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Preparation of Activated Carbon Fibres from Electrospun Polyacrylonitrile Fibre Mat and Characterisation of Their Chemical and Structural Properties

Abstract

Activated carbon fibres with a diameter lower than 1 µm from electrospun polyacrylonitrile (PAN) fibre mat were obtained at developed optimal conditions of stabilisation, carbonisation and chemical activation processes. The fibres obtained were subjected to structural and chemical tests. A nonwoven fabric of activated carbon submicrofibres made from fibres electrospun from PAN/DMSO solution is characterised by very high nitrogen adsorption exceeding 900 cm^3/g within the range of specific micropores, supermicropores and small mesopores, while the total volume of pores exceeds 1.4 cm^3/g and the pore specific surface 2600 m^2/g. The adsorption-desorption isotherm of nitrogen shows easy sorbate access to the inside of pores and its easy removal at the same time.

Key words: *activated carbon fibres, electrospinning, polyacrylonitrile fibres.*

Introduction

The properties of activated carbon depend on the form and properties of the initial raw material or precursor as well as on the conditions of carbonisation and activation, i.e. the type of activator, atmosphere, time, temperature and temperature reaching rate. The properties of activated carbon obtained determine its possible applications. Its excellent sorption properties allow to use activated carbon for adsorption from gaseous or liquid phase, for water and effluent treatment and the protection of the respiratory tract against toxic substances.

A particular form of activated carbon is represented by active carbon fibrous materials with a large specific surface, showing good filtration and sorption properties. The specific surface of conventional activated carbon fibres with a diameter of several µm can be increased if these fibres are made from nanofibres as precursor, i.e. fibres with a diameter smaller than 100 nm, 500 nm [1] or 1000 nm [2].

In literature concerning the production of goods containing activated carbon nanofibres (ACNs), one can find only few items on the preparation of ACNs by electrospinning polymer solution of polyacrylonitrile in dimethylformamide (PAN/DMF) [3 - 7] and polybenzimidasole in dimethylacetamide (PBI/DMAc) [8 - 10].

According to Chronakis [11, 12], controlling the parameters of making precursor nanofibres that affect their properties, one can control the properties of the final carbon nanofibres. Fibre fineness depends on the applied voltage during electrospinning and the viscosity of the spinning solution. The distance between electrodes, the collector shape, the capillary setting angle and potential fibre orientation during the manufacturing process influence the properties of the fibres produced. The carbon content in fibres depends on the heating temperature. In the case of carbon nanofibres made from PAN nanofibres, the degree of graphitisation increases with the carbonisation temperature.

Electrospun PAN nanofibres are used as precursors of carbon fibres as well as activated carbon fibres [13]. Carbonised and activated PAN nanofibres constitute an attractive material, among others, as electrodes for supercapacitors, for catalysis etc. It is the diameter of precursor fibres that affects the conditions of carbonisation and activation processes, the structure and properties of final carbon fibres as well as the size of pores and pore size distribution in a product of activated carbon fibres.

Kim and Yang [14] demonstrated the electro-chemical properties of activated carbon nanofibres based on PAN nanofibres as electrode materials with reinforced capacity and electrical properties of supercapacitors.

Usually PAN-based activated carbon nanofibres are prepared by electrospinning from polymer solution in dimethylformamide, stabilisation, carbonisation and activation by steam [3, 4, 6, 7]. The specific surface of such fibres is about 1403 m^2/g [7].

According to the Textile Research Journal [15], activated carbon nanofibres can be produced by chemical activation of a membrane of electrospun PAN fibres by means of potassium hydroxide at a temperature of 800 °C. In this case the specific surface of such fibres measures about 900 m^2/g.

Patent specification EP 1666649 [16] describes a process of making activated carbon fibres, in the form of nonwoven, with a fibre specific surface ranging from

100 to about 2200 m²/g, and a diameter of 500 nm or smaller. In this process, PAN fibres are impregnated, e.g. with an aqueous solution of KOH with a concentration ranging from 1 to 50%, followed by heating from 550 to 800 °C for 1 s to 1 min.

