

EFFECT OF CARBONIZATION TEMPERATURE ON ELECTRICAL CONDUCTIVITY OF BIOCARBON WATER HYACINTH COMPOSITES

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Abstract— This paper presents the usage of water hyacinth as the base material of activated carbon or biocarbon, using carbonization temperature 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C. The SEM results show that the pore structure of the biocarbon is shaped like a coral, the higher the temperature the more open pores and the smaller the diameter, with about 2µm at 1000°C. The XRD results show that the carbonization temperature affects the percentage of the carbon crystal of the biocarbon formed, the higher the temperature the percentage increases by reaching 14% at 1000°C, the carbon crystals are rhombohedral and graphite-like. The structure of the pores and the percentage of the carbon crystal has the effect of the value of electrical conductivity, the higher the temperature resulting in an increase in the value of the composite electrical conductivity, the water hyacinth biocarbon composite has a range of electrical conductivity values ranging from $2,697 \times 10^{-6} \text{ S / cm}$ (500°C) to $1,767 \times 10^{-2} \text{ S / cm}$ (1000°C) is included in a suitable conductive composite applied to the sensor and EMI (Electromagnetic Interference) Shielding.

Keywords— Biocarbon; Temperature; Electrical conductivity; Hyacinth Composites

1. INTRODUCTION

Composites are engineered materials comprising mixtures of two or more components of binders and fillers, each having different chemical and physical properties, with the aim of improving the particular properties of each component [1]. Generally, composites are materials that are electrical and thermal insulators and to make them conductive, composite fillers can be metal, fiber and carbon [2]. In recent years, carbon fillers such as carbon black (CB), graphite, activated carbon (CA), carbon nanofiber (CNFs), carbon nanotubes (CNTs) and graphene are widely used as fillers because of good electrical conductivity. Some composite applications that have good electrical conductivity are used as energy storage, biosensing, antistatic and electromagnetic shielding material (EMI) [2, 3]. In its development, carbon-active carbon type fillers can be obtained from natural materials. This is because the activated carbon is amorphous carbon [4], which can be produced from ingredients that contain cellulose and lignin, in which cellulose and lignin are organic compounds that contain many chemical elements of C. Some natural materials used as activated carbon for composite fillers with applications of

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electromagnetic wave shields (EMI) such as rice husk [5], bamboo [6], coconut husk [7], coconut shells [8], and wood [9].

Therefore, in order to develop natural materials as the basic ingredients of activated carbon or biocarbon used by conductive composite fillers, this study aims to examine the biocarbon which is based on weed water hyacinth plant as filler of conductive composite.

The rest of this paper is organized as follow: Section 2 presented the proposed experimental design. Section 3 presented obtained results and following by discussion. Finally, Section 4 concludes this work.

2. EXPERIMENTAL DESIGN

2.1. PREPARATION

Water hyacinth is dried, pounded, and powdered with a size of 80 mesh. The carbonization process was then carried out at 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C in a vacuum to obtain a water hyacinth biocarbon, with the carbonization process as follows: water hyacinth powder heated from room temperature to 400°C with a heat boost rate of 7°C min⁻¹, then to temperature 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C with a 3°C min⁻¹ hot rise speed, when it reaches 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C retained for 1 hour, then cooled naturally until it reaches room temperature.

2.2. CHARACTERIZATION

Water hyacinth biokarbon is characterized by X-ray diffraction (XRD) using PHILIPS X-ray diffractometer (ModelPW3050) with monochromatic CuK α radiation at 2 θ . The microstructure of the water hyacinth biocarbon is analyzed using a Scanning electron microscope (SEM) EVO MA 10 with an Energy Dispersive X-ray detector (EDX) system at 20 kV voltage acceleration. On the measurement of electrical conductivity, a composite sample is made of a mixture of water hyacinth biocarbon and 30 wt% phenol formaldehyde resin (PF) (BUEHLER: PhenoCure 640). The mixing process uses ballmill. The mixture is printed using hot-pressed molding with length $\times$ width = 22.86 mm $\times$ 10.16 mm, for each thickness 3 mm, 4 mm and 5 mm. The measurement of the electrical conductivity of the water hyacinth biocarbon composite specimens using HP 4262A (Hawlett Packard) LCR Meter at a frequency of 120 Hz, 1 KHz and 10 KHz at room temperature. The composite sample is clamped between two copper electrodes of conductivity holder as shown in Figure 1. The electrical conductivity value (σ) is calculated using the equation [10]:

$$\sigma = \frac{l}{RA} \quad (1)$$

where

$l = 22,86$ mm

$A = w \times t$, with $w = 10,16$ mm and $t = 3$ mm, 4mm, dan 5 mm

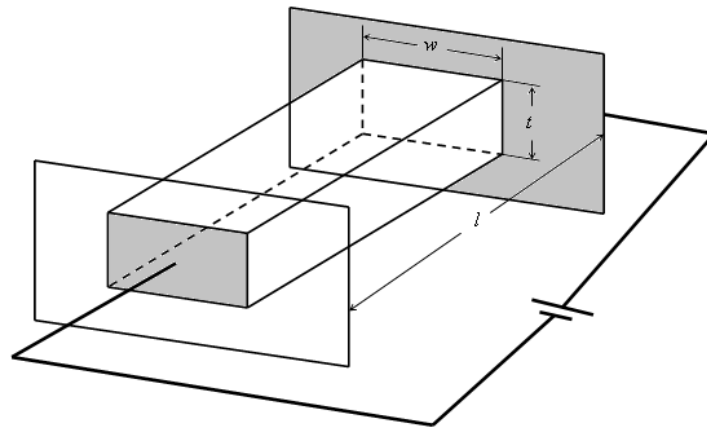


Fig. 1 Measurement of electrical conductivity

3. RESULTS AND DISCUSSION

3.1. SEM TEST RESULTS

The process of carbonization directly from the water hyacinth plant produces porous biocarbon. The process of forming pores already exists at a temperature of 500°C, where at this temperature the pores on the surface have begun to open, but still many are still closed (Figure 2a). Porous biocarbon at 600°C indicates that the pores present on the surface are all open, and the inner pores begin to form (Figure 2b). While at a temperature of 700°C, it produces porous rocky biocarbons or asymmetric sponges. The inner pores are formed with a length of about 10 μm and a width of 5 μm (Figure 2c). With increasing carbonization temperatures, the pores grew smaller, as shown in Figures 2d to 800°C, Figures 2e to 900°C and Figures 2d to 1000°C, with a pore diameter of about 2 μm at a temperature of 1000°C.

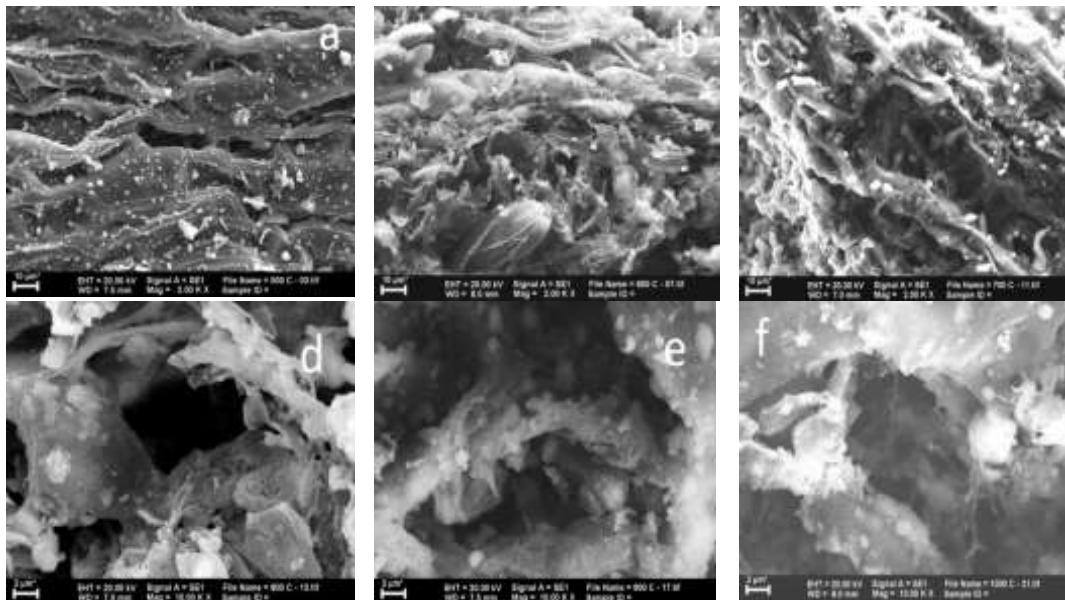


Fig. 2 SEM results with carbonization temperature 500oC (a), 600oC (b), 700oC (c) with 2000x magnification, 800oC (d) 900oC (e), 1000oC (f) with 10000x magnification

Activated carbon has three types of pore structure depending on its size: micropores with size ≤ 2 nm, mesopores meso with size 2 - 50 nm, and macropores macropores with

size ≥ 50 nm [11]. As shown in Figure 3. The water hyacinth biocarbon is activated carbon, therefore carbonization results at 800°C carbonization temperature indicating meso pore formation (Figure 2d).

