As Engenharias frente a Sociedade, a Economia e o Meio Ambiente 3

Henrique Ajuz Holzmann
(Organizador)

Atena Editora
Ano 2019

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Atena Editora
2019

Editora Executiva: Profª Drª Antonella Carvalho de Oliveira
Diagramação: Natália Sandrini
Edição de Arte: Lorena Prestes
Revisão: Os Autores

Atena Editora
Ano 2019

Dados Internacionais de Catalogação na Publicação (CIP)
(eDOC BRASIL, Belo Horizonte/MG)

E57 As engenharias frente a sociedade, a economia e o meio ambiente 3 [recurso eletrônico] / Organizador Henrique Ajuz Holzmann. – Ponta Grossa (PR): Atena Editora, 2019. – (As Engenharias Frente a Sociedade, a Economia e o Meio Ambiente; v. 3)

Formato: PDF
Requisitos de sistema: Adobe Acrobat Reader
Modo de acesso: World Wide Web
Inclui bibliografia
ISBN 978-85-7247-432-0
DOI 10.22533/at.ed.320192506

1. Engenharia – Aspectos sociais. 2. Engenharia – Aspectos econômicos. 3. Desenvolvimento sustentável. I. Holzmann, Henrique Ajuz. II. Série.

CDD 658.5

Elaborado por Maurício Amormino Júnior – CRB6/2422

Atena Editora
Ponta Grossa – Paraná - Brasil
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APRESENTAÇÃO

As obras As Engenharias frente a Sociedade, a Economia e o Meio Ambiente Volume 1, 2, 3 e 4 abordam os mais diversos assuntos sobre métodos e ferramentas nas diversas áreas das engenharias a fim de melhorar a relação do homem com o meio ambiente e seus recursos.

O Volume 1 está disposto em 31 capítulos, com assuntos voltados a engenharia do meio ambiente, apresentando processos de recuperação e reaproveitamento de resíduos e uma melhor aplicação dos recursos disponíveis no ambiente, além do panorama sobre novos métodos de obtenção limpa da energia.

Já o Volume 2, está organizado em 32 capítulos e apresenta uma vertente ligada ao estudo dos solos e aguas, com estudos de sua melhor utilização, visando uma menor degradação do ambiente; com aplicações voltadas a construção civil de baixo impacto.

O Volume 3 apresenta estudos de materiais para aplicação eficiente e econômica em projetos, bem como o desenvolvimento de projetos mecânico e eletroeletrônicos voltados a otimização industrial e a redução de impacto ambiental, sendo organizados na forma de 28 capítulos.

No último Volume, são apresentados capítulos com temas referentes a engenharia de alimentos, e a melhoria em processos e produtos.

Desta forma um compendio de temas e abordagens que facilitam as relações entre ensino-aprendizado são apresentados, a fim de se levantar dados e propostas para novas discussões em relação ao ensino nas engenharias, de maneira atual e com a aplicação das tecnologias hoje disponíveis.

Boa leitura

Henrique Ajuz Holzmann

SUMÁRIO

CAPÍTULO 4

MICROPOROUS ACTIVATED CARBON FIBER FELT FROM BRAZILIAN TEXTILE PAN FIBER: PREPARATION, CHARACTERIZATION AND APPLICATION AS SUPERCAPACITOR ELECTRODE

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RESUMO: Fibras de carbono ativado (FCA) são conhecidas por ser um excelente material adsorvente devido sua elevada cinética de adsorção e facilidade de manuseio. As FCA podem ser produzidas a partir da fibra de poliacriolonitrila e mesmo processo utilizado na fabricação de fibras de carbono, acoplando-se uma etapa de ativação ao final do processo. Neste trabalho é descrito a produção, caracterização química/morfológica e potencial aplicação de feltros de fibra de carbono ativado (FFCA), produzidos a partir de fibra PAN têxtil nacional em um equipamento experimental. Fibras PAN de 5.0 dtex em cabo de 200 mil filamentos foram oxidadas termicamente e utilizadas como matéria prima para a produção de feltro. O feltro de fibra PAN oxidada (FFPO) foi colocada em um porta-amostra, especialmente desenhado, e inserido em um forno tubular elétrico, carbonizado (900 °C) e em seguida ativado em atmosfera de CO_2 a 1000 °C. Todas as etapas foram feitas na maior taxa possível e as caracterizações foram feitas por isotermas de N_2 a 77 K, a química de superfície foi analisada por titulação de Bhoem. Os resultados mostram um material essencialmente microporoso (poros < 3,2nm, concentrados em 1,2 nm) e 1300 m^2 de área específica. O material produzido apresentou forte indicativo de aplicação como eletrodo de supercapacitor.

