INFLUENCE OF THERMAL TREATMENT ON MECHANICAL PROPERTIES OF ACTIVATED CARBON FIBERS

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Introduction

Activated carbon fibers (ACFs) are known as excellent adsorbent materials due to their relatively high adsorption rate, easy handling characteristic and well defined pore structure in their surfaces providing a high adsorption capacity [1]. The ACFs are commercially manufactured by usual carbon fibers (CFs) production process accomplished by an additional activation process, using the standard precursor fibers as raw material [2]. In the current work, the textile PAN fiber was used for ACF production with the main purpose to study the influence of carbonization heating rate on the mechanical property of treated material, as inferred from measurements of tensile strength of the activated carbon fibers.

Experimental

The 5.0 dtex textile PAN fibers were specially spun in 50 ktex tow by Radicifibras Industria e Comércio Ltda. The oxidized PAN fiber was produced in a semi continuous laboratory set up by two steps heat treatment done at 245^{o}C for 40 min and 265^{o}C for 50 min.

Approximately 10g of oxidized fiber, with 43 cm length, was used as sample for carbonization process, which was performed at temperatures between 600^{o}C and 1100^{o}C, in inert atmosphere (argon – 10 sccm), using a static tubular furnace (Fig 1), and four different heating rates: 5, 10, 30 and 100°C/mim.

The carbonization time was fixed at 20min and the cooling was done by removing the quartz tubular chamber from furnace to the room temperature. The carbonization time was chosen by considering results obtained in previous study using the mass spectrometry for analysis of released gases [4].

During the carbonization, a force with 0.3 N magnitude was applied on the sample by a spring fixed at one end of the tow, outside the high temperature zone (details in Fig 1). The activation process based on CO_2-10sccm [3] atmosphere was performed at 1000°C for 30 min using the same apparatus for carbonization.

The activation processes were conducted on selected samples produced by different carbonization temperatures for each heating rate condition, which exhibited maximum moisture adsorption capacity [5]. This choice was shown as reliable methodology for the process of activated carbon fiber production.

The activation process promotes the fiber degradation by thermal oxidation. A set of preliminary experiments have been done before, and we found that after about 40 min of activation, the tow of fiber breaks, indicating that the sample does not support 0.3N anymore. Thus, in order to maintain minimal mechanical propriety of activated fiber the process time was fixed in 30 min for all experiments [4].

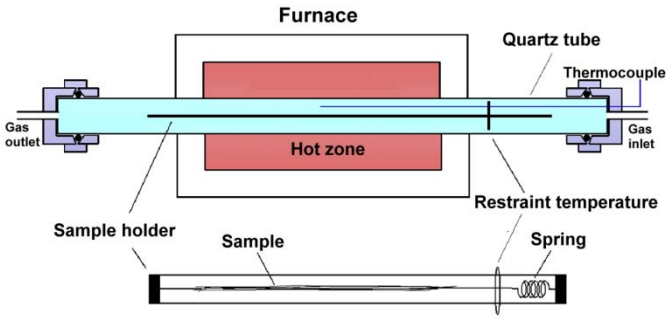

Fig.1 – Carbonization set up used to produce carbon fiber sample from oxidized textile PAN fiber.

Aiming the detection of surface activity of fibers, a 1000 mg/l phenol solution was prepared, and 0.1g of activated fiber sample was inserted into the solution at 25°C, and they were stirred during 1h. The characterization provided as result the amount of phenol removed from original solution. A complete adsorption isotherms study is planned to be done.

The mechanical characterization was performed by Fafegraph HR serie 34110 and applying ISO 11566:1996E method. The moisture content was determined by periodical weighting of 1 g sample, maintained at 110 ^{o}C, during the time enough to complete desorption, which is indicated by mass value reaching a constant value.

Results and Discussion

The Table 1 shows some physical characteristics of the oxidized textile PAN fiber used as raw material in the present work, together with the values of commercial PANOX to provide the comparative analysis of both materials.

The results of moisture content measurement over all carbonized samples are summarized in Fig. 2. The analysis of data reveals a large spread of moisture content produced by carbonization, a common feature observed in all curves of Fig. 2. However, it is possible to clearly detect the existence of a maximum value in each curve. For curves generated by samples treated at 5, 30 and 100 ° C/min heating rate, the maximum moisture is found at 850° C, while for 10°C/min sample this temperature is at 950°C. These range of temperature for maximum moisture values provide the conditions we used for carbonizing the samples for subsequent activation.

