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Solvent Vapour-Sensitive Activated Carbon Submicrofibres Based on Electrospun Polyacrylonitrile Fibre Mat

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Abstract
Precursor polyacrylonitrile submicrofibres with a diameter of about 900 nm, electrospun from polyacrylonitrile/dimethyl sulfoxide (PAN/DMSO) solution, were carbonised and chemically activated. These submicrofibres, characterised by a porous structure more developed than in the case of standard fibres, were investigated as solvent vapour sensors. Sensitivity to vapours of four different fluids: methanol, acetone, benzene & toluene at a low concentration of 200 ppm, was studied directly for fibres and for ones in a prototype textile multilayer system. In experiments the electrical conductivity of carbon fibres was utilised. The response time and sensory coefficient, indicating quantitative changes in electric resistance due to contact with the solvent vapours, were estimated. The activated carbon submicrofibres are characterised by sensitivity, a very short response time not longer than 20 s, stability, and selectivity in relation to vapours of polar and non-polar solvents. The sensitivity to vapours of polar solvents is higher than that to those of non-polar solvents.

Key words: *electrospinning, polyacrylonitrile, activated carbon submicrofibres, vapours, sensitivity.*

Introduction

Materials engineering based on nanotechnology is gaining greater recognition because of the wide opportunities for innovation and competitiveness of products. The use of nanofibres in areas such as medicine or life and health protection is increasingly being used. Nanofibres may have unique properties arising either from the same material, their morphology, or as a consequence of their modification through appropriate agents or processes such as the fluorination treatment of porous nanofibres to increase the capacity of methane storage [1]. Particular application may have nanofibres which can be converted to carbon nanofibres. As precursors of carbon nanofibres, electrospun polyacrylonitrile [2, 3], polybenzimidasole [4], polyvinyl alcohol [5] and polyimide [6] nanofibres are used.

Electrospun polyacrylonitrile (PAN) nanofibres can be used as a precursor of carbon fibres, and even activated carbon fibres [2]. According to Chronakis [4],

the properties of the final carbon nanofibres depend on the properties of precursor nanofibres which result from technological parameters of the electrospinning process. The main parameter of nanofibres, fineness, depends on the voltage applied during the electrospinnig process and on the viscosity of the spinning liquid. Other factors, such as the distance between electrodes, the collector shape, the capillary setting angle and potential fibre orientation during the manufacturing process influence the different properties of electrospun fibres. The porosity of carbon fibres increases through the activation process.

The specific surface of PAN-based activated carbon nanofibres prepared usually by electrospinning from polymer solution in dimethylformamide, stabilisation, carbonisation and activation by steam is about 1403 m2/g [2, 3]. The activation process of electrospun PAN fibres can be carried out by means of potassium hydroxide. The specific surface of such fibres measures about 900 m2/g [7]. PAN fibres 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, are characterised by a specific surface ranging from 100 to about 2200 m2/g, and a diameter of 500 nm or smaller [8].

Such nanofibres that have undergone heat treatment i.e. pyrolysis and activation, are characterised by very high porosity. It has been shown that because of

the extremely extensive surface, they are an excellent adsorbent of toluene [3, 9] benzene [9] and formaldehyde [2].

In the literature concerning functional nanosensors, electrospun nanomaterials, usually in form of nonwoven, are divided into acoustic wave sensors, resistive sensors, photoelectric sensors, optical sensors, and amperometric sensors [10]. Fewer references concern the potential use of carbon nanofibres for making gas or solvent vapour sensors [11]. Considerably more pieces of information concern carbon nanotubes (CNTs) as active materials used in gas or solvent vapour sensors [12 - 15]. Single-wall carbon nanotubes (SWCNTs) are sensitive to NO_2, NH_3 and volatile organic compounds, while multi-wall carbon nanotubes (MWCNTs) show sensitivity to various gases such as NH_3, NO, NO_2, H_2, SF_6, Cl_2 and vapours of organic solvents such as benzene, acetone, toluene and methanol. However, both types of carbon nanotubes as sensor materials show a drawback consisting in long lasting and incomplete regeneration. Moreover working with carbon nanotubes requires extraordinary care as, by reason of insufficient knowledge on the consequences of being in contact with such materials, there is a serious danger for man's health and the environment. A significant issue is also their very high price.

