



Article

Study of the Properties of Zinc Phosphate Composite Cement Modified with Phosphorus Slag

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Abstract

This paper presents an analysis of the physicochemical and biological properties of the developed composite zinc phosphate cement modified with bismuth oxide and phosphorus slag additives. The powder phase was synthesized by sintering a frit with an optimal composition (ZnO, MgO, SiO₂, Bi₂O₃) using phosphorus slag as the active component. The study included an assessment of the microstructure, chemical resistance in aggressive environments (5% NaCl solution, 10% lactic acid, carbonated water), solubility in artificial saliva, and cytotoxicity in human fibroblasts. The addition of phosphorus slag was found to promote the formation of low-melting eutectics, which reduces the sintering temperature by 100 °C and increases the material's whiteness to 97.8%. X-ray diffraction analysis confirmed the presence of zincite, quartz, and periclase phases, forming a dense microstructure without pronounced pores or cracks. The experimental cement demonstrated high acid resistance: the maximum weight loss in lactic acid was 8%, while the leaching of toxic elements (Pb, As, Cr, etc.) remained extremely low (10–67 ppm), confirming the material's environmental safety. Testing of the composite zinc phosphate cement in artificial saliva revealed minimal weight loss compared to similar products. Biological testing showed that the cement's cytotoxicity is dose-dependent; at a 0.3 g dose and a 1:4 dilution, the material loses its toxic properties and becomes safe for living tissue. The developed zinc phosphate composite cement composition offers improved aesthetic and mechanical properties, high chemical stability, and biocompatibility at working concentrations, making it promising for use in clinical dentistry.

Keywords: phosphorus slag; composite material; frit; zinc phosphate cement; chemical resistance; solubility; cytotoxicity



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1. Introduction

Composite materials are modern materials consisting of two or more components combined into a single system while maintaining their properties and used for specific purposes [1]. The production of composite materials essential for dentistry is rapidly

developing worldwide [2]. Zinc phosphate composite cement is one of the oldest and most widely used materials in orthopedic dentistry as a binder for cementing crowns, bridges, and other fixed structures. The main reasons for its widespread use in practice are its excellent properties, including its ability to short setting time [3].

In 2025, the dental cements market size reached US\$1.54 billion. By 2030, this figure is projected to increase by 5.7% and reach US\$ 2.04 billion [4].

Kazakhstan does not produce any types of composite materials for dentistry. Therefore, the country is entirely dependent on imports of these products. This translates into annual costs for the national economy of 3.0–3.5 billion tenge. In 2025, the cost of dental services in the country increased by an average of 13.8%, with total expenditures on dental services by the population of Kazakhstan reaching 209.9 billion tenge, a 1.5-fold increase compared to 2024 [5]. Therefore, developing the composition and organizing the production of zinc phosphate composite cement, widely used in dentistry, is a highly pressing issue.

Many scientists are currently researching ways to improve the quality of zinc phosphate cements. Eslami H. et al. [6] investigated the effects of akermanite and hardystonite nanoparticles in zinc phosphate cement. The scientists synthesized nanoparticles mechanically and then added them to the cement at a concentration of 5–15%. This resulted in a decrease in porosity and an increase in the compressive strength of the material from 90 to 120 MPa. The modified composition showed a 15% improvement in cell survival and a 20% improvement in proliferation, confirming biocompatibility. These results indicate the potential of using nanoceramic fillers to enhance the quality of zinc phosphate cement used in dentistry and orthopedics.

Choe Y.E. et al. [7] studied the properties of zinc phosphate cement with the addition of copper-doped bioglass nanoparticles. Cu-BGn nanoparticles were produced, and their optimal concentration in the cement was determined at 2.5%. Then, the strength and biological properties of the viability of human dental pulp cells were evaluated. The results show that the introduction of copper-doped bioglass Cu-BGn nanoparticles into the cement improved the antibacterial properties and the effect on human pulp cells in vitro. The total hardening time was 2.5–8.0 min. This modification is aimed at expanding the functional capabilities of zinc phosphate cements beyond mechanical fixation.

Choon-Keun Park showed [8] that the addition of 2% SiO₂ to the batch reduces the frit sintering temperature to 1150 °C. It was found that the hardening reaction of zinc phosphate cement depends significantly on the Al³⁺ ion content in the liquid phase. This leads to the formation of amorphous aluminum phosphate and slows crystallization. The reaction involves the interaction of Al³⁺ and H₂PO₄[−], rather than simple neutralization. Hardening is determined by the formation of an amorphous gel, which improves the cement structure [9].

Romanenko A.A. et al. [10] analyzed the effect of the mixing fluid density (H₃PO₄) on the final strength of zinc phosphate cement. With a fluid density in the range from 1.680 to 1.710 g/cm³, the cement had a stable working time of 105 to 115 s, and a hardening time of 300 to 330 s. In the range of fluid density from 1.70 to 1.71 g/cm³, a decrease in the structuring time of the set cement from 330 to 300 s and a decrease in compressive strength from 146.9 MPa to 133.4 MPa were observed. When reaching a fluid density of 1.69 to 1.71 g/cm³, the maximum strength value was recorded.

Dwiputri D.R. et al. [11] conducted a comparative analysis of the influence of the expiration date of zinc phosphate cements on the compressive strength indicators. According to the results, strength cannot correspond to a shelf life of 67.49 MPa at 2 years and 5 months, a shelf life of 66.84 MPa at 11 months, and a shelf life of 57.24 MPa at 2 months. All tested cements performed below the ISO standard (70 MPa). The study confirmed that using zinc

phosphate cement shortly before its expiration date leads to a significant deterioration in its mechanical properties.

Gangavane S. et al. [12] studied two types of cements (zinc phosphate and zinc polycarboxylate) for tensile luting. One hundred freshly extracted maxillary first premolars were used for the study. In the first group, 50 teeth were treated with zinc phosphate cement, while in the second group, 50 teeth were treated with zinc polycarboxylate cement. The obtained results showed that the samples of the first and second groups showed an average tensile strength of 4.52 MPa and 3.56 MPa, respectively. It was found that the average tensile strength of zinc phosphate cement exceeds the tensile strength of zinc polycarboxylate cement.

Chaitra Devi T.M. et al. [13] presented the results of a comparative analysis of the tensile strength of two classic cements—zinc phosphate and zinc polycarboxylate cements. One hundred extracted human maxillary first premolars were used in the experiment. According to the study results, the average tensile strength of zinc phosphate cement was 2.31 MPa, while that of zinc polycarboxylate cement was 2.09 MPa. The results of the study allow us to conclude that zinc phosphate cement provides higher tensile strength of fixation compared to zinc polycarboxylate cement and remains a reliable standard in terms of mechanical stability of fixation.

Li H.L. et al. [14] noted that manual mixing methods affect the compressive strength of dental zinc phosphate cement. The cement was mixed under identical conditions by three nurses. The mixed cement was placed in 5 mm diameter and 10 mm high cylinders, and the compressive strength was measured after hardening. The best value for mixing using the bidirectional rotation method with alternating forward and backward was 106.11 MPa. The strength was lower when mixing using the pull-and-push methods and amounted to 77.57 MPa. The lowest strength, which was 54.41 MPa, was shown when mixing using the unidirectional rotation method.

Safwat E.M. et al. [15] investigated the properties of an experimentally prepared zinc phosphate cement powder. Its working time, setting time, film thickness, compressive strength, and solubility were studied. The experimental results showed good performance comparable to cement requirements, but the compressive strength was significantly lower (42.09 MPa) than the ANSI/ADA specification (70 MPa).

Jabri M. et al. [16] investigated the setting mechanism and mechanical hardness of zinc phosphate cement. They described in detail the stages of raw material preparation, mixing, introduction of the solid phase of cement into the liquid phase, and the cement hardening process. The results showed that powder size affects the setting rate of the cement and demonstrate that aluminum phosphate (AlPO_4) has a significant effect on the hardening and setting time of the cement. X-ray diffraction analysis showed that the main phase of the hydration product is the mineral hopeite. The mineral is present in a mixture of the initial reagents and other secondary products, which are zinc phosphate salts in primary and secondary forms—zinc tetrahydrogen phosphate $\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$.

Tarjányi T. et al. [17] investigated the mechanical properties of various dental cements. They compared six types of cements for the final luting of restorations. The performance of zinc phosphate cement was comparable to that of polycarboxylate and glass ionomer cements (400–800 N), but significantly inferior to composite and hybrid cements, which withstood loads exceeding 1500 N. The average number of cycles to failure was 4507–4567, significantly lower than that of composite materials, most of which withstood 10,500 cycles. Scientists consider the advantages of zinc phosphate cement to be its long history of use and relatively low cost, and recommend its use in areas with low mechanical loads and for unaesthetic restorations.

Mirzaei R. et al. [18] conducted a study to compare the antibacterial effect of three types of Iranian and foreign-made cements (zinc phosphate cement, glass ionomer, and polycarboxylate) and two cariogenic standard bacterial strains, *Streptococcus mutans* and *Subrinus*. The study used the direct contact method of bacterial cultivation in an Agar medium. The effectiveness was assessed by the diameter of the inhibition zone (absence of bacterial growth). In both types of nutrient media containing *Streptococcus mutans* and *Streptococcus subrinus* bacteria, a significant difference was observed in terms of the zone of inhibition of bacterial growth. In general, the average diameter of the inhibition zone was larger for Iranian polycarboxylate compared to its foreign counterpart. Glass ionomer cement demonstrated a better antibacterial effect compared to zinc phosphate and polycarboxylate cements. The three tested cements also demonstrated a greater antibacterial effect against *S. sobrinus* than against *S. mutans*. The Iranian polycarboxylate demonstrated stronger inhibition of both bacteria compared to the foreign-produced product. The antibacterial activity of zinc phosphate cement is associated with a low initial pH at the time of mixing, but this effect diminishes as the acid is neutralized.

