# Structural and functional properties of porous carbon fibers composites

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Porous carbons have a wide application in many fields such as gas separation, purification and storage, as catalyst support, or liquid phase processing. The porous carbon fiber composite is rigidly bonded in an open, permeable structure. The composites were prepared from short pitch based carbon fibers and a thermosetting resin. The composite was carbonized at 650°C. In this paper we study the influence of carbon fibers size versus structural and functional properties of the composite. The carbon fiber composites were characterized by optical microscopy, atomic force microscopy (AFM), X-ray diffraction (XRD), mechanical and thermal measurements.

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## 1. Introduction

Carbon fibers are produced commercially from rayon, PAN and pitch. High performance fibers, such as those with high strength and stiffness, are generally produced from PAN and mesophase pitch. Isotropic fiber applications include: friction materials, reinforcements for plastics, electrically conductive fillers for polymers, filters, paper, hybrid mix, and as reinforced concrete.

Recently, interest has been focused on carbon fibers activated by chemical or physical methods [1-3]. Activated carbon fibers have new properties that make them more attractive than conventional forms (powder or granule carbons) for specific applications. Among possible applications, activated carbon fibers are of interest for the adsorption and recovery of organic vapors, the environment protection, removing $CO_x$, $SO_x$ and $NO_x$ from gases, improve air quality and water treatment [4-6]. Difficulties in using activated carbon fibers can be overcome by incorporating them into a composite, such as woven or nonwoven structure, felt or paper.

We present in this article the preparation of a rigid composite with carbon fibers, which has an open and permeable structure.

## 2. Experimental

The monoliths were prepared from milled pitch based carbon fibers and a phenolic resin. The fibers have an average length of approximately 400 μm, respectively 800 μm. The weight ratio of carbon fibers to phenolic resin is 3:1. The fibers and phenolic resin powder were mixed with water to obtain slurry. The slurry was transferred into a mold and the water was removed under vacuum. The resulting forms were dried in air at 60 °C for 16 hours and after this at 150 °C for 4 hours in order to produce a cured monolith.

The properties of milled carbon fibers are presented in Table 1 and the properties of phenolic resin powder are presented in Table 2. Milled carbon fibers were purchase from Asbury Graphite Mills Inc. (New Jersey, USA).

*Table 1. Physical properties of milled carbon fibers from petroleum pitch.*

| Properties | Carbon fibers type AGM95MF0400 | Carbon fibers type AGM95MF0800 |
|---|---|---|
| Average length, μm | 400 | 800 |
| Fiber diameter, μm | 13 | 13 |
| Density, g/cm$^3$ | 1.54 | 1.54 |
| Carbon, % | min 95% | min 95% |
| Resistivity, μΩm | 60 | 60 |
| Tensile strength, GPa | 0.5 | 0.5 |
| Young's Modulus, GPa | 35 | 35 |

*Table 2. Physical properties of phenolic resin powder.*

| Properties | |
|---|---|
| Appearance | Yellowish powder |
| No tamped volumetric weight, g/dm$^3$ | 350-550 |
| Tamped volumetric weight, g/dm$^3$ | 600-800 |
| pH | 7-8.5 |
| Softening point, °C | 75-90 |
| Melting point, °C | 100-115 |
| Solubility in acetone, ethyl alcohol, ethyl acetate | Soluble |
| Solubility in benzene, carbon tetrachloride, gasoline, water | Partially soluble |
| Rest on the 0.1 wire cloth sieve, % | max 2% |

The obtained monolithic composites (FCN400 and FCN800) were carbonized with a heating rate of 2 ºC/min up to a maximum temperature of 650 °C and held at this temperature for 3 hours in a nitrogen flow of 500 ml/min (Fig. 1).

