v. 3, n. 7, 2023

Journal of
Engineering
Research

CHARACTERIZATION OF ACTIVATED CARBON FELTS WITH DIFFERENT OXIDATIONS FOR APPLICATION AS ELECTRODE IN SUPERCAPACITOR

Jonathan Martimiano Teixeira Santos
PIBIC Scientific Initiation Report, supervised by Dr. Aline Castilho Rodrigues

Abstract: The project aims to study the morphology, structure and electrochemistry of activated carbon fiber. The production of these felts is divided into two stages: thermal oxidation and carbonization. Therefore, samples were prepared with different times (0, 90 and 135 min) of thermal oxidation exposure at 250 °C, which after the oxidation process were carbonized at 900 °C in an argon atmosphere. This way, the project focuses on carrying out tests in a cell with two electrodes of the Swagelok type to simulate a supercapacitor, in which charge/discharge curves are simulated to study how the fiber reacts when receiving and discharging electric charge, cyclic voltammetry for analyze its pseudocapacitive profile and electrochemical impedance of the samples, to analyze the resistances in the device. For the morphological and structure study, Raman and XPS spectroscopy were carried out, thus making it possible to analyze changes in the graphitic and chemical structure of the surface.

INTRODUCTION

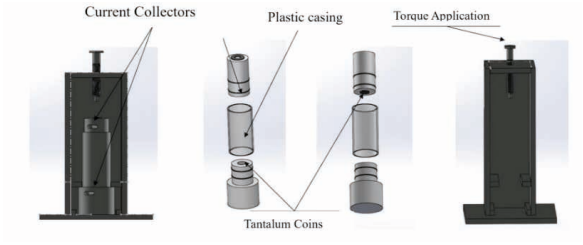
This project focuses on the characterization of different activated carbon felts to be applied as supercapacitor electrodes that can be used in aeronautical and aerospace environments, as well as contributing to the development of this technology in Brazil.

Specifically, the objective is to analyze the properties and potentialities of felts with different oxidations and to identify the differences caused by the variation of oxidations in the capacitance calculation. The aim is also to carry out all the relevant characterizations, morphological, structural and electrochemical, to identify the potential of the activated carbon felt as a carbonaceous material and as a possible supercapacitor electrode. In addition, the work aims to verify the potential of other materials deposited on the activated carbon felt, verifying the adequate closing torque for characterization.

DEVELOPMENT

METHODS AND MATERIALS

Samples of 8 mm diameter activated carbon fiber felts were used, which were dried in an oven at 100 °C for two hours, in order to remove the adsorbed moisture. After this step, the samples were weighed and immersed in the electrolyte for 24 hours(2 mol L^{-1} of H_2SO_4). For the electrochemical analysis, a symmetrical electrochemical cell with two electrodes was used, illustrated in Figure 1.



After setting up the experiment, with the aid of a torquemeter, the cell was closed with a torque of 10 N.cm, 20 N.cm and 30 N.cm, in each of the electrochemical measurements. The electrochemical analyzes performed were 0.0-1.0 V cyclic voltammetry (varying the sweep speed from 2mV s^{-1}-50 mV s^{-1}), 0.0-1.0 V galvanostatic charge/discharge curves (varying current from 1mA-50 mA) and electrochemical impedance spectroscopy.

DISCUSSION OF RESULTS

The Raman shift analysis was carried out with the objective of studying the graphitic structure of the samples with the standard oxidation time and comparing with the spectra of the samples with different times of heat treatment. Thus, it is possible to visualize possible changes in the structure and understand the behavior of the graphitic structure with the increase of carbon bonds inserted by the oxidative process. Figure X0

presents the Raman spectra of the samples with the standard oxidation treatment (called Ox 0) and the samples with longer oxidation times (Ox 90 and Ox 135 min). It is possible to identify two main bands, located at 1350 and 1600 cm-1. Such bands are related to the degree of disorder of the structure (band D), or defects, and to the degree of ordering (band G), or graphitization, respectively. All samples behave classically as a material with amorphous structure (SHIMODAIRA; MASUI, 2002).

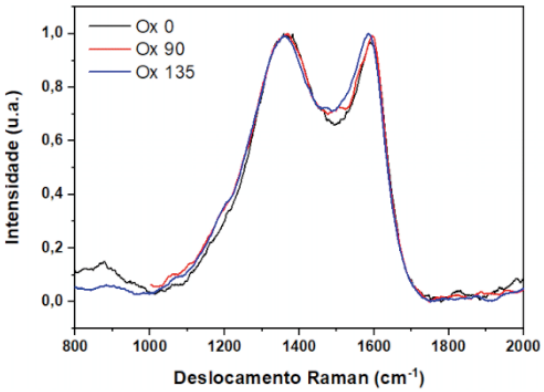


Figure X0 – Raman shift spectrum

In order to analyze possible variations or changes in the surface chemistry of the samples, an analysis of X-ray photoelectron spectra was performed(*X-ray photoelectron spectra - XPS*). The complete (survey) of the samples are shown in Figure X1 below. The XPS analysis was able to identify the main elements and their respective concentrations, present on the surface of each sample.