The aim of this study was to obtain activated carbon fibres (ACFs) with a diameter close to that of nanofibres, i.e. lower than 1 μm and high specific surface, by electrospinning polymer solution of polyacrylonitrile in dimethyl sulfoxide. In accomplishing this objective, the investigation resulted in the development of conditions for making precursor PAN submicrofibres by electrospinning from PAN/DMSO solution, their stabilisation, oxidation, final pyrolysis, and chemical activation. These conditions should make it possible to obtain activated carbon submicrofibres in the form of a nonwoven, showing a high specific surface and very high adsorption of nitrogen within the area of micropores, supermicropores and small mesopores.

■ Experimental

Manufacturing of ACFs
The manufacturing process of activated carbon layer/nonwoven from fibres included the following stages:
1. Manufacturing of a precursor in the form of PAN submicrofibre layer/nonwoven
2. Stabilisation and oxidation
3. Pyrolysis
4. Activation.

Manufacturing of PAN submicrofibre layer/nonwoven
Material
For the production of precursor fibres polyacrylonitrile (PAN) powder including 5% comonomer of acrylic acid and methyl methacrylate (MMA), produced by *Zoltek Rt*, Hungary, was chosen. The intrinsic viscosity of polyacrylonitrile was equal to 1.3 ± 0.02 dl/g. Dimethyl sulfoxide was used as the solvent of PAN as it is a substance used in medicine and does not pose any hazard to man [17]. Pure dimethyl sulfoxide (DMSO) with molecular weight $M_r = 78.13$ g/mol was produced by POCh S.A. Gliwice, Poland.

Preparation of spinning solution
On the basis of our preliminary investigations into carbon nanofibre manufacturing [18, 19] and the necessity of a fur-

ther activation process, the diameter of electrospun fibres was predetermined as about 1 μm. From an earlier study carried out on PAN nanofibre formation [20], 15 wt.% was chosen as appropriate solution concentration for the electrospinning of submicrofibres. In order to obtain a 15% solution of PAN in DMSO, the polymer was immersed in the solvent and left to swell, followed by stirring with a magnetic dipole in a heating aqueous bath at a temperature of 40 °C for about 5 h.

Electrospinning
PAN fibres in the form of nonwoven were spun from spinning solution by a standard (capillary) electrospinning method using a large-size laboratory spinning machine with 32 capillaries and maximal yield of 64 ml/h [20, 21]. In order to obtain fibres with a diameter of about 1 μm from 15 wt.% solution, the technological parameters of the process were as follows: voltage supplied to the system - 15 kV, distance between capillary top and collector - 15 cm, and capillary diameter – 0.9 mm. A flat transporter covered with aluminum foil was used as the receiving element. These conditions ensured a disturbance-free electrospinning process at a temperature of 22 ± 1 °C and relative humidity of 38% under normal atmospheric pressure.

Stabilisation and oxidation
According to the literature [22, 23], as the first stage of the carbon fibre manufacture from polyacrylonitrile, fibres should undergo thermal stabilisation, which prevents the decay of their fibrous form during pyrolysis. The thermal stabilisation of precursor fibres can proceed in an oxygen or neutral atmosphere, but carbon fibres with higher mechanical strength and higher porosity are obtained if the stabilisation is carried out in an oxygen atmosphere, and in suitable conditions.

Appropriate conditions of thermal stabilisation were determined on the basis of our preliminary research results [18, 19] and experiments. From thermogravimetric analysis, microscopic observations of samples, determination of mass loss, area shrinkage, stiffness and preliminary pyrolysis, optimal conditions were chosen.

Precursor fibres in the form of nonwoven with a thickness of 0.27 mm were first stabilised by heating them up to a temperature of 200 °C at a rate of 5 °C/min and thermostated for 6 h, followed by oxidation at 220 °C during a

further 6 h. The whole heating cycle was carried out in a heating chamber with flowing air.

Pyrolysis process
After stabilisation and oxidation, PAN nonwoven was placed in a chamber reactor. After closing and sealing the reactor, air was removed from it to obtain a negative pressure of about 25 mm Hg, followed by filing with carbon dioxide and flushing for 20 min at a rate of 220 l/h. From our experiments came that in the range of 317 – 320 °C the pyrolysis process proceeds slowly, in the range of 320 - 400 °C - intensively with a mass loss velocity of 0.1 %/min, and in the range of 400 - 475 °C - the most rapidly. At higher temperatures the velocity of pyrolysis was still increasing and at 600 °C the mass loss was about 25 wt%. Nonwoven samples after thermal stabilisation were heated up to 600 °C at a rate of 3 °C/min. After reaching that temperature, samples were still heated for 1 h. These conditions ensured the stability and uniformity of the pyrolysis process.