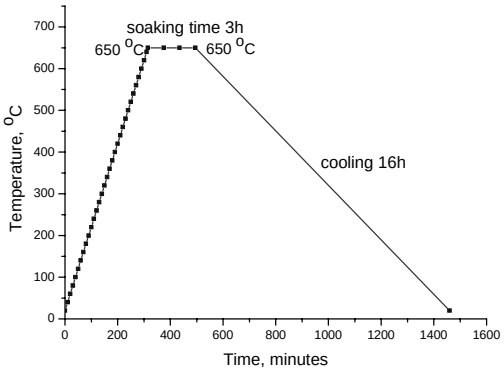


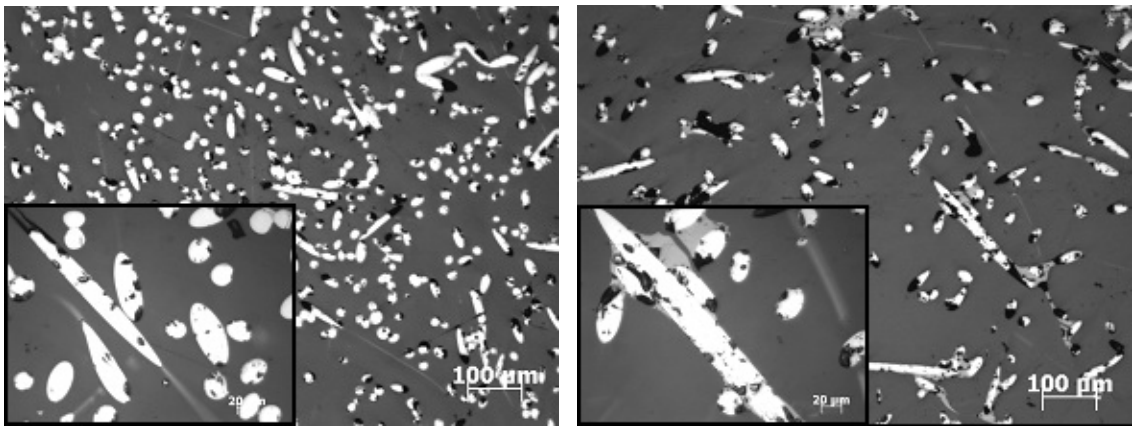

*Fig. 1. Thermal treatment diagram for monolithic composites.*

## 3. Results

The carbon fibers composites were structurally characterized using optical microscopy, atomic force microscopy (AFM) and X-ray diffraction (XRD). The optical properties were studied by using a Zeiss Axiolab microscope. AFM analyses were performed with an Atomic Force Microscope model CP-100-10 VEECO. X-Ray Diffraction analyses were performed with a D8 ADVANCE type BRUKER-AXS Diffractometer, equipped with a Cu target X-ray tube (λ=1.5406 Å) 40 kV/30 mA and Ni $K_\beta$ filter, 0.04° step, measuring time of a point 1 second.

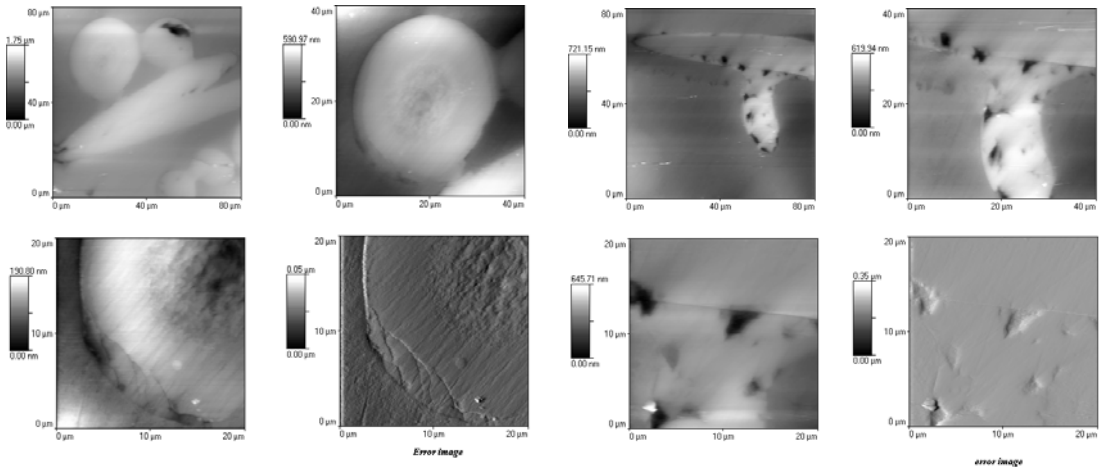
Flexural (three point bending) tests were performed with a testing machine type Zwick TR FR 005 TN at a nominal load of 5 kN. Six test specimens to each composite were used for flexural tests. The specimens have rectangular shape with the dimensions: 4 × 8 × 32 mm.

