Clinical and laboratory evaluation of microstructural changes in the physical, mechanical and chemical properties of dental filling materials under the influence of an electromagnetic field

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Abstract

Restorative filling materials used for dental caries prevention and treatment consist of various components including monomers or oligomers, which play a significant role in forming the main structure of these materials, as well as in characterising their physical, mechanical and chemical properties. The necessity for the development and improvement of structural characteristics of polymeric dental filling materials intended for caries prevention and their life duration increase served as the initiating factor of our research. According to the research purpose and challenges, we studied the changes in the physical, mechanical and chemical properties of composite filling materials with and without electromagnetic field influence. The investigations in vivo include the study of microstructural features of polymeric filling materials by scanning electron microscopy (SEM) and the investigations in vitro include the study of sealed and extracted human teeth chips by using X-ray spectral analysis. We also evaluated the changes in the strength characteristics of dental filling materials with and without electromagnetic field influence. The analysis of the obtained data indicates the presence of structural changes in polymeric dental filling materials, including the material microstructure condensation confirmed by the SEM results, an increase in the strength and adhesion characteristics and certain regularities of the chemical elemental composition concentration change in the area of hard tooth tissue and dental filling material. These scientific data will provide tooth caries prevention and promote the increase of treatment quality.

Keywords Dental polymeric filling materials · Electromagnetic field · Scanning electron microscopy (SEM) · X-ray spectral microanalysis · Physical and mechanical properties · Physical and chemical properties

Background

The incidence rate of dental caries remains high despite the use of modern technology techniques in the diagnosis, prevention and treatment of caries, as well as a wide assortment of dental filling materials. This situation is a serious health problem in most countries of the world, particularly in connection

with a steady increase of restorative treatment costs and complications associated with somatic diseases [1–3].

The success of dental caries treatment depends on the filling material service life, so dental filling material quality improvement is a key problem of dentistry. The search for new effective methods for dental caries treatment promotes a detailed study of the physical, mechanical and chemical properties of dental filling materials [4–6]. Filling materials used for dental caries prevention and treatment consist of various components, including synthetic polymers. All the constituents play a significant role in forming the main structure of these materials, as well as in characterising their physical, mechanical and chemical properties [7–9].

Presently, the development of high technology methods of research and treatment in dentistry results in raised demands on the basic elements of “white” aesthetics, such as colour, form and material durability.

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The qualities that an ideal dental material should have are: to be biocompatible; to resist to the oral cavity medium influences; to provide a strong and permanent connection with the structure of hard tooth tissues; to reproduce their appearance completely; to possess a complex of physical and mechanical properties corresponding to the properties of the restored natural tissues; and, moreover, to promote their recovery and regeneration.

The question of creating an “ideal” filling material for dental hard tissues restoration, which involves a long-term restoration of the anatomical and functional integrity of teeth [10], is currently becoming increasingly important. Therefore, further studies are needed on the stability and property changes of the filling materials under the influence of physical, mechanical and chemical factors. On the way towards the “ideal” filling material creation, new technologies that improve its physical, mechanical and chemical characteristics are being introduced. A special place in the improvement of the physical and mechanical characteristics of polymer-based filling materials is given to the methods of directional change of the morphology of materials by means of their thermal and chemical treatment, irradiation and deformation, as well as by their exposure to magnetic, electric or ultrasonic fields.

Currently, the influence of various physical factors of therapy such as LED and laser exposure, ultrasound and electromagnetic fields on the human tissues, including for accelerating the processes of their regeneration, has been studied [11]. However, the influence of physical factors of therapy on consumable dental products in order to improve their physical, mechanical and chemical properties remains largely uninvestigated.

To date, it has been shown that magnetic and electric fields may cause structural changes of the liquid polymers, subsequently improving their physical and mechanical characteristics [12, 13].

In the available national literature [14], there is a patent which addresses the issue of the electric field effects to increase the antibacterial properties of the material and the use of such a field to polymerise the composite material in the dental inlay manufacturing process [13].