Activation process
The activation process was performed by the chemical method using potassium hydroxide. This method is the most effective way of the activation and development of a porous structure in carbon materials. The final conditions of the activation process were determined on the basis of experiments and investigations carried out earlier [24]. The carbonised nonwoven was immersed in a 20% aqueous solution of KOH placed in a vacuum container and covered with steel wire gauze to keep it under the solution surface. The container was then connected to a vacuum system to degas the nonwoven under a pressure of 3.33 kPa at room temperature for 20 min. Under these conditions the solution boiled slightly. Then the pressure was slowly increased to atmospheric pressure, and under these conditions the sample was soaked for 10 min to completely saturate the fibres with KOH solution. The quantity of KOH incorporated into fibres was corrected by gravimetric draining. The sample was stored under room condition for 5 h to complete the diffusion of KOH into fibre pores and then dried to a constant weight. In dry nonwoven, the carbon:KOH ratio was 1:4.

The nonwoven sample prepared in this way was activated by heating in a ceramic tube reactor (95% Al_2O_3) placed
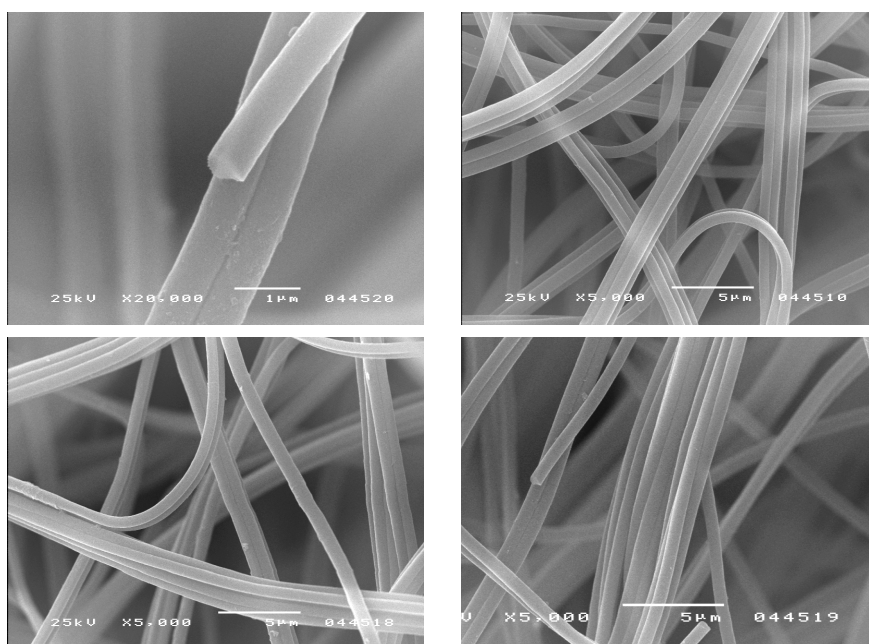
***Figure 1.** SEM images of ACFs obtained from PAN fibres.*

in a resistance furnace at a temperature of 850 °C for 15 min under argon. After activation, the sample was cooled and extracted with a 2% aqueous solution of hydrochloric acid for 5 h and then with deionised water for 5 h in Soxhlet extractors.

Characteristics of ACFs

The diameters of electrospun fibres were determined by means of SEM microphotography (scanning microscope JEOL JSM 5200 LV, *Jeol LTD*., Japan) and Lucia G image analysis software program (*Laboratory Imaging s.r.o*., Czech Republic).

The elementary composition of fibres was examined by CHNS Vario EL III elemental analyser (GmbH, Germany).

An aqueous extract of ACFs was tested for the presence of chlorides, sulfates and starch [25].

