

## ELECTRICAL CONDUCTIVITY OF THE ELECTRODEPOSITED COPPER POWDER FILLED LIGNOCELLULOSIC COMPOSITES

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**Abstract:** This article deals with the synthesis and characterization of electroconductive composite materials prepared by the compression molding of mixtures of lignocellulose and electrochemically deposited copper powder under different pressures, as well as with the investigation of the influence of particle morphology on conductivity and the percolation threshold of obtained composites. Electrodeposited copper powder content varied from 1.9-29.4 vol%. The analysis of the most significant properties of prepared composites and its components included impedance spectroscopy (IS) behavior, the measurements of electrical conductivity, scanning electron microscopy (SEM) and structural analysis. It has been shown that the percolation threshold (PT) depends on both particle shape and the type of spatial distribution. IS measurements and SEM analysis have shown that particles that have pronounced grain boundaries have a bit effect on the appearance of electric conductive pathways thus on the composite conductivity. The packaging effect and more pronounced interparticle contact with copper powder particles lead to the “movement” of PT, which, for the particles <45 μm and the highest processing pressure of 27 MPa, was 7.2% (v/v).

**Keywords:** impedance spectroscopy, electrolytic copper powder, lignocellulose, composite.

### 1. INTRODUCTION

Due to their unique properties and a variety of possible applications, composite materials play a pivotal role in modern industry and are of great importance for material science in general. Attempts to improve the properties of materials by adding fillers, whether inorganic or organic, are not new. For years, the synthetic polymer composites have been used in various industrial fields, equipment, automotive and even aerospace. However, these synthetic materials come from non-renewable sources and are not easily degradable by microorganisms present in nature. In addition, global warming mostly caused by carbon dioxide released during fossil fuels burning is becoming an increasingly important issue, while the disposal of plastic-based petroleum products such as packaging of fast food, dining accessories, containers and bags also creates serious environmental problems [1,2].

The growing environmental awareness around the world has encouraged researchers and industrialists to discuss natural plant fibers as an alternative for reinforcements and fillers to produce composite materials known as biocomposites. Traditional solutions for conductivity in polymers and composites with polymer matrix include polymers with metal filling, substantially conductive polymers and networks, foils and wire laminated structure. A rapidly growing field of nanotechnology has introduced new materials that can increase the conductivity of the polymer composite [3–9]. By combining the insulating polymer with different conductive fillers such as metals and other conductive particles, electrically conductive polymer matrix composites can be obtained. Generally speaking, with an increase in the volume fraction of the conducting filler particles, conductivity of composite increases as the filler particles form the conduction paths through the composite [7–12]. Thus, depen

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ding on the amount of the conductive filler that is incorporated into polymer, the obtained composite can retain electrically insulative qualities of polymer or can become electrically semiconductive or conductive. As the volume fraction of the conducting filler reaches a critical value (the so-called percolation threshold), an infinite conductive cluster (IC) is formed and, consequently, the composite becomes conductive [8–9,13–18]. At low concentrations, below the percolation threshold, the conductivity remains very close to that of the insulating polymer matrix as the electrons still have to travel through the insulating matrix between the conductive filler particles. When a critical volume fraction of the filler, called the percolation threshold, is reached, the conductivity drastically increases by many orders of magnitude. This coincides with the formation of conductive pathways of the filler material forming a three dimensional network, which span the macroscopic sample. The electrons can now predominantly travel along the filler and move directly from one filler to another, increasing the amount of filler material further, levels off the conductivity, the maximum conductivity of the composite [8–9,13–18].

In addition to the conductivity control, electroconducting polymer composites have further advantages of lower cost, ease of manufacture, reduced weight, high flexibility, corrosion resistance and mechanical shock absorption ability, which makes them rather interesting for many fields of engineering [13,19–22].

Numerous studies can be found in literature related to the influence of the type of polymer matrix and filler on the electrical properties of the composites [7–9, 23–26]. The studies suggest that the particle size and shape, the nature of the contact between the conductive filler elements, volume fraction and spatial arrangement of the conducting filler particles, all have a significant influence on the conductivity of filled polymers [9,13–18].

