

Electrical conductivity of poly (L lactic acid) and poly (3-hydroxybutyrate) composites filled with galvanostatically produced copper powder

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Abstract

This manuscript presents experimental studies of composite materials based on poly (L lactic acid) (PLLA) and poly (3-hydroxybutyrate) (PHB) matrices filled with electrolytic copper powder, having a very high dendritic structure. Volume fractions of the copper powder used as filler in all prepared composites were varied in the range 0.5-6.0 vol.%. Samples were prepared by hot moulding injection at 170 °C. Influence of particle size and morphology, as well as the influence of matrix type on conductivity and percolation threshold of the obtained composites were examined. Characterization included: electrical conductivity measurements using AC 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 thresholds of 2.83 vol.% for PLLA and 3.13 vol.% for PHB composites were measured, which is about three times lower than the ones stated in the literature for similar composites. This property is ascribed to different morphologies of the filler used in the present investigation.

Keywords: oily water; electrical conductivity, composite materials, PLLA, PHB, electrolytic copper powder

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1. INTRODUCTION

During the past century, many synthetic polymers made from non-renewable fossil fuels such as polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polyethylene terephthalate (PET), polyamide (PA) and polystyrene (PS) have been used due to their extraordinary properties. These properties include high availability at a relatively low cost, good mechanical performance, such as tensile and impact strengths, good barrier properties, etc. [1]. However, production of these synthetic polymers has to be limited as it leads to depletion of oil resources. The production rate of plastic waste is growing annually. Although plastic material can be recycled, only a small number of these materials can actually be completely recycled [2]. Therefore, biodegradable bioplastics have become important and replace some of the conventional plastic products in the short life cycle. Biodegradable plastic materials have significantly increased the interest of researchers and industry due to environmental problems related to accumulation of the plastic waste. Therefore, biodegradable alternatives are highly desirable. Poly (lactic acid) (PLA) is a biodegradable polymer, which is very suitable for applications in products of a short life cycle, such as packing and containers and cutlery [3]. It can be manufactured as filaments by spinning for subsequent fabrication of preferred textile structures. Medical applications of this polymer arise from its biocompatibility, since the PLA degradation product, lactic acid, is metabolically harmless. PLA fibres can be produced in various shapes and can be used for implants and other surgical applications. Tissue engineering is the newest domain in which poly (lactic acid) is used and is one of the most favourable matrix materials [4].

PLA has numerous advantages including: production of the lactide monomer from lactic acid, significant energy savings in the production process, ability to be recycled back to lactic acid by hydrolysis or alcoholysis; production of

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packaging that is compostable, and the ability to tailor physical properties via material modifications [5]. Drawbacks in PLA applications include a relatively high price, high stiffness and poor moisture barrier properties [6-9].

On the other hand, poly (3-hydroxybutyrate) (PHB) is fully biodegradable and biocompatible polyester synthesized by bacterial fermentation from renewable sources such as sugar cane. This biopolymer attracted recently a lot of attention as an alternative to oil-based polymer materials [10]. PHB is an isotactic linear thermoplastic made of 3-hydroxy butyric acid. It has been already used in small disposable products and packaging materials [11]. However, it possesses several imperfections limiting the commercial use, such as high crystallinity, resulting in inherent brittleness and poor impact resistance, relatively high water vapour permeability, and a low resistance to thermal degradation. Thus, due to these properties PHB can be easily decomposed while having a relatively high melting point (about 175 °C) resulting in a narrow processing window. In order to expand the number of feasible applications, new approaches for practical use of PHB included mixing with other polymers [11,12] or filling with nano-fillers such as coffee waste [13], multilayer carbon nanotubes [14], hydroxyapatite (HA) [15,16], lignocellulose [17], pulp fibres [18] or layered silicates [19]. Improved mechanical performance as well as thermal and barrier properties were obtained by adding small amounts of nanoparticles into this biopolymer matrix, although improvements are still insufficient for industrial applications.

Biocomposites are made from bioplastics that are environmentally friendly and biodegradable, and they can be filled with different materials. Development of these eco-composites is rapid in recent years, mainly due to improvements in process technologies and economic factors [20]. Today, various combinations of fillers and bioplastics have been successfully made resulting in eco-composites with improved mechanical properties, as well as of low cost [20,21].