***Table 1.** Comparison of diameters of precursor PAN fibres and ACFs.*

Fibre diameter	Precursor PAN fibres	ACFs from PAN fibres
Average value, nm	902.2	784.4
Min value, nm	809.6	683.7
Max value, nm	989.8	894.9
S. d., nm	50.57	66.2

***Table 2.** Elementary composition of fibres after pyrolysis and alkaline activation.*

Elementary composition, %	Fibres	
	After pyrolysis	After activation
Moisture	9.20	10.20
Nitrogen	13.96	1.22
Carbon	77.05	80.85
Hydrogen	1.65	1.18
Sulfur	0.00	0.00
Oxygen (from difference)	7.34	16.78
	100.00	100.00

***Table 3.** Chemical compounds in the aqueous extract of precursor PAN fibres and ACFs; pH determined for H_2O 5.51.*

Sample	pH of aqueous extract	Chlorides	Sulfates	Starch
Precursor PAN fibres	6.82	absence	absence	absence
ACFs	7.74			

The chemical constitution of ACFs was examined by means of a monobeam FTIR-8101M spectriphotometer (SHIMADZU, Japan), equipped with a DR-8001 control unit, using the translucent method (tablet technique), reflection technique (ATR) and scattered radiation technique (DRIFFT). Comparative measurements involved precursor PAN fibres and ACFs.

To compare the crystallographic structures of precursor PAN fibres and ACFs, examinations were carried out by the method of wide-angle X-ray scattering by means of an X'Pert PRO diffractometer from Panalytical using CuK radiation and the following diffractometer parameters: accelerating voltage – 40 kV and anode current intensity – 30 mA. An X'Celerator semiconductor counter was used as a detector. The processing of diffractograms was performed by means of the 'WAXSFIT' program [26].

The porous fibre structure was investigated using nitrogen adsorption-desorption isotherms at 77 K, determined in a sorptometer ASAP 2010, manufactured by Micromeritics Instrument Corp. (USA). Prior to isotherm measurements, samples were outgassed at 300 °C for 10 h to a constant vacuum (10^{-4} kPa). The specific surface area was calculated by the BET method (SBET); the total pore volumes (V_{tot}) were determined from the last point of the isotherm at a relative pressure of 0.99 using the BJH model, and the pore volumes and pore size distributions were derived from the adsorption branches of the isotherms (V_{mes}). The micropore volume (V_{mic}) was calculated by the t-plot method.

To obtain a curve representing the distribution of meso- and macropores, measurements were carried out by means of AUTOPORE IV- 9520 apparatus (Micromeritics, USA) by the Hg porosimetry technique.

■ Results and discussion

Characteristics of ACFs

Thickness

The average diameter of ACFs is decreased by about 118 nm in relation to that of precursor PAN fibres and amounts to about 784 nm, which is connected with material shrinkage caused by stabilisation and oxidation. The diameters of precursor fibres and ACFs are given in
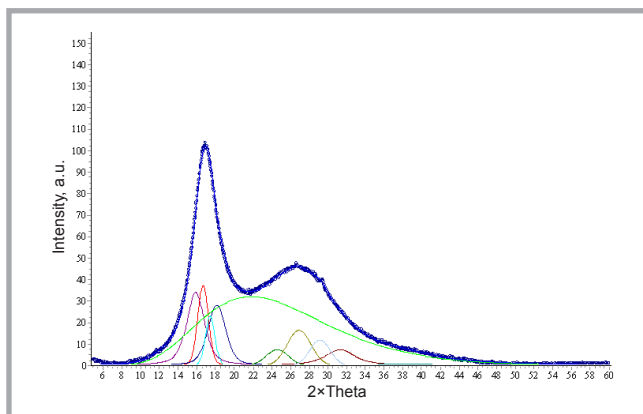
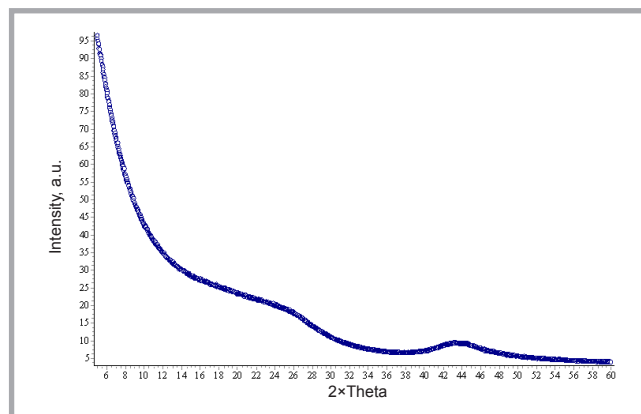*Figure 2. Diffraction pattern of PAN fibres.*

Figure 3. Diffraction pattern of ACFs.

Table 1. Both precursor fibres and ACFs can be called submicrofibres. ***Figure 1*** shows microscope images of ACFs, in which it is seen that fibres are connected with each other in several units. The thickness of precursor nonwoven determined according to Standard PN-EN ISO 9073-2:2002 [27] was 0.27 mm, while after carbonisation and activation, it was increased to 0.5 mm.