Among some foreign authors, there are several papers of particular interest which concern the influence of low-voltage electromagnetic field and antioxidants on the chemical compound (zinc, strontium) of hard dental tissues of rats after its long-term exposure and different variants of its normalisation by using natural immunomodulating agents [15] and the use of pulsed electromagnetic fields on stimulation of the osseointegration process in bone healing [16].

The polymer activation by a magnetic field in a fluid-like state or during its curing leads to a change in its physical and mechanical characteristics [10]. The change in the properties of the polymer is a reflection of the processes taking place in the polymer structure. The changes that occur in the polymer structure under the magnetic field action are caused by the anisotropy of the diamagnetic susceptibility of macromolecules and their parts [12, 17]. During the polymerisation, the

liquid phase of the polymer is reoriented under the magnetic field influence into diamagnetic formations at the moment of their growth.

The aim of this *in vitro* study was to evaluate the structural characteristics of polymeric filling materials and to analyse their physical, mechanical and chemical properties with the use of an electromagnetic field.

Materials and methods

Ethics approval and subject selection

The study was approved by the local committee for ethics in research of the State Funded Educational Institution of Higher Education “Voronezh N.N. Burdenko State Medical University” of the Ministry of Health of the Russian Federation (Protocol no. 1, February 25, 2016). Written informed consent was obtained from all patients. The participants were randomly selected from a pool of patients (18–35 years old) who had sought dental treatment.

This *in vitro* study was conducted to analyse the microstructural features and physical, mechanical and chemical properties of polymeric filling materials under electromagnetic field influence at the VSMU dental clinic base in cooperation with the Voronezh State University and Voronezh State Forestry University named after G.F. Morozov. The adhesive systems Prime & Bond NT, Dentsply, USA; Charisma and Durafile, Heraeus Kulzer, Germany; Spectrum, Dentsply, USA; and Filtek Flow, 3M ESPE, USA, comprising composite fillings, were used for the research study. The investigated materials were divided into two equivalent groups (I: the study group with electromagnetic field influence; II: the control group without electromagnetic field influence).

Examination methods

A special device [10] was used to process polymer materials in a constant electromagnetic field for the implementation of a study concerning electromagnetic field influence on the physical and mechanical properties of dental filling materials.

The base for this installation is an electromagnetic inductor consisting of two movable windings of an electromagnet connected in series on a magnetic yoke. The magnetising coils made of copper cable with double-layer insulation made from glass thread are connected in sequence. A current of up to 12 A is applied to the windings of the electromagnet by the DC power supply unit [12].

The magnetic field intensity is regulated by the amount of applied current and the distance between the electromagnet poles. The installation makes it possible to create a magnetic field with a strength up to 30×10^4 A/m.

Processing of the studied dental filling materials on the device was carried out in the following order. Each material including adhesive system of study group I in the factory package was placed in the working interpolar space for electromagnetic processing of polymeric materials in a constant electromagnetic field. They were influenced by a constant electromagnetic field with magnetic field strengths of 20×10^4 to 24×10^4 A/m for 20 min.

After the electromagnetic field exposure for the microstructure investigation, the dental filling materials and adhesive system samples of cylindrical form with diameter not less than 0.5 cm and not exceeding 1 cm in length of groups I and II (control) were prepared and cured with a blue light polymerisation lamp at a wavelength of 450 nm and a power of 5 W for not less than 40 s. Then, a diamond disc was put at the widest point of the samples to a depth of not more than 0.2 cm. With the help of a dental smoother, the sample was cut along the marked notch so that a chipping of the filling material with a diameter of at least 0.4 cm was obtained [7, 10] (Table 1).

To investigate the microstructure of polymeric filling materials, the chips of obtained filling materials and adhesive system samples were studied by scanning electron microscopy (SEM) using a low-vacuum scanning electron microscope (JEOL JSM-6380 LV model, Japan). The investigation of the filling material microstructure was carried out in the secondary electron emission regime at an accelerating voltage of 20–30 kV and at a magnification of 50,000 $\times$.

For the control of the same procedure, the material of control group II with no preliminary electromagnetic field exposure was cut and examined.