The use of activated carbon nanofibres for making sensors can constitute an

alternative to sensors based on carbon nanotubes.

The use of activated carbon nanofibres as micro sensors for the detection of volatile organic compounds in the air in very small quantities is an essential solution e.g. in the detection of certain diseases in a patient. The detection of volatile organic compounds in breath may signal the beginning of diseases such as diabetes, increased acetone exhalation in the case of lung cancer, and increased ammonia exhalation in the case of kidney damage.. The use of such sensors would create a non-invasive, convenient, patient-friendly method of assessing breath. There are known sensors based on nanotubes composed of tin oxide coated inside with a catalytic coating of uniform nanoparticles of platinum.

On the other hand, active carbon nanofibres could be used to build micro-sensors indicating external gaseous danger. The main requirements for the sensors are those related to vapour sensitive properties, such as sensitivity, selectivity and response time, as well as those related to reliability, such as repeatability and stability.

In previous work, the preparation and characterisation of the structure of activated carbon submicrofibres from electrospun polyacrylonitrile fibre mat were presented [16].

The main aim of this work was to initially evaluate the sensitivity of the activated carbon submicrofibres obtained to noxious vapours. These preliminary investigations were carried out for activated carbon submicrofibres used only as an identifier of the presence of vapours in direct contact with them and in contact with them through the textiles. A prototype sensor structure consisting of an activated carbon submicrofibre layer placed in the middle of the fibrous system was designed. This textile structure could be used in filters and half-masks for individual protection of the human respiratory system in response to the presence of dangerous quantities of toxic vapours in the air.

■ Materials

Activated carbon fibres with a diameter smaller than 1 μm, called submicrofibres, were obtained as a result of three processes: stabilization and oxidation, pyrolysis and activation of electrospun polyacrylonitrile fibre mat. A description of the manufacturing process and precise characteristics of the activated carbon submicrofibres studied are presented in a previous work [16]. In this study it is presented briefly.

Polyacrylobnitrile submicrofibres [16]

A spinning liquid was prepared for the electrospinning process: a solution of polyacrylonitrile (PAN) powder, including 5% of comonomers of acrylic acid and methyl methacrylate (MMA), produced by Zoltek Rt, Hungary, in dimethyl sulfoxide (DMSO) produced by POCh S.A. Gliwice, Poland,. Dimethyl sulfoxide is a substance used in medicine and does not pose any hazard to man [17].

As came from our earlier investigations into activated carbon submicrofibre manufacturing, the diameter of electrospun fibres should be at the level of about 1 mm [18], and to obtain fibres with such a diameter, a 15% solution of PAN in DMSO can be used [19].

The electrospinning process was carried out on a large-size laboratory spinning machine with 32 capillaries at a temperature of 22 ± 1 °C and relative humidity of 38% under normal atmospheric pressure [19, 20]. Technological parameters of the process were as follows: voltage supplied to the system - 15 kV, the distance between the capillary top and collector - 15 cm, and the capillary diameter - 0.9 mm. As the receiving element, a flat transporter covered with aluminum foil was used. Polyacrylonitrile submicrofibres with a diameter of about 900 nm were obtained.

Activated carbon submicrofibres [16]

In the first stage, precursor PAN submicrofibres in the form of fibre mat with a thickness of 0.27 mm were stabilised at 200 °C for 6 h and oxidated at 220 °C for a further 6 h. These processes were carried out in a heating chamber with flowing air.

In a next stage, the fibre mat was put into a chamber reactor, from which air was removed by carbon dioxide. In this chamber reactor pyrolysis was carried out at a final temperature of 600 °C for 1 h, and carbon material was obtained.

The last stage, the activation process, was performed by the chemical method using potassium hydroxide (KOH). Chemical activation is the most effective way of activating and developing the porous structure in carbon materials.

The carbon fibre mat was immersed in a 20% aqueous solution of KOH placed in a vacuum container connected to a vacuum system to degas the sample. When the solution was slightly boiling, 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 sample was stored under room conditions for 5 h and then dried to constant weight. In a dry sample, the carbon: KOH ratio was 1:4.