PALAVRAS CHAVE: Fibra de carbono ativada; feltro ativado; eletrodo supercapacitor

ABSTRACT: Activated carbon fibers (ACF) are known as excellent adsorbent materials due to their fast adsorption rate and easy handling characteristic. The ACF can be manufactured from the polyacrylonitrile fiber, based on an usual carbon fibers (CF) production process accomplished by an additional activation process. The aim of the present work is to describe the production, chemical/morphological characterization and application potentiality of activated carbon fiber felt (ACFF) produced from textile PAN fiber, using a set of homemade equipment. The 5.0 dtex PAN fiber tow with 200 thousand filaments was oxidized and used as raw material for felt production. The oxidized PAN fiber felt (OPFF) was displaced in a special sample holder, carbonized (900 °C) and then activated in CO_2 atmosphere at 1000 °C in an electric tubular furnace. All steps of the process were performed as fast as possible, and characterization was done by 77 K N_2 isotherms, adsorption isotherms in liquid phase, scanning electronic microscope, X-ray diffraction and surface chemistry by Bhoem methodology. The results confirmed the production of essentially microporous (pore < 3.2 nm, centered on 1.2 nm) and 1,300 $m^2 g^{-1}$ surface area. The ACFF produced have demonstrated a strong potential application as electrode supercapacitor.
KEYWORDS: activated carbon fiber; activated felt, supercapacitor electrodes.

1 I INTRODUCTION

Activated Carbons (AC) are one of the most widely used adsorbent materials around the world. Normally, AC is used for drinking and waste water treatment and in many other applications where the removal of generally dispersed contaminant molecules is desired (Marsh 2006). AC also have important applications in energy storage such as a supercapacitors material of and Lithium-ion rechargeable Batteries (Inagaki 2014).

Activated carbon fibers (ACF) have special characteristics when compared with common activated carbons (granular or powder). They can be transformed into fabric, woven or yarn forms which give them self-sustainable characteristics. In addition, ACF show well defined pore structures on their surface which provides a high and fast adsorption capacity for specific components (Solano 2008, Yoon 2000). Facing the application as supercapacitor electrode, ACF have, in comparison with powdered activated carbons, advantages such as a high surface-area, a good electrical conductivity and easy electrode formation and containment (Pandolfo 2006). In spite of all advantages of ACF applications, their use has been limited due to their relatively high cost.

One of the most important characteristics, which makes ACF a very special adsorbent material, is their pore size distribution. The architecture and structure of pores on ACF surface are characterized by a huge amount of micropores localized directly on the surface, leading to a faster and less energetic adsorption mechanism, especially for gases (Mochida 2000)].

The common methods used to produce ACF from a carbon fiber (CF) are not far from those used for AC production. The process can be simply described as a thermal treatment in an oxidant atmosphere at temperatures between 700 and 1000 °C (Solano 2008, Yoon 2000, Carrott 2001).

The ACF are highly quoted for adsorbent use, but it is very brittle and does not have enough mechanical resistance to be applied in normally used textile process. Consequently, it is very difficult to transform ACF in textile form. To solve this problem, oxidized textile PAN fiber was produced and then transformed into oxidized PAN fiber felt (OPFF). The OPFF was carbonized and activated to produce activated carbon fiber felt (ACFF).

2 I EXPERIMENTAL

The commercial 200 k tow of 5.0 dtex textile PAN fibers were oxidized in a laboratory scale oven set built, aiming an experimental production of flame-resistant fibers. About 200 kg of oxidized PAN were produced and transformed in felt (OPFF) with 200 g m^{-2}.