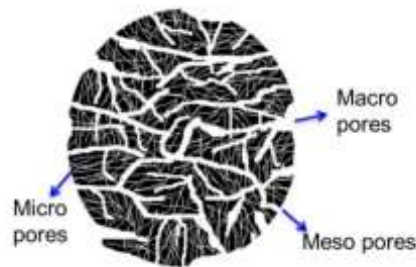


Fig. 3 The pore structure of activated carbon [11]

3.2. XRD RAW MATERIAL TESTING RESULTS

The result of XRD test from biocarbon of water hyacinth was found that carbon crystals were formed with reference code = 01-075-0444 with rhombohedral crystalline system with space group number 166 as depicted in Figure 4. With $a = b = c = 3.6350 \text{ \AA}$ or $3,6350 \cdot 10^{-10} \text{ m}$, angle $\alpha = \beta = \gamma = 36.82^\circ$, mass / volume = calculated density = 2.58 g / cm^3 , and volume of cells = $46.36 \cdot 10^6 \text{ pm}^3$.

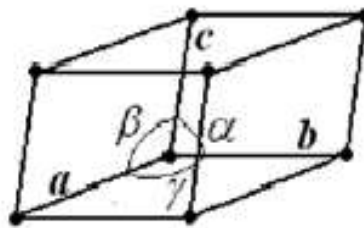


Fig. 4 The rhombohedral lattice is expressed as a primitive unit cell with $a = b = c$ and $\alpha = \beta = \gamma$ [12]

The result of the test of carbon crystal percentage in Figure 5 shows at carbonization temperature 500°C formed by 8%, then increased to 9% at temperature 600°C, 10% at temperature 700°C, 11% at 800°C, 14% at 900°C and 14% at 1000°C. Accordingly, the increase in carbonization temperature causes an increase in the percentage of carbon crystals formed. The formation of carbon crystals during the carbonization process occupies at an angle position (2θ): 26.310° ; 46.502° ; 49.171° ; 54.152° ; 58.957° ; 65.664° ; 82.192° ; 84.288° ; and 86.118° as shown in Figure 6. At an angle of 26.310° has a peak of 100% intensity, accordingly the carbon crystal formed in a water hyacinth biocarbon is graphite-shaped, in which the graphite is at an angle of 27° [13].

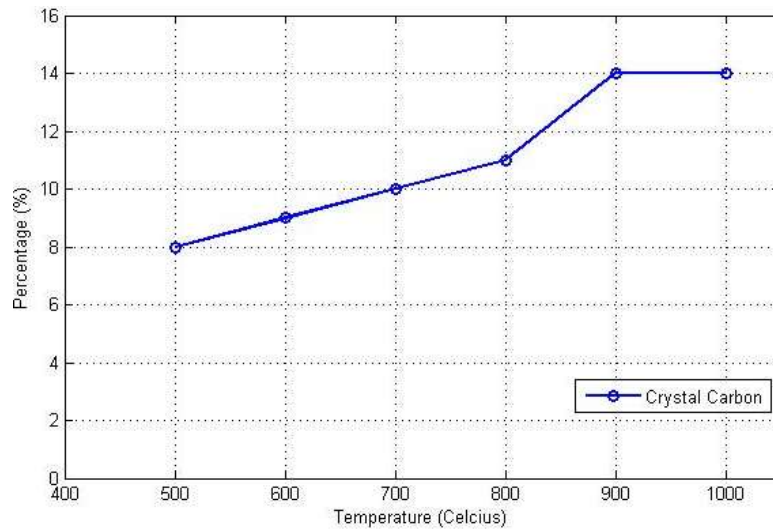


Fig. 5 XRD Test Results Percentage of Carbon Crystals by Carbonization Temperature

