Innovative polymer composites based on biomedical materials
(Rapid communication)

Katarzyna Bulanda[1), *)] (ORCID ID: 0000-0002-5330-5719), **Dorota Bartusik-Aebisher**[2)] (0000-0002-5557-5464), **Małgorzata Oleksy**[3)], **David Aebisher**[4), *)] (0000-0002-2661-6570)

DOI: https://doi.org/10.14314/polimery.2024.5.5

Abstract: The article presents preliminary research on the influence of calcium phosphate (10–40 wt%) on the functional properties of ABS. Maleic anhydride grafted polyethylene was used as a compatibilizer (0.5 wt%). The mass flow rate, tensile properties and hardness were determined. The effect of the filler on the color change of the polymer matrix was also examined. For a composite containing 20 wt% calcium phosphate, the mechanical properties of samples obtained by 3D printing and injection molding were compared, with worse properties obtained by 3D printing. This can be explained by limited adhesion between the printed layers.

Keywords: biomaterials, ABS, calcium phosphate, injection molding, 3D printing.

Innowacyjne kompozyty polimerowe na bazie materiałów biomedycznych *(Komunikat szybkiego druku)*

Streszczenie: W artykule przedstawiono wstępne badania wpływu fosforanu wapnia (10–40% mas.) na właściwości użytkowe ABS. Jako kompatybilizator użyto polietylen szczepiony bezwodnikiem maleinowym (0,5% mas.). Określono masowy wskaźnik szybkości płynięcia, właściwości mechaniczne przy rozciąganiu i twardość. Zbadano także wpływ napełniacza na zmianę barwy osnowy polimerowej. Dla kompozytu zawierającego 20 wt% fosforanu wapnia porównano właściwości mechaniczne próbek uzyskanych metodą druku 3D i formowania wtryskowego, przy czym gorsze właściwości uzyskano metodą druku 3D. Można to wyjaśnić ograniczoną przyczepnością pomiędzy drukowanymi warstwami.

Słowa kluczowe: biomateriały, ABS, fosforan wapnia, formowanie wtryskowe, druk 3D.

Currently, scientists pay attention to topics related to regenerative medicine, in particular materials that can be used to rebuild bone tissue, used as scaffolds, among which ceramic materials and composite materials are widely used [1-7]. Several features are required for these materials, including biocompatibility, bioresorbability, mechanical compatibility with bone, good wettability, osteoinductivity and osteoconductivity [8–11].

Composites based on natural or synthetic polymers are promising materials for the construction of scaffolds. The most popular biostable synthetic polymers are polyolefins (polyethylene, polypropylene), poly(ethylene terephthalate), polytetrafluoroethylene, polyether polyurethane elastomers, polyamide, silicones, polycarbonates, acrylic polymers, acrylonitrile-butadiene-styrene, and polyether ether ketone [12, 13]. The polymers mentioned have sufficient mechanical strength to withstand the necessary mechanical loads. However, the surface of such materials is hydrophobic [14]. Due to this fact, these materials are usually modified using selected additives such as phosphates (hydroxyapatite, tricalcium phosphate). The addition of phosphate particles leads to further improvement of the material's mechanical properties. Moreover, the additive particles on the surface of the material make it more hydrophilic [14].

[1)] Department of Polymer Composites, Faculty of Chemistry, Rzeszow University of Technology, al. Powstańców Warszawy 6, 35-959 Rzeszów, Poland.

[2)] Department of Biochemistry and General Chemistry, College of Medical Science, University of Rzeszów, al. mjr. W. Kopisto 2a, 35-959 Rzeszów, Poland.

[3)] Students English Division Science Club, College of Medical Science, University of Rzeszów, al. mjr. W. Kopisto 2a, 35-959 Rzeszów, Poland.

[4)] Department of Photomedicine and Physical Chemistry, College of Medical Science, University of Rzeszów, al. Rejtana 16c, 35-959 Rzeszów, Poland.

[*)] Authors for correspondence: k.bulanda@prz.edu.pl; daebisher@ur.edu.pl

Composite materials are a promising class of biomaterials for tissue regeneration, especially bone and cartilage regeneration. In recent years, more attention has been paid to various forms of polymer/calcium phosphate composites [15, 16].