In the last few decades there has been a considerable research effort on development of electroconductive polymers filled with metal powders. The addition of a metal base in a polymer matrix allows preservation of mechanical properties of the polymer while supplementing electrical properties of the metal [22]. Conductivity of these polymer composites is largely dependent on the nature of the contact between the filler particles and is critically dependent on the particle volume fraction. These phenomena are well explained by the percolation theory [23-26].

In the last few years there have been numerous reports on different polymers containing dispersed conductive fillers as well as on various production methods. Metal filled polymer composites have found the use in electromagnetic protective shielding and electronic devices, as adhesives, in electronic packaging, switches and overvoltage protection devices [22,27,28]. They have also found numerous applications as self-regulating heaters, chemical and electrochemical catalysts and adsorbents [29]. These polymer based composites exhibit some advantages over pure metals, including a very low price, ease of production, high flexibility, reduced weight, corrosion resistance and conductivity control [22].

However, percolation threshold, electrical conductivity and electrical behaviour of composite systems with fillers, which have highly developed surface area, have not been explored in detail. Also, systems with biodegradable polymer matrices, such as PLLA and PHB, have not been studied in the literature. Therefore, there is a need for more detailed study of synergetic effects of different filler dimensionalities suitable for construction of conductive networks in conductive composites with biodegradable polymer matrices. For this reason, in this work, copper powder was galvanostatically produced with distinct dendritic morphology and a large surface area. The aim was to produce highly conductive PLLA and PHB based composites filled with electrodeposited copper particles, which would retain desirable polymer characteristics, including biodegradability and ease of processing, while exhibiting high conductivity at low cost. The strategy consisted in manipulating the filler morphology, so that high conductivity could be achieved at a low percolation threshold.

2. EXPERIMENTAL

In the experimental part of the work, poly (L lactic acid) (PLLA) and poly (3-hydroxybutyrate) (PHB) were used as matrices. Both polymers were used as commercially available powders supplied by Sigma-Aldrich (Germany). PLLA had the average molecular weight of $\approx 10,000 \text{ g mol}^{-1}$, with a density of 1.25 g cm^{-3} , and the electrical conductivity of about $10^{-12} \text{ S cm}^{-1}$. PHB had the average molecular weight of $\approx 100,000 \text{ g mol}^{-1}$, with a density of 1.25 g cm^{-3} , and the electrical conductivity of about $10^{-12} \text{ S cm}^{-1}$.

Copper powder was produced by electrolysis in the galvanostatic 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^{-1} , temperature of the electrolyte: $t = (50 \pm 2)^\circ \text{ C}$, copper concentration: $c(\text{Cu}^{+2}) = 15 \text{ g dm}^{-3}$ and sulphuric acid concentration: $c(\text{H}_2\text{SO}_4) = 140 \text{ g dm}^{-3}$. In the electrolysis process, both cathode and anode were of copper. All electrolytes were prepared using p.a. grade chemicals obtained from Merck (Germany) and demineralized water.

At the end of precipitation, powder was removed from the electrode by shaking. The powder was then washed, protected from oxidation, stabilized and dried.

Since, the acidic environment promotes rapid powder oxidation during the drying process, the produced copper powder was washed with distilled water at room temperature until all acid traces were removed. After this phase, the copper powder was washed with the aqueous solution of a sodium soap SAP G-30 (Henkel Merima, Serbia) with the aim to protect the powder from succeeding oxidation [34,35]. After drying in a tunnel furnace at 110 – 120° C in the controlled nitrogen atmosphere, the produced copper powder was sieved through a mesh with openings of 45 μm .

Polymer composites filled with the galvanostatically produced copper powder were prepared at the filler volume fractions ranging from 0.5 – 6.0 vol.%. Pure PLLA, PHB and copper samples were prepared as reference materials. Both PLLA and PHB powders were preheated and melted at $t = 170$ °C for 30 min. A previously measured amount of copper powder was added and mixed until the mixture was fully homogenized. Composite samples in the form of a slab (3.9×10.3×13.3 mm) were produced from this homogenized mixture in the molder, Atlas Polymer Evaluation Products LMM Model H30 (producer, country). The samples were then cooled at the room temperature for about 30 min. In order to obtain a flat surface for conductivity measurements, the obtained samples were polished with sandpaper.