A group of researchers [19] developed a new type of cement by converting the mineral hopeite in hardened zinc phosphate cement into biocompatible hydroxyapatite. Pure zinc phosphate cement powder was treated with a calcium nitrate ($\text{Ca}(\text{NO}_3)_2$) solution at various temperatures of 25, 60, and 90 °C. The hardened cement samples were immersed in simulated biological fluid for 2–4 weeks to simulate conditions inside the body. It was found that when exposed to calcium nitrate, hopeite first converted to the mineral scholcite, and then, after 4 weeks, hydroxyapatite crystals were fixed on the surface of the cement. This demonstrated the possibility of converting hopeite in hardened cement into hydroxyapatite, a mineral that forms the basis of human bone.

Japanese scientists presented the results of a unique study, covering an observation period of up to 43 years, dedicated to assessing the longevity of dental restorations cemented with zinc phosphate cement. A total of 454 restorations (inlays, crowns, and bridges) were analyzed. In 53 patients, 189 restorations were made using zinc phosphate cement. The study showed that restorations cemented with zinc phosphate cement were preserved in 43.5% of cases; secondary caries occurred in 29.6% of cases; the need for root canal treatment after cementation was 15.3%; the risk of restoration loss was 10.6%. The obtained results confirm the need to improve the effectiveness of zinc phosphate cement in order to increase the shelf life of screw-retained teeth and prevent critical complications [20].

The use of phosphorus slag in frit powder and the study of its effect on the properties and quality of zinc phosphate cement is a current trend in the development of dental composite materials worldwide, and particularly in Kazakhstan.

The aim of this study is to improve the properties of zinc phosphate cement obtained with the addition of phosphorus slag and to investigate the characteristics of the resulting composite material.

The novelty of this work lies in the creation of a specific dental system, where phosphorus slag acts not simply as a flux but as an active component that forms a biocompatible phosphate matrix in the presence of phosphoric acid. It was the synergy of the fluorides contained in the slag and the mineralizing action of Bi_2O_3 that made it possible to achieve a unique combination of low synthesis temperature and high chemical resistance, which had not previously been described for dental materials based on human-made raw materials from Kazakhstan.

The use of phosphorus slag in composite zinc phosphate cements aims to simultaneously address technical, economic, and environmental issues.

The main hypotheses and scientific premises for improving the properties of zinc phosphate cements with the addition of phosphorus slag are as follows:

1. Phosphorus slag contains trace elements (particularly fluorides), which activate frit sintering processes and subsequently participate in the formation of the zinc phosphate cement structure. Ions released from the phosphorus slag form a complex phosphate matrix.
2. Minor components of slag interact with both the original components of cement and with the products of the interaction of cement with orthophosphoric acid, forming secondary phases.
3. Zinc phosphate cement obtained with the addition of phosphorus slag is characterized by a high degree of sintering, a dense microstructure, and the absence of significant pores and cracks, which contributes to increased chemical resistance when exposed to aggressive environments—5% NaCl solution, 10% lactic acid solution, and carbonated water. Acid resistance is enhanced by the formation of a less soluble amorphous Zn-Al phosphate gel in a highly buffered environment. Furthermore, a small addition of phosphorus slag and Bi_2O_3 to the powder phase promotes the formation of a denser structure with lower porosity and increased resistance to the diffusion of aggressive ions.
4. The use of phosphorus slag, a chemical production waste, not only reduces environmental impact and greenhouse gas emissions but also contributes to the development of a circular economy.

2. Materials and Methods

2.1. Initial Materials

Zinc phosphate composite cement is intended for the luting of inlays, pin structures, metal, plastic, porcelain, and metal–ceramic crowns and bridges, for filling teeth to be covered with crowns, and as an insulating liner during dental fillings. Zinc phosphate composite cement consists of a two-component system: a powder and a liquid [21].

In this study, the powder used was a sintered zinc phosphate cement frit, the composition of which we had previously developed [22]. The experimental frit was a highly dispersed product consisting of 83% zinc oxide, 9% magnesium oxide, 3.5% silicon oxide, 3% bismuth oxide, and 1.5% phosphorus slag, fired at 1000 °C. The whiteness of the resulting experimental zinc phosphate cement powder frit was 97.8%.

A specially prepared phosphoric acid solution was used as a liquid component. The solution was a liquid consisting of 57% phosphoric acid (H_3PO_4) and a partially neutralized solution containing 3% aluminum phosphate (AlPO_4) and 9% zinc phosphate ($\text{Zn}_3(\text{PO}_4)_2$) as dissolved salts, with the remaining 31% being distilled water. This is a classic buffered liquid composition for dental zinc phosphate cement.

Mixing the powder with the liquid causes a chemical reaction accompanied by an exothermic effect, which facilitates the setting of the composite cement. The powder-to-liquid ratio obtained in the experiment was as follows: 2 g of powder to 0.42 mL of liquid. The total mixing time for the composites is 60 ± 10 s. Setting begins after 2–6 min, and the final setting time is 9 min [23].

2.2. Instrumental Methods of Analysis

X-ray fluorescence spectroscopy (XRF), X-ray diffraction (XRD), and electron microscopy were used to conduct this study, as well as methods for determining the quality of the resulting composite zinc phosphate cement.

2.2.1. Thermodynamic Calculation

Thermodynamic calculations were performed using the HSC Chemistry 6 software package. The software package used in the calculations is based on the ideology of the

European consortium SGTE (Scientific Group Thermodata Europe, Stockholm, Sweden), designed to develop, maintain, and disseminate high-quality databases. SGTE includes specialized scientific centers in Germany, Canada, France, Sweden, the United Kingdom, and the United States. The software package database contains information on 22,000 individual substances [24].

2.2.2. X-Ray Fluorescence Spectrometry (XRF)

The chemical composition of the samples was determined using energy-dispersive X-ray fluorescence (EDXRF). Samples were ground to an analytical powder and pelletized, using a 4% aqueous solution of polyvinyl alcohol as a binder. An XL5 spectrometer (Thermo Niton) from Thermo Scientific Portable Analytical Instruments, Inc. (Tewksbury, MA, USA) was used to determine a wide range of elements. The analyzer is equipped with an end-window X-ray tube (Ag anode, U 6–50 kV, I_{\max} 500 μ A, W_{\max} 5 W), a set of 4 primary radiation filters, 3–8 mm collimators, and a geometrically optimized large-area drift detector (GOLDD, count rate over 180,000 cps, resolution up to 130 eV at 5.9 keV). The NDT software (version 4.4) enables automated spectral processing using fundamental parameter methods and Compton normalization. Empirical calibrations obtained by analyzing standard samples with the corresponding matrix and elemental content range can also be used. The total duration of sample analysis was 120 s. Each sample was analyzed in duplicate, and the error under repeatability conditions did not exceed 3%.

Quantitative determination of light elements (Na, Mg, Al, Si, P, S, Cl) was carried out using an analytical complex based on an Ekros XRF-9700 X-ray fluorescence energy-dispersive spectrometer from EKROSKHIM LLC (St. Petersburg, Russian Federation), purchased under the Development Program of Lomonosov Moscow State University. The spectrometer is equipped with an end-window X-ray tube (Rh anode, U 5–50 kV, I_{\max} 2000 μ A, W_{\max} 50 W), a set of 10 primary radiation filters, 1–15 mm collimators, a silicon drift detector (SDD) with a graphene window (count rate over 300,000 cps, resolution up to 125 eV at 5.9 keV), helium and vacuum post (VRD, Value Vacuum pump). Measurements were performed in vacuum at an accelerating voltage of 8 kV, a current of 2000 μ A, for 300 s. Calibration construction and spectra processing were performed in the XRF-PRO software package. The error under repeatability conditions did not exceed 2%. Additionally, the loss on ignition (LOI) of the samples was estimated by ashing a sample portion dried at 105 °C in a muffle furnace at 900–950 °C to constant weight [25].

2.2.3. X-Ray Diffraction (XRD) Analysis

X-ray diffraction data for the zinc phosphate cement frit and stone were obtained using a Rigaku MiniFlex 600 powder diffractometer (Rigaku Corporation, Tokyo, Japan) with Cu $K\alpha$ radiation. Data were collected in continuous scanning mode over a 2θ range of 6° to 70° with a step size of 0.02°. Data processing was performed using the WINXPow software package (version 4.02), and phase identification was performed using the PDF 1 and PDF 2 databases [26].

2.2.4. Scanning Electron Microscopy (SEM) Analysis

Electron microscopy is performed by scanning the surface of the zinc phosphate cement frit and stone sample with an electron probe and recording the broad spectrum of radiation emitted during the process. The signals used to obtain images in an electron microscope include secondary, reflected, and absorbed electrons. Electron microscopy studies were performed using a JSM-6490LV scanning electron microscope (JEOL, Tokyo, Japan). This instrument is equipped with field-effect electron guns operating under ultrahigh vacuum conditions (up to 10^{-8} Pa), which provide sufficient current for a small-diameter

detector (0.15–0.35 nm), with an electron-optical magnification range from $\times 100$ to $\times 60,000$ and an accelerating voltage of up to 100 kV [27].