During the carbonization process, the oxidized PAN loses about 50% in mass and linearly shrinks 10%. The shrinkage is an important parameter and must be controlled (Marcuzzo 2012), and for this purpose, an OPFF sample about 0.7m X 0.25m was cut and displaced in a special sample holder that can control the sample shrinkage in two dimensions.

The set of such sample holder with felt was introduced in an electrical furnace (Figure 1). Both ends of the furnace tube were closed by flanges, which allow the insertion and the purge of argon gas to provide an inert atmosphere condition necessary for carbonization.

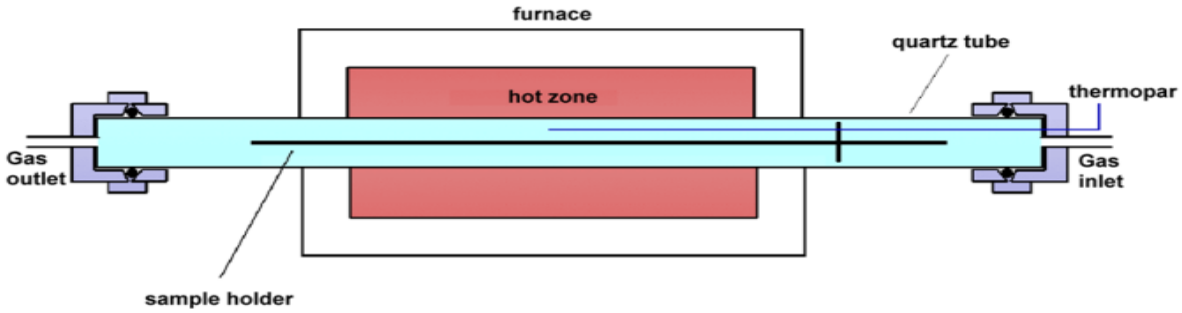

Figure 1. Carbonization and activation set up

The carbonization was performed in argon atmosphere at a final temperature of 900 °C, applying the maximum heating rate attainable (30 °C min^{-1}). The processing

time for maximum temperature was set at 20 min for carbonization. The activation process was performed immediately after the carbonization by changing the argon to carbon dioxide and rising up the temperature to 1000 °C; this temperature was maintained during 50 min. The activation time was defined by a previous essay where the mechanical evaluation was done, and it was fixed at 50 min to guarantee the minimal mechanical characteristic for handling the activated fiber samples.

After finishing the carbonization and activation process, the gas was shifted again from CO_2 to Ar and the furnace was turned off. This condition of inert atmosphere was maintained until the room temperature was achieved inside reactor.

The sample characterization was performed by N_2 adsorption aiming the measurements of surface area and pore sizes distribution function. The nitrogen isotherm was performed at 77 K by Beckman Coulter SA 3100 equipment. The BET method was applied to determine the total surface area; the pore size distribution and the micropore volume were estimated by applying the NLDFT method (Tarazona 1995) over the adsorption isotherm. The burn-off was estimated weighing the sample before and after the activation process.

The methylene blue and iodine adsorption capacities were also determined in liquid media, due to the fact that these characteristics are normally used as a reference in the adsorbate industry. High resolution SEM was used to check surface fiber integrity. The structure of activated carbon fiber was analyzed by X-ray diffraction technique (Cuña 2014) operated with monochromatic incident Cu Kα (λ= 1.5418A˚) ray and an automatic data acquisition system. The carbon surface chemistry was evaluated by the titration method proposed by Boehm (Boehm 1994).