Acrylonitrile-butadiene-styrene (ABS) is one of the most common polymer materials used in extrusion, injection, and 3D printing [17, 18]. Acrylonitrile-butadiene-styrene terpolymer belongs to the group of amorphous plastics. ABS contains a group of monomers acrylonitrile, butadiene, and styrene, usually in amounts of 15–35%, 5–30% and 40–60%, respectively. However, the proportions of individual monomers can change within a very wide range [19]. The combination of materials in ABS gives new, unique properties, such as high impact strength and mechanical strength, as well as appropriate melt fluidity [18–20]. Addition of calcium phosphates (CaP) into the polymer matrix is promising, due to introducing the hemostatic properties, improving biocompatibility, and increasing mechanical strength [21, 22]. The scientific community widely uses calcium phosphate as a biomaterial, especially teams researching and improving bone regeneration processes, due to the chemical and crystallographic similarity of this material to the inorganic components of native bone [21]. Calcium phosphate is one of the few inorganic biomaterials approved for use in many products, such as over-the-counter supplements, toothpaste allowed in clinic trials [23–27].

Medicine, biotechnology, automotive industry, and aviation are one of many fields that use 3D printing technologies to obtain parts from polymer composites [28, 29]. Additive manufacturing technologies are ideal for product development, rapid prototyping, and small-scale production [30-32]. Scientists and doctors use 3D printers every day to create and visualize their ideas, models, and products. The most popular 3D printing methods are technologies involving the deposition of molten polymer material, such as Fused Deposition Modeling (FDM), Fused Filament Fabrication (FFF), Melted and Extruded Manufacturing (MEM), in which a thermoplastic polymer, usually in the form of a continuous filament. During 3D printing filament is heated, extruded in layers to create a 3D object, and cooled to obtain physical object. The devices are easy to use and maintain, making it cost effective and economical [30–32].

New technologies, such as FDM/FFF/MEM, are often compared with traditional methods, in this case with injection molding, to determine the possibility of obtaining elements with similar/sufficient features for given applications using another/alternative production technology. Combining or even replacing traditional technologies with new ones is important for the production process of various functional parts.

Natural bone is a composite made of a polymer (collagen) matrix filled with calcium phosphate nanocrystals in the form of insoluble calcium hydroxyapatite. For this reason, calcium phosphate-based materials are used in medical research on inorganic materials imitating natural bone [33].

Therefore, this study investigated the effects of calcium phosphate (10–40 wt%) on the mass flow rate, tensile properties, and hardness of ABS to obtain artificial bone. To increase the interactions at the phase boundary, polyethylene grafted with maleic anhydride (0.5 wt%) was used as a compatibilizer. It was assumed that these amounts of CaP and compatibilizer would allow obtaining homogeneous composites and would not have a significant impact on their functional properties. All materials used in the work were selected in such a way as to enable the use of the developed polymer composites for medical applications. For selected composites, the mechanical properties of samples obtained by 3D printing and injection molding were compared. Moreover, the influence of calcium phosphate on the color change of the polymer matrix was also examined.

EXPERIMENTAL PART

Materials

Acetonitrile-butadiene-styrene copolymer (ABS) trade name ABS medical (Spectrum Filaments, Pecice, Poland) was used as polymer matrix. ABS is certified USP class VI or ISO 10993-1, which proves biocompatibility for up to 30 days in contact with the human body (topical use) and is approved for contact with food in accordance with EU standards No. 10/20111 and 21 CFR FDA [34]. Calcium phosphate (CaP) with molecular weight of 310.17 g/mol and complexometric titration ≥ 95% was supplied from Thermo Fisher Scientific (Waltham, MA, USA). Polyethylene grafted with maleic anhydride (PE-g-MA) with the trade name Fusabond E926 was purchased from DuPont (Wilmington, DE, USA).

Sample preparation

In the first stage, the artificial bone composite components were homogenized using a twin-screw extruder (HAAKE MiniLab II Thermo Scientific, Waltham, MA USA) with work volume 7 cm^3. The process was conducted at a temperature of 245°C and a screw speed of 50 rpm. In the second stage, samples were obtained by injection molding and 3D printing using MEM technology.

Injection molding

The samples were obtained using a mini-injection molding machine (Haake MiniJet II, Thermo Scientific, Waltham, MA USA) in the form of paddles. The process parameters are listed in Table 1.

