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MORPHOLOGICAL CHARACTERIZATION OF CHEMICALLY CARBON-CONDUCTIVIZED COTTON

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Solutions of green electric energy production are ever more intensively studied, developed and implemented in different activity areas, like textile industry, as a result of climatic changes. Such energy sources are intermittent by their nature; thus, the energy storage systems are necessary to guarantee the availability of supply at interruptions. The electric charge storage structures, like supercapacitors and batteries, are suitable for e-textiles with storage functionality due to the possibility of obtaining in flexible form. In this work, cotton material was used as substrate for a conductive carbonic deposition to obtain electrodes usable inside a further developed supercapacitor. The treatment was applied on a textile material using a carbonic conductive paste synthesized by a chemical route which has used pristine graphite and activated carbon as raw materials. The morphology of electrodes influences the ion mobility, and so the charging and decaying performance. The size of pores is a very important characteristic in such meaning, and its statistics were found based on the measurements performed by scanning electron microscopy (SEM). The chemical analysis was performed by X-ray energy-dispersive spectrometry (X-EDS), both in spectral and mapping mode, revealing the distribution of elements on surface of the finishing product applied on cotton substrate.

Keywords: energy storage, electrode porosity, conductive textiles

INTRODUCTION

The circular economy concept, which involves the reusing of the materials from goods after their lifetime, is also applicable in electricity production by harvesting the environment renewable energy (e.g. solar, wind, etc.) This approach consists of the so-called green technical solutions which aim to stop the growing of climatic changes and of ecosphere pollution (Ahmad *et al*., 2023). These energy sources have variable or interruptible features, a fact which leads to the needing the electricity storage systems. Especially at low power consumption, the most effective way to storage is electrochemically, using batteries or supercapacitors.

There are applications where such systems, both for harvesting and storage, must possess flexibility besides efficiency, e.g. wearable technologies. The textile materials are suitable to compose these structures by virtue of the rigidity lacking (Islam *et al*., 2022).

These storage devices consist of an electrolyte medium placed between two electrodes (cathode and anode), alternatively connected to the direct current generator (G) and to the energy consumer (X), during on the charge or the discharge cycle, as shown in Figure 1.

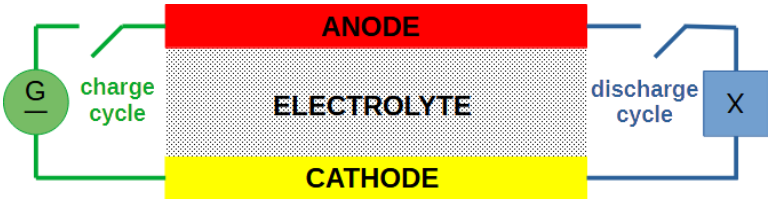

Figure 1. The sketch of electro-chemical storage device

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The electrodes require a porous morphology to allow inside a good impregnation of electrolyte, which increases the area of the electrolyte-electrode interface, thus a high storage capacity is available (Salleh *et al*., 2023). Also, this favorizes enough ion mobility which enhances the charge-discharge power. The structure of textile materials is very indicated to be involved in manufacturing such electrodes. The conductive finishing based on carbon compounds (graphite, graphene, nanotubes, carbon nanofibers, activated carbon) has shown an enhanced electrical performance (Paleo *et al*., 2018; Maher *et al*., 2021; Rădulescu *et al*., 2023). The activated carbon presents the most usability from such compounds (Maher *et al*., 2021).

In this work, textile electrodes were fabricated using cotton material as substrate for a conductive carbonic paste previously prepared. The electrode morphology was investigated by SEM followed by the statistical analysis of measurements. The elemental composition of the conductive treatment was performed by X-EDS on an electrode sample, both in spectral and mapping mode.

MATERIALS AND CHARACTERIZATION METHODS

Preparation and Functionalizing

In a one-liter Berzelius beaker (Figure 2), the following reagents were mixed using a glass rod: 45 g graphite powder, 45 g activated carbon powder, 10 g chitosan and 750 mL of 1% aqueous acetic acid solution. Firstly, the acidic solution and chitosan were mixed until the polymer dissolved. Then, the remaining ingredients were added to the obtained solution.