For electrochemical characterization, a two-electrode Swagelok TM-type cells having two tantalum rods as current collectors were used. The electrodes (6.3 mg) were prepared by a rectangular cut of the ACFF, with cross-section areas of 0.8 cm^2 and thickness of 0.0015 m. A glassy microfiber paper (Whatman 934 AH) was used as separator, and 2M aqueous H_2SO_4 electrolyte was used. Galvanostatic charges and discharges were performed at a current density in the range 1-10 mA cm^{-2}, and at the voltage range of 0-1 V. The cross-section area was used to determine the current density. The specific capacitance was determined according to the equation: , where is the current applied, is the discharge time, is the voltage range during the discharge, and me is the mass of the electrode. The volumetric capacitance was determined according to the equation: , where ρ is the bulk density of the ACFF. The bulk density was determined by measuring the weight and the geometrical dimensions of each electrode. The impedance measurements were carried out in the frequency range from 1 mHz to 100 kHz with a signal of amplitude of 15 mV. All the electrochemical measurements were carried out at room temperature and 2M of sulfuric acid as the electrolytic solution and the setup was VoltaLab PGZ301 equipment.

3 I RESULTS AND DISCUSSION

The structural morphology of ACFF was studied by X-ray diffraction and scanning electronic microscopy. For pore structure analysis nitrogen gas adsorption at 77K was determined. In additional, iodine and methylene blue isotherm were used to the determine adsorption capacity. For surface chemistry, Boehm methodology was applied [11].

3.1 N_2 Isotherm

Figures 2 and 3 show, respectively, the nitrogen gas adsorption isotherm at 77 K and pore size distribution function.

The nitrogen adsorption isotherm at 77K (Figure 2) corresponds to a type I curve, with no hysteresis, and it shows that the gas saturation occurs at 0.2 P/Po, indicating that this activated material is predominantly populated with micropores. The pore size distributions curve performed by NLDFT (Non local density function theory) methodology is shown in Figure 3, and it clearly shows that the maximum pore width presented is around 3.2 nm and the predominant pores are sized at around 1.2 nm. This technique does not give information about pores less than 1.0 nm in diameter, due to the N_2 penetration limit, but it can be clearly observed in this curve that the distribution in the region for a size width less than 1 nm is ascendant in the direction of origin. This fact infers that the actual micropore volume and surface area of this material may be larger than those calculated by using these isotherms.

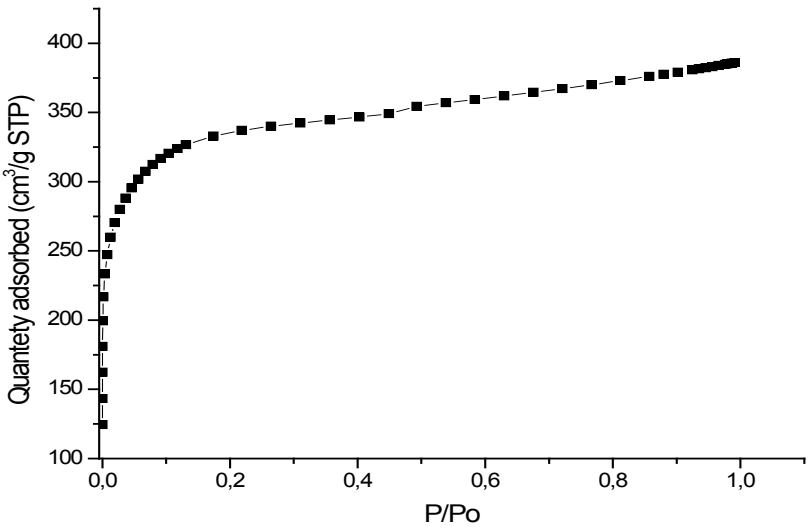

Figure 2. Nitrogen isotherm at 77 K.