Electrical conductivity was measured by AC impedance spectroscopy (IS). Experiments were performed in the potentiostatic mode using all prepared composites. Instrumentation involved Bio-Logic® SAS Instrument, model SP-200 (producer, country), guided by EC-Lab® software (producer, country). Samples were placed between two metal plates and the response to the input sinusoidal potential signal of ± 10 mV (rms) amplitude was monitored. The instrument geometry of the contacts is such that it minimizes edge effects so that they can be neglected. Experimental IS data were fitted by ZView® software (producer, country).

The sample thickness (necessary for conductivity calculation) was determined by using a micrometre, to an accuracy of 0.01 mm. Several thickness measurements were performed per sample and then averaged.

Scanning electron microscopy (SEM) analysis of PLLA and PHB composites and constituents was performed on a Tescan Mira 3 XMU FEG-SEM instrument (producer, country). Composite samples were broken by applying of vertical force; they were mounted on the carrier with broken side facing up and plated with gold. Energy-dispersive X-ray spectroscopy (EDS) of the composites was performed on a Jeol JSM 5800 SEM instrument (producer, country) with a SiLi X-Ray detector (Oxford Link Isis series 300, UK).

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

3. RESULTS AND DISCUSSION

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

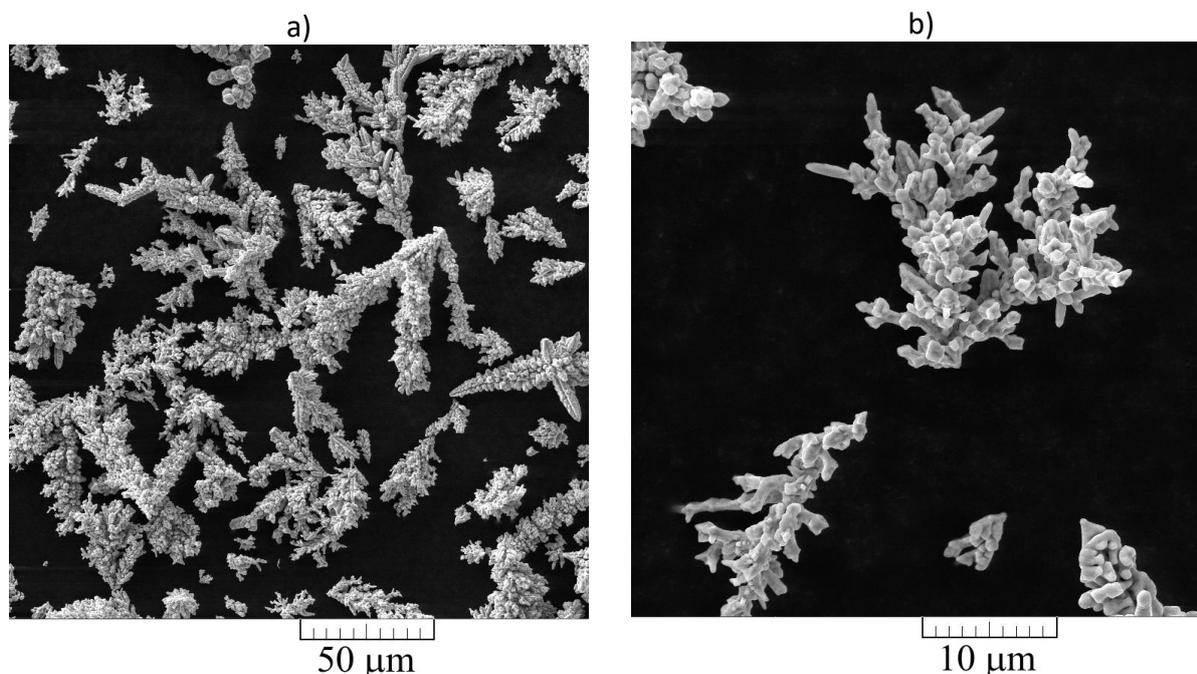


Figure 1. SEM micrographs of Cu powder particles obtained by electrolysis in the galvanostatic regime and sieved through a mesh <math> < 45 \mu\text{m}</math>: a) lower magnification image, scale bar: 50 μm b) higher magnification image, scale bar: 10 μm

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 formation of a larger number of interparticle contacts for achievement of conductive properties as well as lowering the percolation threshold. Also, this very branchy structure of the particles enables formation of multiple contacts with neighbouring particles at lower filler volume fractions.