A cotton woven were cut in round substrates which were scoured before functionalizing, having the specific mass of 326 g/m^2, the thickness of 0.73 mm, and the warp and weft yarn densities of 340 yarns/10 cm and of 220 yarns/10 cm (Rădulescu *et al*., 2023).

The obtained paste was applied two times on a cotton substrate using a spatula, on both sides, followed by heating at 105ºC for 30 min to dry and fix the conductive treatment.

Figure 2. The prepared conductive carbonic paste

Morphological Characterization

The investigation of porous structure is essential to evaluate the suitability of a material for electrode use. While the materials show a variation in pore sizes, a statistical analysis of these measurements is necessary.

The characterization of porous morphology was performed by SEM using FEI Quanta 200 equipment. The image acquisition was made at 15 kV electron acceleration voltage, in top and transversal sample view modes.

The pore sizes were measured using an image processing tool (Scandium software) applied on the images from Figure 3.

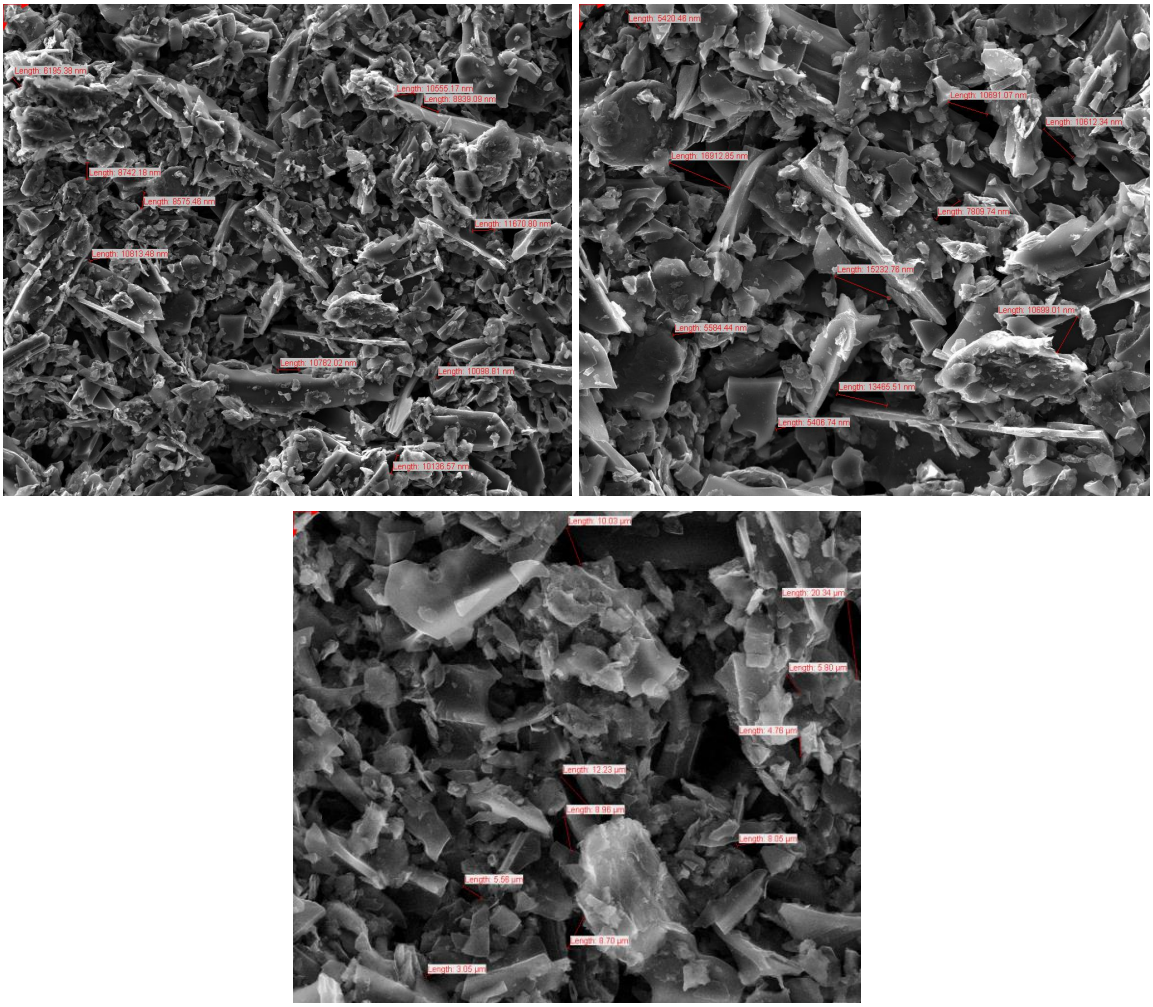


Figure 3. SEM images with porous morphology (pore size measurements)

The porous morphology on electrode surface is revealed in the acquired SEM images, generated by the granular structure of the dried applied paste. The statistical parameters found on the set of the 30 size values from the preceding images are 9.5 µm for average and 3.7 µm for standard deviation (STD), with the corresponding histogram from Figure 4.

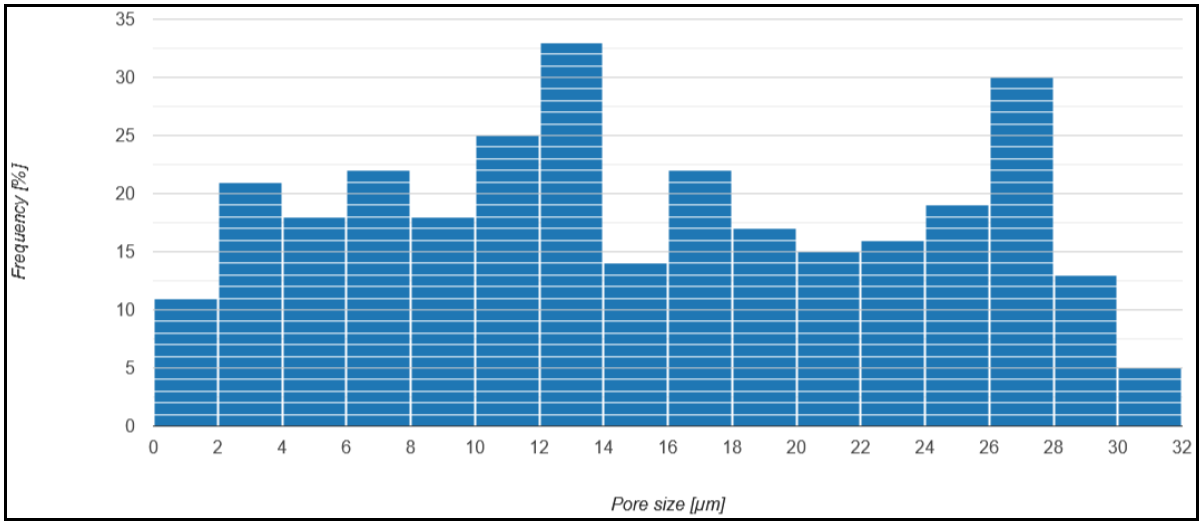


Figure 4. The histogram of pore sizes

The impregnation percentage of carbonic paste inside the textile substrate was evaluated by SEM in transversal section mode measuring the thicknesses of the layers formed after the conductive functionalizing (Figure 5).