Furthermore, the increasing environmental awareness combined with an increase in crude oil price has opened investigations of eco-friendly and sustainable alternatives to fossil fuel-based synthetic polymers and stirred up recent investigations towards biopolymers i.e. biodegradable polymers [19–26]. Accordingly, a large number of these biodegradable polymers are currently commercially available [27].

Biopolymers, such as lignocellulose (LC), are a good alternative to polymers based on petrochemical industry due to their environmental benefits like biodegradability and renewability. Biocomposites based on raw materials derived from natural resour-

ces are of great interest and have been a major subject in a large number of publications [7–12,28–29]. Produced by wooden plants and having a large range of properties, lignocellulose can be used for applications like packaging, agriculture, hygiene, and automotive industry.

Lignocellulosic biomass is a nonstarch, fibrous part of plant material and is an attractive resource because it is renewable and abundant [10,11]. Lignocellulose-based fibers are the most widely used since, intrinsically, these fibers have a number of interesting mechanical and physical properties [12,28]. The structural and chemical composition of lignocellulosic feedstocks is highly variable because of genetic and environmental influences and their interactions [10]. Chemical composition of lignocellulosic feedstocks is a key factor affecting the properties of this biodegradable polymer and its composites [28].

Lignocellulose can be obtained by physical treatment such as high pressure homogenizer and grinder treatment, using high mechanical shear force to generate bundles of microfibrils called cellulose microfibril or microfibrillated cellulose with diameters from tens of nm to  $\mu\text{m}$  [7–9].

Conducting polymer composites with metal fillers have found applications as conducting adhesives in electronics packaging, underfill for flip chips, in electromagnetic shielding of computers and electronic equipment, cold solders, switching devices, static charge dissipating materials and devices for surge protection [28,30–31]. They also found numerous technological applications as photothermal optical recorders, chemical and electrochemical catalysts, self regulating heater, direction finding antennas, chemical detecting sensors used in electronic noses and adsorbents [12,32–36].

The primary objective of this study was to investigate the electrical properties of lignocellulose matrix composites filled with galvanostatically produced copper powders. The main focus of this paper is to examine the conductive properties of composites and the relationship between the matrix and filler in the composite in order to better understand the behavior of the electrical conductivity of these materials. This approach involves the characterization of the classical percolation characterization, along with a number of physical tests of material properties of the composites. The analysis included measurements of electrical conductivity, structural investigations and impedance spectroscopy (IS) characterization.

Impedance spectroscopy (IS) has been gaining a more important role in fundamental and applied electrochemistry and materials science in the last years. In a number of respects it is a method of choi-

ce for characterizing the electrical behavior of systems in which the overall system behavior is determined by a number of strongly coupled processes, each proceeding at a different rate [9,37]. Impedance spectroscopy is a relatively new and powerful method for characterizing many of the electrical properties of materials and their interfaces with electronically conducting materials. It may be used to investigate the dynamics of bound or mobile charge in the bulk or interfacial regions of any kind of solid or liquid material: ionic, semiconducting, mixed electronic-ionic, and even insulators (dielectrics). It can be used for measurements in a wide range of materials from solid electrolyte materials (amorphous, polycrystalline and single crystal), solid metallic electrodes, fused salts, aqueous electrolytes and composite materials [37].

## 2. EXPERIMENTAL

Copper powder was galvanostatically produced with distinct dendritic morphology and a large specific area [38–40], with all the same parameters of electrolysis and deposition times as stated in [7–9]. Electrolytic copper powder has been produced from electrolyte containing 140 g/dm<sup>3</sup> sulfuric acid and 15 g/dm<sup>3</sup> copper ions, using an electrolyte temperature (50±2) °C. The powder was deposited galvanostatically at current density of 3600 A/m<sup>2</sup> and the deposition time (the time of powder removal by brush) was 15 min, whereby the experimental setup was the same as in [7–9]. The wet powder was washed several times with a large amount of demineralized water until the powder was free from traces of acid, at room temperature, because acid promotes rapid oxidation of powder during drying. Obtained copper powder was washed afterwards with sodium soap SAP G-30 solution to protect the powder against subsequent oxidation, which was prepared and used as in previous work [7–9]. After drying in tunnel furnaces at 110 – 120 °C in a controlled nitrogen atmosphere, the copper powder was sieved through a mesh with 45µm openings.

