JOURNAL OF ENGINEERING SCIENCES

Volume 10, Issue 1 (2023)

Lebedev V. V., Miroshnichenko D. V., Nyakuma B. B., Moiseev V. F., Shestopalov O. V., Vyrovets S. V. (2023). Design of inorganic polymer composites for electromagnetic radiation absorption using potassium titanates. Journal of Engineering Sciences, Vol. 10(1), pp. C1-C8, doi: 10.21272/jes.2023.10(1).c1

Design of Inorganic Polymer Composites for Electromagnetic Radiation Absorption Using Potassium Titanates

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Article info:

Submitted:	February 24, 2023
Received in revised form:	May 8, 2023
Accepted for publication:	May 15, 2023
Available online:	May 19, 2023

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Abstract. This paper investigated the synthesis of inorganic polymer composites for electromagnetic radiation absorption using potassium titanates. The selected polyamide 6 and potassium polytitanate materials contain TiO_2, K_2CO_3, and KCl obtained by charge sintering. Results showed that modification of polyamide 6 with sintering products in the form of a fine powder of potassium polytitanate that contains different phases $K_2O \times 2TiO_2$, $K_2O \times 4TiO_2$, and $K_2O \times 6TiO_2$ which increased their strength properties. With increased potassium titanates (PTT) synthesis, a gradual transition from di to potassium hexatitanates occurs $K_2O \times 2TiO_2 – K_2O \times 4TiO_2 – K_2O \times 6TiO_2$. The optimal content of potassium polytitanate was over 20 % by mass. To fully ensure the reinforcing effect due to the filling of potassium polytitanate polyamide 6, it is necessary to use whiskers $K_2O \times 6TiO_2$, which can be collected by the additional crystallization of the amorphous charge sintering product. By designing experimental-statistical mathematical models in equal regressions, mathematical optimization of inorganic polymer composites for electromagnetic radiation absorption using PTT was carried out.

Keywords: polymer, composite, potassium titanates, synthesis, electromagnetic radiation, absorption, strength properties.

1 Introduction

Nowadays, the production of various radio-electronic equipment is actively expanding. The main direction is radio-electronic equipment improving properties: reducing the size and energy losses, better accuracy, and speed, among others.

Many technologies are already in transit to a higher frequency range. So, there is a need for materials with appropriate electromagnetic characteristics, which can match and protect various electromagnetic ranges components.

Scientific progress and technological development have significantly increased electromagnetic pollution (EP) with significant costs to human health, safety, and the environment. This challenge is exacerbated by new sources of electromagnetic radiation (EMR) and energy transmission, such as cellular, satellite radio communication, navigation and radar systems, radio engineering installations, medical devices, household appliances, and other technical equipment [1, 2].

The EMR generated by various sources also causes equipment and machinery malfunctions [3,4]. It should be noted that the presented division by types is conditional since most materials are systems that combine features of functioning different types [5, 6]. By their composition, most known radioabsorbing materials (RAM) are composite materials that include a dispersed absorber of electromagnetic waves and a dielectric matrix that creates absorber particles with volumetric distribution [7].

The absorber use can solve the problem of EMR dissipation in the material structure by one of two possible mechanisms: due to dielectric type losses (conductive fibers, dispersed graphite, and carbon black) and dielectric and magnetic simultaneous losses (e.g., ferromagnets and ferrites).

There are various measures aimed at protecting biological and technical objects from EMR exposure, which can be divided into several types [1-3]:

– organizational, such as distance protection (location at the maximum allowable distance from the EMR area action); time protection (restriction staying in the EMR zone); quantity protection (the EMR sources capacity must be the minimum necessary);

– therapeutic and preventive – increasing the body's resistance to EMR exposure; sanitary and preventive provision; treatment in emergencies;

– engineering and technical – particular protective materials use; individual and collective protection use; structures improvement.