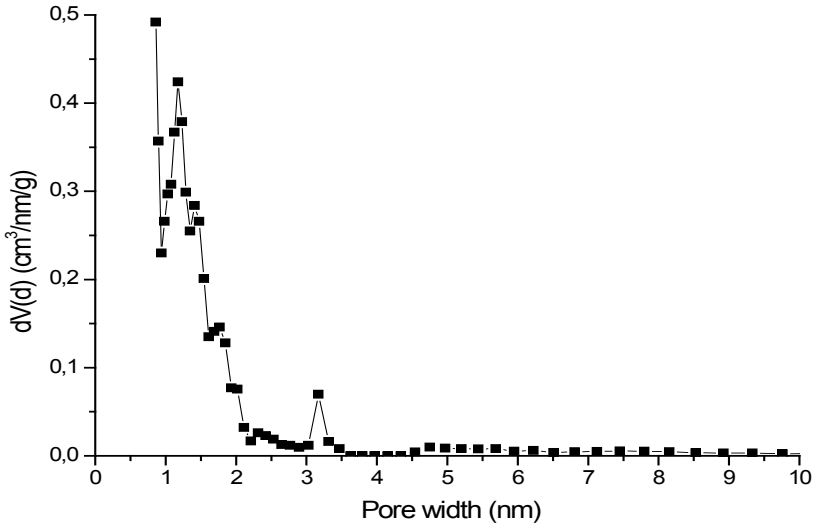

Figure 3. Pore size distribution by NLDFT

Table 1 presents the surface characteristics of activated carbon fiber felt obtained from textile PAN fiber, and the burn-off value of the activation process.

Sample	Burn off (w %)	S_{BET} ($m^2 g^{-1}$)	$V_{micropore}$ ($cm^3 g^{-1}$)	V_{Total} ($cm^3 g^{-1}$)
ACFF	35	1260	0,53	0,59

Table 1. Surface characteristics of ACFF

3.2 Iodine and methylene blue Isotherm

Iodine and methylene blue adsorption capacity of activated materials are parameters normally used to classify them as water pollutant organic compounds removing materials. Moreover, the adsorption capacity of iodine is associated to the presence of micropores sized between 0,5 and 1,5 nm (Fernandez-Colinas 1991), while the methylene blue adsorption capacity is related to small mesopores (Baçaoui 2001). For common activated carbon, the typical value of Iodine adsorption is around 1000 mg g^{-1} and methylene blue is between 100 and 350 mg g^{-1}.

The iodine content for ACFF was determinate by ABNT/MB-3410. The total Iodine adsorption was estimated in 1800mg g^{-1}. There isn't a standard to Methylene blue measurement, the common methodology to determine methylene blue in activated materials is by using spectrophotometer (665 nm) with silica sells of length 1cm. This methodology shows a 700 mg g^{-1} of methylene blue as ACFF adsorption content.

The amount of methylene blue and iodine ACFF adsorption is significant bigger then the value found for activated carbon on literature. This fact suggests that the ACFF is more efficient then activated carbon for small molecular compounds.

3.3 Scanning electronic microscopy

The carbonization heating rate used in this work is a rate not usually applied in the production of a material that essentially contains micropores. The usual heating rate is in the range of 1 to 10 °C/min (Ronaldo 2007), while in this work it was used the maximum heating rate allowed for the furnace which was 30 °C min^{-1}. The choice of such high value for this process parameter has been assigned to the production of macropores, cracks and other surface damages, however the SEM analysis of fiber surfaces showed the surface integrity of activated samples.

Figure 4 shows the felt fibers distribution overview. The fibers are not broken for a small length. This is associated to their mechanical characteristics handling.

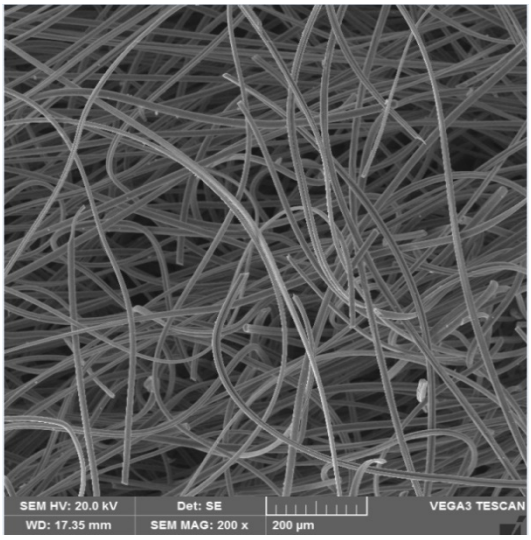

Figure 4. Filament structure ACFF overview.

Figure 5 shows details of the carbon fiber filament. It can be observed that macropores, cracks, collapsed filaments and any other macroscopic surface impairment are absent. In addition, it can be said that the surface of activated fibers is clear and smooth.