To study the changes in the physical and mechanical properties of the filling materials Charisma, Durafil, Spectrum and Filtek Flow after the electromagnetic field exposure, strength tests were carried out in accordance with the National Standard 31574-2012 (Dental resin restorative materials. Technical requirements. Test methods), which is equal to ISO 4049:1988* Dentistry - Resin-based filling materials, ISO 10477:1992 Dentistry - Polymer-based crown and bridge materials in the following paragraphs: 5.1; 6.0; 6.1.1; 6.1.3; 6.1.4; 6.1.5; 6.1.6; 6.1.7; 6.2.1; 6.2.2; 6.2.6; 6.3.2 and ISO 11405:1994 Dental materials - Guidance on testing of adhesion to tooth structure in paragraph 6.3.1 and included determination of the strength for a diametrical fracture, bending and shear, which characterises the resistance and determines their strength limits at various loads. For the comprehensive assessment of the physical and mechanical properties of the composite filling materials, we also conducted tests to determine the compressive strength, which is not included in the National Standard. The testing samples have been prepared in accordance with the National Standard 31574–2012 [18]. For each strength parameter, such as compressive strength, strength for a diametrical fracture, bending and shifting, investigations used 20 samples of filling materials for each group. The tests to determine the

ultimate strength of the composite filling materials were carried out on the testing machine IR 5040-1.

For analysis of the tooth enamel and dentin permeability for the chemical components of the filling material and adhesive system with and without electromagnetic field influence, 32 teeth that had been removed according to the orthodontic indications were filled according to the generally accepted technique with the diagnosis of “medium caries”.

The freshly removed teeth were randomly divided into two groups before sealing. In the first study group ($n = 17$), the filling was performed with a material pre-treated with an electromagnetic field, and in the second control group ($n = 15$), the filling was performed with a material without prior electromagnetic exposure.

Preparation of the chippings for the study consisted of the following: the removed sealed teeth were washed out with running water, dried with filter paper, then the diamond disc was put to a depth of 0.3 cm. Using a dental smoother, the sample was cut along the marked notch to give a chipped tooth with a border of the filling material and the visible surface area was up to 0.5 cm.

The prepared chippings of the sealed teeth were fixed on a holder, placed in the JEOL JSM-6380 LV scanning electron microscope chamber and then exposed to a stationary thin-focused electron beam unfolding into the raster on the sample surface. The images were obtained in the secondary electron emission regimes and back-scattered electrons in a low-vacuum scanning electron microscope. The research analysis of X-ray radiation gave qualitative and quantitative information about the elemental composition of the object under study.

The distribution of chemical elements in the filling material–dentin border area was studied by X-ray spectral analysis with mapping of lateral chips using the INCA-250 energy dispersive analysis system. The distribution of chemical microelements in the filling material–tooth tissue border area on the X-ray chart is marked with a colour (C - red, Ca - green, Si - blue) and characterises the mutual penetration of the filling material and adhesive system components into dentin.

Statistical analyses

For statistical processing of the study results, a standard package of STATISTICA 8.0 software programs by Statsoft was used. Before statistical analysis of the study results, a particular copy of the program was verified on the test set with known properties and results [19]. Within the framework of descriptive statistics, the following parameters were calculated: mean and mean square deviation, median and the standard error of the mean, kurtosis, asymmetry, minimum and maximum values of the data series, quantile value and quantile range. To compare the groups, we used a non-parametric Mann–Whitney *U*-test, Kruskal–Wallis test and a median test; the effect of multiple comparisons was taken into account.

Table 1 Distribution of polymeric restorative materials samples into the two investigation groups (with and without electromagnetic field influence) for scanning electron microscopy (SEM) and strength tests

Groups	Filling materials	Quantity of samples		Number of packages
		SEM	Strength tests	
I: study group (with electromagnetic field influence)	Charisma	1	20	1
	Durafil	1	20	1
	Spectrum	1	20	1
	Filtek Flow	1	20	1
	Prime & Bond NT	1	5	1
II: control group (without electromagnetic field influence)	Charisma	1	20	1
	Durafil	1	20	1
	Spectrum	1	20	1
	Filtek Flow	1	20	1
	Prime & Bond NT	1	5	1
Total		10	170	10

When processing statistical data, the differences were considered significant at $p < 0.05$.