In the next stage, a series of tests of the functional properties of the obtained composites were performed. Based on the obtained test results, material was selected for further treatment.

T a b l e 1. Injection molding parameters

Parameter	Injection temperature °C	Mold temperature °C	Injection time s	Plasticizing time s	Injection pressure bar	Post-injection pressure bar
Value	245	80	5	80	600-550	550-500

T a b l e 2. 3D printing parameters

Nozzle diameter mm	Layer height mm	Filling %	Fill pattern °	Extrusion temperature °C	Worktable temperature °C	Print speed mm/s
0.4	0.2	100	45	245	80	70

3D printing using MEM technology

Filaments with a diameter of 1.75±0.05 mm were obtained from the selected material, using a designed line for obtaining fibers, made according to the design of Gamart S.A. (Łańcut, Poland). The view of the mentioned line was included in previous works [35-37]. During manufacturing fibers, the engine speed was 160 rpm, extrusion speed was 170 mm/s, speed of placing the filament on the spool was 140 mm/s and temperature of individual zones in the range of 210–245°C. Pure ABS and the filament were used to obtain paddle- and bar-shaped forms using a TierTime Up Box+ printer (Beijing, China) using MEM technology. The optimal printing parameters were selected in accordance with literature reports. The process parameters are listed in Table 2. The process flow is shown in Figure 1.