Then the sample was activated by heating it in a ceramic tube reactor (95% Al_2O_3) at a temperature of 850 °C for 15 min under argon. After activation, the sample was cooled and extracted at the beginning with a 2% aqueous solution of hydrochloric acid for 5 h and then with deionized water for 5 h in Soxhlet extractors. Activated carbon submicrofibres in the form of nonwoven were obtained [16].

The average diameter of these submicrofibres 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 microscope images in *Figure 1* show that activated carbon submicrofibres are connected with each other in several units. The thickness of the precursor nonwoven was 0.27 mm, while after the above processes, it was increased to 0.5 mm.

The total volume of pores in these activated carbon submicrofibres exceeds 1.4 cm^3/g, and the specific surface amounts to 2630 m^2/g, which is a better result than the highest value reported in literature (2200 m^2/g) [8]. The pores comprise micropores and supermicropores as well as small mesopores. Nitrogen adsorption is high, exceeding 900 cm^3/g. Based on the adsorption and desorption isotherm, one can conclude that sorbates should have very good access to the inside of pores.

The activated carbon submicrofibres produced from the electrospun polyacrylonitrile fibre mat were characterised by a
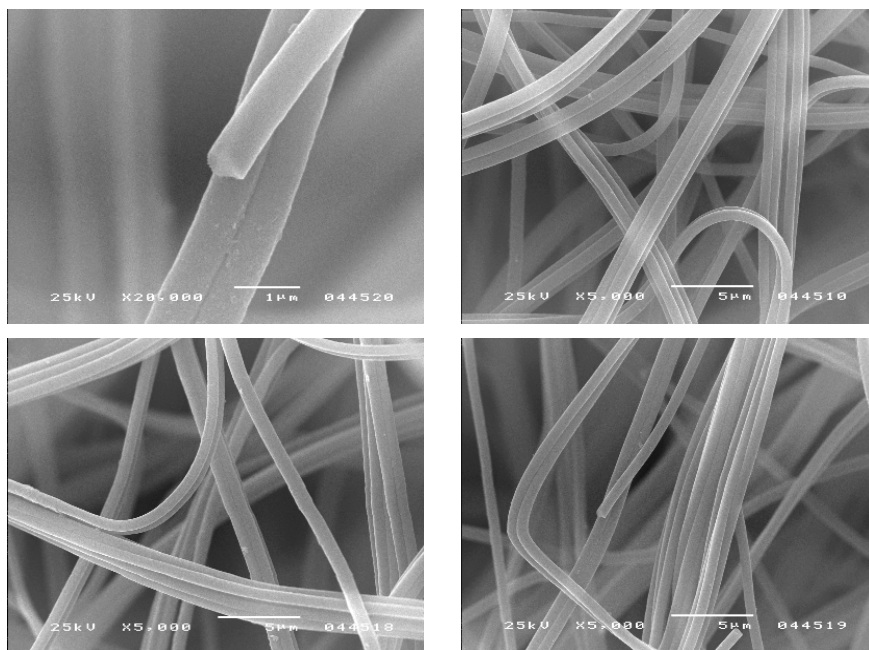
Figure 1. *SEM images of activated carbon submicrofibres obtained from PAN submicrofibres.*

porous structure more developed than in the case of standard fibres, with the total volume of pores being 1.409 cm^3/g, the V_{mic}/V_{mes} ratio - 0.603, and the average size of pores - 2.15 nm. Such a developed porous structure and nanometer dimensions of pores give favourable conditions for very fast penetration of the sorbat, used in small concentration, to the inside of pores.

Methods

The thickness of nonwovens was determined according to Standard PN-EN ISO 9073-2:2002.