Results

The SEM results confirm significant changes in the microstructure of polymeric restorative materials under electromagnetic field influence: particles enlargement and a reduction in the distance between the particles of the material polymer matrix (Figs. 1 and 2) (Table 2).

Figures 1 and 2 show that the electromagnetic field influence results in a more ordered structure of the material sample, it is more dispersed and has certain morphological changes not observed in the material without such an influence. The morphology of polymeric filling materials exposed to the electromagnetic field becomes densified due to the enlargement of the polymer matrix particles and the reduction of the distance between them; the difference is obvious when compared with the material of the control group. The control group materials have larger gaps between the particles, which are stress concentrators that reduce the strength of polymer compounds, while the material exposed to the electromagnetic field has more uniform and homogeneous structure.

To confirm the obtained results, we analysed the changes occurring in the microstructure of polymeric filling materials after electromagnetic field exposure, including determination of the particle size and the distance between them. The data are presented in Table 2 and Figs 1 and 2.

The data of Table 2 and Fig. 1a, b representing the microstructure of the filling material of Charisma before and after electromagnetic field influence indicate the particles enlargement and a reduction in the distance between the particles in the study group material exposed to the electromagnetic field in comparison with

the material of the control group without electromagnetic field influence. The differences between the study groups on account of this feature are statistically significant ($p < 0.01$).

The same tendency was observed in the dental filling materials Durafil, Spectrum, Filtek Flow and Prime & Bond NT after electromagnetic field exposure (Table 2, Figs. 1c–f and 2a–d). The differences between the study groups on account of these characteristics are statistically significant ($p < 0.01$).

The tests carried out to study the physical and mechanical properties of the filling materials positively resulted in an increase of the material compressive strength and its strength for a diametrical fracture, bending and shifting after electromagnetic field exposure (Table 3).

The data of Table 3 and Figs. 5, 6, 7 and 8 representing the strength parameters of the composite materials before and after electromagnetic field influence indicate statistically significant differences between the study and control groups in relation to the strength increasing in the study group ($p < 0.01$). The same tendency was observed in all investigated composite materials (Charisma, Durafil, Spectrum and Filtek Flow) after the electromagnetic field exposure.

In examining the sealed teeth samples by X-ray microanalysis with mapping after the preliminary action of the electromagnetic field on polymeric dental filling materials and adhesive systems, the secondary electron image demonstrates a reliable increase in the concentration of carbon, calcium and silicon in the filling material–dentin border area. This was confirmed by the penetration of the filling material and adhesive system components into the interprismatic spaces of the tooth crown dentin. Carbon, as a specific organic component of the bond, becomes visually accumulated in the filling material–dentin adhesion zone. The filling material–dentin contact area is presented in the form of a thick belt along the entire length, with the elements of the filling material and adhesive

Fig. 1 Scanning electron microscopy (SEM) results of the dental filling materials, at 50,000 $\times$ magnification. **a, b** Charisma, Heraeus Kulzer, Germany before (**a**) and after electromagnetic field exposure (**b**). **c, d** Durafil, Heraeus Kulzer, Germany before (**c**) and after electromagnetic field exposure (**d**). **e, f** Spectrum, Dentsply, USA before (**e**) and after electromagnetic field exposure (**f**). An increase in the particle size and a decrease in the gaps between them were observed

system penetrating into dentin after electromagnetic field influence (Fig. 3).

At the enamel–dentine border in the area of moderate caries in accordance with the World Health Organization (WHO) classification of dental diseases (ICD-DA, WHO, Geneva, 1995), classified as dentine caries (caries media), the enamel–dentine border area with the filling material Charisma was scanned using X-ray spectral microanalysis.