Thermal diffusivity of composites was measured using a device type LFA 447 NanoFlash – NETZSCH.




a) b)

*Fig. 2. Optical micrographs of composites: a) FCN400, b) FCN800, optical zoom 100x, 500x.*



a) b)

*Fig. 3. AFM images of composites: a) FCN400, b) FCN800.*

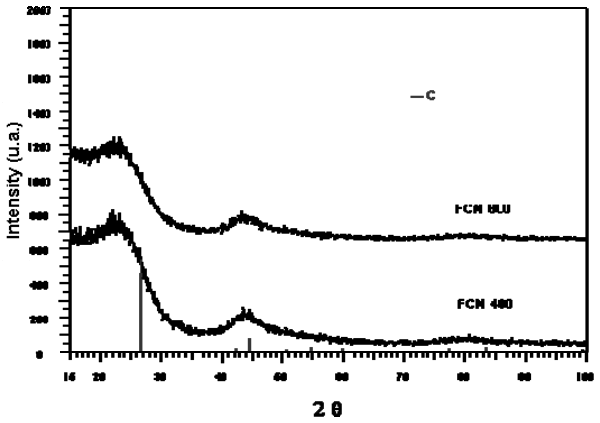



*Fig. 4. XRD pattern of composites FCN400 and FCN800.*

*Table 3. Cell parameters obtained from XRD patterns.*

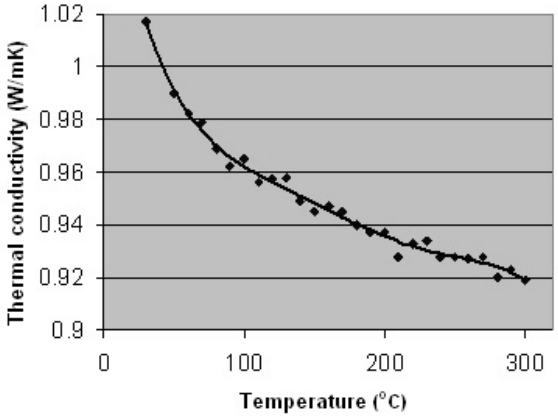
| Sample | a, [Å] | c, [Å] |
|---|---|---|
| FCN400 | 2.403 | 7.727 |
| FCN800 | 2.410 | 7.767 |
| Turbostratic graphite (index 03-065-6212) | 2.464 | 6.711 |

*Table 4. The average size of crystallites calculated with Debye-Scherrer formula.*

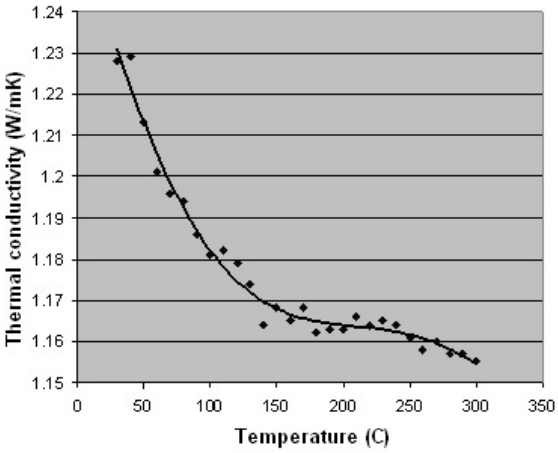
| Sample | hkl | D, [nm] |
|---|---|---|
| FCN400 | (002) | 1.4 |
| | (101) | 3.0 |
| FCN800 | (002) | 1.7 |
| | (101) | 3.0 |