The sensitivity of activated carbon submicrofibres in the form of nonwoven to vapours of chemical fluids – solvents was studied. For evaluation of vapour sensitivity, the electrical conductivity of the carbon fibres was utilized [21]. The laboratory stand used by the authors, constructed by the Department of Material and Commodity Science and Textile Metrology of Lodz University of Tech-
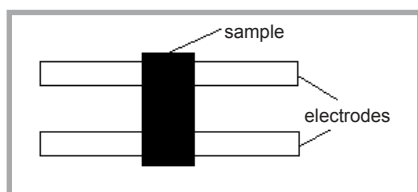

Figure 2. *Scheme of lay-out of two electrodes and sample.*

nology, Poland, is dedicated for measurements carried out in small concentrations of noxious vapours. It consists of two chambers made of glass. In the main, bigger chamber, fluids dropped into the small glass cell evaporate, making vapours. In the second, smaller chamber two measuring rectangular electrodes of 4 × 0.5 cm arranged in parallel at a 1 cm distance relative to each other are situated. Across these electrodes, the sample examined is placed under a 10 g load, *Figure 2*.

The measuring electrodes are connected to a Keithley multimeter (USA) coupled with a computer. During the experiment, vapours are forced by a pump from the gaseous chamber to the measuring chamber [22]. The surface resistance of the sample was measured according to Standard EN 1149-1:2006 "Protective clothing - Electrostatic properties - Part 1: Test method for measurement of surface resistivity". The measurements were carried out at a voltage of 10.7 V. In order to check the selectivity of the activated carbon submicrofibres, four solvents (methanol, acetone, benzene, toluene) with different physical and chemical properties were chosen according to Standard ISO 6529:2013 – "Protective clothing – Protection against chemicals – Determination of resistance of protective clothing materials to permeation by liquids and gases", *Table 1*. These solvents represent two categories of solvents, i.e. polar and non-polar. Moreover polar sol-

vents can be protic such as methanol, or aprotic such as acetone. Non-polar solvents are only aprotic, and two solvents in this category, i.e. benzene and toluene, were chosen comparatively.

Kinetics of the resistance changes of the activated carbon submicrofibres due to vapour sorption were registered and analysed.

A liquid substance was dropped into a small glass cell situated inside a gaseous chamber with air and then evaporated during 3 h at 80 °C. In order to check the sensitivity, the vapour concentration was at a level of 200 ppm, as the lowest value causes a toxic effect [15]. The toxic influence on the human body depends on the solvent vapours and their concentration. For methanol the minimum hazardous concentration is 300 ppm, for acetone and benzene 500 ppm, and for toluene it is 200 ppm.

The sample was placed in the measuring chamber with air directly on the two metal electrodes distant from each other by 1 cm. Registration of the material resistance was then started. After a time of about 30 s vapours were forced into this measuring chamber for about 400 s. On the graph of the resistance-time dependence, the response time and stability of the materials studied were observed. For each kind of vapour five tests were carried out.

To express the quantitative changes in the material's electric resistance due to contact with vapours of selected organic fluids, the sensority coefficient was calculated from the following formula [22]:

$$S_s = \Delta R/R_o \times 100\%$$

where, S_s – sensority coefficient,
ΔR – absolute change in electric resistance of the material, $\Delta R = R – R_o$,
R – resistance of the material under the influence of external stimulus,
R_o – resistance of the material before the action of the stimulus.

Investigations of sensitivity were carried out for several samples of activated carbon submicrofibres in direct contact with vapours and in contact with them through textiles, i.e. for the prototype sensor structure. For each kind of vapour the characteristic electric resistance – time dependence was analysed, the results of which are presented for representative samples.

A textile prototype sensor structure consisting of an activated carbon submicrofibre layer placed in the middle of the fibrous system was designed. An activated carbon submicrofibre layer of 1 × 2.5 cm area was situated inside the system between two polypropylene meltblown nonwoven layers of 2.0 × 3.5 cm area. The mass per unit area of the activated carbon submicrofibre layer was equal to 7 g/m². The mass per unit area of the meltblown nonwoven layer was typical for that nonwoven used in the half-mask and was equal to 50 g/m². In the bottom layer of meltblown nonwoven, two rectangular holes were cut to make the submicrofibres investigated come into contact with the two electrodes, *Figure 3*. In all cases the samples of activated carbon submicrofibres were cut from one nonwoven layer.

■ Results and discussion

The sensitivity to vapours of the four different solvents: acetone, benzene, methanol and toluene, at a low concentration of 200 ppm, was studied directly for fibres and for ones in a prototype textile multilayer system, *Figures 4 - 11* (see page 100).

