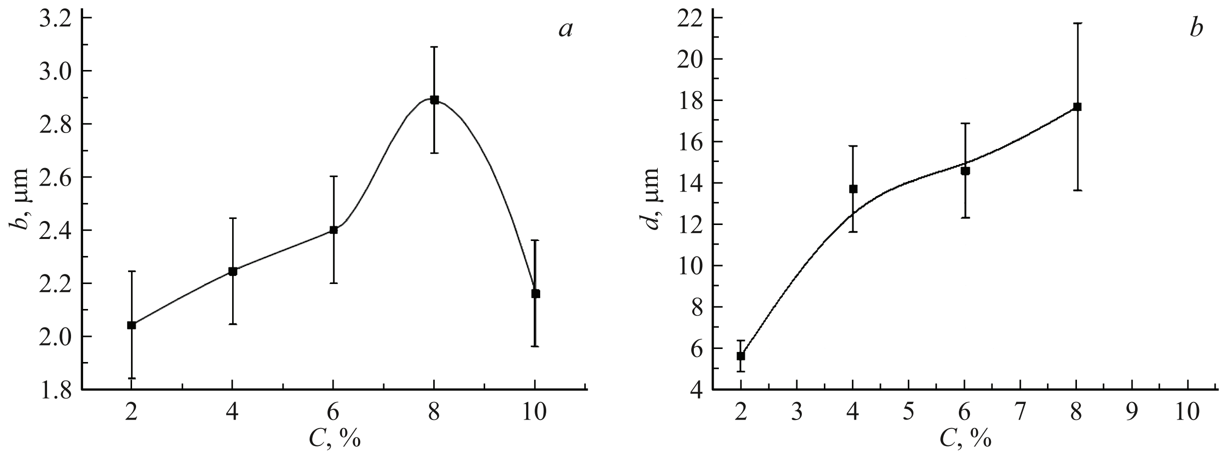


**Figure 4.** Concentration dependences: *a* — the average lengths ($b$) of the major axis of the elliptic dispersed particles; *b* — the linear sizes ($d$) of the agglomerates.

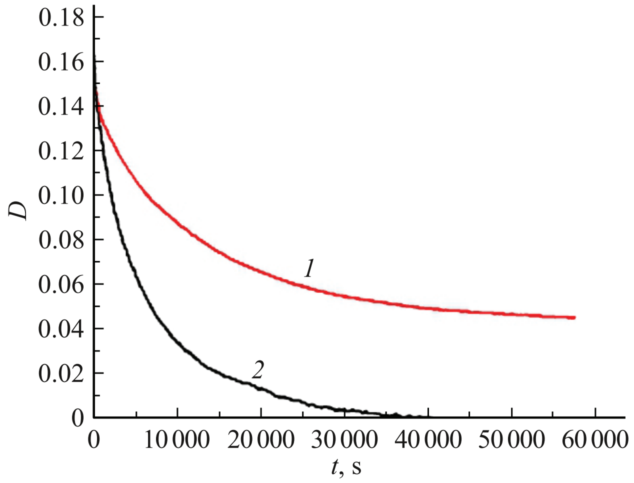


**Figure 5.** Curves of variation of absorbance of the samples: *1* — the sedimentation curve for the particles produced in the solution with the PMMA concentration of 10%; *2* — the sedimentation curve for the particles produced in the solution with the PMMA concentration of 8%.

Data of approximation of the dielectric spectra

| Process | | $C$=2,% | $C$=4,% | $C$=6,% | $C$=8,% | $C$=10,% |
|---|---|---|---|---|---|---|
| 1 process | $\Delta\varepsilon_1$ | 0.407 | 0.418 | 0.69 | 0.734 | 1.02 |
| | $\tau_1$, c | 0.013 | 0.0109 | 0.0114 | 0.0096 | 0.00493 |
| | $\alpha_1$ | 0.33 | 0.329 | 0.317 | 0.338 | 0.346 |
| | $\varepsilon_\infty$ | 2.56 | 2.572 | 2.57 | 2.517 | 2.49 |
| 2 process | $\Delta\varepsilon_2$ | 0.097 | 0.103 | 0.108 | 0.21 | 0.31 |
| | $\tau_2$, c | 8.54e-7 | 7.632e-7 | 8.357e-7 | 5.054e-7 | 5.85e-7 |
| | $\alpha_2$ | 0.341 | 0.373 | 0.41 | 0.249 | 0.219 |
| Jonscher function | B, 1/c | 0.0125 | 0.001 | 0.001 | 0.0041 | 0.0421 |
| | N | 0.803 | 0.185 | 0.188 | 0.161 | 0.444 |

the index 1 corresponds to the first process described by the second term of the expression (1), and the index 2 corresponds to the second process described by the third term of the expression (1). Data of approximation of the dielectric spectra for the samples under study are provided in Table.