Elementary composition

The results of the elementary composition of fibres after pyrolysis and activation are listed in ***Table 2***. The percentage weight content of particular elements in fibres concerns a dry, ash-free sample.

The carbon content in fibres after pyrolysis is typical of PAN carbonisate and amounts to 77.05%. Their considerable nitrogen content, i.e. 13.96% originates from cyclized CN groups (ladder structure) formed during the oxidation and pyrolysis of imine groups (N-H), amine groups (-NH$_2$), hydrogen cyanide and ammonia. The oxygen content, amounting to 7.34%, originates from the oxidation of polymer with air and the formation of carbonyl and hydroxyl groups.

As a result of further fibre treatment at a temperature of 850 °C and oxidation with alkalis during activation, the nitrogen content was radically decreased to 1.22% due to the formation of graffene structures consisting mainly of condensed carbon rings, while nitrogen was emitted in the form of ammonia and hydrogen cyanide. On the other hand, the oxygen content is considerably increased, i.e. up to 16.78%, which is a feature of the activation method. During the action of KOH (precisely K$_2$O after KOH decomposition), considerable amounts of oxygen are incorporated into the structure of the carbon matrix in the form of carboxyl, carbonyl and hydroxyl groups.

Aqueous extract

Chemical compounds in aqueous extracts of both precursor PAN fibres and ACFs were determined for comparative purposes (***Table 3***).

The aqueous extract of ACFs is more alkaline than that of precursor fibres and contains no chlorides, sulfates and starch.

Chemical structure

The spectrum of fibres electrospun from PAN/DMSO solution was compared with that of powdered PAN used for the preparation of this solution. No new absorption bands indicating DMSO residues were observed.

The DRIFFT spectrum of fibres after carbonisation and activation shows the presence of CH and CH$_2$ groups (the presence of inorganic nitrates is also possible) – band ~1380 cm^{-1} – and OH groups (band ~3400 cm^{-1}).

The ATR spectrum of fibres after carbonisation and activation shows CN groups (bands ~1640, 2150 cm^{-1}) and CH and CH$_2$ groups (band ~2850 - 2950 cm^{-1}).

Crystallographic structure

The crystallinity degree of PAN fibres determined on the basis of the diagram shown in ***Figure 2*** was 42.55%. After carbonisation and activation, fibres became amorphous, as shown in ***Figure 3***.

Structure

The nitrogen isotherms determined (***Figure 4***) indicate a very high sorption (over 900 cm^3/g) within the area of micropores, supermicropores and small mesopores. Such a high value of nitrogen sorption for ACFs allows to include the porous system among the mixed structural type despite the fact that the adsorption in large mesopores is not high. The adsorption isotherm practically shows no hysteresis loop. In the case of considerable amounts of mesopores, this indicates very good absorbate access to the inside of pores, suggesting the occurrence of first or second order pores. These pores are relatively short with no narrowings that would retard gas sorption and desorption, which largely results from the small diameter of fibres. Such a structure facilitates a high adsorption rate, especially in the case of low sorbate concentrations and regeneration, which is of paramount importance for maintaining the stability of sorbent performance.

ACFs contain a particularly great number of supermicropores (the largest ones) for p/p$_o$ 0.1 < 0.42. The total volume of pores exceeds 1.4 cm^3/g and their specific surface amounts to 2630 m^2/g, which is a better result than the highest one, 2200 m^2/g, cited in literature [16]. It is mainly formed by supermicropores and small mesopores. The results obtained are listed in ***Table 4*** and illustrated in ***Figures 5*** and ***6*** (see page 46).

Within the area of micropores, ACFs possess mainly pores with a size below 1.25 nm. The shape of the differential curve of the mesopore volume (***Figure 5***) indicates the existence of considerable amounts of smaller pores than 1.1 nm. These are largely supermicropores (considerable sorption within the area: p/p$_o$ 0.1 – 0.4, shown in the adsorption isotherm).