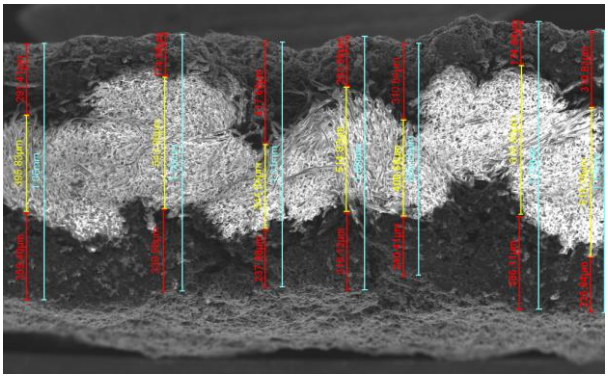


Figure 5. SEM image with layers thicknesses measurements (transversal view)

The transversal structure consists of three layers with variable thickness: one top and one bottom layer formed by impregnation of cotton substrate, and one middle layer of the substrate portion remained unimpregnated. In Table 1, the thickness values measured are shown. The total thickness was separately measured, not by the addition of the layers' thicknesses.

Table 1. Layers thicknesses

	Top layer thickness [µm]	Middle layer thickness [µm]	Bottom layer thickness [µm]	Total thickness [µm]
Average [µm]	269	491	301	1071
STD [µm]	90	111	66	83

Figure 6 shows the SEM images from which the cotton fibre dimensions were measured. In transversal section were found the values of 12 µm, 10 µm and 10 µm for width, and 22, 18 and 16 µm for length. In longitudinal view, the width and length are 8 µm and 16 µm, respectively.

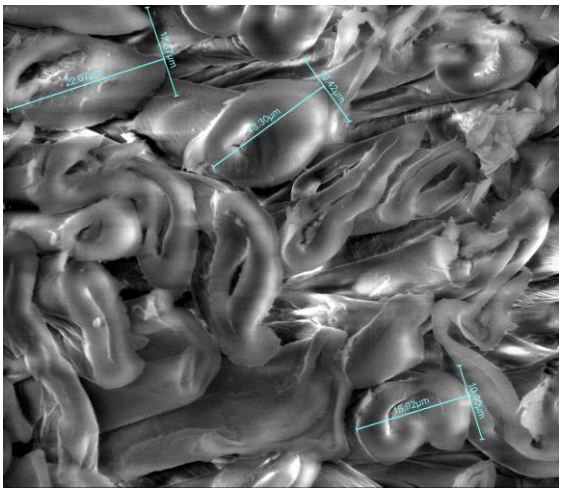
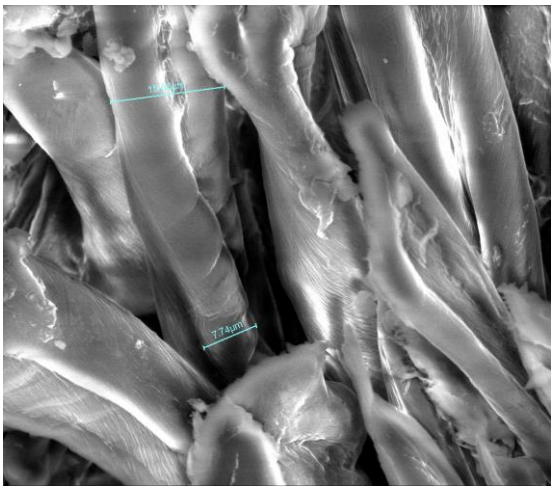
A)  B) 

Figure 6. Cotton fibres SEM images: A) transversal section; B) longitudinal

Chemical Analysis

The elemental composition of the compounds used in the preparation of the applied treatment was determined by X-EDS in spectral mode (Figure 7). The concentration of elements, in percentage, agrees with the atomic composition of the ingredients used and of the cotton substrate: 90.23% carbon, 7.34% oxygen, and traces under the detection limit of the nitrogen, aluminum and silicon which were measured to verify the impurities missing.