The dependence of the volume portion $\phi$ on the PMMA concentration in the solution is shown in Fig. 6, *a*. The concentration dependences of ratios of the real

$$\alpha = \varepsilon'(80\,^\circ\mathrm{C})/\varepsilon'(20\,^\circ\mathrm{C})\big|_{f=0\,\mathrm{Hz}}$$

and the imaginary

$$\beta = \varepsilon''(80\,^\circ\mathrm{C})/\varepsilon''(20\,^\circ\mathrm{C})\big|_{f_{\max}}$$

part of permittivity are shown in Fig. 6, *b*, where $f_{\max}$ is a value of the frequency, which corresponds to the maximum in the frequency dependence of the imaginary part of permittivity [34].

An increase of the volume portion $\phi$ of the filler in the polymer films with the increase of the concentration indicates an increase of efficiency of dispersion. The obtained result confirms high efficiency of dispersion of the CuO particles in the samples with $C = 10\,\%$. Since the films were poured only from the solution within the cuvette volume, a portion of the filler in the films is determined by a respective portion of the dispersed particles in this volume. It seems that efficiency of dispersion of the particles is increased due to the higher thickness of the polymer shell on the minireactors surfaces when $C = 10\,\%$.

Interaction of the dispersed particles with the polymer matrix limits mobility of polymer macromolecules [35], including one related to thermal motion, thereby causing better temperature stability of indicators of their dielectric characteristics. Absence of the agglomerates in the sam-

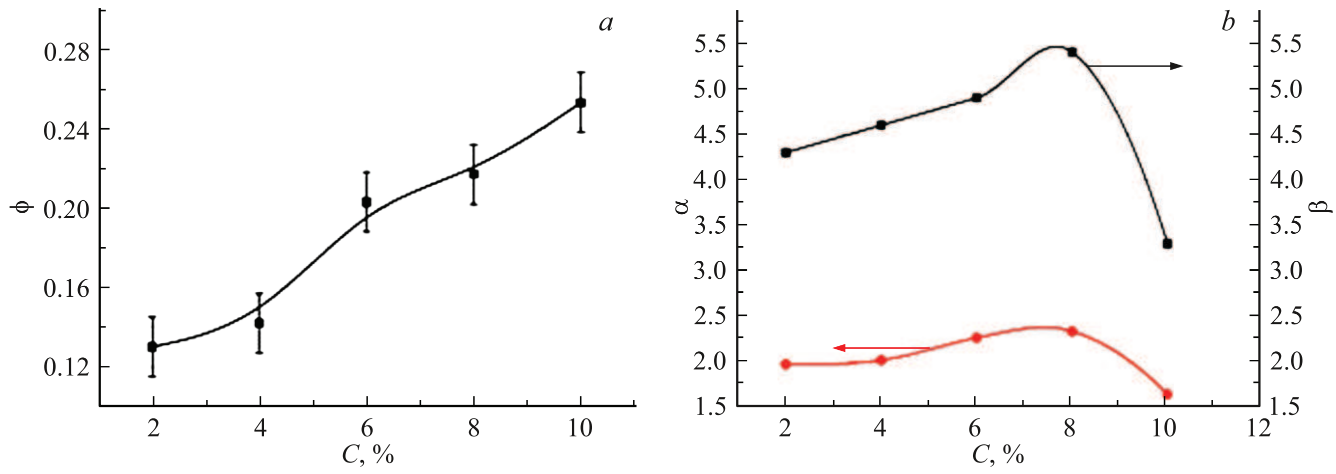


**Figure 6.** Results of investigation of the samples of the polymer films produced from the solutions, by dielectric spectroscopy: $a$ — the dependence of the volume portion of the particles on the polymer concentration $(C)$ in the solutions; $b$ — the concentration dependences of the indicators $\alpha$ and $\beta$.