When samples of activated carbon submicrofibres in the form of nonwoven were exposed to the vapours, their electrical resistance changed rapidly, *Figures 4, 6, 8 & 10*, confirming the changes proceeding in the material as a result of vapour action. For all kinds of vapours the immediate response was observed. It means that the sorption of vapour particles proceed very quickly, because from the moment when the vapours were released, changes in the electrical resistance were observed in a time not longer than 20 s. After this time up to 500 s of the experiment, no meaningful changes were observed, which suggests saturation by the vapours. Such a fast reaction of fibres is probably connected with their developed porous structure. The sorption of vapours can proceed deep in the fibre structure. The high values of the total volume of pores and specific surface favour better sorption.

Similar situations were observed for activated carbon submicrofibres as a layer in the textile prototype structure, *Figures 5, 7, 9 & 11*. The response time is very short, lower than 20 s, and then the signal

Table 1. *Characteristics of solvents, [23].*

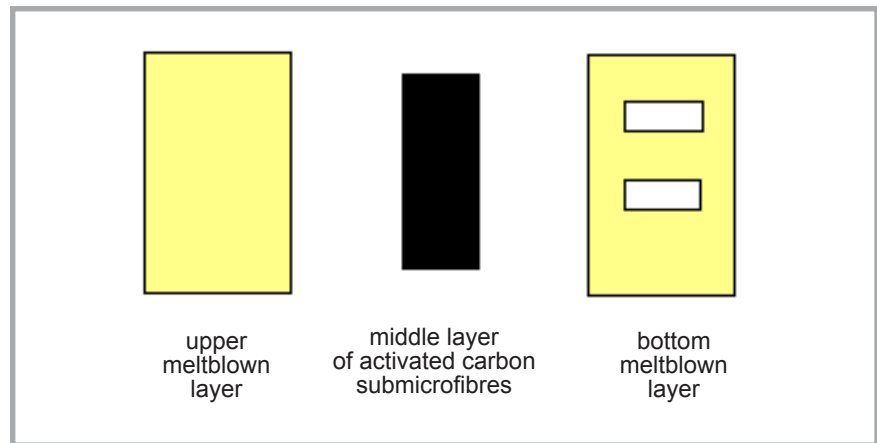| Solvent		Boiling point, ºC	Dielectric constant	Density, g/cm³	Chemical properties
Polar	methanol	65	33	0.791	protic
	acetone	56	21	0.786	
Non-polar	benzene	80	2.3	0.879	aprotic
	toluene	111	2.4	0.867	

Figure 3. Scheme of layers in prototype structure.

of electrical resistance is nearly constant, without distinct extremes.

For other textile materials, which are solvent vapour sensitive the reaction time presented in literature [14, 15] is longer, and depends on the material structure. For 98% poly(lactic acid) nonwoven modified with 2% carbon nanotubes the reaction time is in the range of 30 - 60 s, if the fibre diameter is about 50 μm and the apparent density of the nonwoven is about 120 - 150 kg/m³, and more than 60 s if the fibre diameter is about 20 μm and the apparent density of the nonwoven is about 120 kg/m³. If the textile material is covered (printed) by a paste with 0.5 - 1.5 wt% of carbon nanotubes, its reaction time is long, e.g. near 100 s for benzene and ethanol vapours, for example.

To compare the sensitivity of activated carbon submicrofibres in direct contact with vapours and activated carbon submicrofibres as a layer in the prototype structure to vapours, the sensority coefficient was calculated, *Table 2*. R_o was calculated as the mean value of resistance in the air environment. As the R parameter, the value of resistance measured at the time of response to vapours was chosen.

The values of sensority coefficient indicate that in the case of activated carbon submicrofibres being in direct contact

with vapours, the sensitivity to vapours of all solvents are higher than in the case of activated carbon submicrofibres being in the middle of the textile prototype structure. The difference in the sensority coefficient is similar for all solvents, i.e. a value of 5% for methanol, acetone and benzene, and a value of 6% for toluene. The melt-blown nonwoven barrier leads to a decrease in values of the sensority coefficient.