The characterization results of carbonized and activated fibers are all together presented in Table 2.

Table 1. Oxidized 5.0 dtex PAN fiber and PANOX® *

Properties	Oxidized PAN	PANOX®
Moisture content (%)	5	9
Density (g/cm^3)	1.36	1.35
Tensile strength (MPa)	216	210
Tensile Modulus (GPa)	5.2	ND

* Type LB C076 SSC

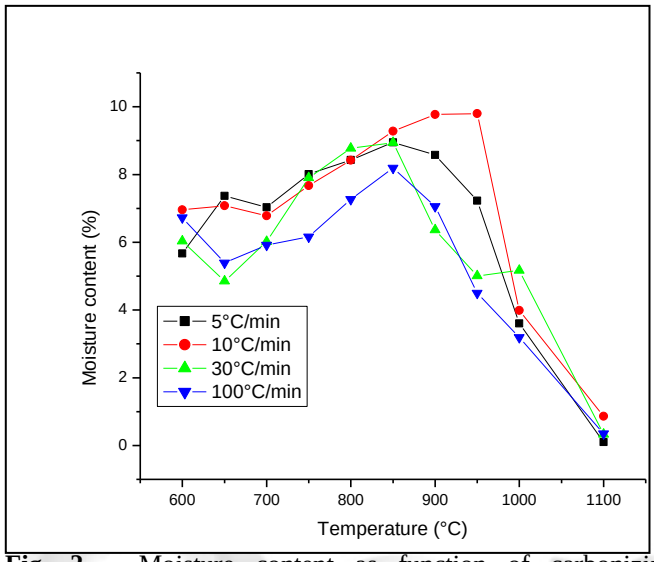

Fig. 2 – Moisture content as function of carbonizing temperature for fibers treated at different heating rates.

The phenol adsorption results for carbonized fibers are not presented in this table due to the fact that all measurements gave nil value. The same measurements done with activated fibers gave results ranging from 20 to 49 mg of adsorbed phenol for 1 g of activated fiber. These results lead us to conclude that the fibers treated in this work are actually ACF.

On the other hand, the moisture content percentages are between 8 and 9% for carbonized fibers, while 5 to 11% are for activated fibers. These results indicated that, as far as the adsorption of different molecules is concerned, the surface pore structures produced by different process parameters may lead to different adsorption effectiveness. The main noticeable feature depicted in this characterization was the great decrease of moisture content for activated fiber produced at fastest carbonization (100°C/min).

The mass loss due to the volatilization during the carbonization decreases when heating rate was raised up, while the tensile strength of fiber (σ) slightly varies. The tensile modulus (E) presented high fluctuation without well defined tendency related to the heating rate of carbonization.

The burn off percentages of activation process increased while the tensile strength decreased with heating rate of carbonization. This fact indicates that the activation by CO_2 produces structural damage of a carbonized fiber while creates activated sites for phenol adsorption. In other hand, the phenol adsorption capacity, which is absent for a carbonized fiber, turn out to be significant, i.e., in the range of 20 – 49 mg/g for an activated fiber. Its variation respective to the heating rate has similar behavior as the burn off. As the phenol adsorption methodology applied is only a preliminary study, it is worth to note that a complete isothermal adsorption study would be necessary to accomplish the characterization of activated fibers.

Table 2. Carbonized and activated samples characteristics

	Carbonized fiber			
	5°C/min	10°C/min	30°C/min	100°C/min
Moisture (%)	9.0	9.3	9.0	8.2
Mass loss (%)	44	42	37	38
σ (MPa)	482	472	545	503
E (GPa)	30.0	25.6	33.8	24.0
	Activated fiber			
Moisture (%)	11.2	10.9	11.3	4.9
Burn off (%)	10	22	24	25
σ (MPa)	289	180	208	87
E (GPa)	28.0	20.0	16	7,5
Phenol (mg/g) adsorption	20	49	46	45

Conclusion

ACF produced by CO_2 thermal oxidation can be used as phenol adsorbent material.

The activated carbon fiber produced by 5°C/min presents highest tensile modulus (E) and tensile strength (σ), compared with values of other ACFs, but it is a poor phenol adsorbent fiber.

The highest heating rate (100°C/min) of carbonization produces higher burn off and inferior mechanical properties when activation process is accomplished.

The best results of phenol adsorption and mechanical characteristics were produced by carbonization at 10 and 30°C/min heating rates.

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