Electrical conductivity of the obtained PLLA and PHB composites as a function of filler content for all prepared samples was measured and calculated from impedance characteristics of the composite by using the equation:

$$\sigma = \frac{1}{\sum_{i=1}^7 R_i} \frac{l}{S} \tag{1}$$

where σ is the electrical conductivity, R_i is the resistivity of a layer in the equivalent circuit, l is the length and S is the cross-section area of the sample.

Registered impedance data were simulated by equivalent electrical circuits based on a transmission line model. The equivalent electrical circuits which gave the best fitting results (*chi-squared* based on modulus calculated was below 0.001, while the relative error of parameter values of the elements did not exceed 20 %) are schematically presented in Figure 2.

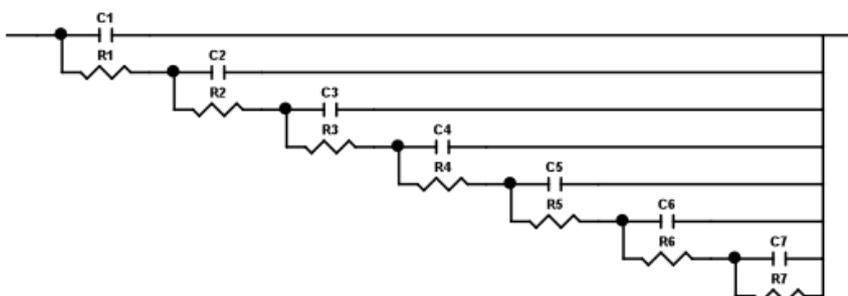


Figure 2. The most suitable equivalent electrical circuits used to simulate the IS data of PLLA-Cu and PHB-Cu composites

These transmission line circuits of the composites represent distributed RC time constants as a measure of accessibility of different parts of the internal surface of a layer. Circuits include the ohmic resistance in the outer layer, R_1 , and subsequent resistances, R_n ($n=2-7$), related to conductivity in the inner layers. Number of resistors related to the layer resistance depends on the filler morphology and particle size. Capacitances of the overall composite surface, available through the layer resistances, are represented by a network of capacitors of the capacitances C_n .

Conductivities of both PLLA and PHB composites are showing a typical S-shaped dependency with three distinct regions: dielectric, transition and conductive, as expected (Fig. 3).

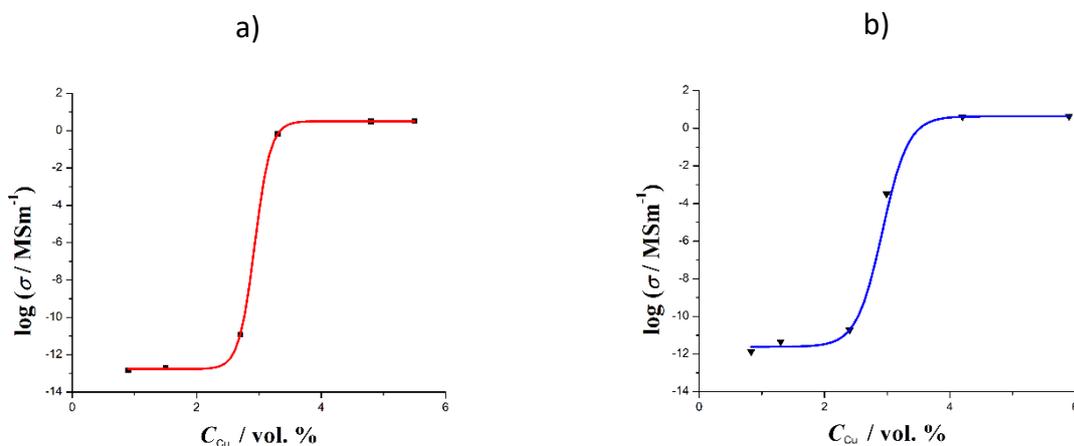


Figure 3. Electrical conductivity as a function of the filler volume fraction for a) PLLA composites and b) PHB composites filled with copper powder