Within the area of large mesopores (***Figure 6***), one can observe a considerable number of pores with a size of about

Table 4. *Parameters of the porous structure of ACFs; 1) Single-point for $p/p_o = 0.97$ (< 77 nm), 2) t-plot, 3) BJH method 17 – 300 nm.*

Volume of pores, cm³/g			V_{mic}/V_{mes}	Specific surface of pores, m²/g	Size of pores, nm	
V_{total}[1]	V_{mic}[2]	V_{mes}[3]		S_{BET}	d_{av}	d_{mes}
1.409	0.530	0.879	0.603	2630	2.15	2.57
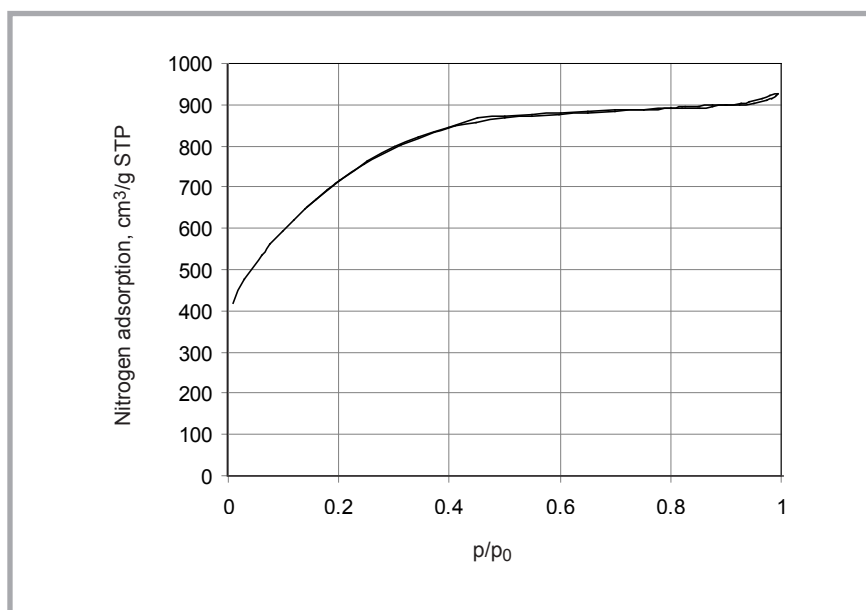
Figure 4. *Nitrogen adsorption isotherms for ACFs.*

50 nm (total curve of pore volume distribution). Taking into account the diameters of submicrofibres, these are the largest possible pores occurring inside fibres. The considerable increase in the pore volume within the range of 85 – 130 nm is a measure of nitrogen adsorption in the inter-fibre spaces, in which capillary phenomena occur mainly between the crossing of single fibres. These observations confirm the test results of Hg porosimetry, **Figure 7**.

The size and number of these pores is of importance in filtration processes, where molecules are arrested in inter-fibre spaces.

■ **Conclusions**

To obtain ACFs with a diameter lower than 1 µm from PAN submicrofibres electrospun in the form of nonwoven from PAN/DMSO solution, it is required to use appropriate process parameters of electrospinning, and then stabilisation, oxidation, pyrolysis and activation. To impart submicrofibres a high pore specific surface and high nitrogen adsorption, the precursor fibres should be heated to a temperature of 200 °C for 6 h at a heating rate of 5 °C/min, and then to 220 °C and thermostated for a further 6 h. The temperature of pyrolysis should be 600 °C. The activation process should be performed by the chemical method using potassium hydroxide as an activator at a temperature of 850 °C for 15 min, with a carbon : KOH ratio of 1 : 4.

The diameter of fibres after pyrolysis and activation is decreased. Moreover the nitrogen content is decreased and oxygen content increased in ACFs showing the presence of CH, CH_2 and CN groups and no residue of DMSO. The aqueous extract of ACFs is more basic than that of precursor fibres with no chloride, sulfate and starch. PAN submicrofibres have a crystalline structure (crystallinity degree amounts to about 42.55%), while after pyrolysis and activation they become amorphous.