The most significant scientific and practical interest is engineering and technical measures. Design and creation of special protective materials for shielding can help us. Two types of shielding are generally considered: shielding EMR sources from humans and shielding humans from EMR sources [6]. The screen's protective properties are based on the tension-weakening effect and electric field distortion in the space near the grounded metal object. Protection against the industrial frequency magnetic field is possible only at the product development or object design stage.

The field level reduction is achieved due to vector compensation since other shielding industrial frequency magnetic field methods are highly complicated and expensive [1]. Various radio reflectors (RR) and radio-absorbing materials are used for EMR shielding in radio frequency ranges. RR materials include various metals – iron, steel, copper, brass, and aluminum.

To protect against UHF, thin or perforated sheets, conductive films, metalized fabrics, or metal meshes are used, which have sufficient attenuation but differ from sheet materials in lower weight and cost.

Many materials that provide a high EMR shielding level have been designed to create flexible screens, protective clothing, and covers to ensure biological protection and electromagnetic compatibility of radio-electronic equipment [1, 7].

The RR materials' disadvantages for human protection include EMR reflection from curved surfaces of the protected object leads to interference of waves with different amplitudes and phases and, as a result, individual body parts irradiation [1]. This circumstance necessitates the RR materials used to reduce the EMR impact.

The radio-absorbing material reduces the overall EMR level reflection in the radio range due to the electromagnetic energy conversion [8].

EMR energy dissipation in its propagation in the material is due to the conduction, magnetization, and polarization processes [2]. From a technological point of view, radio-absorbing materials can be divided into two large groups.

The first group is materials or radio-absorbing coatings (RAC), and the second is structural radio-absorbing materials (RAM). Most often, the initial components composition, the intended structure, and the structural RAC and RAM purpose are equal [2].

According to the operation principles, the representative class of designed RAC and RAM can be conditionally divided into the following types [1, 8, 9]:

1) gradient materials, the outer layer of which has radiophysical properties as close as possible to the free space characteristics, and the magnetic and dielectric permeability change values continuously in the direction of increase from the material surface layer to the depth;

2) materials, which action is based on the electrodynamic swamp principle, the distinguishing feature of which is uniformity, that is, the radiophysical characteristics practically do not change in the direction from the material surface to the depth (cellular, fibrous, and foam composites can be classified as such materials);

3) multiplanar, which are structures consisting of a large number of thin conductive or ferromagnetic films, separated by dielectric and interference-absorbing layers, which action is based on the total or partial mutual quenching of the electromagnetic wave incident and reflected from the metal substrate.

In most cases, anthropogenic electromagnetic pollution sources use energy in the entire UHV range of wavelengths, and absorbing materials should reduce the incident EMR reflection in a wide frequency range. In contrast, the effective reflection level should not exceed minus 15 dB (3 % of incident EMR capacity).

At the same time, an essential factor is the reduction of RAM's weight and size characteristics and the increase of resistance to environmental impact [10]. These requirements implementation is an urgent and complex scientific and applied task.

Today, a lot of radio-absorbing coatings and materials have been designed based on polymer binders and dispersed conductive (semiconductor) carbon-containing, metal (alloys and oxides), and magnetic fillers [11]. As a rule, multi-component systems are used to achieve the best radiophysical properties. It can be elastomers filled with dielectric and absorbing fillers. A metalized surface (substrate) is required for RAC to function. At the same time, a layer or several layers of RAC must be applied directly to the protected object, which is a complex and time-consuming technological process. Individual protective elements can be made from structural RAMs, produced separately from the object [1].

Depending on the purpose, such materials can be conventionally divided into rigid and flexible (elastic). The rigidity or flexibility degree will depend on the polymer matrix type and structure [11].

Rigid screens are used in mobile screens construction to locate service personnel in repair places, limit local UHF sources' impact, and separate or shield individual elements or blocks inside radio technical devices [12].

Flexible materials are used for the equipment covers and capes production and protective clothing for household and particular purposes production. It is essential that all materials, regardless of their composition and construction, are designed based on the specified requirements achievement [13].