Celgran<sup>®</sup> C fraction of the corn cob produced by Maize Research Institute „Zemun Polje” was used as a source of lignocellulose. It was milled and then sieved, with particle sizes below 45 µm. Lignocellulose prepared and obtained in this manner was used as a natural polymer matrix.

Investigated composites of lignocellulose matrix filled with copper powder were prepared with filler contents in the range 2.0% (v/v) – 29.8% (v/v), while pure lignocellulose samples were prepared as reference materials. Thoroughly homogenized com-

posite powder mixtures were pressed into 16 mm diameter pellets under pressures of 10, 20 and 27 MPa at ambient temperature (t = 25 °C)

Impedance spectroscopy (IS) experiments were performed in potentiostatic mode on all prepared composites. Instrumentation involved Bio-Logic<sup>®</sup> SAS Instrument, model SP-200, guided by EC-Lab<sup>®</sup> software. Samples were put between two metal plates and they responded to the potential input sinusoidal signal of ±10mV (rms) amplitude. The geometry of the instrument contacts used is such that it minimizes the edge effects, thus it can be assumed that they do not exist. Experimental IS data were fitted by ZView<sup>®</sup> software [41].

Sample thickness (necessary for the calculation of conductivity) was determined using a micrometer, at an accuracy of 0.01 mm. Several thickness measurements were taken per sample and then averaged.

The morphology of electrochemically deposited copper powder and lignocellulose, as well as their composites was investigated by scanning electron microscopy using a VEGA TS 5130MM microscope (Tescan).

## 3. RESULTS AND DISCUSSION

As previously mentioned, the conductivity of filled polymers is strongly dependent on the nature of the contact between the conductive filler elements, particle size and shape of the conductive particle filler. This was shown in [7–9] where highly dendritic copper powder was used, and the percolation threshold was moved to a lower filler content. Electrical properties of lignocellulose polymer matrix are altered by addition of conductive copper powder as the filler. The electrical conductivity of polymer composites does not increase continuously with increasing electroconductive filler content. Instead it has a rapid increase in conductivity when the percolation threshold of the conductive filler content is reached. Therefore, the copper powder was galvanostatically produced since it should have a distinct dendritic morphology and a large specific area [38–40]. This feature can be seen in Figure 1a, which shows typical copper powder particle obtained by constant current deposition. Copper powder particles have pronounced dendrite branching and, hence, this powder is a good prerequisite for formation of more interparticle contacts between conductive copper particles and lowering the percolation threshold. Figure 1b shows SEM photomicrograph of used lignocellulose with distinct layered structure.