The total volume of pores in ACFs made from PAN submicrofibres with a diameter of 900 nm exceeds 1.4 cm³/g, and the specific surface of ACFs amounts to 2630 m²/g, which is a better result than the highest value reported in literature (2200 m²/g) [16]. The pores in ACFs comprise micropores and supermicropore as well as small mesopores. The nitrogen adsorption is high, exceeding 900 cm³/g. Based on the adsorption and desorption isotherm, one can conclude that sorbates
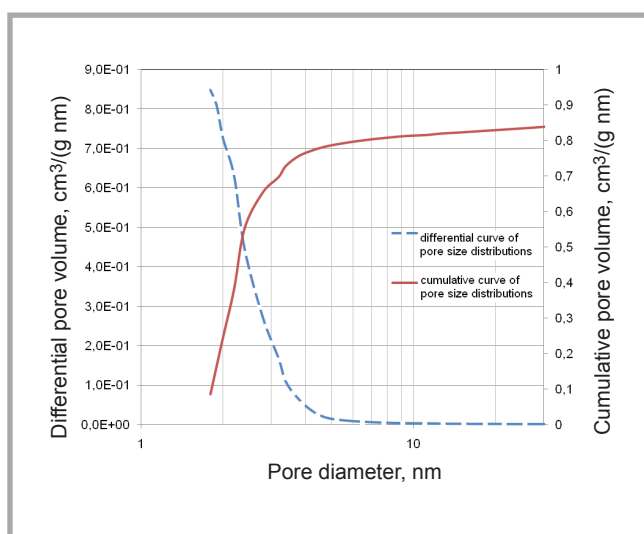

Figure 5. *Differential and cumulative curves of mesopore volume distribution by size in ACFs: pore diameter < 11 nm.*
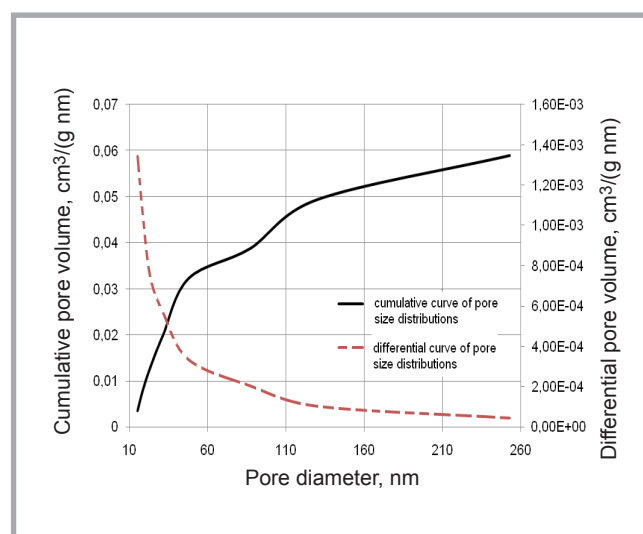

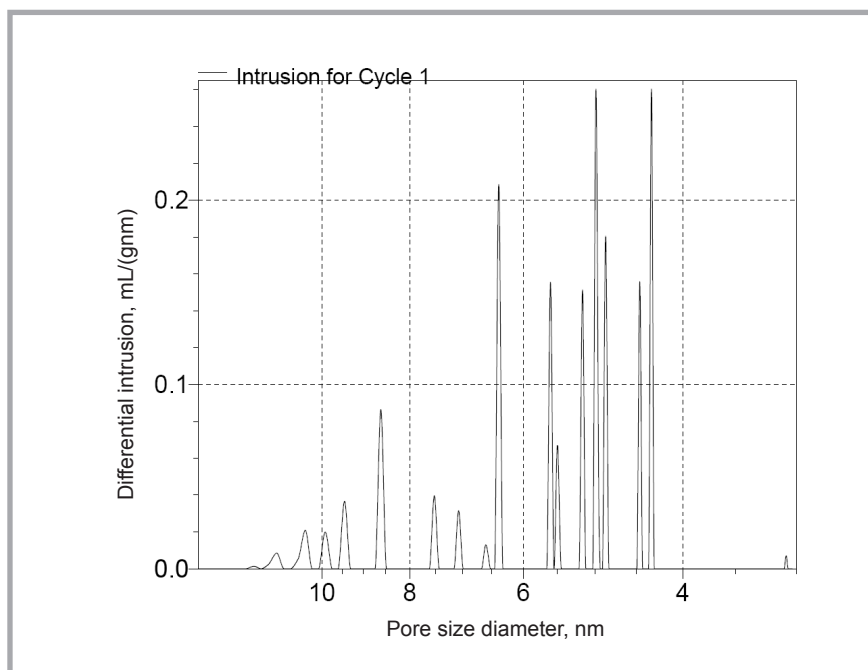**Figure 6.** *Differential and cumulative curves of mesopore volume distribution by size in ACFs: pore diameter >11 nm.*

Figure 7. Pore size distribution recorded by the technique of mercury porosimetry.

should have very good access to the inside of pores.

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