Specified requirements are defined by the frequency band within which the effective level of EMR absorption is achieved. Different RAM types are well-known, but their receiving often challenging technological processes with low productivity. Thus, in works [2], RAM was received by the polymer melt extrusion method with structure fixation by cooling. A polyethylene terephthalate and polypropylene mixer modified by different conductive fillers was used as a polymer matrix.

Modern technologies are implemented on composite materials with different filler combinations, as no single-component material could achieve a high absorption capacity in a wide frequency band. Such composite materials are used for: biological object protection, electromagnetic device compatibility [14], and electromagnetic wave reflection reduction [15].

Various composite materials are actively used to protect against electromagnetic radiation, which corresponds to the sustainable development concept.

The main feature of composite materials is that they consist of two or more constituent components, which possess properties different from the component materials. Composites are easy to produce and provide new and unique physical-chemical properties (e.g., flexibility, high strength, and elasticity) unattainable for traditional materials.

2 Literature Review

According to the literature, typical materials for absorbing electromagnetic radiation are [3, 4]:

1) conductive powdered materials (coal, carbon black, graphite, metals – steel, cast iron, iron, aluminum, cobalt, lead, zinc, tin, copper, and metal salts) with spherical, cylindrical, flaky, and shaped particles.

2) conductive carbon, metal, and metal-carbon fibers, carbon fabrics, metal threads, plates, foil strips, wire scraps, meshes of a complex shape, gratings, resonant elements in the form of cross-shaped dipoles or closed conductors (rings) [6, 7];

3) metalized carbon and polymer fibers, fabrics, films, and microspheres;

4) magnetic fillers – ferrites of different chemical composition (mostly magnetically soft), as well as magnetic powders of metals and amorphous Fe alloys, Fe-Co-Ni alloys, and perm);

5) dispersed semiconductors – metal oxides, carbides and sulfides, silicon carbide, ferroceramics, charred organic silicon fabrics, and fibers;

6) dielectrics, in particular, easily polarized organic substances (retinyl Schiff salts) and biopolymers (chitin).

Many compositions of ferrite components used as absorbers of various ranges are described in modern scientific literature. In addition, such absorbers have an insufficiently wide band of operating frequencies [5].

By varying fillers concentration, it is possible to achieve the required electromagnetic properties such as high absorption coefficient, complex dielectric, magnetic permeability, and shielding level. In modern scientific sources, there is much research on receiving materials for protection against electromagnetic radiation.

Most research concerns the design of effective materials for protection against electromagnetic radiation based on mixtures and composites of fillers with magnetic properties (such as ferrites [3,4], metals [5,6]) and various dielectrics – ceramic, polymer, and other matrices [7].

As an active phase (filler) of composites, carbon-containing materials (carbon nanotubes, graphite, carbonyl iron, and fullerenes) [7, 8] and ferrite powders [9, 10] are used widely. The composite materials research based on ferroelectrics and multiferroics is actively developing [11, 12].

Thermo-reactive polymer composites containing silicon carbide [12], carbon fibers [13], carbon black [14], and carbon nanotubes [15, 16] are also effective. The materials act as radio shielding materials and coatings due to the combination of absorption mechanisms.

This is due to their natural ferromagnetic resonance, the resonance of domain boundaries movement, multiple imprints, eddy current losses, and repolarization, among others. Such materials effectively protect against electromagnetic radiation, but their disadvantages include the complexity of synthesis and high cost.

Therefore, polymer composites for protection against electromagnetic radiation based on various thermoplastic matrices (polyvinyl butyral [17], polypropylene [18], and polyvinylidene chloride [19]) have been recently designed. Inorganic ferromagnetic fillers are used in polymer composites due to their better technological, operational, and economic characteristics.