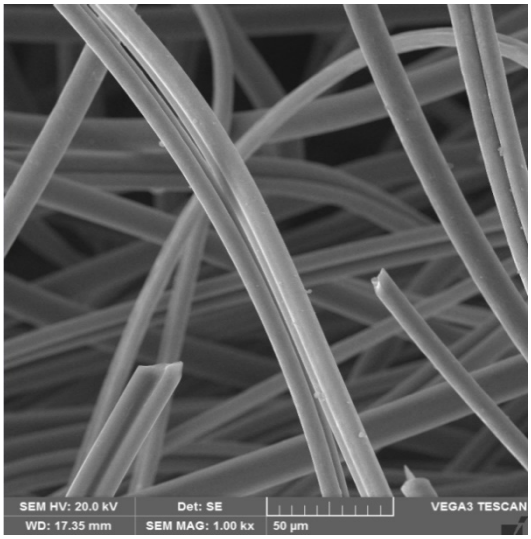

Figure 5. Detail of activated carbon fiber in the filament structure of activated carbon fiber felt.

As the ultimate surface damage analysis, the high-resolution SEM image, with 150 k magnification, is also provided and presented in Figure 6. This micrograph shows that the activated carbon fiber surface is exempted of macro-damages, which in turn indicates that the high heating rate does not have any relationship with macropores or damage production on the activated carbon fiber surface.

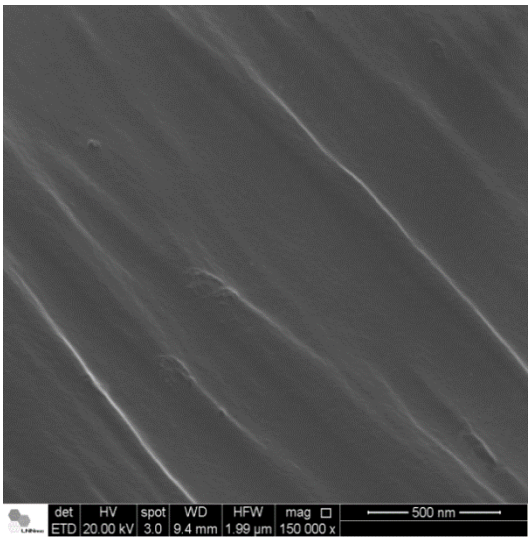

Figure 7. Magnification of 150 K on the activated carbon fiber.

3.4 X-ray diffractogram (XRD)

XRD of the activated carbon fiber felt is shown in Figure 7. The amorphous character of this fiber was confirmed. Two broad peaks appear at approximately 2θ = 25º and 44 º. The first peak is associated with *(002)* line, while the second at 2θ = 44 º is *(100)* line. The presence and the shapes of these two bands indicate that the activated carbon fiber (prepared by carbonization at 900 °C) has a more organized aromatic structure with marked sp^2 bonding character than other amorphous carbon, reported in the literature. These materials when prepared at lower temperature, such

as the one used in this work, present higher sp^3 bonding characteristic (Cuña 2014, Celorrio 2011, Kennedy 2004, Mochidzuki 2003).

Based on these results, it is expected that this fiber presents a relatively good electrical conductivity, what is an important characteristic for applications such as supercapacitor electrode manufacture [5, 10, 18].

