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ELECTRIC PROPERTIES OF BIODEGRADABLE COPPER FILLED POLY(LACTIDE-CO-GLYCOLIDE) COMPOSITES

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Abstract

The results of experimental studies of the properties of composite materials based on Poly(lactide-co-glycolide) (PLGA) matrix filled with electrolytic copper powder, having very high dendritic structure, are presented in this manuscript. Copper powder volume fractions used as filler in all prepared composites were varied in the range of 0.4-7.2 vol. %. The samples were prepared by hot moulding injection at 140°C. Influence of morphology on the conductivity and percolation threshold of the composites was examined and characterization included: Electrical conductivity measurements using Impedance Spectroscopy (IS), Scanning Electron Microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDS) and Fourier-transform Infrared Spectroscopy (FTIR). Presence of three dimensional conductive pathways was confirmed. The obtained percolation threshold was 2.72 vol.%, which is about three times lower than the one stated in the literature for similar composites. This property is ascribed to different morphology of filler used in investigation.

Key words: *electrical conductivity, biodegradable composite material, Poly(lactide-co-glycolide), electrolytic copper powder, FTIR, hardness*

Introduction

Electronic devices represent essential parts in everyday lives in modern society. Although these electronic devices offer vigorous and consistent performance with a long service lifetime, the ever fast enhancement along with the rapid propagation leads to increasing electronic waste. This e-waste is larger than 50 million tons every year (Heacock et al., 2016; K. Zhang, Schnoor, & Zeng, 2012). In recent years, recycling and disposal of e- waste have become major topics and of global concerns (Robinson, 2009; Widmer, Oswald-Krapf, Sinha-Khetriwal, Schnellmann, & Böni, 2005). One of the potential solutions to the growing e- waste issue lies in so called "transient electronics" (Bauer & Kaltenbrunner, 2014; Cheng, 2016; Fu, Wang, Dai, Carter, & Hu, 2016; Huang, 2018; X. Yu, Shou, Mahajan, Huang, & Pan, 2018). Transient electronic devices, unlike conventional devices that are built to last, are physically disintegrable after a short and controlled lifetime when they are subjected to specific environment elements such as water and other biofluids (Huang, 2018; Hwang et al., 2013, 2012; X. Yu et al., 2018), moisture (Gao, Zhang, et al., 2017) light (Hernandez et al., 2014) and heat (Gao, Sim, et al., 2017; Lee et

al., 2015; Park et al., 2015; X. Zhang & Bellan, 2017). This transiency is manifested by diminishing of physical and electronic properties, and it is mostly programmable by material selections (Çınar, Jamshidi, Chen, Hashemi, & Montazami, 2016; Gao, Zhang, et al., 2017).

Bioresorbable electronics refer to a new class of advanced electronics that can completely dissolve or disintegrate with environmentally and biologically benign byproducts in water and biofluids. Bioresorbable materials such as biodegradable polymers, dissolvable conductors, semiconductors, and dielectrics are extensively studied, enabling massive progress of bioresorbable electronic devices (Hwang et al., 2014; X. Yu et al., 2018). Processing and patterning of these materials are predominantly relying on vacuum-based fabrication methods so far.

Most of the devices are designed for biomedical applications, including temporally implantable diagnostic/therapeutic devices and conformal biosensors. Their applications lie in physiological sensing (Kang et al., 2016; K. J. Yu et al., 2016), health monitoring (Boutry et al., 2015), drug delivery (Son et al., 2015; Tao et al., 2014), etc.

Poly(lactide-co-glycolide) (PLGA) is biodegradable copolymer usually synthesized by means of ring-opening co-polymerization of glycolic acid (GA) and lactic acid (LA). Depending on the ratio of LA to GA used for the polymerization, different forms of PLGA can be obtained (X. Yu et al., 2018). These are usually identified in regard to the molar ratio of the monomers used (Félix Lanao et al., 2013; Gentile, Chiono, Carmagnola, & Hatton, 2014). Poly(lactic-co-glycolic acid) (PLGA) is the most often used biocompatible and biodegradable synthetic polymer. A large number of medical devices comprising PLGA have been approved for clinical use in humans by the American Food and Drug Administration (Félix Lanao et al., 2013). As compared with the homopolymers of lactic acid poly(lactic acid) and poly(glycolic acid), the co-polymer PLGA is much more versatile with regard to the control over degradation rate (Shen, Hu, Bei, & Wang, 2008; L. Yu & Ding, 2008). As a material for bone regeneration, the use of PLGA has been extensively studied for application and is included as either scaffolds, coatings, fibers, or micro- and nanospheres to meet various clinical requirements. The crystallinity of PLGA varies from fully amorphous to fully crystalline depending on block structure and molar ratio (Swider et al., 2018). Different PLGA typically show a glass transition temperature in the range of 40-60 °C. In living systems PLGA slowly degrade into natural metabolites, such as lactic and glycolic acids. Although many published studies have demonstrated that unmodified PLGA is a poor substrate for cellular attachment and growth because of its low surface energy and high hydrophobicity (Yang et al., 2002; H. Zhang, Wang, Wang, & Xu, 2018), PLGA has been most widely used among the various available biodegradable polymers for bone screws, drug delivery and vascular grafts. PLGA-coated bioalloys or bioceramics have been reported in many research studies (Xi et al., 2010).