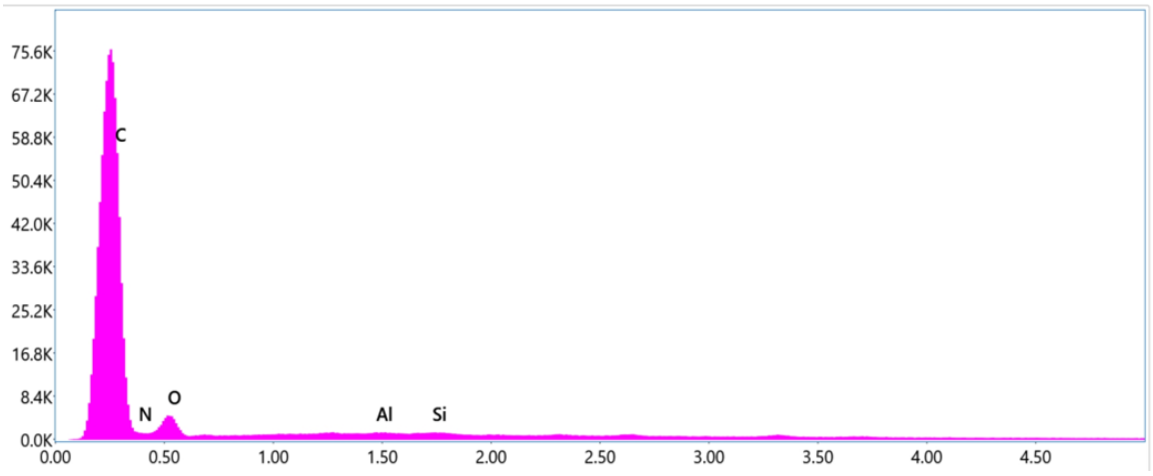
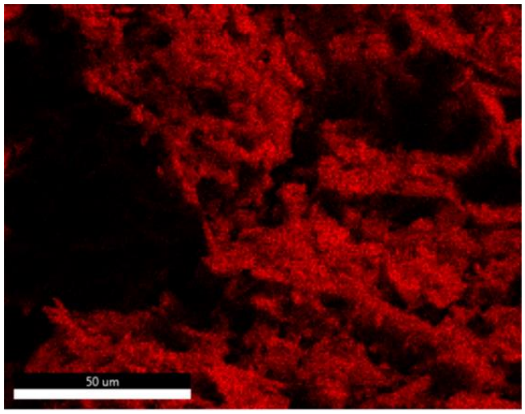


Figure 7. X-EDS spectrum

The X-EDS analysis in mapping mode was performed to visualize the distribution of chemical elements (Figure 8 shown this for the predominant elements of spectrum). Oxygen follows the carbon map because of the organic nature of substrate fibres and of the carbon-based and organic composition of the conductive treatment.

A) 

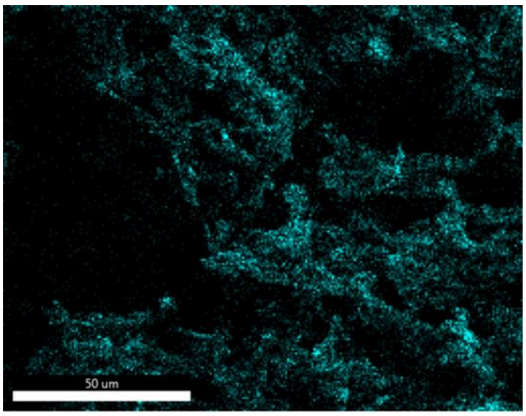
B) 

Figure 8. X-EDS maps for carbon (A) and oxygen (B)

CONCLUSIONS

The applied finishing shows an average impregnation depth, inside the textile substrate, of ~285 µm and a pore size of 9.5 µm average, both with acceptable variations. Therefore, a good filling of the porous structure with an electrolyte solution could be allowed in a further step toward the supercapacitor or battery fabrication, leading to an ion mobility and interface area enough for a performance comparable with non-flexible devices. The found interval of fibre dimensions is placed between 8 µm and 22 µm, thus revealing that the porous structure of conductive layer could only be formed inside the inter-yarns and, at most, inter-fibres spaces. This fact is concluded from the relatively comparable of the fibre dimension interval with the pore size in STD limits.

The electrode presents a clean chemical composition, the elements which could provide from the migration of the environmental mineral contaminants to the carbonic paste are missing in the acquired spectrum.

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