2.2.5. Study of the Chemical Resistance of Zinc Phosphate Cement Under Aggressive Environments

To determine the chemical resistance of zinc phosphate cement under aggressive environments, three $20 \times 20 \times 20$ mm specimens are prepared. The specimens are prepared using 100 g of frit powder and 21 mL of an aqueous solution of orthophosphoric acid, which acts as a binding agent for the frit powder. After stirring for 60 s, the mold is filled with cement paste. To seal the paste and prevent the formation of gaps and pores, the mold is filled with zinc phosphate cement in large portions, compacted with a spatula, and then the specimens are pressed. Excess cement paste is removed from the top of the mold. One hour after molding, the ends of the cylindrical specimens are ground to obtain a smooth surface perpendicular to the longitudinal axis of the specimen. Then, 50 mL of the corresponding solutions are poured into 100 mL beakers. Three samples are simultaneously immersed in containers containing a 10% lactic acid solution, a 5% sodium chloride solution, and carbonated water for 1 h. After the specified time, the samples are removed, rinsed with distilled water, and assessed visually and using an optical microscope. Following visual and microscopic analysis, the samples are dried in a drying oven for 1 h at 37 °C, after which they are tested for compressive strength using a PGM-100MG4 press [28].

2.2.6. Study of the Solubility of Zinc Phosphate Cement in Artificial Saliva

To determine the solubility of zinc phosphate cements in artificial saliva, six samples measuring $20 \times 20 \times 20$ mm were prepared. The sample preparation method is presented in Section 2.2.4. After hardening, the samples were weighed on an electronic scale with a measurement accuracy of 0.0001 g. The samples were then placed in a container with artificial saliva, the chemical composition of which was prepared by dissolving the following reagents in 1 L of distilled water:

- Sodium chloride (NaCl)—0.4 g;
- Potassium chloride (KCl)—0.4 g;
- Calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$)—0.795 g;
- Sodium dihydrogen phosphate dihydrate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$)—0.78 g;
- Sodium bicarbonate (NaHCO_3)—1.0 g.

The pH of the prepared artificial saliva was 6.9.

The test cube-shaped cement samples were removed from the artificial saliva after 24 and 48 h, and also after 5 days. After removal, the samples were carefully dried on filter paper and weighed on the same scale used for the initial weighing. This technique allows one to determine the presence of material dissolution, both during the crystallization stage and after final curing. The solubility of the cements in artificial saliva was expressed as the difference in the mass of the samples before placement in the medium and after their removal at specified time intervals [29].

2.2.7. Cytotoxicity Assay of Zinc Phosphate Composite Cement Research Methodology

Preparation of Dermal Fibroblasts for the Cytotoxicity Assay (MTT Assay). Human dermal fibroblasts (Gibco, Grand Island, NY, USA) were used for the MTT assay. They were pre-cultured in standard DMEM (Dulbecco's Modified Eagle Medium) supplemented with 10% fetal bovine serum (FBS) and 1% antibiotic/antimycotic solution. The cells were cultured at 37 °C and 5% CO_2 in a humidified incubator. Cultivation was continued until the desired cell density was reached, after which the cells were removed from the

surface of the culture flask by treatment with trypsin-EDTA solution. After incubation with trypsin, the cell suspension was resuspended in fresh culture medium. Cells were counted using an automated cell counter. After obtaining the required cell suspension concentration, fibroblasts were seeded into 96-well plates at a density of 7000 cells per well. This parameter was optimized to ensure uniform cell attachment to the well surface and to obtain representative data for viability analysis. After seeding, the cells were incubated for 24 h under conditions that ensured their attachment to the surface of the culture vessel. After the attachment stage was complete, the culture medium was carefully removed to minimize possible mechanical damage to the cell layer and replaced with fresh medium.

In each experimental series, phosphate-buffered saline (PBS), in which dental cement had been pre-incubated, was added to each well. The first well of each series was supplemented with PBS pre-incubated with zinc phosphate composite cement of different weights: 0.3 g and 0.7 g. Subsequent wells were supplemented with PBS obtained by serial dilutions of 1:2, 1:4, and 1:8. This approach allowed us to study the dose-dependent effect of zinc phosphate composite cement on cell viability. After adding PBS, the plates were again placed in an incubator under standard culture conditions to ensure interaction of the cells with the substances contained in the dental cement. The incubation duration and solution concentrations were selected based on preliminary experiments aimed at identifying optimal conditions for cell viability analysis. The plates prepared in this manner were used for the subsequent MTT assay [30].

Cytotoxicity Assay

The cytotoxicity assay was performed using the commercial MTT Assay Kit (Abcam, Cambridge, UK), a highly sensitive instrument for assessing cell proliferation and viability. The assay was performed on human dermal fibroblasts, pre-prepared and seeded in 96-well plates at a density of 7000 cells per well.

On the day of the experiment, a 12 mM MTT solution was prepared by dissolving 5 mg of MTT in 1 mL of sterile PBS. The solution was mixed using a vortex mixer or sonicated until completely dissolved. The prepared solution was stored at 4 °C, protected from light. An SDS-HCl solution was also prepared by dissolving 1 g of SDS in 10 mL of 0.01 M HCl, mixing by inversion or sonication until completely dissolved.

The culture medium in the plates was replaced with fresh medium (100 µL per well). For adherent cells, the old medium was removed by gentle aspiration, while for suspended cells, the pellet was centrifuged and resuspended in fresh medium. After replacing the medium, 10 µL of 12 mM MTT solution was added to the cells. As a negative control, only cell-free culture medium with the same volume of MTT solution was added to individual wells. The plates were incubated at 37 °C for 4 h in a humidified atmosphere with 5% CO₂.

After incubation, 100 µL of SDS-HCl solution was added to each well, ensuring uniform mixing by pipetting. The plates were incubated again at 37 °C for 4–18 h in a humidified atmosphere to dissolve the precipitated formazan. After incubation, the well contents were further mixed, after which the optical density of the samples was measured at a wavelength of 570 nm using a spectrophotometer. The obtained values were used to assess cell viability based on comparison with the negative control and statistical analysis of the data. Cytotoxic reactions were assessed as severe (30%), moderate (30–60%), mild (60–90%), and non-toxic (>90%) [31]. The *p*-value was calculated using MedCalc statistical software (Version 23.4.9) from MedCalc Software Ltd. (Ostend, Belgium) [32].

3. Results and Discussion

The process of producing a composite frit of zinc phosphate cement powder using phosphorus slag involves several key steps, beginning with the preparation of the starting

materials, component dosing, grinding and homogenization of the composition, and finally, frit firing at a high temperature of 1000 °C.

The thermodynamic analysis carried out in the ZnO–MgO–SiO₂–Bi₂O₃—phosphorus slag system showed that the formation of phases such as willemite (Zn₂SiO₄), forsterite (Mg₂SiO₄), and gahnite (ZnAl₂O₄) at temperatures of 1000–1100 °C has a high thermodynamic probability (negative Gibbs free energy ΔG values from –8.3 to –109.3 kcal). This scientifically substantiates the mechanism of matrix strengthening due to the formation of complex silicate and aluminate bonds [33]. Due to its fluoride content (F, CaF₂), the slag promotes the formation of low-melting eutectics, which allows for a reduction in sintering temperature by 100 °C (to 1000 °C). The high SiO₂ content in the slag (37.6%) ensures transparency and increases whiteness to 97.8%. Ca²⁺ and Al³⁺ ions from the slag participate in the formation of additional phosphate phases, compacting the structure. Bismuth oxide (Bi₂O₃) improves the smoothness and homogeneity of freshly mixed cement. In large quantities, it increases setting time, acting as an activator, accelerating strength gain during hardening.

The resulting frit was subjected to chemical composition determination. Chemical analysis of the frit composition was determined using an X-ray fluorescence spectrometer at Lomonosov Moscow State University. The chemical composition of the zinc phosphate composite cement frit (powder) is presented in Table 1.

Table 1. Chemical composition of zinc phosphate composite cement frit.

Material	Chemical Composition, wt.%														
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	MnO	F	P ₂ O ₅	ZnO	Cl	Total
Frit powder	2.56	0.52	0.025	1.03	7.05	0.02	0.76	0.01	0.014	0.004	0.02	0.69	82.93	0.011	95.644

According to the analysis results, the zinc phosphate cement frit contains more than 82.9% zinc oxide, indicating the formation of a large amount of the mineral zincite, more than 7.0% magnesium oxide, and more than 2.5% silicon oxide. The P₂O₅ content is 0.69%, and the chloride ion content does not exceed 0.1%. The concentration of trace elements in the zinc phosphate composite cement frit was also determined (Table 2).

Table 2. Concentration of microelements in the frit composition.