The research effort on electroconductive polymers filled with metal powder has had a good development in the last few decades. The addition of a metal base in a polymer matrix allows the mechanical properties of the polymer to be retained while exploiting the electrical properties of the metal (Poblete, Alvarez, & Fuenzalida, 2009). Conductivity of these polymer composites is largely dependent on the nature of the contact between the filler particles and is critically dependent on the volume fraction of the conductive filler particles. This is well explained by the percolation theory (Janković et al., 2018; E. P. Mamunya, Davidenko, & Lebedev, 1996; Y. P. Mamunya, Davydenko, Pissis, & Lebedev, 2002; Pavlovic et al., 2012; Pavlović et al., 2014).

However, percolation threshold, electrical conductivity and electrical behavior of composite systems with fillers which have highly developed surface area have not been explored in details. Also, systems with biodegradable polymers matrices, such as PLGA, have not been studied in the literature. Therefore there is need for more detailed study of real synergetic effects of different fillers dimensionalities suitable for construction of conductive networks in conductive polymer composites with biodegradable matrix. For this reason, the copper powder was galvanostatically produced with distinct dendritic morphology and large surface area.

The need for highly conductive polymer-based materials has been the motivation to develop poly

(lactide-co-glycolide) (PLGA) composites filled with electrodeposited copper powder particles which would retain desirable polymer characteristics, including biodegradability, ease of processing, and that would obtain high conductivity at low cost. The strategy consisted in manipulating the morphology of the filler so that high conductivity could be achieved at a low percolation threshold and the results of electrical conductivity of PLGA composites filled with galvanostatically produced copper powder particles are shown in this paper.

Experimental Part

In the experimental part of the work, ester terminated Poly(D,L-lactide-co-glycolide) (PLGA) with lactide to glycolide ratio of 85:15 was used as matrix for prepared composites. PLGA polymer was used as commercially available powder supplied by Sigma-Aldrich. PLGA had average molecular weight of $M_w \sim 190,000\text{--}240,000$, with a density of 1.27 g/cm^3 , and the electrical conductivity of about 10^{-12} S/cm .

Copper powder was produced by the galvanostatic electrolysis regime under following conditions: current density: $j = 3600 \text{ A/m}^2$, time of powder growth: $\tau_r = 15 \text{ min}$, electrolyte flow: $Q = 1$ change of the cell volume/h, temperature of the electrolyte: $t = (50 \pm 2) \text{ }^\circ\text{C}$, concentration of copper: $c(\text{Cu}^{+2}) = 15 \text{ g/dm}^3$ and concentration of sulfuric acid: $c(\text{H}_2\text{SO}_4) = 140 \text{ g/dm}^3$. Both cathode and anode were made out of copper. All electrolytes were prepared from p.a. chemicals obtained from Merck and demineralized water.

At the end of the precipitation, the powder was removed from the electrode by brushing it. The powder was then washed, protected from oxidation, and the stabilization and drying processes were performed.

Produced copper powder was washed with distilled water at the room temperature until the powder was acid free, and then it was washed with 1 g/dm^3 aqueous solution of a benzoic acid (Sigma-Aldrich) with the aim to protect the powder from succeeding oxidation. After drying in a tunnel furnace at $130 \text{ }^\circ\text{C}$ in controlled nitrogen atmosphere, the produced copper powder was sieved through mesh with openings of $45 \text{ }\mu\text{m}$.