In studying the energy spectrum with the use of the Charisma material, the quantitative parameters of the microelemental composition of the filling material–dentin contact zone were determined and served as indicators of the filling material adhesion to the tooth tissues (Fig. 4, Table 4).

The quantitative analysis presented in Table 4 shows a reliable increase in the weight coefficient of oxygen, aluminum, phosphorus and calcium, which indicates an increase in the concentration of these elements in this area after the electromagnetic field influence on the material, whereas the

weight coefficient of carbon and barium is higher in a non-exposed sample, which may indicate their energetic participation in the adhesion (Table 4, Fig. 4).

Discussion

A new direction in dentistry can be considered as the development of a method using the influence of an electromagnetic field on polymeric dental materials, allowing modifying the substance structure, to obtain a modification of the polymer with increased adhesion and strength characteristics in order to prevent secondary caries and increase the service life of fillings.

By means of SEM, we carried out a comparative analysis of the microstructural features of restorative polymeric materials used to treat dental caries before and after electromagnetic field influence in vitro. The studies were carried out with magnifications of 2000 $\times$, 3000 $\times$, 30,000 $\times$, 50,000 $\times$ and

Fig. 2 SEM results of the Flow dental filling material and adhesive system, at 50,000× magnification. **a, b** Filtek Flow, 3M ESPE, USA before (**a**) and after electromagnetic field exposure (**b**). **c, d** Prime & Bond NT, Dentsply, USA before (**c**) and after electromagnetic field exposure (**d**). An increase in the particle size and a decrease in the gaps between them were observed

100,000×. As a result of the research, a significant increase in the particles of the polymer matrix of restorative materials and a decrease in the distance between them after electromagnetic field action ($p < 0.05$) was revealed.

We evaluated the physical-mechanical, strength and adhesion properties of polymeric restorative materials before and after electromagnetic radiation in vitro. By means of the strength parameters determination, the physical-mechanical and adhesive properties of the material, including those of the sealed tooth after electromagnetic field influence, were found to be enhanced. The data obtained are confirmed by the statistical data presented in Table 3 and on the graphs shown in Figs. 5, 6, 7 and 8).

Thus, from Table 3 and Figs. 5, 6, 7 and 8, which characterise the strength parameters of the composite material Charisma under compression, it follows that, in the control group without electromagnetic field exposure, the average value of the load applied to the sample to destroy it was 3192.995 (3021.800/3429.900) H, and in the study group, it was 4552.500 (3950.200/4791.400) H; when testing for a diametrical fracture, the average value of the load in the control group was 923.245 (858.100/998.200) H, and in the study group, it was 1028.610 (991.200/1082.600) H; in bending testing, the average value of the load in the control group was 20.870 (19.400/22.400) H, and in the study group, it was 22.250 (20.400/23.900) H; in shift testing, the average

Table 2 Changes of the polymeric restorative materials microstructure after electromagnetic field exposure

Groups	Dental filling material	II: control group ($n = 15$) $M \pm m$	I: study group ($n = 15$)
Particle size (in microns)	Charisma	$0.036 \pm 0.002^*$	$0.104 \pm 0.005^*$
	Durafil	$0.044 \pm 0.004^*$	$0.114 \pm 0.005^*$
	Spectrum	$0.04 \pm 0.004^*$	$0.216 \pm 0.015^*$
	Filtek Flow	$0.118 \pm 0.008^*$	$0.216 \pm 0.017^*$
	Prime & Bond NT	$0.099 \pm 0.005^*$	$0.165 \pm 0.006^*$
Distance between the particles (in microns)	Charisma	$0.038 \pm 0.003^*$	$0.025 \pm 0.001^*$
	Durafil	$0.028 \pm 0.002^*$	$0.016 \pm 0.001^*$
	Spectrum	$0.227 \pm 0.005^*$	$0.127 \pm 0.005^*$
	Filtek Flow	$0.1996 \pm 0.011^*$	$0.098 \pm 0.005^*$
	Prime & Bond NT	$0.077 \pm 0.004^*$	$0.021 \pm 0.002^*$