Material	Elements, ppm																	%		
	Cr	V	Co	Ni	Cu	Rb	Sr	Zr	Ba	U	Th	Y	Nb	Pb	As	Mo	Bi		La	Total
Frit powder	22	12	5	105	37	5	24	13	33	5	5	5	5	5	5	9741	33,523	10	43,560	4.356

As can be seen from the data in Table 2, the bismuth concentration is 3.35%, and the total concentration of 17 elements in the frit is only 1.006%, which does not negatively impact its subsequent use. X-ray diffraction analysis of the zinc phosphate composite cement frit was performed using a powder diffractometer at Lomonosov Moscow State University. The X-ray diffraction results are presented in Figure 1.

Figure 1 shows that the main minerals in the frit sample are zincite (ZnO), quartz (SiO₂), and periclase (MgO).

Electron microscopic analysis of the zinc phosphate composite cement frit was performed using a JEOL JSM-6490 LV microscope at M. Auezov South Kazakhstan University. A micrograph of the frit is shown in Figure 2.

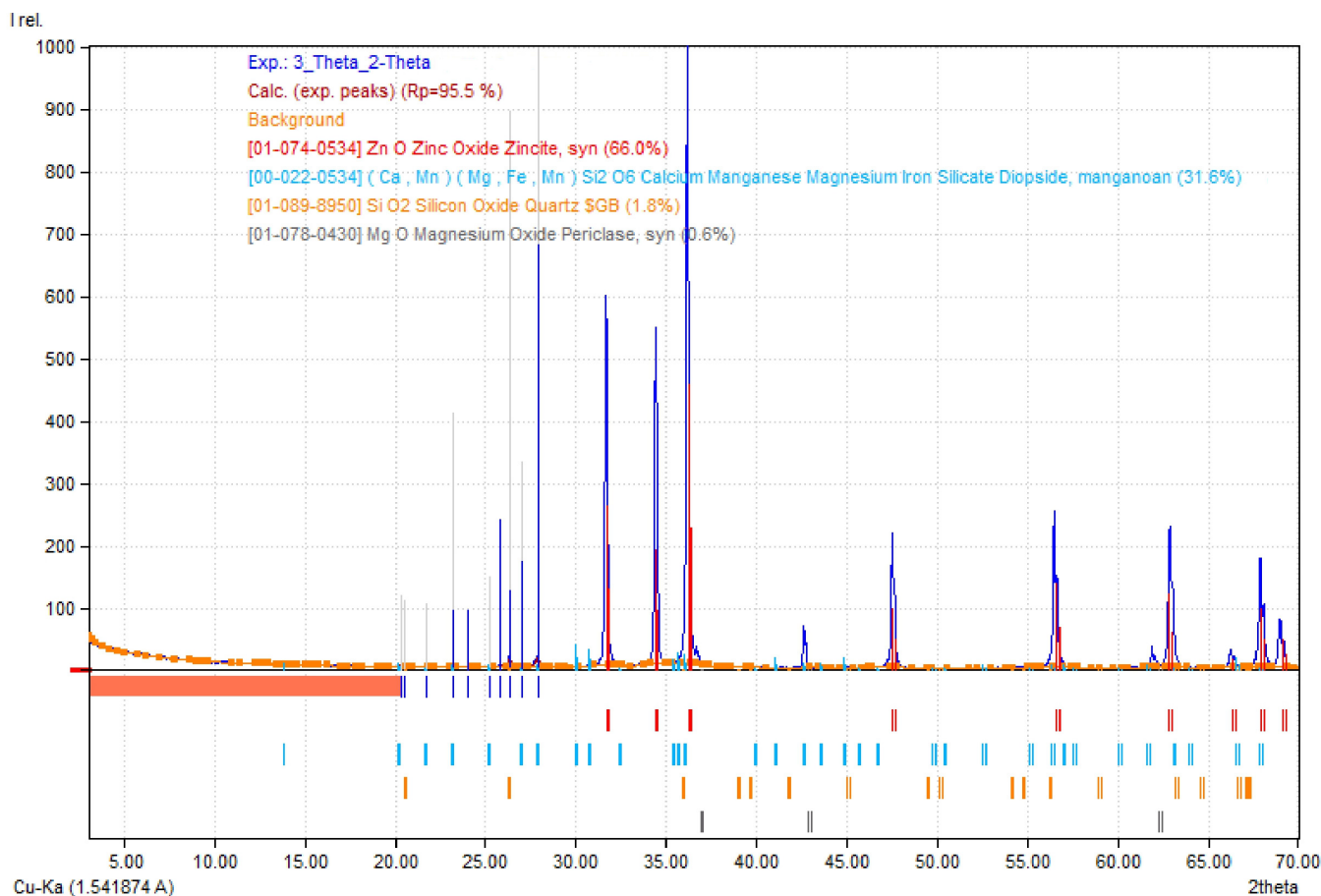


Figure 1. X-ray diffraction pattern of zinc phosphate composite cement frit.

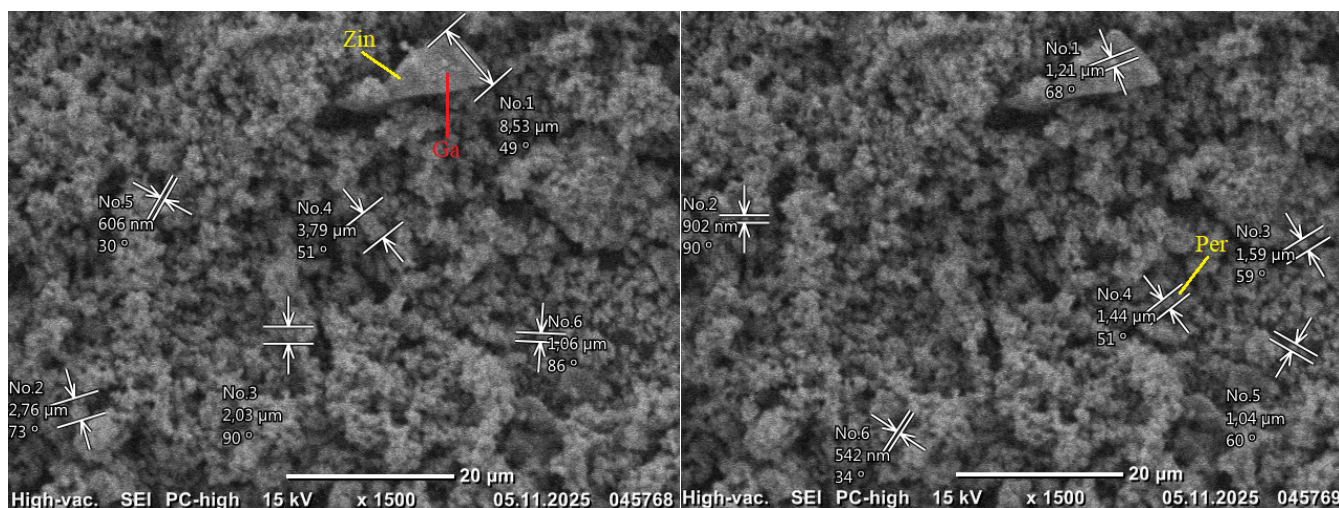


Figure 2. Electron microscopic analysis of zinc phosphate composite cement frit. Zin—zincite; Per—periclase; Ga—ganit.

The obtained results demonstrate that the zinc phosphate cement frit exhibits a high degree of sintering, a dense microstructure, and the absence of significant pores and cracks. This indicates sintering efficiency due to the participation of low-melting euthetics formed by Bi_2O_3 and phosphorus slag components (CaF_2 , F , SiO_2 , CaO). The addition of phosphorus slag and bismuth oxide ensured a significant reduction in the sintering temperature and the formation of a denser structure compared to traditional ZnO-MgO compositions, which require temperatures of 1100–1300 °C. The particle size is optimally in the range

from 0.5–4 μm to 10–20 μm, indicating efficient liquid-phase sintering processes. The main coarse-grained mineral is zincite, measuring 8.53 μm. Among the zincite minerals, there is the brightly colored gahnite, measuring 1.21 μm. The mineral periclase is represented by dark black rounded oval crystals with a size of 1.04–2.76 μm.

Thus, the use of phosphorus slag—a byproduct of phosphate fertilizer production and other chemical processes related to phosphate mining and processing—promotes not only the production of high-quality zinc phosphate composite cement, but also the development of a circular economy—a concept of renewable production and consumption that maximizes resource utilization, eliminating waste and reducing environmental impacts.

The resulting zinc phosphate cements were then tested to determine their chemical resistance in aggressive environments. A general view of the samples is shown in Figure 3.

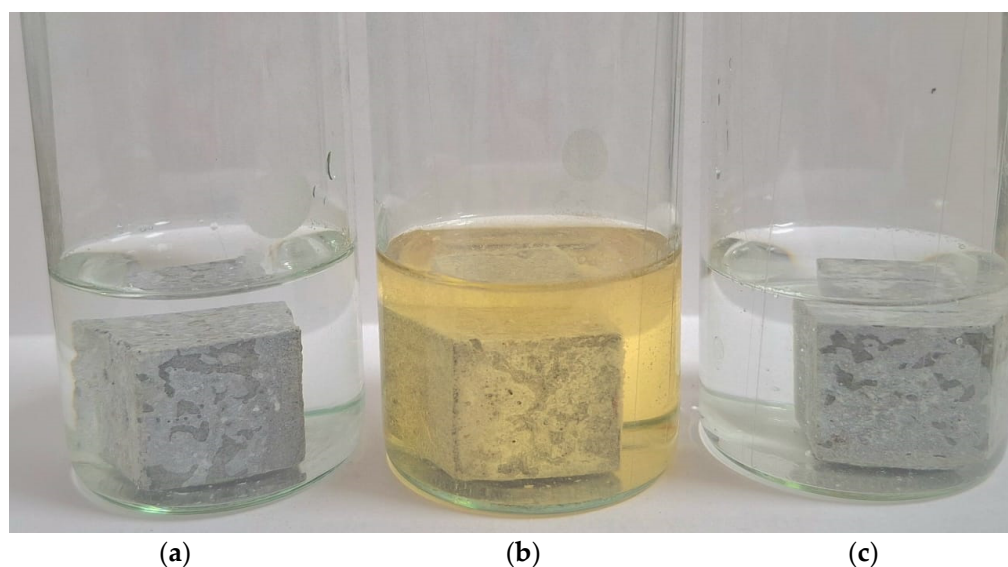


Figure 3. Zinc phosphate cement samples placed in solutions: (a) 5% sodium chloride; (b) 10% lactic acid; (c) carbonated water.