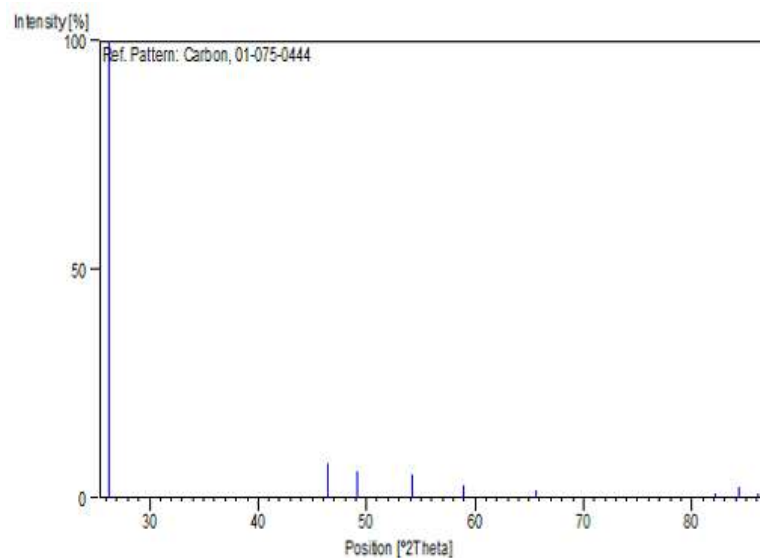


Fig. 6 XRD Results Carbon Crystal Position With Code 01-072-154044 On Water Hyacinth Biocarbon

3.3.ELECTRICAL CONDUCTIVITY TEST RESULTS

After carbonization process with temperature variation from 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C, we made a composite water hyacinth biocarbon composite composition with 70% water hyacinth biokarbon and 30% by weight phenol formaldehyde (PF). The composite mixing process uses ballmill to produce a uniform mixture, then printed using hot press with length $\times$ width = 22.86 mm $\times$ 10.16 mm, and variation of 3 mm, 4 mm, and 5 mm specimen thickness. There were 3 composite specimens for each thickness, so that 9 composite specimens were obtained for each carbonization temperature. So the total of 9×6 composite specimens = 54 specimens. After that, the test of biocarbon composite specimen with LCR Meter HP 4262A was done. With variation of measurement frequency: 120 Hz, 1 KHz, and 10 KHz. The result of the measurement of resistance based on the frequency variation is taken average, then taken the average resistance value of each thickness. With the results as shown in Table I and Figure 7.

Table I. Results Measurement of Resistance Value of Water Hyacinth Biocarbon Composites

Temperature	Resistance (Ω)			
	3 mm	4 mm	5 mm	Average
500	$4,620 \times 10^6$	$1,53 \times 10^6$	$1,171 \times 10^6$	$2,44 \times 10^6$
600	$621,7 \times 10^3$	$274,91 \times 10^3$	$217,09 \times 10^3$	$371,23 \times 10^3$
700	$7,808 \times 10^3$	$6,708 \times 10^3$	$4,213 \times 10^3$	$6,243 \times 10^3$
800	$1,867 \times 10^3$	$1,554 \times 10^3$	$1,22 \times 10^3$	$1,549 \times 10^3$
900	790,667	554,333	420,333	588,444
1000	381,222	320,333	230,889	310,815

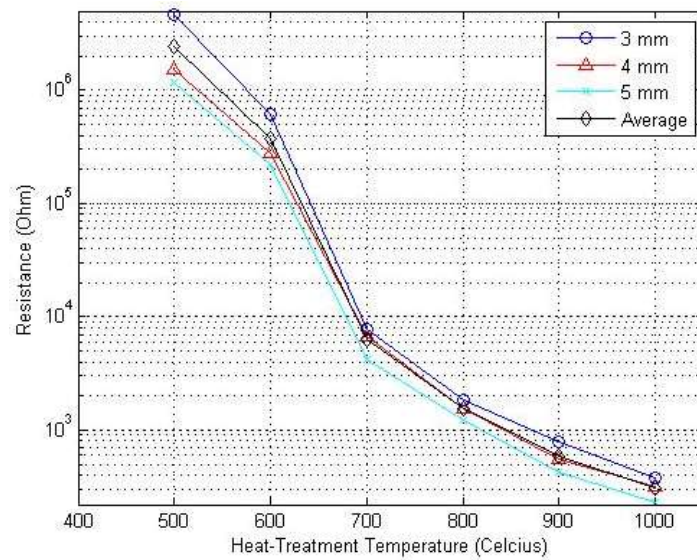


Fig. 7 Results of Resistance Test of Water Hyacinth Biocarbon Composite

Table II. Result of Measurement of Electrical Conductivity Value of Water Hyacinth Biocarbon Composite