*Table 5. The flexural strength of carbon fiber composites FCN400 and FCN800*

| No. | Sample | Flexural strength, [MPa] | Mean of measurements, [MPa] |
|---|---|---|---|
| 1. | FCN400 | 0.78 | 1.11 |
| 2. | | 0.80 | |
| 3. | | 1.15 | |
| 4. | | 1.06 | |
| 5. | | 1.27 | |
| 6. | | 1.62 | |
| 7. | FCN800 | 2.37 | 1.59 |
| 8. | | 1.10 | |
| 9. | | 1.17 | |
| 10. | | 2.03 | |
| 11. | | 1.91 | |
| 12. | | 0.95 | |




a)




b)

*Fig. 5. Thermal conductivity of carbon fiber composites: a) FCN400, b) FCN800*

## 4. Discussion

Fig. 2 (a, b) presents optical micrographs of carbon fiber composites FCN400, respectively FCN800. We can observe that the carbon fibers are bonded in composites in a tridimensional structure due to carbonized phenolic resin. The carbon fibers are randomly oriented in the structure. Carbonized phenolic resin provides to the carbon fibers composite a porous and unitary structure.

From the optical micrographs analysis, we can see a homogeneous dispersion of carbon fibers with lengths of 400 μm in composite FCN400, better than in composite FCN800.

Fig. 3 (a, b) shows topographic images obtained by atomic force microscopy for composites FCN400 and FCN800. It can be seen that the carbon fibers and phenolic resin have a good compatibility. The carbonized resin provides the link between carbon fibers. The composites have a porous open structure.

Fig. 4 shows the XRD patterns of FCN400 (a) and FCN800 (b). Classical analysis of XRD pattern obtained on

graphite type carbon materials is based on planar disordered network model, called turbostratic model (Biscoe and Warren 1942). In agreement with this model, carbon atoms form stacks of flat graphenic equidistant layers, parallel planes randomly translated and rotated around an axis perpendicular to the plane. Turbostratic structure can be considered as a two-dimensional structure in which graphenic planes scatter radiation independently.

XRD patterns of FCN400 and FCN800 relieve the presence of (002) line which is characteristic of turbostratic graphite. Their structure is similar to the hexagonal structure of graphite, having elementary cell parameters presented in Table 3. Table 4 presents the average size of crystallites calculated with Debye-Scherrer formula.

Table 5 contains the values of flexural strength for the carbon fiber composites. We can observe that the mean value of flexural strength is 1.59 MPa for composite FCN800, greater that 1.11 MPa for composite FCN400. However, we see a greater heterogeneity of the composite FCN800, mechanical strength ranging from 0.95 to 2.37 MPa, while the mechanical strength of FCN400 ranging from 0.78 to 1.62 MPa. Although greater length of carbon fibers increases the mechanical strength, their weaker dispersion leads to inhomogeneity of the sample.

Fig. 5 shows the dependence of thermal conductivity of composites with the temperature. The device used for measurements is equipped with a furnace operating at temperatures between room temperature and 300 °C. The temperature rise on the other side of the sample and it is measured using a detector type InSb. Acquisition and evaluation of results is made using a software package. The data obtained were used to calculate the thermal conductivity of the composites.

As it is noticed, FCN800 sample has a better thermal conductivity than FCN400 sample, the values decreasing with the increasing of temperature. Changes in thermal conductivity FCN800 temperature varies much more steeply than for FCN400 sample, which has a smoother variation. Better thermal conductivity of the composite FCN800 is determined by carbon fibers greater length.

Values for thermal conductivity are very good, of 1.017 W/m·K for the composite FCN400 and 1.228 W/m·K at 30°C for composite FCN800, since the thermal conductivity of the granular activated carbon packed beds is limited to 0.14 to 0.17 W/m·K, as specified in the literature [7, 8]. Conductivity of composites FCN400 and FCN800 decreases with the increasing of the temperature, at a temperature of 300°C reaching values of 0.919 W/m·K for composite FCN400, respectively 1.155 W/m·K for composite FCN800.

## 5. Conclusions

The porous carbon fibers composites studied in this article have an interesting structure. A monolithic porous carbon structure was obtained using the phenolic resin as binder for carbon fibers. After the heat treatment, the phenolic resin is converted to carbon that provides mechanical and electrical connection between the milled carbon fibers.

The porous monolithic composites obtained have a mechanical strength up to 2.37 MPa and a thermal conductivity up to 1.2 W/m·K at room temperature (30°C).

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