After the specified time, the samples were thoroughly washed, dried, and press tested. The results of the zinc phosphate cement compressive strength tests under aggressive environments are presented in Table 3.

Table 3. Properties of zinc phosphate cement under the influence of aggressive environments.

Name of Cement	Tensile Strength, MPa	Compressive Strength After Exposure to Aggressive Environments, MPa		
		5% Sodium Chloride	10% Lactic Acid	Carbonated Water
Zinc phosphate cement	107.4	100.1	98.8	104.4

An analysis of the strength properties revealed that, compared to the control sample (107.4 MPa), 10% lactic acid exhibited the greatest aggressiveness toward zinc phosphate cement. After exposure to this solution for 1 h, the compressive strength of the material decreased by 8%, reaching 98.8 MPa. Exposure to a saline solution resulted in a 6.8% decrease in strength, reaching 100.1 MPa. The least significant effect was observed when the samples were exposed to carbonated water, with a 2.8% decrease in strength and a final value of 104.4 MPa. It has been established that only two-thirds of the material’s final strength is achieved within the first hour after cementation. Exposure to oral fluid during this period can lead to increased permeability and structural degradation. Strength testing

after an “acid attack” during this period simulates the extreme conditions of early service. Even under these conditions, the experimental cement retained a strength of 98.8 MPa, significantly exceeding the standard requirement of 70 MPa.

The obtained data indicate varying degrees of destructive impact of the studied environments on the structure of zinc phosphate cement, with the most pronounced decrease in mechanical strength observed under conditions of exposure to an organic acid.

Following compression testing, all set cement samples were subjected to chemical composition determination using an X-ray fluorescence spectrometer. The chemical composition and elemental concentrations of set zinc phosphate cement after exposure to aggressive environments are presented in Tables 4 and 5.

Based on the chemical composition of zinc phosphate cement exposed to aggressive environments, it was found that lactic acid causes the greatest phosphorus (P_2O_5) leaching, from 27% to 23.21%, and a relative enrichment of ZnO to 56.97%. The matrix loses its phosphate component, leaving behind more zinc oxide and insoluble Zn compounds. CaO increases to 1.27%, resulting in the formation of more stable calcium phosphate phases or the accumulation of calcium from phosphorus slag. Lactic acid exhibits the lowest loss on ignition—12.56%—and fewer volatile components. 5% NaCl and carbonated water have a much milder effect—their compositions are almost identical. Neutral and slightly acidic environments do not affect the chemical composition of the set cement. Losses are minimal, confirming its high resistance to saline and slightly carbonic environments. ZnO is relatively stable, and the basic matrix is preserved. CaO is not washed out of the phosphorus slag, but rather accumulates in the acid (0.21%). CaO and fluorides from the slag, as well as Al^{3+} and Zn^{2+} buffers from the liquid, significantly reduce leaching of the phosphate matrix, which explains the low strength loss (6.8–8.0%).

The concentration of toxic elements (Pb, As, Cr, Ni, Th, etc.) remains very low (10–67 ppm) and does not increase after exposure. The material is safe in terms of heavy metal leaching (Table 5).

The results of X-ray diffraction analysis of set zinc phosphate cement after exposure to aggressive environments are presented in Figure 4.

Figure 4 shows that the main minerals in the samples are hopeite ($Zn_3(PO_4)_2 \cdot 4H_2O$), zinc hydrogen phosphate trihydrate ($Zn_3(HPO_4) \cdot 3H_2O$), amorphous Zn-Al phosphate gel, amorphous silicate glass residues ($SiO_2 \cdot nH_2O$), and magnesium-containing phases.

Scanning electron microscopy results of set zinc phosphate cement confirm these findings (Figure 5).

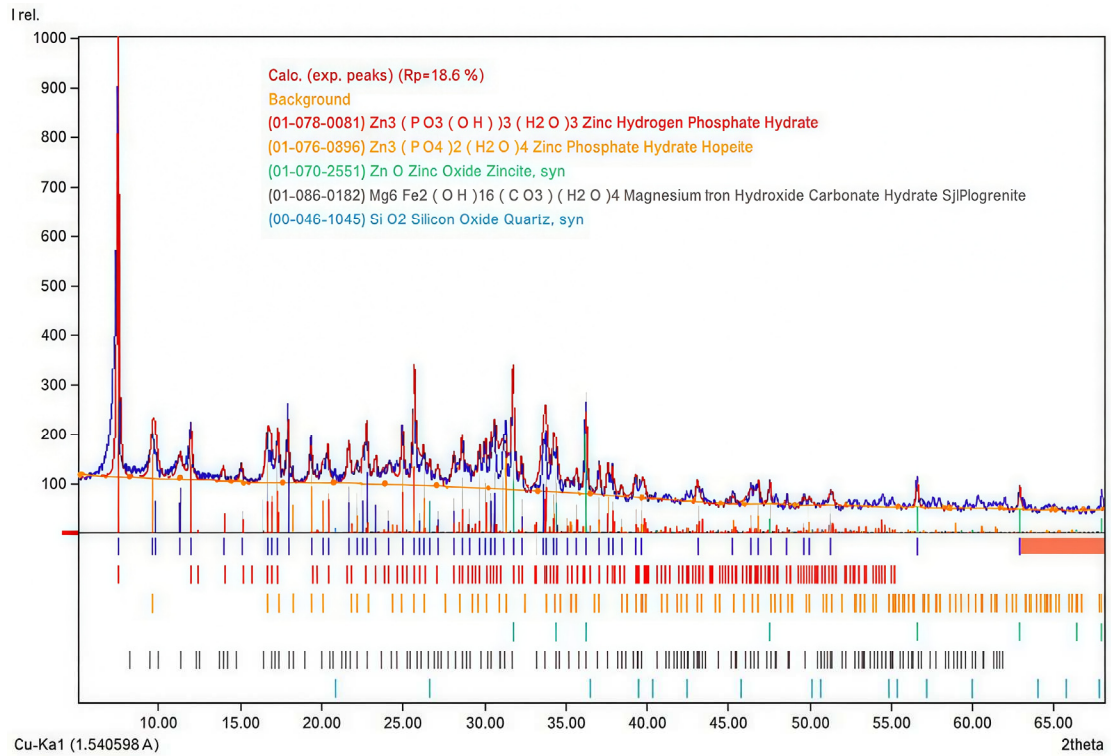
The presented micrographs show the surface of hardened cement paste, consisting of a typical zinc phosphate cement structure: unreacted ZnO covered by a matrix of amorphous Zn-Al phosphate gel and crystalline phases. The matrix consists of amorphous zinc–aluminum phosphate gel—a dense, glassy gel that fills the spaces between particles. The gel appears as a continuous background with a fine-grained texture. Hopeite crystals ($Zn_3(PO_4)_2 \cdot 4H_2O$) are clearly distinguishable as elongated, plate-like, or prismatic crystals (size 0.5–9 μm , most often 1–4 μm). They have a characteristic lamellar/acicular morphology, often with sharp edges and corners (angles measured 30–90°). The crystals protrude above the surface or lie in the plane of the matrix. Amorphous remnants of silicate glasses (from $SiO_2 \cdot nH_2O$ and phosphorus slag components) appear as small, rounded, or irregular inclusions 0.5–2 μm in size, with a smoother surface than hopeite. Magnesium-bearing phases appear as small (0.5–1.5 μm), rounded or submicron particles, often in the form of agglomerates within the gel or on the surface of unreacted ZnO. Unreacted ZnO cores are large (up to 8–9 μm), angular or subrounded particles with a smoother surface, covered by a thin layer of gel and crystals. The surface is generally dense, with low visible porosity (pores < 1 μm , rare).

Table 4. Chemical composition of set zinc phosphate cement after exposure to aggressive environments.