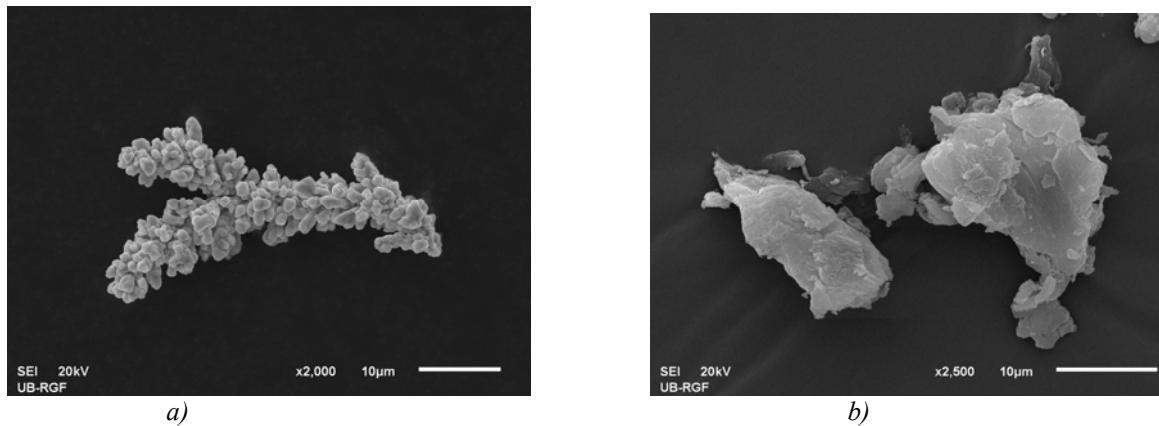


Figure 1. SEM photomicrographs of a) copper powder particles obtained in constant current deposition and b) lignocellulose matrix used in composite preparation

The results of the percolation threshold (obtained from the maximum of the derivative of the conductivity as a function of filler volume fraction) measured from the experimental data of impedance spectroscopy are in good agreement with the previous results [7–9].

At an interface between two phases in the composite, physical properties like crystallographic, mechanical, compositional, and, particularly, electrical change precipitously and heterogeneous charge distributions (polarizations) reduce the overall electrical conductivity of a system. The morphology of used copper powder (Figure 1) shows that greater interparticulate contact of smaller, highly porous, highly dendritic particles with high values of specific area move the percolation threshold towards lower filler content.

It should be mentioned that the resistance of the composites increases due to an increasing contribution of a part of internal surface of polymer matrix as frequency decreases. In a certain frequency range, capacitance and resistance values are similar.

The electrical conductivity of all the particle size composites as a function of filler content for all the samples was measured from impedance characteristics of a composite having the filler of characteristic morphology. Electrical conductivity was determined according to the following relation:

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

where  $\sigma$  is electrical conductivity,  $R_i$  – resistivity of a layer in equivalent circuit,  $l$  – length and  $S$  – cross-section area of the sample. Measurements of electrical conductivity as a function of filler content for all the samples are presented in Figure 2.

Due to the packaging effect and more pronounced interparticulate contact with smaller, highly porous,

highly dendritic particles with high values of specific area lead to „movement” of percolation threshold towards lower filler content. This feature can be observed in Figure 3. The conductivity of the composite is obtained through conductive pathways of the filler that form in the composites. These pathways are formed in 3D in a pure random order. SEM images of the inner structure of the composites are prepared after composite preparation and its braking.

It should be mentioned that copper powder filler was sieved through the mesh with the openings of 45  $\mu\text{m}$ , and the conductive pathways are at much larger scale which can be observed in Figure 3 [42]. The results of quantitative microstructural analysis for the sieved powders are shown in Table 1. The symbols used have the following meanings:

- $A$  (area) – Feature area (the total number of detected pixels within the feature).
- $D_{\max}$  – Diameter (length) of the largest (longest) particle.
- $D_{\min}$  – Diameter (length) of the smallest (shortest) particle.
- $L_p$  (perimeter) – The total length of the boundary of the feature which is calculated from the horizontal and vertical projections, with an allowance for the number of corners.
- $f_A$  (form area) – A shape factor of area. This is calculated from the ratio:

$$f_A = \frac{4A}{\pi \cdot D_{\max} \cdot D_{\min}} \quad (2)$$

and for a circle and ellipse is equal to 1.

–  $f_L$  (form perimeter) – A shape factor of perimeter. This is calculated from the ratio:

$$f_L = \frac{4\pi A}{L_p^2} \quad (3)$$

and for a circle and ellipse is equal to 1.