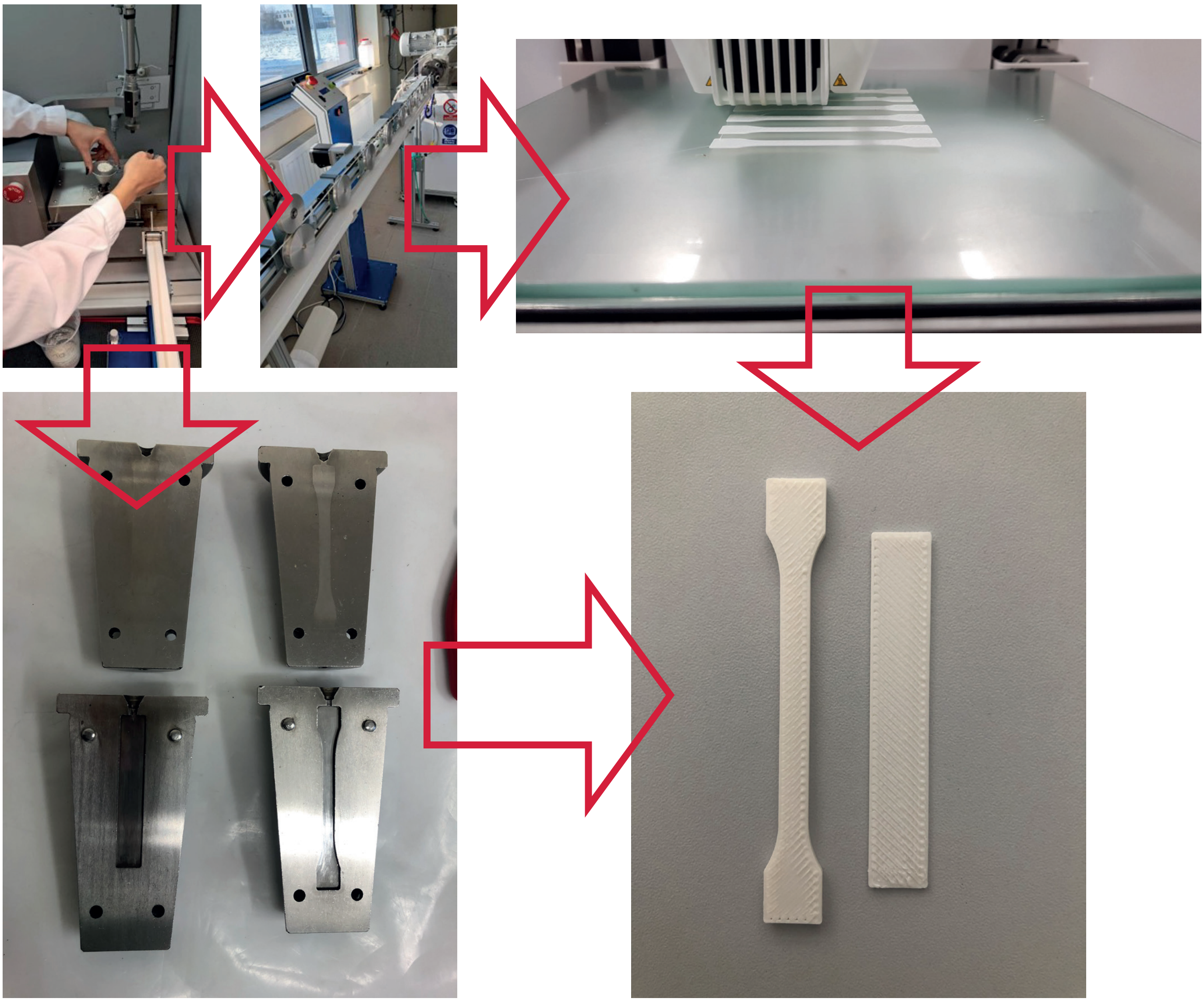

Fig. 1. Homogenization in a twin-screw extruder and obtaining filament; test samples obtained using 3D printing and injection molding methods

T a b l e 3. MFR of the composites

Property	CaP, wt%				
	0	10	20	30	40
MFR, g/10 min	9.6±0.2	7.5±0.1	6.8±0.4	5.5±0.1	4.5±0.1

Methods

The mass melt flow rate (MFR) was determined according to the ISO 1133 standard using a Dynisco 4781 plastometer (Kayeness Inc., Honey Brook, PA, USA) at a temperature of 245°C, with a load of 2.16 kg. Rockwell hardness was measured according to the PN-EN ISO 6508 standard using a Zwick/Roell hardness tester from Zwick GmbH & Co. (Ulm, Germany) at ambient temperature. Ten determinations were conducted for each series. Tensile tests were conducted according to the ISO 527 standard, on a universal testing machine (Instron 5967, Norwood, PA, USA) at ambient temperature. The crosshead speed for Young's modulus was 5 mm/min (until 1% tensile strain was obtained), after which the speed was increased to 50 mm/min. Five measurements were made for each series of materials. The color test was conducted using a spectrophotometer (CM-5 Konica Minolta Business Solutions Polska Ltd., Warsaw, Poland) according to the PN-EN ISO 13655 standard. The measurement was conducted for samples obtained by injection molding.

RESULTS AND DISCUSSION

From the processing point of view mass flow rate is an important parameter because materials with greater fluidity will more easily fill the entire volume of the injection mold and will also allow for denser printing of elements made with 3D printing technology. Table 3 shows the effect of the amount of filler used on the MFR of ABS.

Unmodified ABS is characterized by the highest mass flow rate, and therefore the best processability in injection and 3D printing processes (9.63 g/10 min). The introduction of the filler into the polymer matrix resulted in a lower MFR but did not cause technological problems in subsequent processes. It can be noticed that the higher the degree of filling the polymer with calcium phosphate, the lower the fluidity of the material, because the introduced additive increases the material flow resistance, which is also confirmed by other literature reports [38, 39].

Based on the results obtained regarding the rheological and mechanical properties, the ABS/CaP 80/20 composite was selected as the material with the highest tensile strength among the tested composites and with MFR allowing to obtain a material thread of constant size. For this reason, a composite containing 20 wt% calcium phosphate was used to obtain 3D printing filament, which made it possible to compare the results of samples obtained by injection molding and those obtained using MEM technology.

Fig. 2 shows the results of Rockwell hardness measurements for samples obtained using injection molding technology and MEM technology.

Analyzing the mechanical properties of composites obtained by additive manufacturing and, for comparison, by injection, samples obtained by 3D printing are characterized by worse mechanical properties. The observation is widely described in the literature and results from the greater homogeneity of samples obtained by injection molding.