The highest values of sensority coefficient were evaluated in the case of vapours of polar aprotic solvent, i.e. acetone vapours. For activated carbon submicrofibres in direct contact with vapours, the mean value of the sensority coefficient is at a level of 19%.

Very similar sensitivity, lower by 1%, was presented in the case of vapours of polar protic solvent, i.e. methanol vapours.

Table 2. *Sensority coefficient for activated carbon fibres and activated carbon fibres as a layer in the textile prototype structure.*

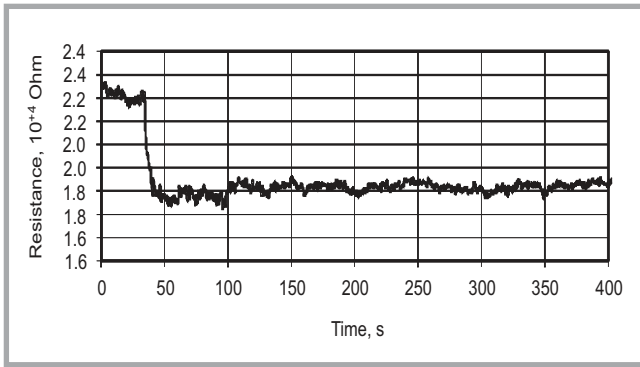| Vapours	Sensority coefficient S_s, %	
	Fibres	Prototype structure
Methanol	18	13
Acetone	19	14
Benzene	13	8
Toluene	14	8

Figure 4. *Electric resistance – time dependence for activated carbon submicrofibres in contact with methanol vapours.*
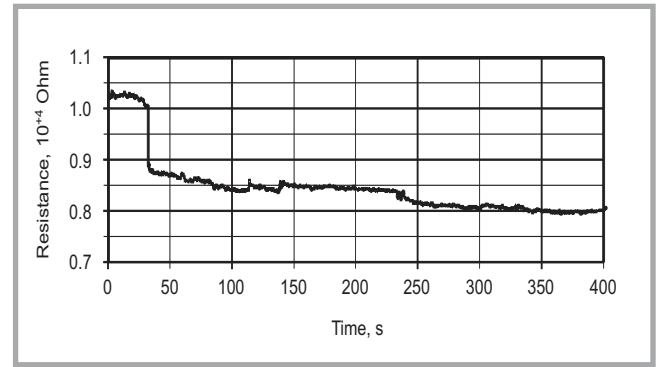

Figure 5. *Electric resistance – time dependence for activated carbon submicrofibres as a layer in the prototype structure in contact with methanol vapours.*
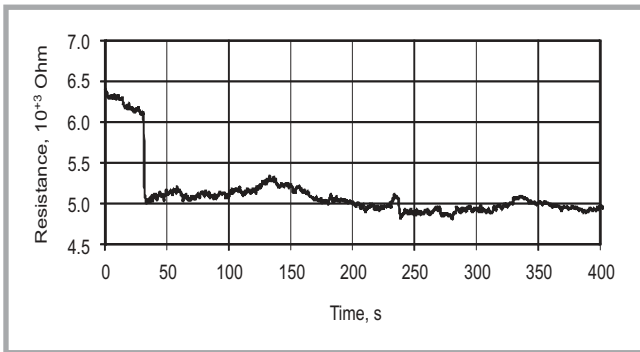

Figure 6. *Electric resistance – time dependence for activated carbon submicrofibres in contact with acetone vapours.*
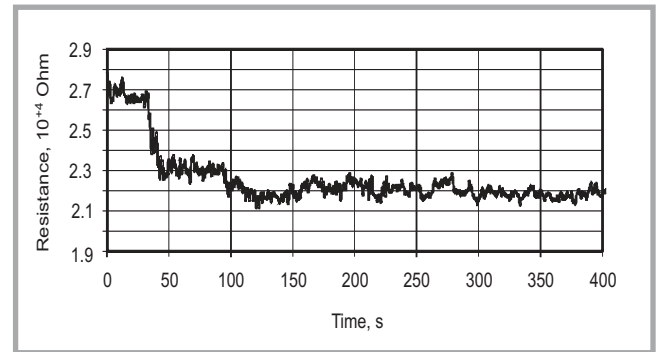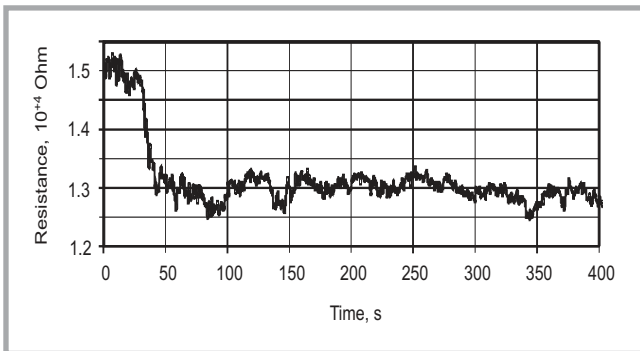