–  $f_R$  (roundness) – A shape factor which gives a minimum value of unity for a circle. This is calculated from the ratio of perimeter squared to area:

$$f_R = \frac{L_p^2}{4\pi \cdot A \cdot 1.064} \quad (4)$$

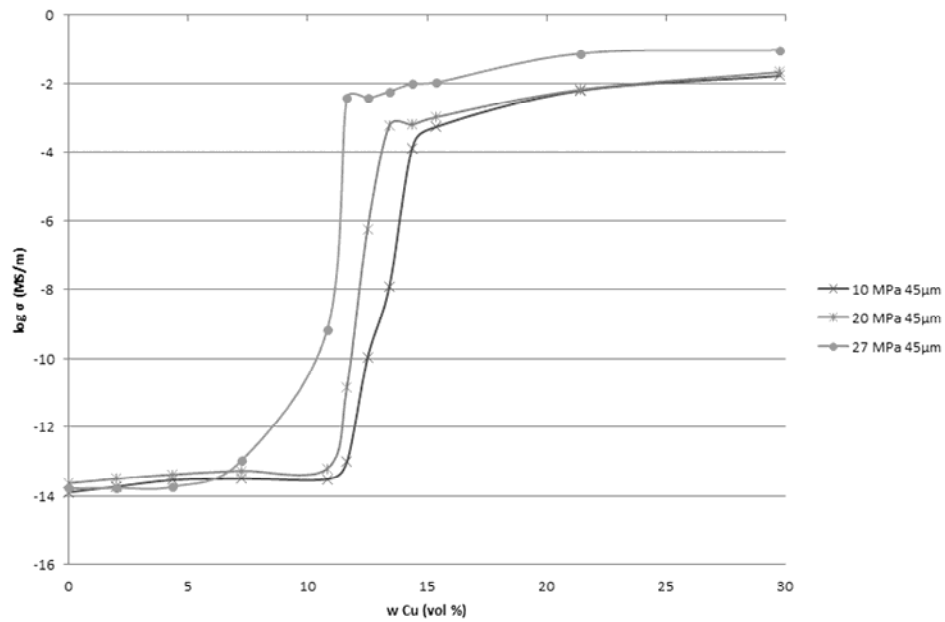


Figure 2. Comparison of electrical conductivities, as a function of filler content, of lignocellulose composites filled with copper powder under different processing pressures.