Percolation threshold value was calculated as the maximum of the derivative of conductivity as a function of the filler volume fraction. As it can be seen from Figure 3, as well as from calculated value, the percolation threshold was at 2.83 vol.% for PLLA composites and 3.13 vol.% for PHB composites. These low values that occur are most likely due to the filler shape, *i.e.* powder particles, which were very dendritic with high-developed free surface area, and hence less filler was needed to form a conductive network throughout the composite volume. Namely, a more regular, rounded shapes of the copper powder filler, results in higher values of the percolation threshold. Experiments have shown that the morphology of particles plays a crucial role for the percolation threshold level. Besides the filler morphology, the polymer matrix type, as well as the preparation method influence the percolation threshold, which further moves towards lower values.

The obtained values of the percolation threshold are about three times lower than the ones stated in literature for composites with the same filler but different (PMMA) matrix [22]. These data can be used as a comparison since the reported system is the closest to the ones presented in this manuscript, while it is for the first time that PLLA and PHB polymers are used as matrices.

For investigation of electrical conductivity, as well as for morphology examination of PLLA and PHB composites, the samples at the percolation threshold were cross-sectioned perpendicular to the surfaces at which the electrical conductivity was measured. The cross sections were 3.9×13.3 mm in size, and polished before further investigation. Figure 4 shows SEM images of the cross sections of PLLA and PHB composites at the percolation threshold in which two different phases can be distinguished. However, the mechanism of the electrical conductivity, as well as interparticle contacts could not be clearly concluded from the Figure 4. For this reason, EDS measurements were performed on the same cross sections of composites (Fig. 5).

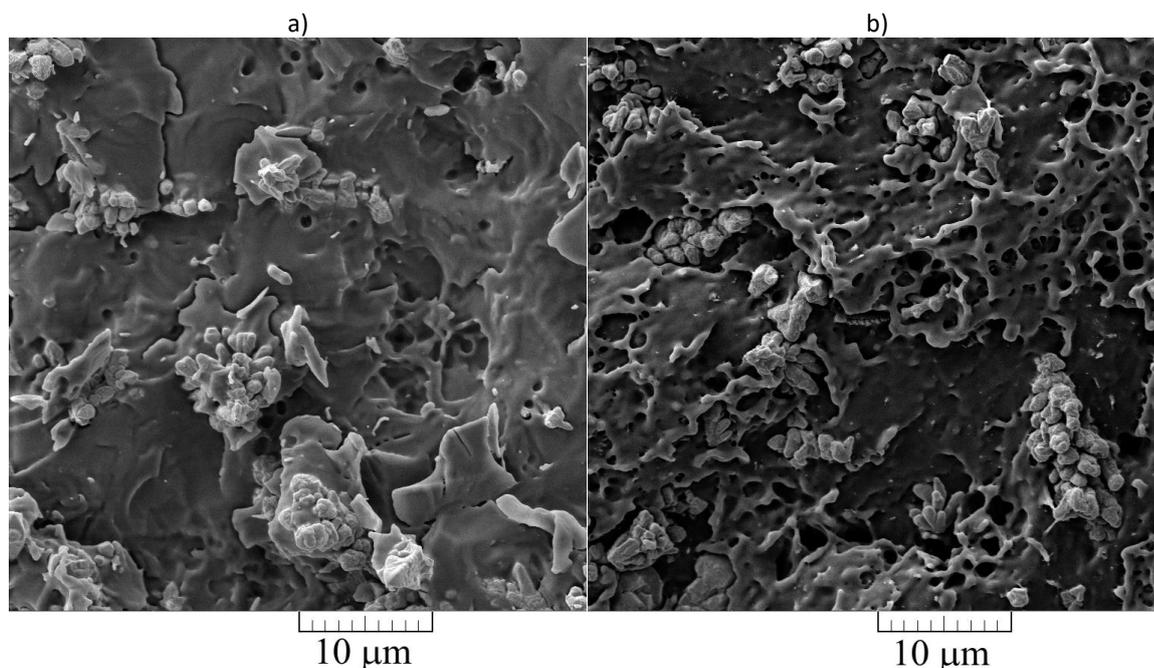


Figure 4. SEM image of the cross section of a) PLLA composite and b) PHB composite filled with copper powder at the percolation threshold