Temperature	Conductivity (S/cm)			
	3 mm	4 mm	5 mm	Average
500	$1,439 \times 10^{-6}$	$3,221 \times 10^{-6}$	$3,432 \times 10^{-6}$	$2,697 \times 10^{-6}$
600	$1,111 \times 10^{-5}$	$1,947 \times 10^{-5}$	$1,865 \times 10^{-5}$	$1,641 \times 10^{-5}$
700	$9,3186 \times 10^{-4}$	$8,968 \times 10^{-4}$	$10,105 \times 10^{-4}$	$9,463 \times 10^{-4}$
800	$3,730 \times 10^{-3}$	$3,294 \times 10^{-3}$	$3,264 \times 10^{-3}$	$3,429 \times 10^{-3}$
900	$9,760 \times 10^{-3}$	$9,420 \times 10^{-3}$	$9,711 \times 10^{-3}$	$9,630 \times 10^{-3}$
1000	$1,876 \times 10^{-2}$	$1,632 \times 10^{-2}$	$1,793 \times 10^{-2}$	$1,767 \times 10^{-2}$

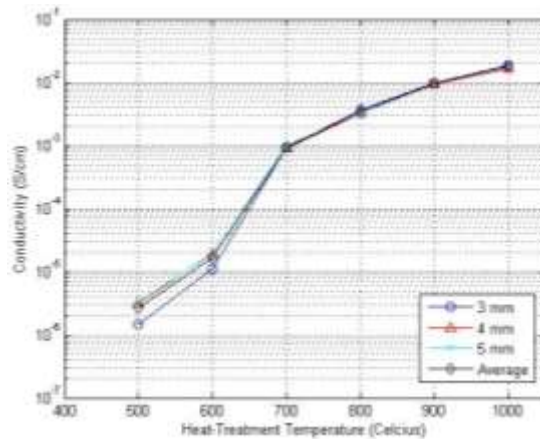


Fig. 8 Results of Electrical Conductivity Test of Water Hyacinth Biocarbon Composites

Based on Table I and Figure 7, it shows that the increase of carbonization temperature has an effect of decreasing the resistance value, which is about 2.4 MOhm at 500°C to about 300 ohm at 1000°C. From the value of the resistance of Table I, it is used to obtain the electrical conductivity value of the water hyacinth biocarbon composite specimen using equation (1), then the electrical conductivity value as shown in Table II and Figure 8. Where the electrical conductivity value increases with the temperature of carbonization from value of 2.697×10^{-6} S/cm at a temperature of 500°C to a value of 1.767×10^{-2} S/cm at a temperature of 1000°C. This indicates that the increase in carbonization temperature causes more open pores and the greater percentage of the resulting carbon crystals causing an increase in the value of electrical conductivity. The relationship between electrical resistivity and electrical conductivity is indicated in the equation [14,15]:

$$\sigma = \frac{1}{\rho} \quad (2)$$

Figure 9 shows the application of a conductive polymer composite with various resistivity ranges or electrical conductivity; accordingly, the electrical conductivity values resulting from the water hyacinth biocarbon composite ranging from $2,697 \times 10^{-6}$ S/cm (carbono temperature 500°C) to $1,767 \times 10^{-2}$ S/cm (carbonization temperature 1000°C) is in a conductive composite classification, where the application of a conductive composite is suitable for use as a sensor and EMI (Electromagnetic Interference) Shielding.

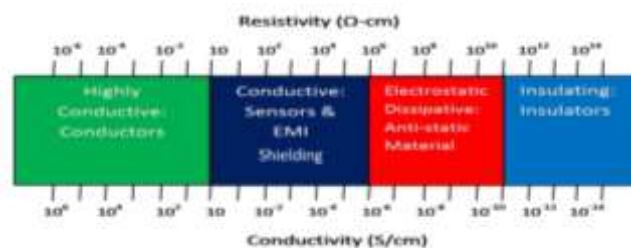


Fig. 9 Classification of Conductive Polymer Composites Applications Depending on Resistivity or Electrical Conductivity [16,17]

4. CONCLUSION

This paper has examined the biocarbon which is based on weed water hyacinth plant as filler of conductive composite. In summary, the following conclusions can be drawn: (1) The higher the carbonization temperature, the more open pore water hyacinth biocarbon

and the smaller pore diameter is 2 μ m at 1000°C than other temperature, with the shape of the pore-like structure of the coral; (2) The higher carbonization temperature results in an increase in the percentage of carbon crystal biocarbon with water hyacinth 14% at 1000°C, with its carbon crystals rhombohedral and graphite-like; (3) The pore structure and percentage of carbon crystals have an effect on the value of electrical conductivity, so the higher the carbonization temperature of the water hyacinth biocarbon leads to an increase in composite electrical conductivity with a value of $2,697 \times 10^{-6}$ S/cm (500°C) up to $1,767 \times 10^{-2}$ S/cm (1000°C); (4) A suitable water hyacinth biocarbon composite is included in a suitable conductive composite applied to the sensor and EMI (Electromagnetic Interference) Shielding.