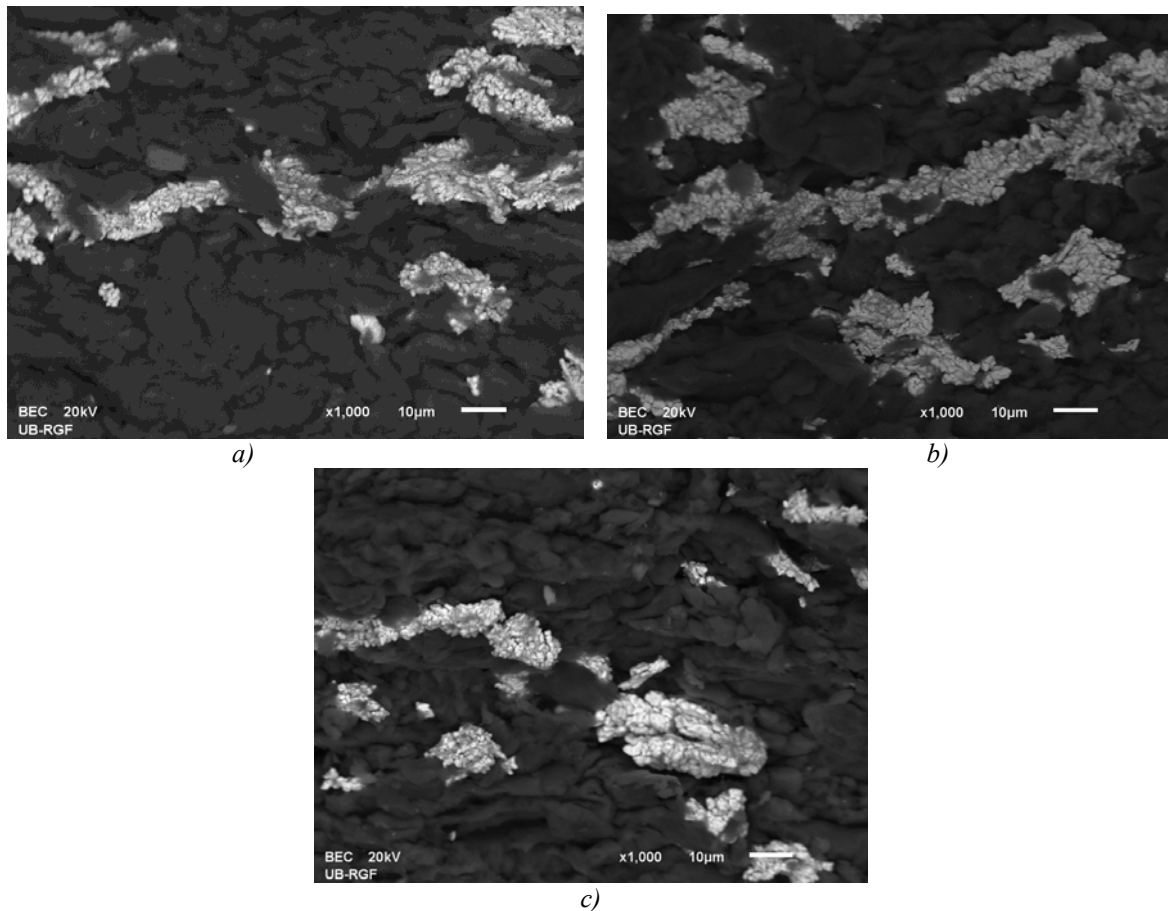


Figure 3. SEM images of the conductive pathways in the composites at percolation threshold. Composites are prepared under a) 10 MPa, b) 20 MPa and c) 27 MPa. All photographs are prepared at magnification x350

The adjustment factor of 1.064 corrects the perimeter for the effect of the corners produced by the digitization of the image.

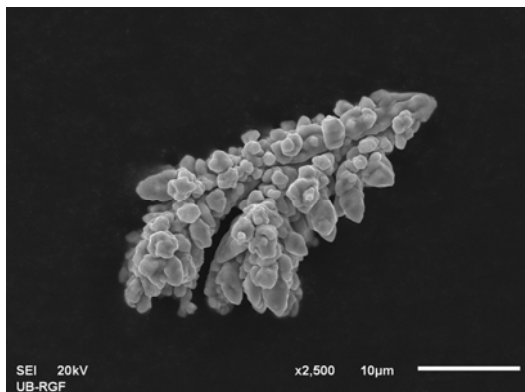
–  $f_w$  – This is the ratio of the length of the polygon circumscribing the feature formed by tangents to its boundary (very similar to the length of a piece of string stretched around the feature) to perimeter.

Table 1. Parameters which characterize sieved fraction ( $\leq 45\mu\text{m}$ ) of electrolytic copper powder deposited galvanostatically at current density of  $3600\text{ A/m}^2$  [42]

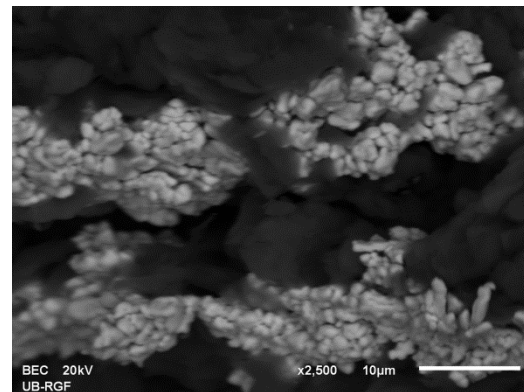
	Max	Min	Mean
$A$ (area) ( $\mu\text{m}^2$ )	578.32	5.71	91.76
$L_p$ (perimeter) ( $\mu\text{m}$ )	181.34	0.98	57.82
$D_{\text{max}}$ ( $\mu\text{m}$ )	50.47	2.91	28.34
$D_{\text{min}}$ ( $\mu\text{m}$ )	12.94	0.98	6.81
$f_A$ (form area)	1.00	0.37	0.75
$f_L$ (form perimeter)	0.87	0.12	0.50
$f_W$	1.00	0.73	0.89
$f_R$	3.99	1.22	2.12