Figure 7. *Electric resistance – time dependence for activated carbon submicrofibres as a layer in the prototype structure in contact with acetone vapours.*

Figure 8. *Electric resistance – time dependence for activated carbon submicrofibres in contact with benzene vapours.*
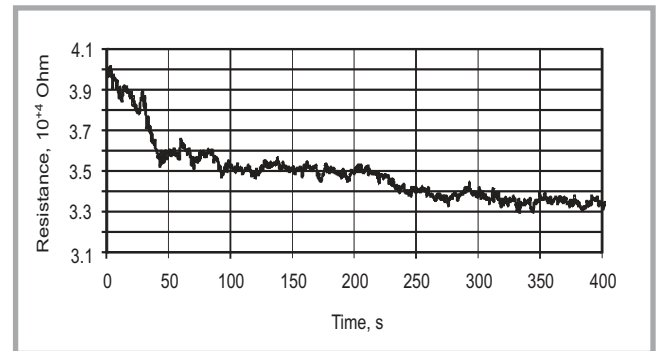

Figure 9. *Electric resistance – time dependence for activated carbon submicrofibres as a layer in the prototype structure in contact with benzene vapours.*
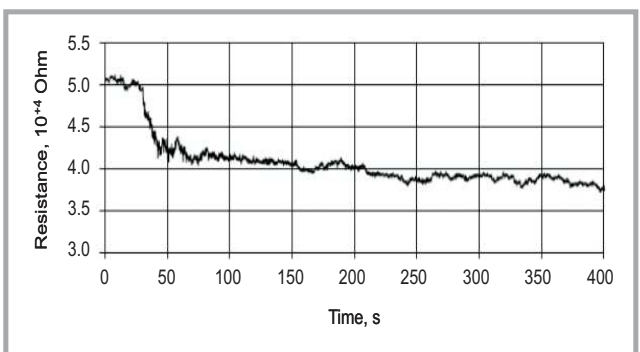

Figure 10. *Electric resistance – time dependence for activated carbon submicrofibres in contact with toluene vapours.*
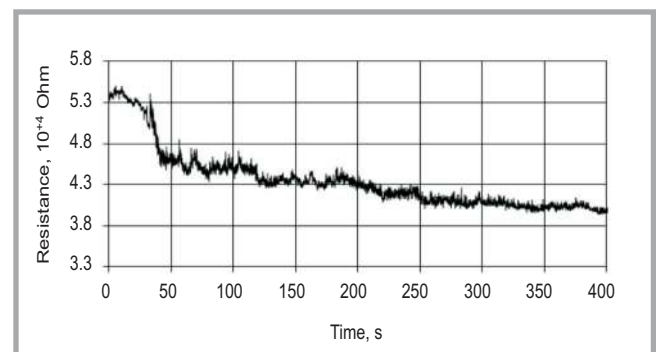