Polymer composites filled with galvanostatically produced copper powder were prepared with the filler volume fraction ranging from $0.4 \text{ } \%$ (v/v) – $7.2 \text{ } \%$ (v/v). Pure PLGA and copper samples were prepared as reference materials. PLGA was preheated and melted at $t = 140 \text{ }^\circ\text{C}$ for 30 min. Previously measured amount of copper powder was added afterwards and mixed until the mixture was fully homogenized. Samples were produced from this homogenized mixture in the molder, Atlas Polymer Evaluation Products LMM Model H30, having size $3.9 \times 10.3 \times 13.3 \text{ mm}$. After composite preparation samples were cooled at the room temperature for about 30 min. In order to obtain flat surface for conductivity measurements, samples were polished with sandpaper.

Electrical conductivity was measured by impedance spectroscopy (IS). Experiments were performed in potentiostatic mode on all prepared composites. Instrumentation involved Bio-Logic® SAS Instrument, model SP-200, guided by EC-Lab® software. Samples were put between two metal plates and they responded to the potential input sinusoidal signal of $\pm 10 \text{ mV}$ (rms) amplitude. Geometry of the instrument contacts used is such that it minimizes edge effects thus it can be assumed that they do not exist. Experimental IS data were fitted by ZView® software.

Scanning Electron Microscopy (SEM) analysis of PLA and PHB composites and constituents was performed on Tescan Mira 3 XMU FEG-SEM. Energy-dispersive X-ray spectroscopy (EDS) of the compacts was performed on a Jeol JSM 5800 SEM with a SiLi X-Ray detector (Oxford Link Isis series 300, UK).

Fourier transform infrared spectroscopy (FTIR) was recorded on stabilized copper powder, PLGA and composites. Michelson MB Series Bomen FTIR was used (Hartmann Braun), scanning from 500 to 4000 cm^{-1}

Results and Discussion

Figure 1 shows morphology of galvanostatically produced copper powder particles, from which can be noticed very dendritic 3D (three-dimensional) structure of the obtained powder.

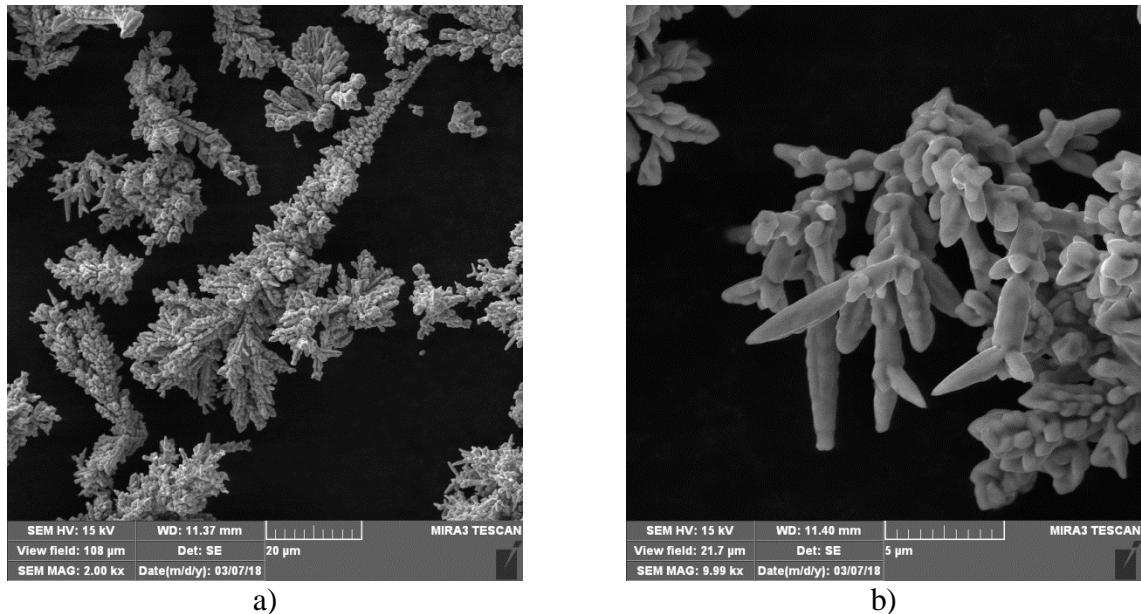


Figure 1. SEM microphotographs of Cu powder particles obtained by the galvanostatic regime of electrolysis and sieved through mesh < 45 μm: a) general view and b) single particle view.

It can be seen from image scales on Figure 1 that typical copper powder particle is < 45 μm in size. The high dendritic character of the obtained particles is a good prerequisite for the formation of a larger number of interparticle contacts between the conductive powder particles and lowering of percolation threshold. Also, this very branchy structure of the particles enables formation of multiple contacts with neighbouring particles at lower filler volume fractions.