The introduction of calcium phosphate as a filler into the ABS matrix in an amount of up to 30 wt% caused an increase in hardness. The ABS/CaP 70/30 composite was characterized by the highest hardness, which was 14% higher than ABS. It should be noted that the addition of 10–30 wt% filler allowed to obtain a similar hardness. It

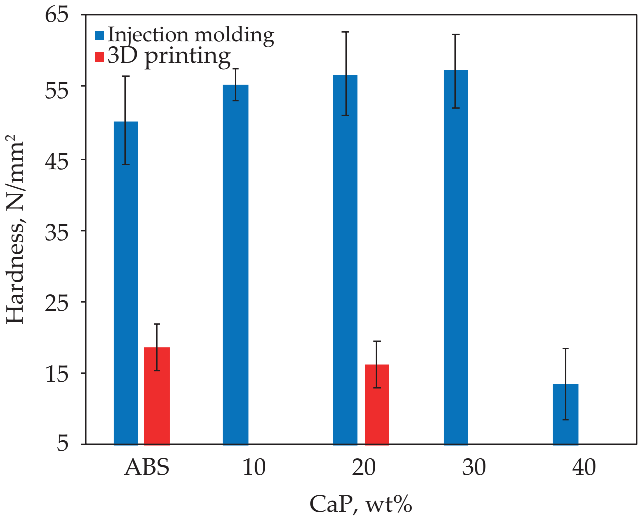

Fig. 2. Hardness of ABS and ABS/CaP composites

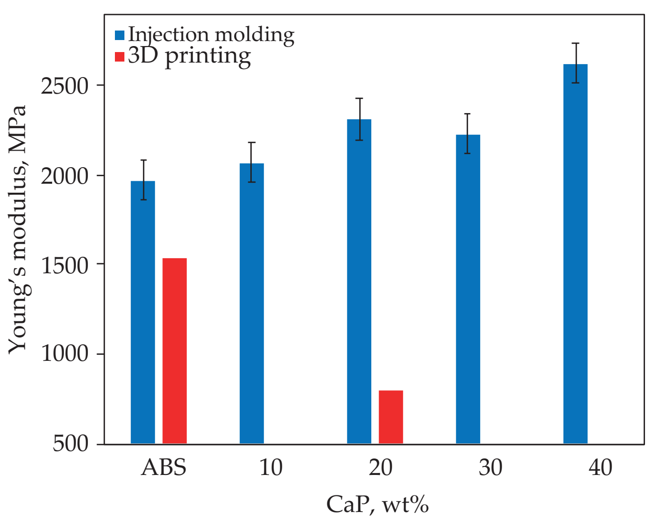

Fig. 3. Young's modulus of ABS and ABS/CaP composites

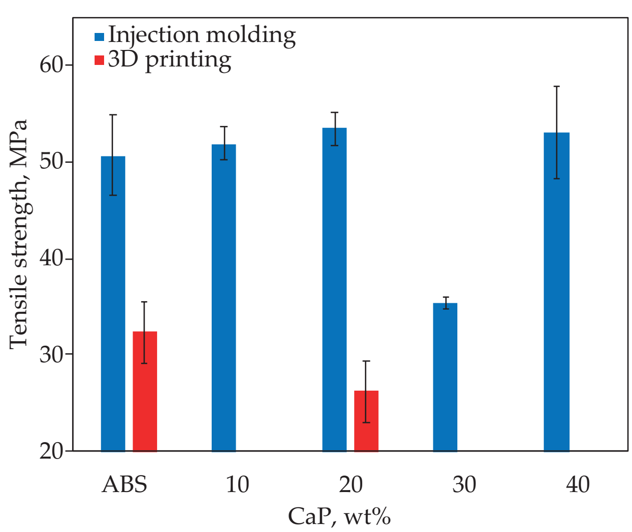

Fig. 4. Tensile strength of ABS and ABS/CaP composites

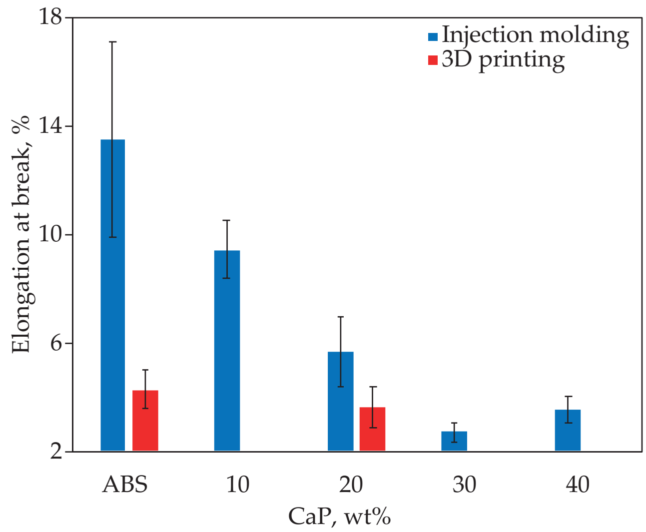

Fig. 5. Elongation at break of ABS and ABS/CaP composites

has been shown in the literature that adding more phosphate filler increases the resistance to dent damage [40]. The ABS/CaP 60/40 composite showed the lowest hardness, which may be due to the uneven mixing of a large amount of filler in the polymer matrix, which in turn could have led to the formation of agglomerates and aggregates of the filler in the polymer matrix. However, the samples obtained by 3D printing are characterized by good hardness compared to ABS.

In the case of samples obtained by injection molding, the higher the filling level, the greater the Young's modulus, which is associated with the greater stiffness of the composites (Fig. 3) and is consistent with the hardness results (Fig. 2).

It was observed that ABS/CaP composites have better mechanical properties compared to ABS. However, the properties of samples obtained by 3D printing are worse than those of ABS. This can be explained by weak interactions at the filler-polymer interface and limited adhesion between printed layers [41].

Moreover, CaP had no effect on the tensile strength, with significantly lower strength obtained by 3D printing (Fig. 4). The exception was the composite containing 30 wt% CaP. Other scientists have also reached similar conclusions [41].

Figure 5 shows the effect of the filler on the elongation at break of ABS. The presented results closely coincide with those discussed earlier, because as the amount of filler increases, the elongation at break decreases, which indicates greater stiffness of the composites. However, in the case of samples obtained using 3D printing technology, no significant difference was observed between the results obtained for the composite and ABS.

T a b l e 4. Color change data of ABS and ABS/CaP 80/20 composite

CaP, wt%	L	a	b
0	72.77±4.27	0.23±0.11	10.36±0.24
20	83.16±7.35	-2.28±1.77	10.62±0.18