Creating inorganic polymer composites (PIC) for electromagnetic radiation absorption using potassium titanates presents a unique perspective. Potassium titanates (PTT) with the general formula $K_2Ti_nO_{2n+1}$, consisting of titanium oxide layers and cations interlayers, form unique layered and tunnel crystal structures due to their high ion exchange, intercalation capacity, and photocatalytic activity properties. Depending on synthesis conditions and chemical composition, PTT is either an amorphous compound with a layered (flaky) structure or crystalline submicron- and nanofibers (potassium tetra-, hexa-, or octatitanate).

The layered structures of PTT are characterized by low interlayer shear energy and high reflection and absorption values in the visible and infrared spectral regions. Fibrous materials have high mechanical strength, low thermal conductivity, and good reflectivity in a wide spectral range. These properties make PTT a prospective material for producing functional materials, including fillers for polymeric materials.

Polyamide 6 hybrid composites reinforced with PTT whiskers $K_2Ti_6O_{13}$ and liquid crystalline polymer have been reported in the literature [20]. The static tensile measurements showed that the tensile strength and modulus of the composite increased with increasing whisker content.

As potential fillers for composite materials that can absorb electromagnetic radiation, PTT with amorphous layered structures is exciting due to their low interlayer shear energy and high values of [21-24].

Other characteristics of PPT include reflection coefficient and absorption radiation coefficient in the visible and infrared spectral regions as well as high intercalation ability and catalytic activity.

PTT's ability to absorb ultraviolet radiation or visible light with photosensitized modifications enhances its potential application in solar photovoltaic manufacture. Photoelectric characteristics of Nb-doped titanates were investigated in [25]. There is a high potential for using PTTs with various morphologies and structures to obtain composite materials for electromagnetic radiation absorption.

Researchers have designed and investigated polymer composites using complex filler systems such as graphite and humic substances [16, 26]. Similarly, our previous work [27] examined polymer composites for absorbing electromagnetic radiation based on thermoplastic polyamide 6 and silicon carbide. The proposed composite was relatively transparent in the millimeter frequency range and characterized by a small absorption coefficient. Hence, it is essential to consider using PTT for effective composite materials development for electromagnetic radiation absorption.

The article aims to design and characterize inorganic polymer composites for electromagnetic radiation absorption using PTT. To achieve this aim, the following tasks were set:

– to research sintering products of titanium oxide TiO_2,potassium carbonate K_2CO_3 and potassium chloride KCl at different temperature conditions;

– to determine the influence of the modification of polyamide 6 with PPT on their strength properties;

– to design experimental-statistical mathematical models of strength properties of inorganic polymer composites for electromagnetic radiation absorption using PTT.

3 Research Methodology

The following materials were used to design the inorganic polymer composites for electromagnetic radiation absorption:

– polyamide 6 (Grodnamid PA6-L-211/311). It is the baseline injection moulding polymer composite material on the base of PA-6 with modifying additives that improve its injection properties: surface quality of moulded articles, filling of the mould, and easier demoulding of finished articles;

– titanium oxide TiO_2;

– potassium carbonate K_2CO_3;

– potassium chloride KCl.

Polymer inorganic composites for electromagnetic radiation absorption were synthesized by extruding pre-prepared raw materials in a single-screw laboratory extruder at 170-200 °C and a screw speed of 30–100 turns per minute.

The study of impact strength and breaking stress during the bending of the samples was carried out on a pendulum head according to ISO 180 and ISO 178, respectively, without notching at a temperature of 20 °C.

Microscope analysis was performed on the Olympus GX Inverted Microscope LECO Corporation (USA).

PTT was received by charge sintering that contains TiO_2, K_2CO_3, and KCl The TiO_2:K_2O was used in the ratio (in moles) from 4:1 to 7:1 with the addition from the mass S TiO_2:K_2O 20 % wt. KCl.

The materials were first crushed, mixed, passed through a sieve of 60-300 μm and then heated in a muffle furnace under oxidative conditions (Air) for 1-3 hours at 900 °C (product diameter is 0.005-0.100 μm and a length is 1-10 μm).