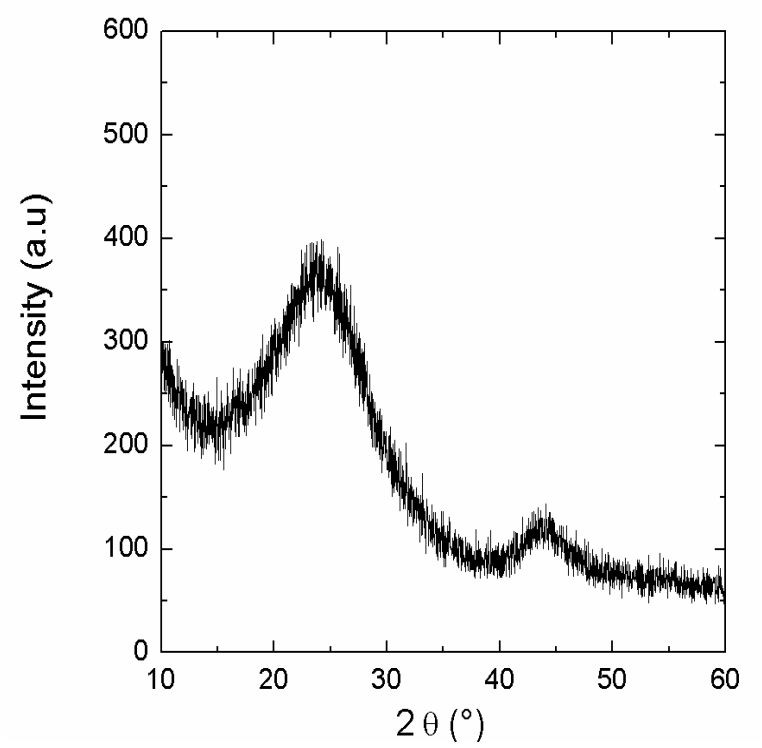

Figure 7. X-ray diffractogram of ACFF

3.5 Chemical surface by Boehm

The well know Boehm methodology [11] was used to investigate the activated carbon fiber felt surface chemistry. By using the Boehm titration, it is possible to determine the amount of main acid groups as: carbonyl, carboxyl, phenolic, lactone, and also basic groups that there aren't detectable per species.

The results are presented on Table 2. It is possible to observe only two different species; however, the presence of carbonyl groups is much less prevalent than the basic groups that shows a considerable amount. Consequently, the activated carbon fiber felt surface has basic characteristics. This result indicates a basic behavior for aqueous applications.

3.6 Electrochemical characterization as supercapacitor electrode

Figure 8 shows the specific capacitance Cs, and volumetric capacitance Cv, as a function of the current density. The ACFF has a high Cs at low current densities (200 F g^{-1} at 1 mA cm^{-2}), comparable to those reported for other carbon materials using the same electrolyte [5, 10, 18]. This was expected by its the high specific surface area

(1260 m^2 g^{-1}) which gives a high double layer capacitance. A current density increase produces a drastic drop in the capacitance, which is mainly due to the high electrical resistance of the cell.

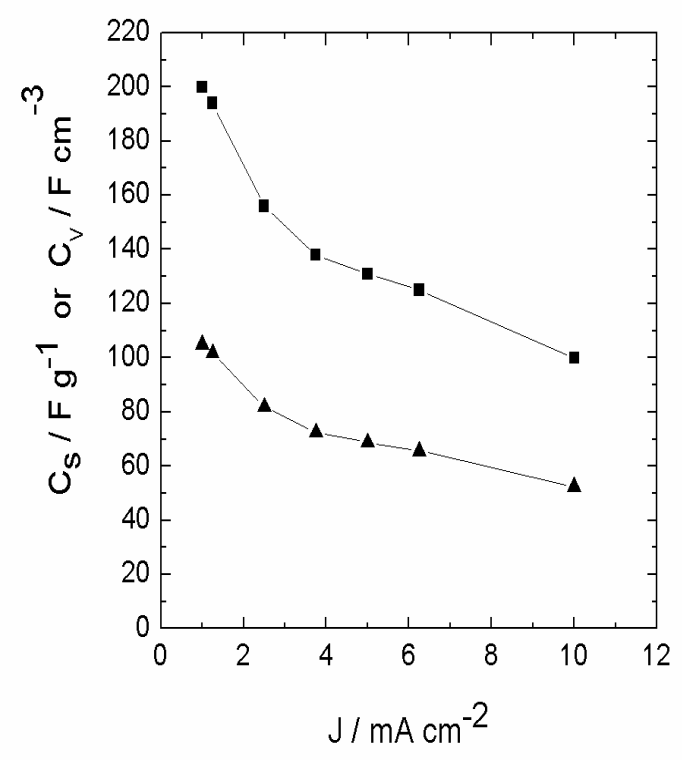

Figure 8. Variation of the specific capacitance Cs (squares) and the volumetric capacitance CV (triangles) with the current density.