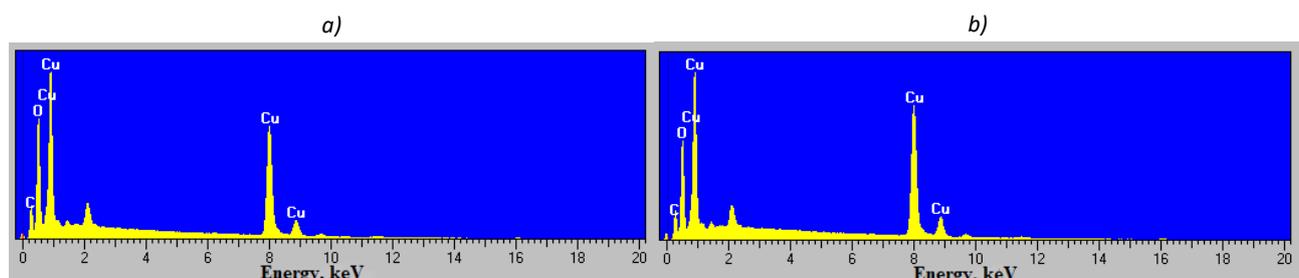


Figure 5. EDS spectrums of cross sections of a) PLLA composite and b) PHB composite filled with copper powder at the percolation threshold

Results of the EDS analysis show presence of all major components of the composites on the cross-section surface, where Cu powder forms interparticle connections throughout the surface of both PLLA and PHB composites. From the measurements of electric conductivity and knowing that the presented sample is conductive and that it is at the percolation threshold, it can be concluded that conductive pathways are formed throughout the surface of the composite. This conclusion is drawn also from the fact that size of the copper phase present in the figures ($>200\ \mu\text{m}$) is much larger than the size of copper powder particles ($<45\ \mu\text{m}$, Fig. 1) used for composite preparation. Clearly, composites conduct electricity through conductive pathways that are formed in 3D in pure random order.

Figure 6 shows FTIR spectra of the stabilized copper powder, PLLA, PHB and resulting composites with 5.0 vol.% copper powder content. The FTIR spectrum of the Cu powder shows three distinct peaks at $3423\ \text{cm}^{-1}$ characteristic for O-H alcohol groups, $2921.9\ \text{cm}^{-1}$ characteristic for C-H methyl and methylene groups and $1623.2\ \text{cm}^{-1}$ characteristic for C=C alkene bonds. This is in good agreement with experimental conditions that copper powder was stabilized with sodium soap SAP G-30, which contains 78% of total fatty acids, in order to protect the powder against subsequent oxidation. It can be concluded that chemical reactions do not occur between PLLA, PHB and copper powder, and that the influence of O-H, C-H and C=C groups on the spectra is amplified. There is a slight or a negligible shift in the wavenumbers among the studied samples.