Next, the temperature was ramped to 1000 °C (product diameter 0.1-0.6 μm and length 5-60 μm) and then finally at 1100 °C (product diameter 0.6-2.0 μm and length 60-600 μm). The salt matrix (KCl) separated from unreacted particles by leaching with a slightly acidic solution with stirring or deionized water at 90 °C.

The precipitate was then filtered and washed until the chloride ion was removed entirely. Lastly, it was dried at 105-120 °C to a residual moisture content of 0.5-1.0 % wt.

4 Results and Discussion

Figures 1, 2 show the batch sintering stage, the resulting sintering products, and the finished product after drying.

Figure 1 – Photo of the charge sintering process (A) and the finished product after drying (B)

As observed in Figure 2, the sintering product after drying is a fine powder of PTT with a particle size of 1-5 μm.

Figure 3 shows a microscope image of PTT received by different temperature sintering of composition TiO_2, K_2CO_3, and KCl. From microscopic images of PTT obtained at different temperature conditions, it can be seen that with an increase in the synthesis temperature, a crystalline phase is formed along the edges of PTT particles in the form of filamentous crystals.

Figure 2 – Photo of the finished product of PTT after synthesis (A) and after drying (B)

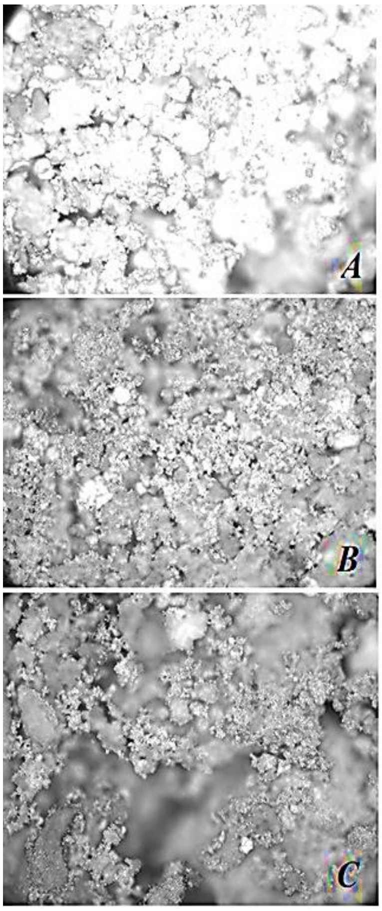

Figure 3 – Microscope image (the degree of increase – 100) of PTT obtained at different sintering temperatures for TiO_2, K_2CO_3, and KCl compositions: A – 900 ºC; B – 1000 º C; C – 1100 º C

This observation could be ascribed to the direct formation of an amorphous phase in the PTT, which also correspondent with data in the articles [21, 22]. With an increase in the process of PTT synthesis, a gradual transition from di to potassium hexatitanates occurs $K_2O \times 2TiO_2 – K_2O \times 4TiO_2 – K_2O \times 6TiO_2$ [24, 25].

Next, the influence of PTT and the introduction of the polyamide 6 strength properties complex was examined, as shown in Figures 4-5.

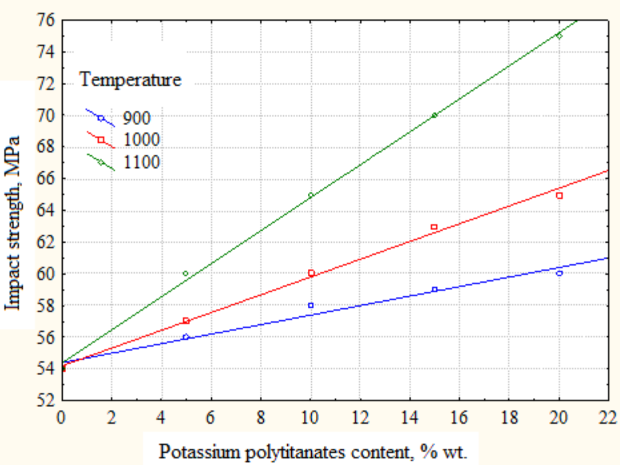

Figure 4 – Effect of on impact strength of polyamide 6 PPT particles at different temperature conditions with regression equations: Impact strength$_{900}$ = 54.4 + 0.3·PPT; Impact strength$_{1000}$ = 54.2 + 0.56·PTT; Impact strength$_{1100}$ = 54.4 + 1.04·PTT