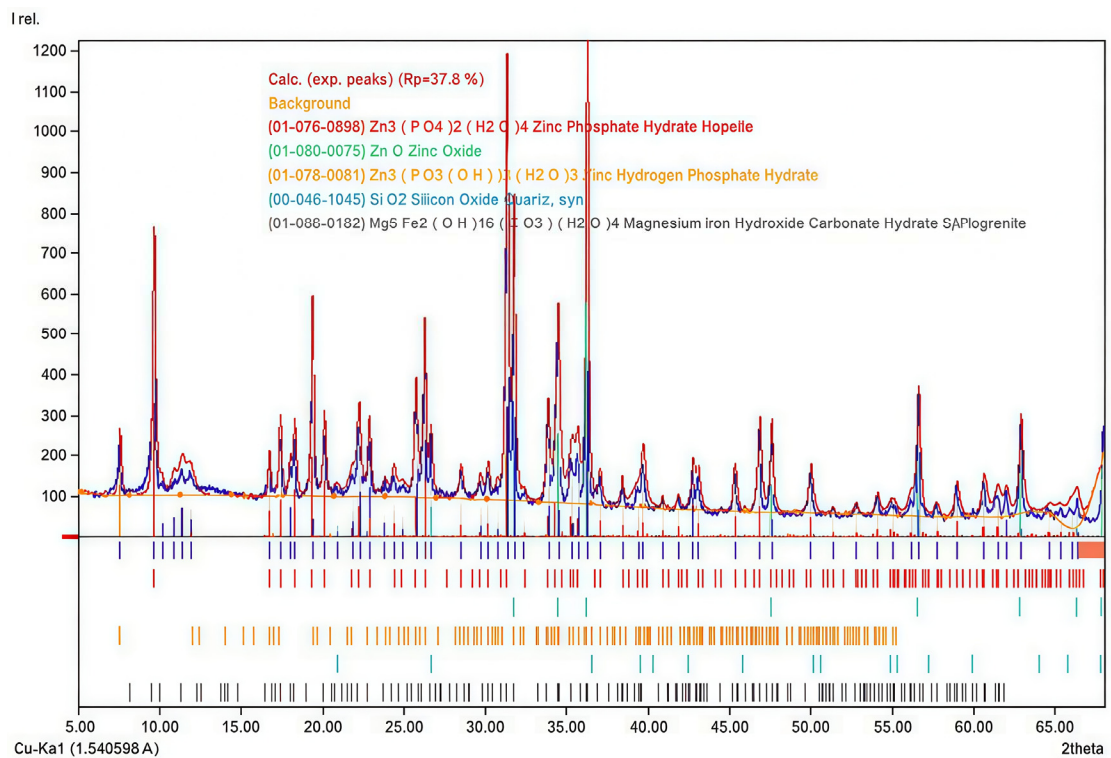
Material	Chemical Composition, wt.%															
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	MnO	F	P ₂ O ₅	ZnO	Cl	LOI	Total
5% sodium chloride	2.21	0.21	0.11	0.71	3.10	0.10	-	0.012	0.005	0.010	0.02	27.11	50.54	0.008	15.82	99.965
10% lactic acid	2.25	0.11	0.14	1.27	3.32	0.08	-	0.012	0.005	0.012	0.02	23.21	56.97	0.008	12.56	99.967
carbonated water	2.25	0.15	0.14	0.66	2.99	0.10	-	0.012	0.005	0.010	0.02	26.72	51.02	0.008	15.88	99.965

Table 5. Concentration of elements in set zinc phosphate cement after exposure to aggressive environments.

Material	Elements, ppm																		%	
	Cr	V	Co	Ni	Cu	Rb	Sr	Zr	Ba	U	Th	Y	Nb	Pb	As	Mo	Bi	La		Total
5% sodium chloride	17	14	10	43	30	10	19	10	50	10	10	10	10	10	10	10	10	67	350	0.035
10% lactic acid	20	24	10	53	30	10	29	10	50	10	10	10	10	10	10	10	10	14	330	0.033
carbonated water	17	13	10	43	30	10	23	10	50	10	10	10	10	10	10	10	10	64	350	0.035



(a)



(b)

Figure 4. Cont.

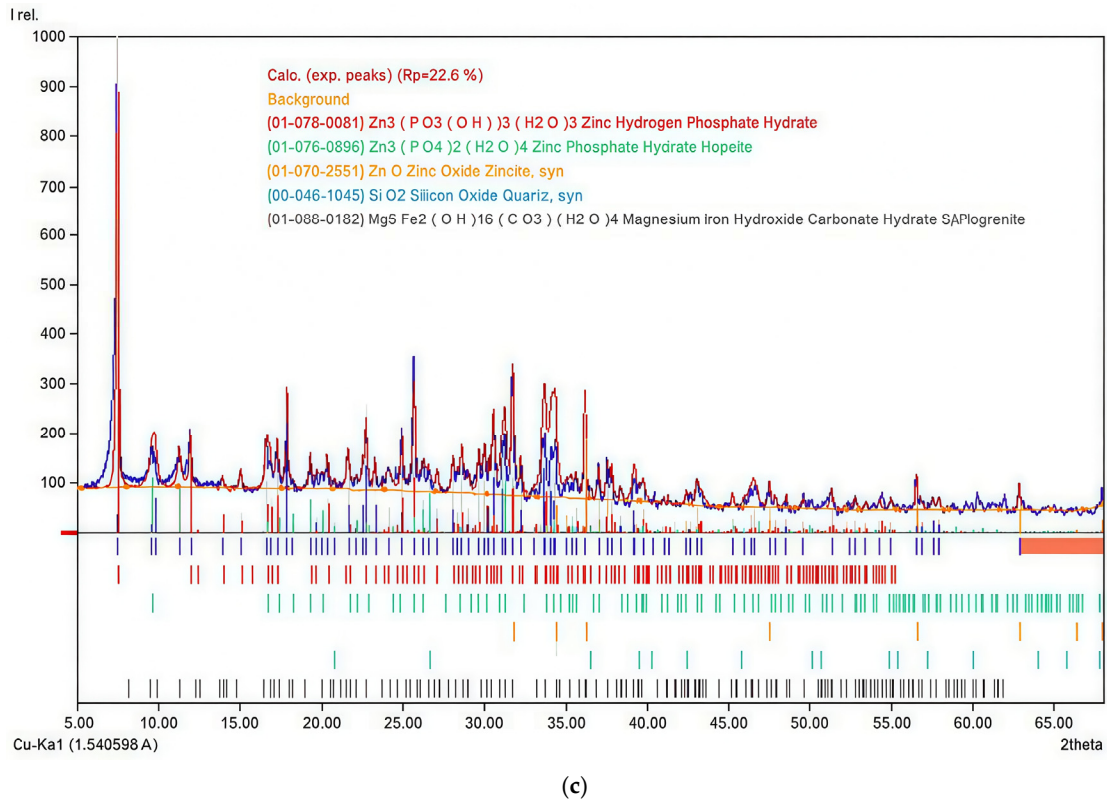


Figure 4. X-ray diffraction patterns of zinc phosphate cements after exposure to aggressive environments: (a) 5% sodium chloride; (b) 10% lactic acid; (c) carbonated water.

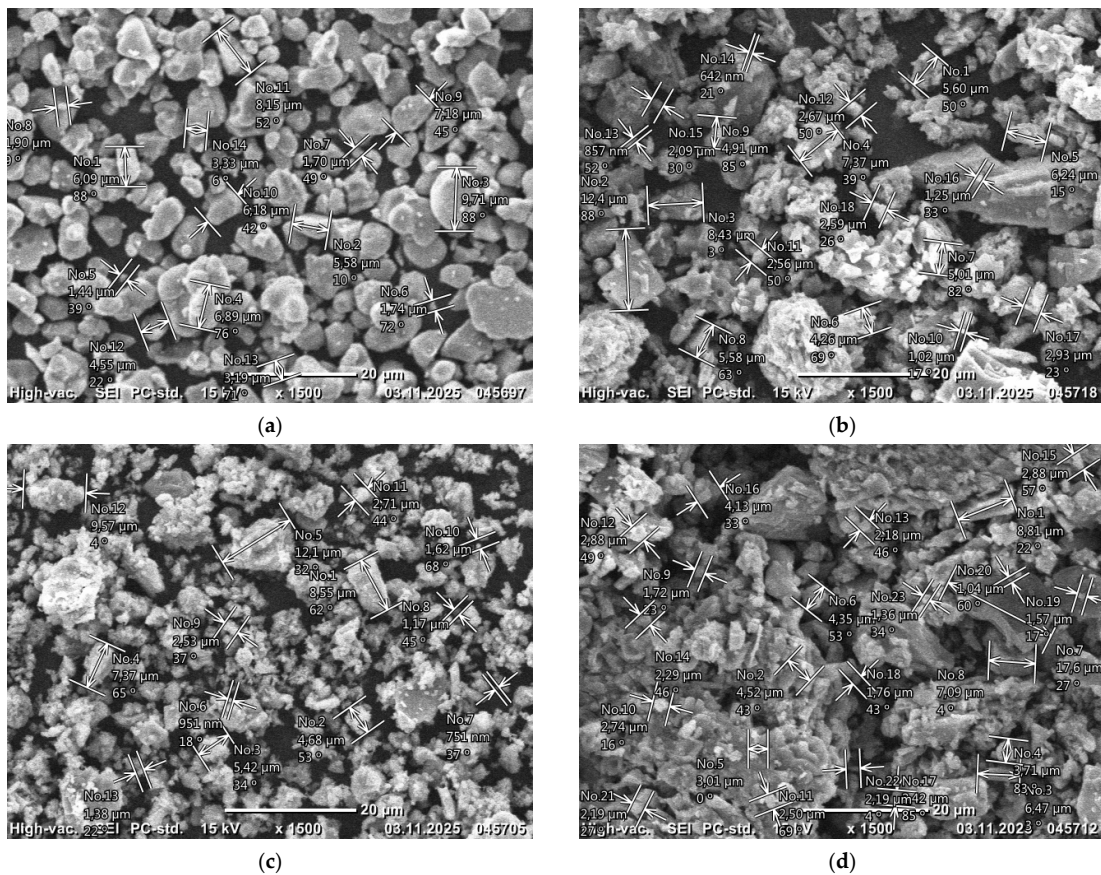


Figure 5. Micrographs of set zinc phosphate cement: (a) control sample; (b) 5% sodium chloride; (c) 10% lactic acid; (d) carbonated water. Magnification: 1500×.