The electrical conductivity of the PLGA composites as a function of filler content for all prepared samples was measured as stated in Experimental part. The electrical conductivity of composites as a function of filler content for all the samples was measured and calculated from impedance characteristics of a composite using Equation (1):

$$\sigma = \frac{1}{\sum_1^7 R_i} \cdot \frac{l}{S} \quad (1)$$

where σ is electrical conductivity, R_i – resistivity of a layer in equivalent circuit, l – length and S – cross-section area of the sample. The conductivity of PLGA composites, showing typical S-shaped dependency with three distinct regions: dielectric, transition and conductive, as expected, is shown on Figure 2.

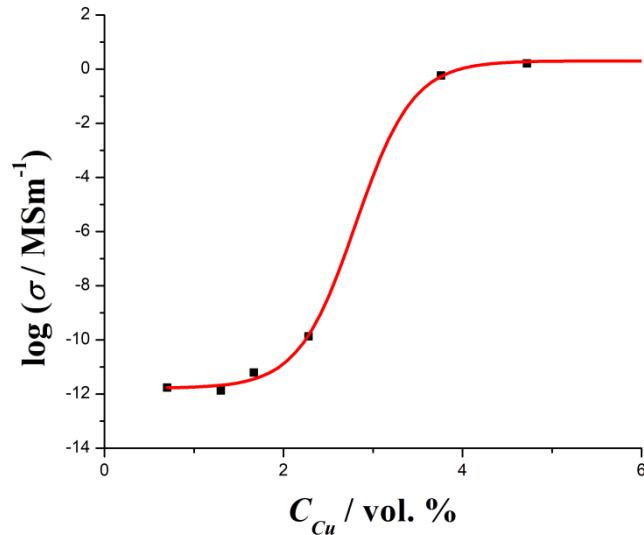


Figure 2. Change in electrical conductivity as a function of filler volume fraction for a) PLA composites and b) PHB composites filled with copper powder

Percolation threshold value was obtained from the maximum of conductivity derivative as a function of volume fraction of filler. As it can be seen from Figure 2, as well as from calculated value, the percolation threshold was at 2.72 vol.% (v/v) for PLGA. This low value that occur is most likely due to filler shape, *i.e.* powder particles, which were very dendritic with high-developed free surface area, and hence less filler was needed to form conductive network throughout the composite volume. Namely, more regular, rounded shapes of copper powder filler, obtain higher values of percolation threshold. Experiments have shown that the morphology of the particles plays a crucial role for the percolation threshold appearance. Besides the filler morphology, polymer matrix type, as well as preparation method influences the percolation threshold, which further moves towards lower values.

For investigation of electrical conductivity, as well as for morphology examination of PLGA composites, cross sections of the samples at percolation threshold perpendicular to the surfaces at which the electrical conductivity was measured, were made. This cross section was 3.9×13.3 mm in size. Figure 3 shows SEM image of the cross section of PLGA composite filled with copper powder at percolation threshold. Two different phases can be seen on Figure 3. However, mechanism of the electrical conductivity, as well as interparticle contacts could not be clearly concluded from the Figure 3. For this reason EDS measurements were performed on same cross section of PLGA-Cu composite. These results are shown in Figure 4.

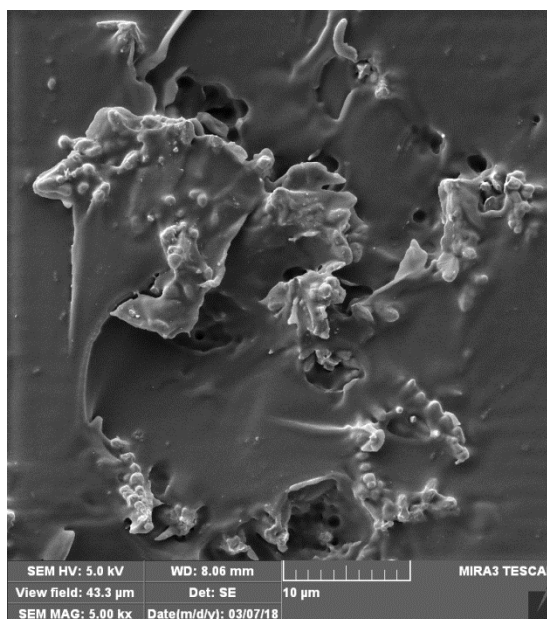


Figure 3. SEM image of cross section of PLGA composite filled with galvanostatically produced copper powder at percolation threshold. Cu powder particle size <math>< 45 \mu\text{m}</math>.