The color change after introducing the filler into the ABS polymer matrix is presented in Table 4. Color was determined using an independent CIE Lab model, where "L" describes luminance (brightness from black to white), "a" describes color from green to purple, and "b" describes color from blue to yellow. The composite had a lighter color compared to ABS, which showed a slight red shift, while ABS/CaP 80/20 had a green shift. However, the materials have similar values of the "b" parameter, which suggests some similarity in shades of yellow.

CONCLUSIONS

Calcium phosphate-filled ABS composites (10–40 wt%) were successfully homogenized using a mini lab twin-screw extruder. The addition of calcium phosphate had a significant impact on the processability, hardness and tensile properties of ABS. Due to good fluidity and the highest tensile strength, a composite containing 20% CaP was selected for 3D printing. The composites were characterized by better mechanical properties compared to ABS. However, the properties of samples obtained by 3D printing were worse than those obtained by injection molding and even ABS. This can be explained by weak interactions at the filler-polymer interface and limited adhesion between printed layers. A change in the color of ABS after adding the filler was also observed. The ABS/CaP 80/20 composite had a lighter color compared to the unmodified polymer matrix.

Further research will include, among others: morphology, thermal resistance, susceptibility to the sterilization process and in vitro degradation time. The conducted research will determine the application potential of the obtained polymer composites.

ACKNOWLEDGMENTS

Katarzyna Bulanda would like to thank Prof. Ph.D. Sylwester Czopek, Rector of the University of Rzeszów for enabling the research internship at the University of Rzeszów, as well as the scientific supervisor Dr hab. n. med. Eng. Dorota Bartusik-Aebisher, Prof. UR and Dr hab. n. med. David Aebisher, Prof. UR for your kindness and time to work together at the Center for Innovative Research in Medical and Natural Sciences.

Authors contribution

K.B. – conceptualization, methodology, formal analysis, investigation, writing-original draft, visualization; D.B-A. – writing-review and editing; M.O.: investigation; D.A. – writing-review and editing. All authors have read and agreed to the published version of the manuscript.

Funding

This research received no external funding.

Conflict of interest

The authors declare no conflict of interest.

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Received 28 III 2024.
Accepted 18 IV 2024.

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