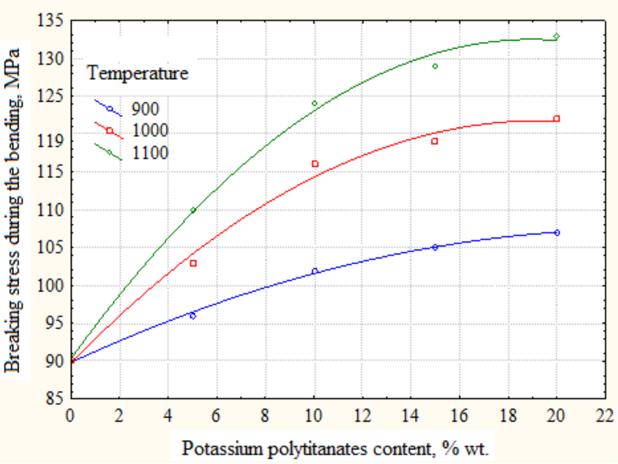

Figure 5 – Effect of breaking stress during the bending of polyamide 6 PPT particles at different temperature conditions with regression equations: Breaking stress during the bending$_{900}$ = 89.8 + 1.49·PTT – 0.031·PTT^2; Breaking stress during the bending$_{1000}$ = 89.7 + 3.31·PTT – 0.086·PTT^2; Breaking stress during the bending$_{1100}$ = 90.3 + + 4.44·PTT – 0.117·PTT^2

From Figures 4, 5, polyamide 6 with PPT modification increased their strength properties. Concurrently, the reinforcing effect of PTT concerning the strength properties of polyamide 6 increased with higher PPT synthesis temperatures due to the formation of crystalline filamentous phases, which also corresponds with data in the article [28]. It was also observed that the optimal PTT content is over 20 % wt.

It is necessary to introduce a dispersed conductive filler into its composition to provide inorganic polymer composites for electromagnetic radiation absorption high strength properties [15]. Different geometric shapes and sizes of particles are used as conductive fillers. The particles' shapes are spherical (soot, metal powders, colloidal graphites, metalized microsclospheres), lamellar (expanded graphite, metal powders after attrition treatment, metalized mica), and needle-like (carbon fibers, metalized fibers, needles) [1, 2].

Figures 6, 7 present the 3D plot of the mathematical model based on regression equations developed in MathCad Prime 6.0. For this model, theoretical calculations of the predicted impact strength and breaking stress values during bending inorganic polymer composites for electromagnetic radiation absorption were performed. The results are presented in 3D graphics for forecasting the strength properties of inorganic polymer composites for electromagnetic radiation absorption using PTT in different synthesis temperatures.

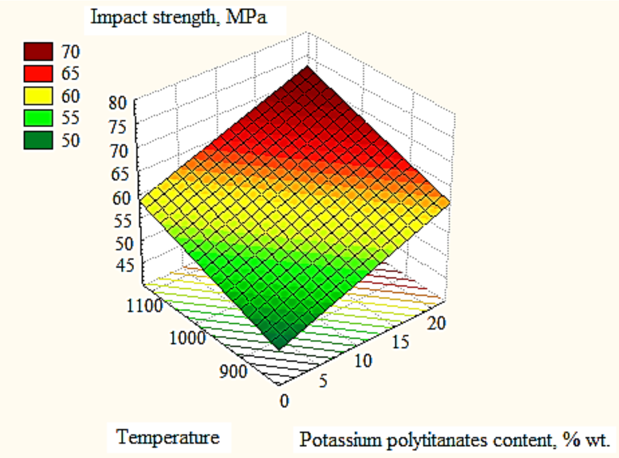

Figure 6 – The 3D plot for forecasting the impact strength of inorganic polymer composites for electromagnetic radiation absorption using PTT with regression equations: Impact strength = 17.3 + 0.63·PTT + 0.037·Temperature