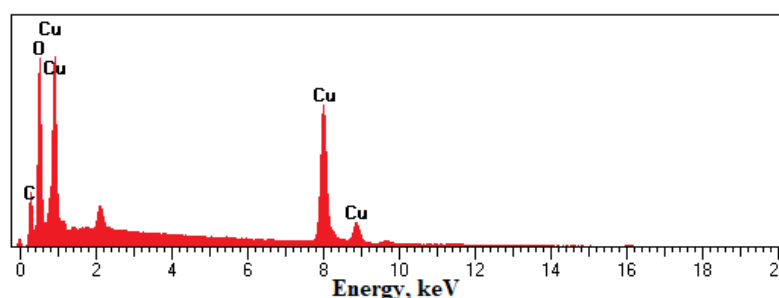


Figure 4. EDS spectrums of cross section of PLGA-Cu composite surface with analyses of different phases.

Full surface EDS analysis was performed on the samples cross section. Results of surface area EDS analysis are shown in Table 1.

Table 1. Elemental analysis of cross sections of PLGA composite filled with Cu powder at percolation threshold

Element type	Atomic %
C	48.12
O	43.11
Cu	8.77
Total	100

From the measurements of electric conductivity, and knowing that presented sample is conductive and that it is at percolation threshold, it can be concluded that conductive pathways are formed throughout the surface of the composite, since the size of copper phase present in the figures (> 80 μm) is much larger than size of copper powder particles (< 45 μm from Figure 1) used for composite preparation. Clearly, composites conduct electricity through conductive pathways that are formed in 3D in pure random order.

Figure 5 shows results of the FTIR spectra of the experiments performed on stabilized copper

powder, PLGA and its composite with 4.8% (v/v) copper powder content. FTIR spectrum of the Cu powder shows three distinct peaks at 3431 cm^{-1} characteristic for O-H stretching vibrations, 2924 cm^{-1} characteristic for C-H stretching vibrations and 1625 cm^{-1} characteristic for C=C aromatic ring stretching vibrations. This is in good agreement with statement that copper powder was stabilized with benzoic acids, in order to protect the powder against subsequent oxidation. It can be concluded that there is no chemical reaction occurring between PLGA and copper powder, and that the influence of the O-H, C-H and C=C groups on the spectra is amplified. There is slight or no shift in the wavenumber between the studied samples.

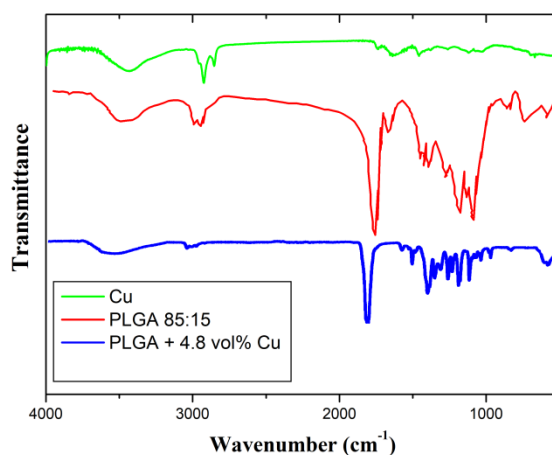


Figure 5. FTIR spectra of the pure stabilized copper powder, PLGA and its composite at 4.8 vol. % Cu content.

Conclusions

In this article, experimental study about the effects of electrodeposited copper powder content on the electrical conductivity of PLGA composites filled with that powder has been described. Results have shown that the powder has very high surface area and it has pronounced dendrite branching with well-developed primary and secondary dendrite arms. The conductivity measurements showed S-shaped dependency with percolation transition from non-conductive to conductive region, typical for such polymer composite materials. The results showed that the shape and morphology of the copper powder, and filler at all, play a significant role in the phenomenon of electrical conductivity of the prepared samples and the appearance percolation threshold. Conductivity measurements have shown that percolation threshold is at 2.72 vol. % Cu. The results showed that conductivity of PLGA composites is much improved comparing to similar composites filled with more regular structure fillers that can be find in the literature. Morphology of the samples showed presence of conductive pathways throughout the sample, which was proven by EDS measurements. Clearly, it was shown that composites conduct electricity throughout conductive pathways that are formed in 3D in pure random order. FTIR measurements have shown that there is no chemical reaction occurring between PLGA and copper powder.

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