* $p < 0.01$

Table 3 Comparative characteristics of the strength properties in polymeric restorative materials with and without electromagnetic field influence

Dental filling material	Group M ± m/M (min/max)	Compression (H)	Diametrical fracture (H)	Bending (H)	Shifts (H)
Charisma	II: control group (without influence)	3192.995 ± 27.981 (3021.800/3429.900)	923.245 ± 11.715 (858.100/998.200)	20.8700 ± 0.174 (19.400/22.400)	57.5750 ± 2.462 (41.800/74.000)
Charisma	I: study group (with influence)	4552.500 ± 41.452* (3950.200/4791.400)	1028.610 ± 6.317* (991.200/1082.600)	22.25000 ± 0.224* (20.400/23.900)	101.9750 ± 2.734* (81.600/123.000)
Durafil	II: control group	3209.945 ± 36.649 (3020.700/3471.200)	958.215 ± 4.815 (924.900/991.800)	21.13000 ± 0.186 (19.800/22.700)	66.1000 ± 0.586 (61.100/68.900)
Durafil	I: study group	4503.120 ± 41.666* (4098.100/4781.400)	1061.260 ± 5.730* (1010.200/1091.300)	23.18500 ± 0.190* (21.900/25.100)	128.6250 ± 1.194* (118.100/135.200)
Spectrum	II: control group	3868.755 ± 18.385 (3695.200/4028.800)	1090.395 ± 13.113 (1001.700/1191.800)	23.740 ± 0.633 (19.100/28.700)	83.245 ± 0.459 (80.100/87.400)
Spectrum	I: study group	4174.890 ± 48.606* (3910.100/4901.700)	1315.875 ± 17.701* (1201.900/1465.000)	29.270 ± 1.019* (21.800/34.800)	101.140 ± 0.703* (97.100/108.300)
Filtek Flow	II: control group	3450.375 ± 42.206 (3194.900/3724.400)	1277.495 ± 10.750 (1191.800/1321.400)	32.970 ± 0.426 (29.100/35.100)	59.925 ± 0.255 (58.100/62.100)
Filtek Flow	I: study group	3910.645 ± 44.571* (3641.100/4295.600)	1292.445 ± 8.544 (1201.100/1324.600)	33.36500 ± 0.441 (29.500/35.800)	108.5600 ± 0.962* (103.100/115.100)

* $p < 0.001$

value of the load in the control group was 57.575 (41.800/74.000) H, and in the study group, it was 101.975 (81.600/123.000) H, which indicates an increase in the material strength. The differences between the study group with electromagnetic field influence and the control group without such an influence on account of this characteristic are statistically significant ($p < 0.001$).

A tendency to an increase in the material strength was also observed in the filling material Durafil. In the control group without electromagnetic field exposure, the average value of the load applied to the sample to destroy it was 3209.945 (3020.700/3471.200) H, and in the study group, it was 4503.120 (4098.100/4781.400) H; when testing for a diametrical fracture, the average value of the load in the control group was 958.215 (924.900/991.800) H, and in the study group, it was 1061.260 (1010.200/1091.300) H; in bending testing, the

average value of the load in the control group was 21.130 (19.800/22.700) H, and in the study group, it was 23.185 (21.900/25.100) H; in shift testing, the average value of the load in the control group was 66.100 (61.100/68.900) H, and in the study group, it was 128.625 (118.100/135.200) H, which indicates an increase in the material strength. The differences between the study groups on account of this characteristic are statistically significant ($p < 0.001$).