There is an influence of the pressure on electrical conductivity of the composites. For the pressure of 10 MPa the percolation threshold was lowered to 12.5% (v/v), for 20 MPa it was 11.6% (v/v), and for 27 MPa it was 7.2% (v/v) which is the difference of 5.8% (v/v) in the filler content. At higher pressures less filler is needed to form conductive pathways through a composite. However, at the pressure of 27 MPa it can be observed that the starting surface features of electrodeposited copper powder is a little deformed, although it is still forming conductive pathways (Figure 4). Further tests, at higher pressures, will show the future impact of increased pressure on composite conductivity, and whether it starts to decrease after a certain point.

Flandin et al. [43] state that typical values of 20–40% (v/v) for percolation threshold for spherical filler particles. Hence, electrodeposited copper powder particles represent an excellent choice as conductive composite fillers.



a)



b)

Figure 4. SEM images of the a) electrodeposited copper powder used as a filler and b) copper powder in the composite prepared at 27 MPa. Magnification x2500

#### 4. CONCLUSIONS

In this article we describe the experimental study about the effects of particle morphology of electrodeposited copper powder on the electrical conductivity of lignocellulose composites filled with this powder. It was shown that a particle shape of copper powder with very high surface area and pronounced dendrite branching plays a significant role on electrical conductivity of the prepared samples. Electrical conductivity was calculated from the results of impedance spectroscopy measurements and it is in agreement with previously published results [7–9]. The layered electrical conductivity through the samples was found, where the resistance of the inner layers is a guiding process of the overall

resistance of the composite. Grain boundary plays a significant effect. The response is an elongated coupled resistance with constant impedance „tail” towards lower frequencies. It can be pointed out that resistance increases due to the increasing contribution of the part of internal surface of polymer matrix as frequency decreases. The internal space charge that is created at the phase boundaries leads to a significant increase in the concentration of mobile defects, hence giving the circuit response with purely geometric reductions in the conductivity with respect to the single crystal and thus showing that grain boundary plays a significant effect. This response is elongated coupled resistance with constant impedance „tail” towards lower frequencies. Polycrystallinity, high surface area, bigger number

of interparticle contacts and grain boundary effect of the copper powder are key factors influencing the percolation threshold in the LC-Cu composites. This research has undeniably shown that particle morphology of galvanostatically obtained copper powder and lignocellulose matrix plays a significant role in electrical conductivity of the composites.

## 5. ACKNOWLEDGEMENT

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#### ЕЛЕКТРИЧНА ПРОВОДЉИВОСТ ЛИГНОЦЕЛУЛОЗНИХ КОМПОЗИТА ПУЊЕНИХ ЕЛЕКТРОЛИТИЧКИМ БАКАРНИМ ПРАХОМ

**Сажетак:** У овом раду приказана је синтеза и карактеризација електропроводних композитних материјала на бази лигноцелулозе и електрохемијски добијеног



бакарног праха добијених хладним пресовањем на различитим притисцима, као и испитивање утицаја морфологије праха на проводност и перколациони праг добијених композита. Запремински удео бакарног праха је варирао 1.9–29.4% (v/v). Анализа најзначајнијих особина припремљених композита обухватала је импедансну спектроскопију (IS), скенирајућу електронску микроскопију (SEM), мерења електричне проводности и структурну анализу. Показано је да перколациони праг (PP) зависи како од облика честица тако и од њиховог просторног уређења. IS мерења и SEM анализе су показали да морфологија честица са изражено великом границом зрна има велики утицај на појаву електричних проводних слојева. Ефекат паковања и израженији међучестични контакт са честицама бакарног праха доводи до „померања” PP, који је за честице <45  $\mu\text{m}$  и највећи притисак од 27 МПа износио 7.2% (v/v).

**Кључне речи:** импедансна спектроскопија, електролитички бакарни прах, лигноцелулоза, композит.