The control sample (without aging) is characterized by the densest and most homogeneous structure. Hopeite crystals are numerous, 0.5–3 μm in size, well-formed, and protruding above the surface. The amorphous gel is continuous, filling the entire space. Magnesium phases and silicate residues are uniformly distributed. The surface is smooth, with no signs of erosion.

The structure of the sample after aging in 5% NaCl is almost identical to the control. Hopeite crystals retain their shape and size (0.5–5 μm), and the same plate-like crystals with sharp edges are visible. The amorphous gel remains dense. There is a slight increase in visible roughness or small depressions (localized leaching), but no pronounced erosion. Silicate residues and magnesium phases are unchanged. The overall state of preservation is high; a neutral saline environment has virtually no effect.

The most noticeable changes are visible in the sample structure after storage in 10% lactic acid; the surface becomes rougher and more porous. Hopeite crystals are partially dissolved or reduced in size (many crystals are 0.5–2 μm in size, with smoothed edges and some disappearing). The amorphous gel appears corroded, with small depressions and cavities (1–5 μm in size) appearing, exposing ZnO cores. The number of small crystalline fragments and debris increases (possibly remnants of hopeite or zinc hydrogen phosphate trihydrate phases ($\text{Zn}_3(\text{HPO}_4)\cdot 3\text{H}_2\text{O}$)). Magnesium-containing phases and silicate residues are better preserved (they are more stable in acid). The overall effect is surface erosion, but without deep destruction (strength loss of only 8%).

The structure of the sample after storage in carbonated water is characterized by minimal changes and is similar to the structure of the sample stored in NaCl. Hopeite crystals and gel retain their original morphology. They exhibit slight roughness and occasional small depressions. The structure remains dense, without noticeable porosity or dissolution.

A further study on the solubility of set zinc phosphate cement in artificial saliva was conducted in the Composite Materials Laboratory at the E.A. Buketov Karaganda National Research University.

Determining the solubility of set zinc phosphate cement in artificial saliva is an important step in the study, as the low solubility of cements helps reduce the risk of secondary caries and prevents the accumulation of microorganisms and organic debris at the cement–tooth interface. Zinc phosphate cement reaches full strength 24 h after mixing, with approximately two-thirds of its final strength developed within the first hour after cementation. Exposure to oral fluid during this period can increase cement permeability. However, prolonged waiting periods during tooth cementation are virtually impossible, making this study relevant. The solubility of zinc phosphate cements in artificial saliva was assessed by comparing the changes in sample weight after exposure to the solution. Zinc phosphate cement of the “Unicem” brand was used as the reference cement. The results of the change in the mass of set zinc phosphate cement samples depending on the exposure time are presented in Table 6 and Figure 6.

When determining the stability of “Unicem” zinc phosphate cement in artificial saliva, it was found that the average initial mass of the cement samples was 14.4682 g, and after 24 h of exposure, the average mass of the samples decreased to 14.4663 g, corresponding to a mass change of 0.0019 g (0.013%). After 48 h, a decrease in the average initial mass was recorded from 14.6377 g to 14.6341 g, with a mass change of 0.0036 g (0.025%). After 5 days of exposure, the average initial mass of the samples decreased from 14.6964 g to 14.6041 g, corresponding to a mass change of 0.0923 g, which is equivalent to 0.63% of the initial value.

Table 6. Change in weight of set zinc phosphate cement depending on time.

No of the Sample	Initial Weight of Samples, g	Weight After 24 h, g	Weight After 48 h, g	Weight After 5 Days, g
“Unicem” zinc phosphate cement				
1	14.4581	14.4562	-	-
2	14.4783	14.4764	-	-
3	14.7113	-	14.7082	-
4	14.5641	-	14.5601	-
5	14.7587	-	-	14.5792
6	14.6342	-	-	14.6291
Experimental zinc phosphate cement				
1	15.3328	15.3241	-	-
2	15.3469	15.3373	-	-
3	15.4974	-	15.4485	-
4	15.3998	-	15.3803	-
5	15.4117	-	-	15.3571
6	15.4451	-	-	15.3799

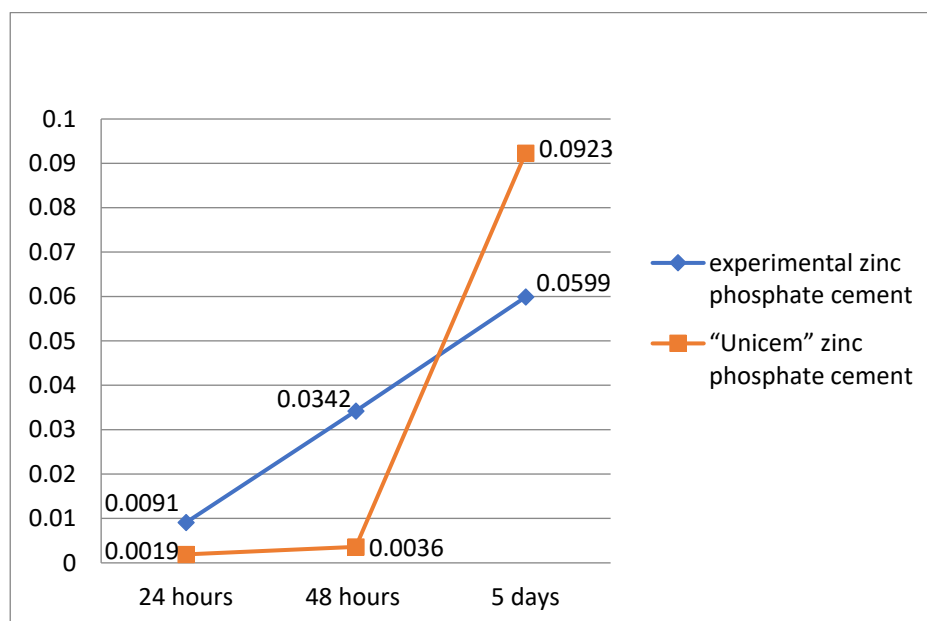


Figure 6. Change in mass of cement samples depending on time.

For the experimentally produced zinc phosphate cement, the average initial sample weight before exposure to the artificial saliva solution (pH 6.9) was 15.3398 g. After 24 h of exposure, the average weight decreased to 15.3307 g, representing a weight change of 0.0091 g (0.059%). After 48 h, the average sample weight decreased from 15.4486 g to 15.4144 g, corresponding to a weight change of 0.0342 g (0.22%). After 5 days (120 h) of exposure, the average sample weight decreased from 15.4284 g to 15.3685 g, representing a weight change of 0.0599 g, corresponding to 0.39% of the initial sample weight. This is 35% lower than the commercial standard “Unicem” (0.63%). This demonstrates the high resistance of the phosphate matrix to long-term leaching in a physiological environment.

The properties of zinc phosphate cement obtained with and without the addition of phosphorus slag are presented in Table 7. The studies were conducted in compliance with strict methodological requirements. To determine mechanical strength, 10 samples were prepared, of which the six most defect-free ones were selected for final testing (in accordance with GOST protocols and guidelines) [34].

Table 7. Properties of dental cements.

Indicators	Dimension	The Value of the Indicators for Cement	
		Zinc Phosphate Cement Brand "Unicem"	Experimental Zinc Phosphate Cement
Grinding fineness	cm ² /g	3280	3345
Whiteness	%	95.2	97.8
Powder to liquid ratio	g/mL	2 g—to 0.35 mL	2 g—to 0.42 mL
Setting time, start/end	min	4/8	6/9
Compressive strength	MPa	101.8	107.4
Water solubility	%	0.16	0.18
Film thickness	µm	25.1	25.5
Erosion	mm/h	0.116	0.144
Adhesiveness	kg/cm ²	14.2	12.6
pH level	1 h	4.2	4.4
	24 h	6.3	6.5
	48 h	6.8	7.0

The absolute strength values (101.8–111.9 MPa) obtained in the study meet standards; their clinical significance is revealed through their resistance to aggressive environments simulating oral conditions. A key indicator is the retention of high strength (98.8 MPa) after exposure to 10% lactic acid (a bacterial metabolic product), representing only an 8% loss from the initial value. This demonstrates the exceptional stability of the phosphate matrix under bacterial attack.

The setting times for the cements were an initial setting time of 6 min, and the final end time was 9 min. These setting times provide the clinician with a more comfortable working time compared to the prototype (4 and 8 min, respectively). The experimentally determined film thickness of the experimental zinc phosphate cement was 25.5 µm, slightly exceeding the best international standards (the norm is up to 25 µm). This is achieved due to the high dispersion of the powder (average particle size 5.8 µm). The adhesive capacity of the experimental cement was 12.6 kg/cm². This high adhesion is due to the dense microstructure and the synergy of bismuth oxide and phosphorus slag.