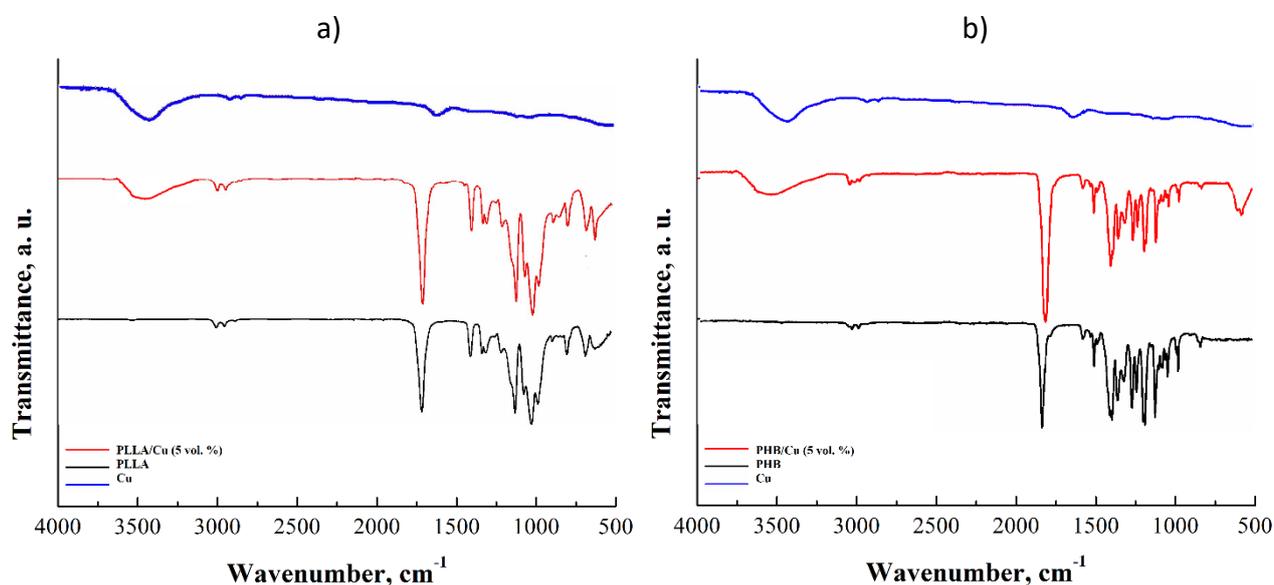


Figure 6. FTIR spectra of: a) pure stabilized copper powder, PLLA and the PLLA/Cu composite at 5 vol.% Cu content and b) pure stabilized copper powder, PHB and PHB/Cu composite at 5 vol.% Cu content.

4. CONCLUSION

In this manuscript, an experimental study is described focusing on the effects of electrodeposited copper powder content on the electrical conductivity of composites made of PLLA and PHB matrices and the powder as filler. Results have shown that the powder has a very high surface area and pronounced dendrite branching with well-developed primary and secondary dendrite arms. Conductivity measurements showed S-shaped dependency with percolation transition from the non-conductive to conductive region, typical for such polymer composite materials. The results have shown that the shape and morphology of the copper powder, and generally a filler, play a significant role in the phenomenon of composite electrical conductivity and the percolation threshold appearance. Conductivity measurements have shown that percolation threshold was at 2.83 vol.% Cu for PLLA and at 3.13 vol.% Cu for PHB composites. Thus, conductivities of the investigated composites are significantly improved as compared to similar composites containing fillers of a more regular structure that can be found in the literature. Morphology of the samples showed the 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 chemical reactions do not occur between PLLA, PHB and copper powder.

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SAŽETAK**Električna provodnost kompozita poli(L mlečne kiseline) i poli(3-hidroksibutirata) punjenih galvanostatski dobijenim bakarnim prahom**

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(Naučni rad)

U ovom rukopisu su predstavljeni rezultati eksperimentalnih ispitivanja svojstava kompozitnih materijala na bazi matrica poli(L mlečne kiseline) (poly (L lactic acid) - PLLA) i poli(3-hidroksibutirata) (PHB) punjenih elektrolitičkim bakarnim prahom, sa izrazito razvijenom dendritičnom strukturom. Zapreminski udeli frakcija bakarnog praha koji se koristio kao punilac u svim pripremljenim kompozitima su varirali u opsegu od 0,5-6,0 vol.%. Uzorci su pripremljeni toplim injektovanjem-moldovanjem na 170° C. Utvrđeni su uticaj veličine i morfologije čestica, kao i uticaj tipa matrice na perkolacioni prag i provodnost kompozita, a karakterizacija je uključivala: merenja električne provodnosti spektroskopijom elektrohemijske impedancije (IS), skenirajuću elektronsku mikroskopiju (SEM), energetska disperziona spektroskopiju X zračenja (EDS) i infracrvenu spektroskopiju sa Furijeovom (Fourier) transformacijom (FTIR). Potvrđeno je prisustvo trodimenzionalnih provodnih puteva. Izmeren je perkolacioni prag od 2,83 vol.% za PLLA i 3,13 vol.% za PHB kompozite, što je oko tri puta niža vrednost od vrednosti navedene u literaturi za slične kompozite. Ova osobina se pripisuje morfologiji punioca koji se koristio u istraživanjima.

Ključne reči: električna provodnost, kompozitni materijali, PLLA, PHB, elektrolitički bakarni prah