INVESTIGATION ON THE DEGRADATION RATE OF PCL POLYMER SCAFFOLDS WITH BIOGLASS AND GRAPHENE ADDITIVES USING COMPUTER MICROTOMOGRAPHY

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Introduction

Scientific research describing the phenomenon of degradation of bioresorbable polymers intended for medical applications such as PLLA, PGLA or PCL is carried out on the basis of the ISO 10993 standard for medical devices. These tests are based on the measurement of both changes in the material (mainly related to the surface characteristics or mass change) and the medium in which the material is incubated. Depending on the material tested, surface changes used by optical microscopy or SEM can be observed after several months of incubation. Due to the resolution obtained with the use of non-destructive computer microtomography technique (up to 0.5μmm), volumetric changes of the tested samples can be observed earlier, which makes it possible to better understand the phenomena related to the degradation of the material.

Materials and Methods

The research material consisted of four groups of scaffolds: PCL, PCL with bioglass, PCL with bioglass and zinc, and PCL with graphene produced by 3D printing, FDM technique [1,2]. Original Prusa® i3 MK3 printer, working in the FDM technique, was used for printing. The bed temperature to increase the adhesion for the first layer was 60°C and the extruder temperature was 190°C. The duration of the printout was about 14-15 minutes. The base material was pure poly (ε-caprolactone) PCL, (Sigma Aldrich) with a molecular weight of 80 kDa. Measurement samples with dimensions of 10x19.7x3 mm were prepared from three layers that are arranged alternately at an angle of 90°. The height and width of each layer are 1 mm and the distance between the tracks is 0.7 mm. For each of the studied groups, 5 measurement samples were prepared. Samples were incubated in PBS solution. Measuring the weight of scaffolds and the pH value, conductivity and concentration of Na^+, K^+, Ca^{2+} ions in the incubation fluid, as well as qualitative and quantitative measurement of structural properties using computer microtomography assessed the *in vitro* degradation of materials.
The registration of samples with a resolution of 10μm and lamp parameters 40kV/250μA, was performed using a SkyScan 1172 computer microtomograph by Bruker®.
A series of tests was carried out over a period of 22 weeks, the first stage was 14 days later and each subsequent stage was 30 days apart, according to standard 10993-13.

Results and Discussion

Research with the use of microtomography and dedicated programs such as DataViewer and CtAn (Bruker) allowed for the qualitative and quantitative assessment of changes occurring at individual stages of polymer degradation. The possibility of comparing and submitting several reconstructions of the same samples obtained in different stages of degradation allowed to obtain pictures of the differences of the samples before and after 22 weeks of incubation, which was 6 months. These effects for exemplary PCL samples with bioglass and graphene are shown in FIG. 1.

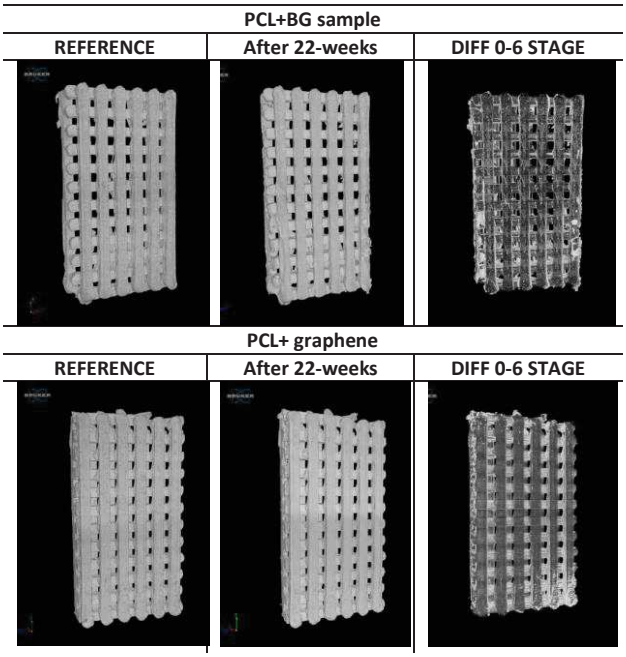

FIG. 1. Comparison of the reconstruction of sample samples: PCL with bioglass and PCL with graphene, along with a representation of the differences of these samples before and after 6 months of incubation

Conclusions

In all the scaffolds tested, a similar pattern of degradation can be seen. The comparative analysis of the individual stages allowed the observation that the corners of the samples change first, only then the center. A characteristic "cross" is formed. On the other hand, quantitative studies showed the greatest changes in the tested parameters (pH, mass and conductivity) between the 4th and 5th stage of incubation. The structural changes were associated with a decrease in the concentration of Na^+, K^+ and Ca^{2+} ions in the fifth stage of degradation. The research presented here covers a period of 6 months, but it is only a fragment of the designed experiment, which is assumed to last 18 months, thanks to which it will be possible to obtain information on a longer degradation time.

References

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