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REFERENCES

- [1] Ezquerra, T. A., Connor, M. T., Roy, S., Kulescza, M., Fernandes, N. J., & Balta, C. F. J. (2001). Alternating-current electrical properties of graphite , carbon-black and carbon- fiber polymeric composites. *Composites Science and Technology*, 61, 903–909.
- [2] Jing, L. ., Yern, C. ., Gan, S. N., Rozali, S., & Julai, S. (2016). Effects of Oil Palm Empty Fruit Bunch Fiber on Electrical and Mechanical Properties of Conductive. *BioResources*, 11(1), 913–928.
- [3] Khan, S. ud D., Arora, M., Puri, C., Wahab, M. A., & Saini, P. (2014). Synthesis and characterization of acrylic resin / activated carbon composites. *Indian Journal of Pure & Applied Physics*, 52(April), 251–254.
- [4] Barranco, V., Lillo-Rodenas, M. A., Linares-Solano, A., Oya, A., Pico, F., Ibañez, J., ... Rojo, J. M. (2010). Amorphous carbon nanofibers and their activated carbon nanofibers as supercapacitor electrodes. *Journal of Physical Chemistry C*, 114(22), 10302–10307.
- [5] Liu, S., Chen, X., Zhang, A., Yan, K., & Ye, Y. (2014). Electromagnetic Performance of Rice Husk Ash. *BioResources*, 9, 2328–2340.
- [6] Wu, K. H. (2008). Science and Electromagnetic and microwave absorbing properties of Ni 0 . 5 Zn 0 . 5 Fe 2 O 4 / bamboo charcoal core – shell nanocomposites. *Composites Science and Technology*, 68, 132–139.
- [7] Suliyanti, M. M., Yudasari, N., Indayaningsih, N., Tresna, W. P., Wahyu, Y., & Puspipitek, K. (2012). Pembuatan rf absorber berbasis karbon lokal untuk aplikasi radar. In *InSinas* (pp. 137–140).
- [8] Liu, Q., & Zhang, W. (2012). Biomorphic porous graphitic carbon for electromagnetic interference shielding. *Journal of Material Chemistry*, 22, 21183–21188.
- [9] Velez, P. N., Nenkova, S. K., & Kulevski, M. N. (2012). Polymer composites on the basis of lignocellulose containing copper sulfide for electromagnetic wave protection. *Bulgarian Chemical Communications*, 44(2), 164–171.
- [10] Rani, A., Nam, S.-W., Oh, K.-A., & Park, M. (2010). Electrical Conductivity of Chemically Reduced Graphene Powders under Compression. *Carbon Letters*, 11(2), 90–95.
- [11] Hao, W. (2014). Refining of hydrochars/ hydrothermally carbonized biomass into activated carbons and their applications. Stockholm University.
- [12] Reventós, M. M., Rius, J., & Amigó, J. M. (2012). Mineralogy And Geology: The Role Of Crystallography Since The Discovery Of X-Ray Diffraction In 1912. *Revista de La Sociedad Geológica de España*, 25(3–4), 133–143.
- [13] Zhang, K., Zhang, Y., & Wang, S. (2013). Enhancing thermoelectric properties of organic composites through hierarchical nanostructures. *Scientific Reports*, 3(1), 3448. <https://doi.org/10.1038/srep03448>
- [14] Heaney, M. B. (2004). Electrical Conductivity and Resistivity. In *Electrical Measurement, Signal Processing, and Displays* (pp. 7–1–7–14).
- [15] Nam, I. W., & Lee, H. K. (2015). Image Analysis and DC Conductivity Measurement for the Evaluation of Carbon Nanotube Distribution in Cement Matrix. *International Journal of Concrete Structures and Materials*, 9(4), 427–438.
- [16] Mittal, G., Rhee, K. Y., & Park, S. J. (2016). The effects of cryomilling CNTs on the thermal and electrical properties of CNT/PMMA composites. *Polymers*, 8(5).
- [17] Pang, H., Xu, L., Yan, D. X., & Li, Z. M. (2014). Conductive polymer composites with segregated structures. *Progress in Polymer Science*, 39(11), 1908–1933.