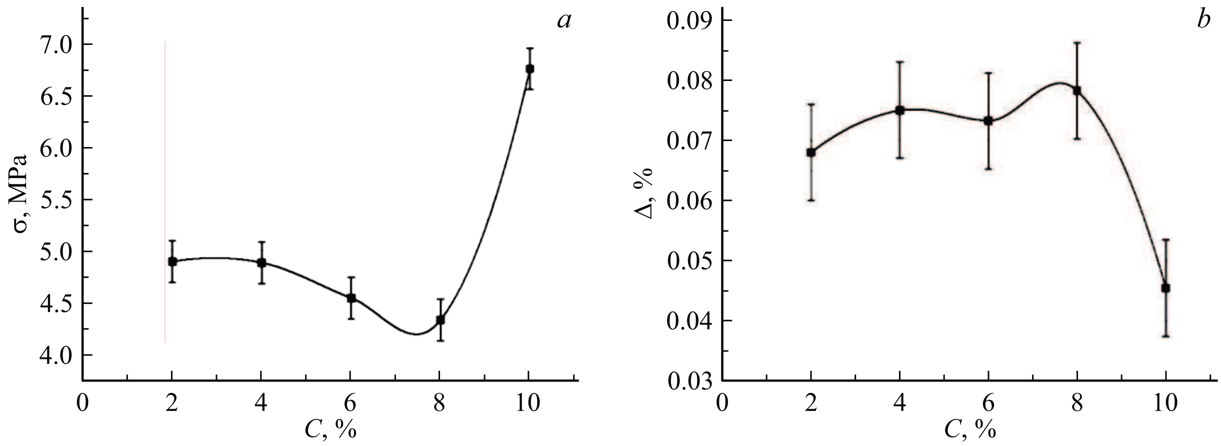


**Figure 7.** Mechanical characteristics of the films in the dependence on the polymer concentration in the solutions: $a$ — strength; $b$ — ultimate strain.

ples with $C = 10\,\%$ explains high temperature stability of permittivity (Fig. 6, $b$).

It should be noted that strength $(\sigma)$ of the films produced from the 10% polymer solution (Fig. 7) significantly increases with simultaneous reduction of ultimate strain $(\Delta)$.

The value of strength of the polymer film produced from the solutions is by about an order smaller than a typical value for PMMA [36,37]. Probably, it is caused by film porosity due to evaporation of the solvent [38] as well as by incomplete evaporation of the solvent.

Due to their needle-like shape and interaction with the polymer matrix, the dispersed copper (II) oxide particles limit mobility of the macromolecules by bonding them between each other with physical cross links [39]. It should be noted that in case of agglomeration of the filler particles the strength characteristics of the respective polymer composite decrease [10]. At the same time, the absolute values of strength and ultimate strain depend on the portion of the agglomerates, the type of the polymer matrix and the method of composite production.

## Conclusion

The study has investigated a technology of formation of the films of the dispersion-filled polymer composite from the PMMA solution in toluene with *in situ* production of the dispersed copper (II) oxide particles. The copper (II) oxide particles are formed in a reaction of thermal decomposition of copper oxide due to impact by the microwave field of the 2.45 GHz frequency and power $(720 \pm 10)$ W. The experimental results obtained confirm the mechanism of particle dispersion due to an explosive nature of collapse of the minireactors, which are droplets with the polymer shell, inside which copper hydroxide is originally held. As it is heated, copper hydroxide first transits into copper (II) oxide and water with subsequent formation of bubbles with water vapor. As internal pressure increased, the bubbles were ruptured with dispersion of the copper (II) oxide particles. The best efficiency of dispersion takes place in the saturated solution with the PMMA concentration of 10 %. It is caused by the maximum thickness of the PMMA shell on the minireactors surfaces due to displacement of the polymer from the saturated solution by water. Absence of the agglomerates of the dispersed particles in the respective samples is confirmed by the methods of electron microscopy (TEM) and photometry. The particle deposition time constant in the solutions prepared for sedimentation is in $\sim 5$ times higher for the particles produced in the solution with the 10 % concentration as compared to the 8 % solution. The best efficiency of particle dispersion in the saturated 10 % solution made it possible to produce the DPCM films with the dispersed phase's volume portion $\sim 0.25$. The increase of the dispersed phase's portion as well as absence of the agglomerates of the particles results in the significant increase of strength of these DPCM films. It can be explained by an increased number of physical cross links between the polymer macromolecules, which is confirmed by increased temperature stability of the indicators of the dielectric properties of the films produced from the 10 % PMMA solution.

These films can be used when creating polymer electrets with high temperature stability of their polarizability: the dispersed particles limit mobility of polar polymer macromolecules oriented in the external electric field, thereby contributing to an increase of the relaxation time of the polymer electrets.


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### Conflict of interest

The authors declare that they have no conflict of interest

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