The same tendency was also observed in the filling material Spectrum. In the control group without electromagnetic field exposure, the average value of the load applied to the sample to destroy it was 3868.755 (3695.200/4028.800) H, and in the study group, it was 4174.890 (3910.100/4901.700) H; when testing for a diametrical fracture, the average value of the load in the control group was 1090.395 (1001.700/1191.800) H, and in the study group, it was 1315.875 (1201.900/1465.000) H; in

Fig. 3 X-ray map of the planar distribution of the chemical elements [C (carbon), Ca (calcium), Si (silicium)] on the filling material–dentin border without (a) and with (b) electromagnetic field exposure, at 500 times magnification

Fig. 4 X-ray map of X-ray microanalysis of the chemical elements distribution: the filling material–dentin border scanning zone with (a) and without (b) electromagnetic field action, at 250 times magnification

bending testing, the average value of the load in the control group was 23.740 (19.100/28.700) H, and in the study group, it was 29.270 (21.800/34.800) H; in shift testing, the average value of the load in the control group was 83.245 (80.100/87.400) H, and in the study group, it was 101.140 (97.100/108.300) H, which indicates an increase in the material strength. The differences between the study groups on account of this characteristic are statistically significant ($p < 0.001$).

This definite tendency was also observed in the filling material Filtek Flow. In the control group without electromagnetic field exposure, the average value of the load applied to the sample to destroy it was 3450.375 (3194.900/3724.400) H, and in the study group, it was 3910.645 (3641.100/4295.600) H; in shift testing, the average value of the load in the control group was 59.925 (58.100/62.100) H, and in the study group, it was 108.560 (103.100/115.100) H, which indicates an increase in the material

strength. The differences between the study groups on account of this characteristic are statistically significant ($p < 0.001$).

However, when testing for a diametrical fracture, the average value of the load in the control group was 1277.495 (1191.800/1321.400) H, and in the study group, it was 1292.445 (1201.100/1324.600) H; in bending testing, the average value of the load in the control group was 32.970 (29.100/35.100) H, and in the study group, it was 33.365 (29.500/35.800) H. The differences between the study groups on account of this characteristic are not statistically significant ($p < 0.5$).

X-ray spectral microanalysis made it possible to assess the state of the mineral and organic components of the tooth and to define the qualitative and quantitative characteristics of metabolic processes in the enamel and dentine of the tooth sealed with regard to medium caries with the material exposed to the electromagnetic field and not exposed to it in vitro.

Table 4 Quantitative elemental chemical composition of enamel in marginal gaps between the filling material and the tooth with and without electromagnetic field influence

Element	Weight coefficient, %		Atomic ratio, %	
	Tooth-filling material border without exposure $M \pm m$	Tooth-filling material border with exposure	Tooth-filling material border without exposure	Tooth-filling material border with exposure
C	31.493 ± 0.449	25.428 ± 0.505	43.416 ± 0.404	34.99 ± 0.475
O	43.556 ± 0.472	52.464 ± 0.462	44.547 ± 0.459	54.262 ± 0.474
Al	1.823 ± 0.188	2.401 ± 0.168	1.342 ± 0.113	1.646 ± 0.132
Si	12.319 ± 0.339	12.441 ± 0.363	7.478 ± 0.294	7.745 ± 0.343
P	0.344 ± 0.067	1.113 ± 0.077	0.209 ± 0.057	0.681 ± 0.074
Ca	0.491 ± 0.06	1.521 ± 0.079	0.219 ± 0.024	0.742 ± 0.041
Ba	8.228 ± 0.283	6.266 ± 0.237	1.077 ± 0.04	0.831 ± 0.037
Total	100.00		100.00	

Fig. 5 Comparative characteristics of the strength properties (compression) in Charisma material without and with electromagnetic field influence

Based on the conducted studies, a certain influence of the change in the microstructure of restorative material on the activation of enamel metabolic processes and the level of its chemical adhesion to hard tooth tissues after the electromagnetic field action in vitro was revealed.

The use of SEM and X-ray spectral microanalysis made it possible to follow the ion exchange occurring in the enamel and dentine under the influence of filling materials during treatment. When filling with the electromagnetic field treated materials, there were no cases of unsatisfactory filling, namely the formation of microcracks.

Minimum disturbances and rapid stabilisation of mineral metabolism in hard tooth tissues were noted when using sealing materials treated preliminarily with an electromagnetic

field. The most pronounced adhesion intensity was detected by the method of energy dispersive analysis (EDA) in 32 chipped teeth performed with the chemical analyser of the scanning electron microscope JEOL JSM-6380 LV, where, in the automatic mode, the studied chemical elements are marked. The planar spectroradiographs clearly demonstrated the presence of a thick colour band between the filling and hard tooth tissues, as well as areas not marked with a colour when using the materials without electromagnetic field exposure, which indicated the presence of microcracks in this area.