This is demonstrated in the Nyquist plot (Figure 9) obtained from the electrochemical impedance measurements. In this plot, Rs is the ionic resistance of the electrolyte bulk and Ra is the arc resistance (Figure 9 inset). The total resistance of the cell can be defined as Ra+Rs. Ra can be linked to the electric resistance of the electrode plus the resistance of the electrode/current collector contact (Garcia 2010). For the ACFF studied in this work, Ra has a very high value (125 Ω), highest than the Rs (2.7 Ω). As discussed in section 3.3, it is expected that the activated fiber has a high electrical conductivity, but the poor connectivity between the fibers in the ACFF determine a high electrode resistance and electrode/current collector contact resistance [5].

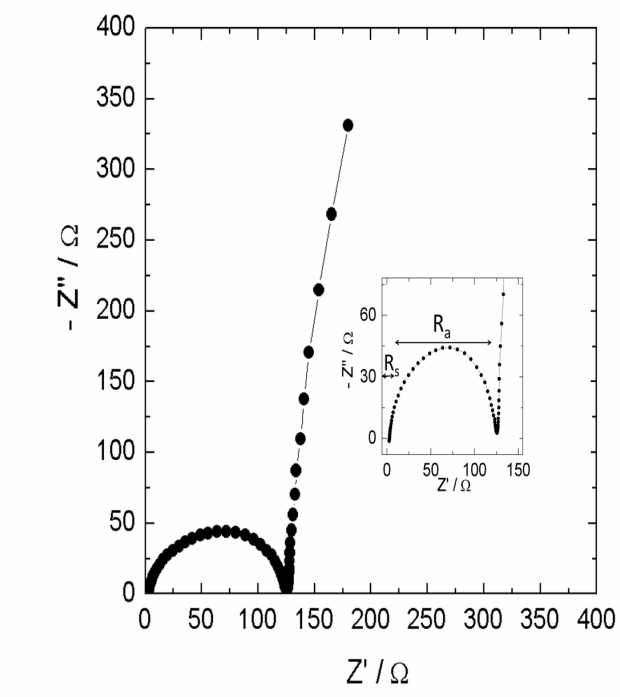

Figure 9.Nyquist diagram for the ACFF cell. Inset: magnification of the high-frequency region showing the arc. Ra is the arc resistance and Rs is the electrolyte bulk resistance.

The bulk density of the ACFF was 0.552 g cm^{-3}. Given this value, Cv of the ACFF was determined for different current densities. At 1 mA cm^{-2}, Cv was 104 F cm^{-3}, slightly larger than those reported for bio carbon monoliths (Cuña 2014) but significantly less than that reported for commercial carbon monoliths treated by different methods (Kunowsky 2014). The biggest disadvantage of the ACFF is the drastic drop in capacitance at the highest current density.

4 I CONCLUSION

It was proved that Oxidized PAN fiber felt can be transformed in a relatively high surface specific area activated material, with 1300 $m^2 g^{-1}$. Moreover, the heat treatment and the activation treatment parameters can be adjusted in a way that they drastically reduce the time and energy consumption for the manufacturing process. The surface characterizations showed that the produced activated carbon fiber felt is a material populated essentially by micropores sized at 1.2 nm, and with minor amount of 3.2 nm mesopores More work must be performed in the future to better describe the pore structure with sizes below 1 nm, which was not accessed by nitrogen. XRD analysis shows that the activated carbon fibers do not present an ordered two-dimensional structure. The activated carbon fiber felt produced from textile PAN fiber, is an interesting material for supercapacitor electrode application, it could be successfully used as a supercapacitor electrode material if its high electrical resistance can be reduced.

5 I ACKNOWLEDGMENT

The authors want to acknowledge ITA for the conventional SEM images; the CNPEM/ LNNano for the high-resolution SEM images; the Multivácuo Aeroespacial Ltda. for the laboratorial infrastructure and FAPESP Process 12/51087-6 and 09/52989-0for the financial support.

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Agência Brasileira do ISBN
ISBN 978-85-7247-432-0