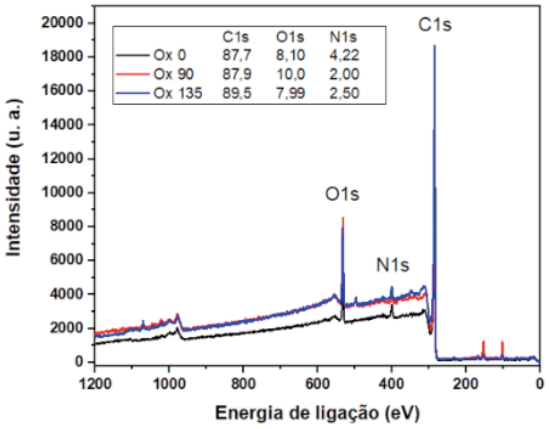


Figure X1 – Full Spectrum Survey XPS

Analyzing the concentrations, it is noted that there is a variation in the concentrations of the identified elements. Thus, it is possible to state that the variation in the oxidation time is sufficient to modify the chemical bonds and functional groups on the surface of the material. For the application of the material as a supercapacitor electrode, the element of greatest interest is nitrogen, since, depending on how it is connected to the carbonic structure, it will contribute to energy storage processes. Figure X2 relates the nitrogen concentration in the samples with the possible functional groups of nitrogen.

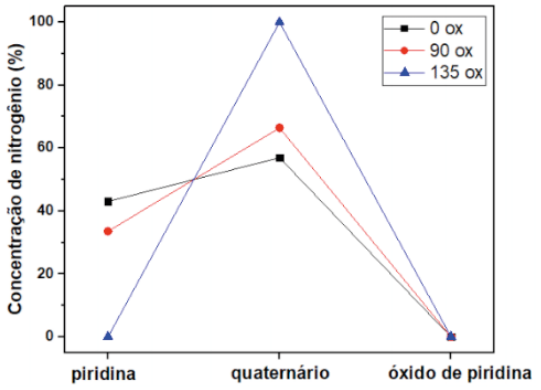


Figure X2 – Nitrogen concentration by nitrogenous functional groups

In the literature, certain nitrogenous functional groups directly influence the

capacitive response of energy storage devices (LIU; TUFA; LEE, 2019; RODRIGUES et al., 2019). The main groups are: N-pyridine, N-pyrrole, N-quaternary and pyridine N-oxide (WEIDENTHALER et al., 2006). Based on the XPS spectra, it was possible to identify possible nitrogenous groups present in the samples. It is also noted that the concentration of these groups varies with the time of oxidation.

CONCLUSION

We can conclude that when studying the structure of the samples varying the oxidation times using Raman spectroscopy, it is possible to visualize possible changes in its structural form and understand the behavior of the graphitic structure with the increase of carbon bonds inserted by the oxidative process. The samples have classic characteristics of carbonaceous material, with a similar behavior between them. In the XPS study, it is noted that there is a variation in the concentrations of the identified elements, and it is possible to state that the variation in the oxidation time is sufficient to modify the chemical bonds and the functional groups on the surface of the material, with nitrogen being the element that presents better results for its application as a supercapacitor.

Due to the lack of time to use the laboratories, caused by the quarantine due to the Covid-19 pandemic, it was not possible to complete the electrochemical tests that were planned.

REFERENCES

LIU, D.; TUFA, L. T.; LEE, J. N-doped microporous carbon hollow spheres with precisely controlled architectures for supercapacitor. **Electrochimica Acta**, v. 313, p. 389–396, 2019.

RODRIGUES, A. C. et al. Nactivated carbon fiber produced by oxidation process design and its application as supercapacitor electrode. **Journal of Porous Materials**, n. 0123456789, 2019.

SHIMODAIRA, N.; MASUI, A. Raman spectroscopic investigations of activated carbon materials. **Journal of Applied Physics**, v. 92, n. 2, p. 902–909, 2002.

WEIDENTHALER, C. et al. X-ray photoelectron spectroscopic studies of PAN-based ordered mesoporous carbons (OMC). **Microporous and Mesoporous Materials**, v. 88, n. 1–3, p. 238–243, 2006.