When using the electromagnetic field treated materials, the mutual penetration of microelements fixed by a colour code as well as the uncertainty of the boundaries between the bond, filling material and tooth tissues, i.e. a full chemical adhesion,

Fig. 6 Comparative characteristics of the strength properties (diametrical fracture) in Charisma material without and with electromagnetic field influence

Fig. 7 Comparative characteristics of the strength properties (bending) in Charisma material without and with electromagnetic field influence

was observed. Colour-marked microelements from the filling material were detected in hard tooth tissues and vice versa, which contributes to predicting the filling durability.

Our complex of research methods, SEM and X-ray spectral microanalysis, made it possible to assess objectively the electromagnetic field influence on polymeric restorative materials, as well as to estimate the quality of adaptation and adhesion of filling materials to tooth tissues before and after the electromagnetic field influence in vitro.

The composition of chemical elements at the filling material–tooth tissues border was defined with the use of targeted X-ray spectral microchemical analysis.

The obtained data allow to state the increase in the weight coefficient of oxygen (up to 52.464 ± 0.462), aluminum (up to 2.401 ± 0.168), phosphorus (up to 1.113 ± 0.077) and calcium (up to 1.521 ± 0.079) after electromagnetic field influence, while the carbon and barium content is reduced, up to 25.428 ± 0.505 and up to 6.266 ± 0.237 ($p < 0.05$), respectively. The weight percentage of silicon (12.441 ± 0.363) after the electromagnetic field action is not significantly different ($p < 0.05$) (Table 4, Fig. 4).

Scanning of the enamel–dentine border with the filling material revealed a rather high weight percentage of macroelements characterising the mineral components of hydroxyapatite:

Fig. 8 Comparative characteristics of the strength properties (shifts) in Charisma material without and with electromagnetic field influence

phosphorus up to 1.113 ± 0.077 , calcium up to 1.521 ± 0.079 , oxygen up to 52.464 ± 0.462 , aluminum up to 2.401 ± 0.168 and silicon up to 12.441 ± 0.363 after the electromagnetic field action ($p < 0.05$). Carbon, as an indicator of the organic components of hard tooth tissues, is increased in the sample untreated by an electromagnetic field, up to 31.493 ± 0.449 , apparently due to the presence of metabolic products of microorganisms, in contrast to the content of this element in the samples of sealed teeth after electromagnetic field influence, being 25.428 ± 0.505 (Table 4, Fig. 4).

Conclusions

The analysis of the obtained data indicates the presence of structural changes in the physical, mechanical and chemical properties of polymer-based filling materials after electromagnetic field exposure, including certain microstructural transformations of the material due to the polymer matrix particles size increase and the reduction in the distance between them, which is confirmed by the results of scanning electron microscopy (SEM). X-ray spectral microanalysis studies have made it possible to clarify the qualitative and quantitative composition of the tooth tissues, to reveal in detail the concentration of micro- and macroelements of organic and inorganic nature, to trace the changes occurring in dentin in tooth sealing with the material that has been exposed to an electromagnetic field.

The tests carried out to study the physical and mechanical properties of the materials resulted in a reliable increase in the adhesive strength of the materials after electromagnetic field action.

The results obtained in the course of the study promote further investigation of the regularities of filling material physical and mechanical properties changing under electromagnetic field influence. These findings may contribute to increasing the service life of filling materials and preventing the occurrence of secondary caries, which positively affects the quality of treatment on the whole.

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Author contributions NSM substantially contributed to the conception and design, acquisition of the data, and analysis and interpretation of the data, carried out the scientific studies, participated in the sequence alignment, conceived the study, participated in its design and coordination and drafted the manuscript. AAK participated in its design and coordination and drafted the manuscript. All authors read and approved the final manuscript.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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