The results indicate that depending on the PTT content, the impact of synthesis temperature is the most effective in terms of increasing the polymer inorganic composites for electromagnetic radiation absorption strength properties. Forecasting researches on definition the most effective structure of the polymer inorganic composites for electromagnetic radiation absorption are carried out.

The possibility of forming various products of inorganic polymer composites for electromagnetic radiation absorption is shown, with particular attention to its deserved composition with a content of PTT over 20 % wt.

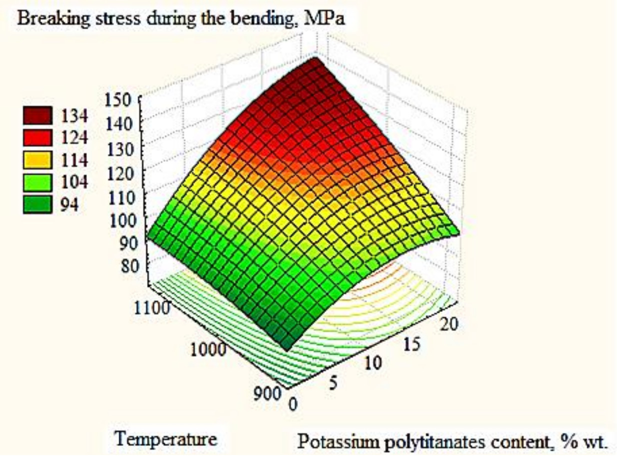

Figure 7 – The 3D plot for forecasting the breaking stress during the bending of inorganic polymer composites for electromagnetic radiation absorption using PTT with regression equations: Breaking stress during the bending = –73.1 – – 3.12·PTT + 0.304·Temperature – 0.0781·PTT^2 + + 0.0062·PTT·Temperature – 0.0001·Temperature^2

The increase of breaking stress during bending also indicates the manufacturability of the inorganic polymer composites for electromagnetic radiation absorption. In addition, it even slightly «softens» the original rather rigid polyamide 6. The simulation allowed obtaining models for forecasting the strength properties of inorganic polymer composites for electromagnetic radiation absorption depending on their chemical composition, which can be adapted to any content and temperature synthesis of PTT.

5 Conclusions

The study proposed using inorganic polymer composites for electromagnetic radiation absorption using PTT. The charge-sintered PTT containing TiO_2, K_2CO_3, and KCl were utilized as fillers in the polyamide 6 polymer composite. The sintering products were found to consist of a fine powder of potassium polytitanate containing different phases $K_2O \times 2TiO_2$, $K_2O \times 4TiO_2$, and $K_2O \times 6TiO_2$. With an increase in the PTT synthesis process, a gradual transition from di to potassium hexatitanates occurs $K_2O \times 2TiO_2 – K_2O \times 4TiO_2 – K_2O \times 6TiO_2$.

Modifying polyamide 6 with PTT increased their strength properties, whereas the optimal content of PTT was over 20 % by mass. By designing experimental-statistical mathematical models in equal regressions, mathematical optimization of strength properties of inorganic polymer composites for electromagnetic radiation absorption using PTT was performed. The results are presented in the 3D plot to forecast the strength properties of inorganic polymer composites for electromagnetic radiation absorption using PTT at different synthesis temperatures. The simulation 3D graphic allowed to forecasting the strength properties of polymer inorganic composites for electromagnetic radiation absorption depending on their chemical composition, which can be adapted to any content and temperature synthesis of PTT.

Perspective directions for future research are to determine the degree of absorption of electromagnetic radiation developed polymer composites based on polyamide 6 and PTT and also to explore the potential applications of these composites in various industries.

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