A direct comparison with the "Unicem" brand showed that the solubility of our composition in artificial saliva is 35% lower (0.39% vs. 0.63% over 5 days), which is critical for preventing secondary caries. A whiteness of 97.8% is significantly higher than that of standard samples, providing an aesthetic advantage. The material not only "meets the required specifications", but also offers an improved combination of properties (high acid resistance, low solubility, and increased whiteness) while simultaneously reducing the production temperature by 100 °C, making it more cost-effective and technologically efficient than imported analogues.

Thus, based on the results of a study of the solubility of cements in artificial saliva, it was established that the experimentally obtained zinc phosphate cement samples exhibited

the lowest mass loss, indicating low solubility of the material and, consequently, higher resistance to the aggressive oral environment.

Dermal fibroblasts were used to determine toxicity. The use of human dermal fibroblasts is a generally accepted standard for primary screening of medical materials for cytotoxicity, as these cells are highly sensitive to changes in the chemical composition of the environment. The choice of fibroblasts allowed for a standardized comparison with a commercial standard under identical conditions. This is confirmed in the study by Kontonasaki E. et al. [35], which demonstrated that zinc phosphate cements exhibit acceptable biocompatibility with osteoblast-like cells capable of colonizing their surface. Arun et al. [36] also examined the biological evaluation of zinc phosphate cement specifically for potential contact with bone tissue.

To determine cytotoxicity, “Unicem” brand zinc phosphate composite cement and an experimental zinc phosphate cement prepared in different ratios were used. Cement beads weighing 0.3 and 0.7 g were prepared and then immersed in 1 mL of PBS for 24 h in an incubator at 37 °C. After 24 h, the dental cement was removed, and the extracts were used for further analysis. The extracts were filtered through PES membrane filters (pore size 0.22 µm). The sterile extracts in which the cemented samples were soaked for 24 h were added to human fibroblasts to determine their cytotoxicity (Figure 7).

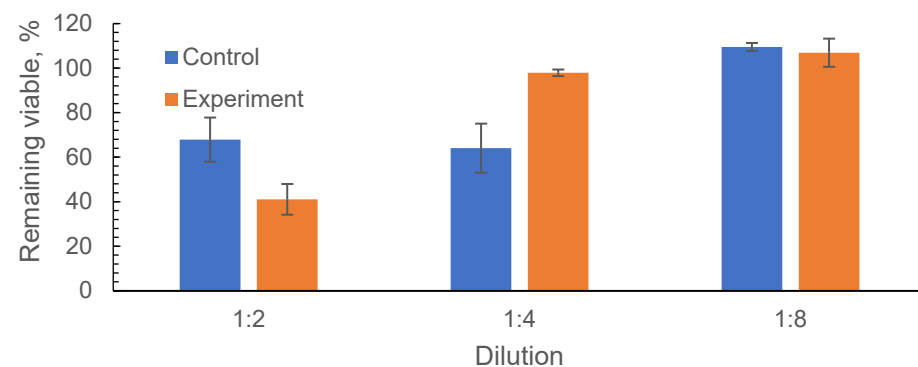


Figure 7. Cytotoxic analysis of bio cement at 0.3 g/mL. (Control) zinc phosphate cement “Unicem”, (Experiment) experimental zinc phosphate cement.

“Unicem” brand zinc phosphate cement is produced without exotic additives (phosphorus slag and Bi_2O_3). The primary focus is on bactericidal properties and an antimicrobial effect. Compressive strength is less than 100 MPa. The material is characterized by relatively rapid setting, which ensures accelerated fixation.

According to study results, zinc phosphate composite cement at a 0.3 g concentration exhibits low cytotoxicity towards human fibroblasts, with cell viability reaching 68%. Meanwhile, the experimental zinc phosphate cement exhibits moderate cytotoxicity at this concentration, resulting in only 41% of cells remaining viable (Figure 7). The statistical significance level for these values is $p = 0.0184$, confirming the reliability of the analysis results. Increasing the concentration to 0.7 g increases the toxicity of the cement components to human fibroblasts. At a dilution of 1:4 with the control sample, 85.78% of the cells were viable, whereas with the experimental dental cement only 32.47% of the cells remained viable; the reliability of p is 0.0085. At a dilution of 1:8 for a 0.7 g sample of cement, no cytotoxicity towards human fibroblasts was detected (Figure 8).

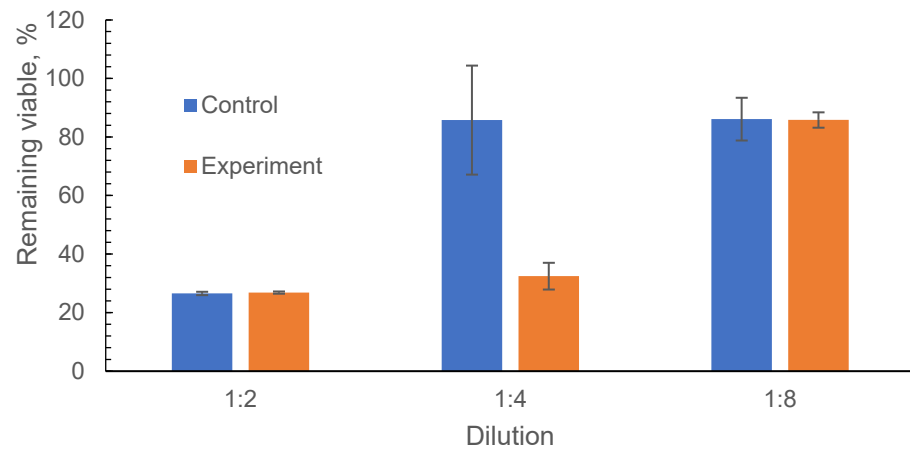


Figure 8. Cytotoxic analysis of bio cement at 0.7 g/mL. (Control) zinc phosphate cement “Unicem”, (Experiment) experimental zinc phosphate cement.

Thus, a comparison of the cytotoxicity of dental cement on human fibroblasts showed that toxicity was dose-dependent for both cement types. Analysis of the data obtained from the cement dilutions showed that both cements differed slightly in toxicity. However, the zinc phosphate composite cement was slightly more biocompatible than the experimental zinc phosphate cement. Thus, both dental cements studied at a 0.3 g dose and a 1:4 dilution were non-toxic.

4. Conclusions

Based on the conducted research, the following conclusions can be drawn:

1. The developed optimal composition of the zinc phosphate cement powder frit is as follows: ZnO is the main source of Zn^{2+} ions and reacts with H_3PO_4 to form zinc phosphates and an amorphous gel; MgO is a modifier for frit sintering that forms a magnesium phosphate matrix; SiO_2 is a filler that improves sintering and imparts vitreousness; Bi_2O_3 is a mineralizer that improves frit smoothness and accelerates strength gain; phosphorus slag forms low-melting eutectics, reduces sintering temperatures by 100 °C, and improves mechanical strength and increases whiteness to 97.8%. The main minerals in the fired frit sample are zincite (ZnO), quartz (SiO_2) and periclase (MgO). Zinc phosphate cement is characterized by a high degree of sintering, a dense microstructure and the absence of significant pores and cracks. The addition of phosphorus slag and bismuth oxide ensured a noticeable reduction in sintering temperature and the formation of a denser structure compared to traditional ZnO-MgO compositions.
2. Chemical resistance was studied under the influence of aggressive environments: a 5% NaCl solution, a 10% lactic acid solution, and carbonated water. The maximum mass loss of the sample in lactic acid was 8%, indicating the low solubility of the phosphate matrix. In a 5% NaCl solution, the loss was 6.8%, and in carbonated water, the decrease was minimal—2.8%, indicating virtually no reaction of the cement with weak acids. It was established that zinc phosphate cement exhibits high acid resistance due to the formation of a less soluble amorphous Zn-Al phosphate gel in a highly buffered environment. Furthermore, a small addition of phosphorus slag and Bi_2O_3 to the powder phase promotes the formation of a denser structure with lower porosity and increased resistance to the diffusion of aggressive ions. Chemical analysis of zinc phosphate cement after exposure to aggressive environments revealed that lactic acid causes the greatest phosphorus (P_2O_5) leaching, from 27% to 23.21%, with a relative enrichment of ZnO reaching 56.97%. The matrix loses its phosphate component,

leaving behind more zinc oxide and insoluble Zn compounds. The concentration of toxic elements (Pb, As, Cr, Ni, Th, etc.) remains very low (10–67 ppm), making the developed material safe for heavy metal leaching.

3. Determination of the solubility of set zinc phosphate cement in artificial saliva showed that the experimentally obtained samples of zinc phosphate cement are characterized by the lowest mass loss, which indicates low solubility of the material and, accordingly, higher resistance to the aggressive environment of the oral cavity.
4. The cytotoxicity of zinc phosphate composite cement and experimental zinc phosphate cement was determined. The experimental zinc phosphate cement was shown to have moderate cytotoxicity, with only 41% of cells remaining viable. A comparison of the cytotoxicity of dental cement on human fibroblasts revealed that the experimental zinc phosphate cement exhibited dose-dependent toxicity. Analysis of the data obtained from the dilution of biocements showed that zinc phosphate composite cement at a weight of 0.3 g and a dilution of 1:4 was nontoxic.

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Conflicts of Interest: The authors declare no conflicts of interest.

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