Figure 11. *Electric resistance – time dependence for activated carbon submicrofibres as a layer in prototype structure in contact with toluene vapours.*
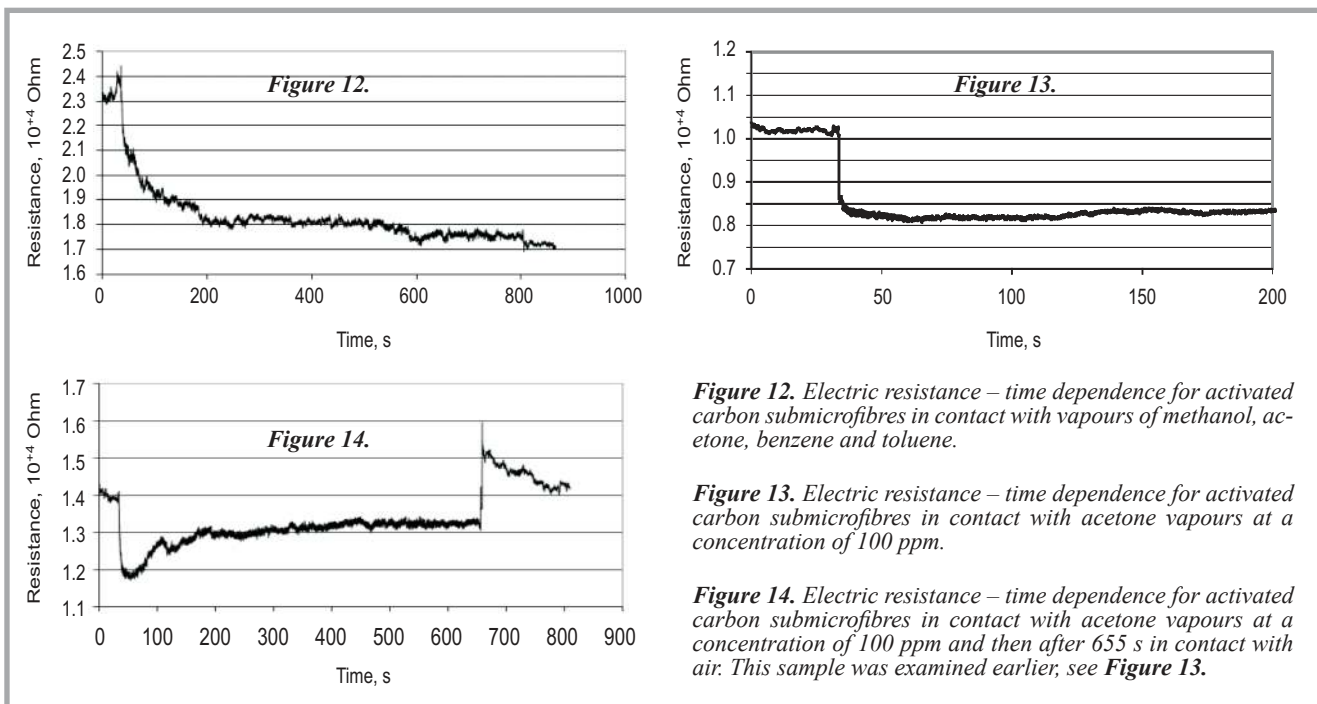
Figure 12. *Electric resistance – time dependence for activated carbon submicrofibres in contact with vapours of methanol, acetone, benzene and toluene.*

Figure 13. *Electric resistance – time dependence for activated carbon submicrofibres in contact with acetone vapours at a concentration of 100 ppm.*

Figure 14. *Electric resistance – time dependence for activated carbon submicrofibres in contact with acetone vapours at a concentration of 100 ppm and then after 655 s in contact with air. This sample was examined earlier, see **Figure 13**.*

The sensority coefficient is the lowest for toluene and benzene vapours. For activated carbon submicrofibres in direct contact with toluene vapours, the value of the sensory coefficient is at a level of 14%, and with benzene vapours - 13%. Both solvents give vapours acting on the activated carbon submicrofibres with a similar effect, suggesting that vapours of non-polar aprotic solvents, which are characterised by a very low dielectric constant, act on the activated carbon submicrofibres studied less than vapours of polar solvents with a high dielectric constant. The difference in the sensority coefficient is at a level of 4 - 5%.

On the basis of these four different solvents, we conclude about selectivity concerning vapours of polar and non-polar solvents.

In comparison with other textile materials, which are solvent vapour sensitive but containing carbon nanotubes [14, 15], the activated carbon submicrofibres sensitive materials developed are characterized by shorter response time, lower sensority coefficient and a sensitivity of different character. In all cases, i.e. for the materials studied, as well as for those presented in literature, the sensory properties are better for polar vapours.

The reaction of activated carbon submicrofibres being in direct contact with vapours of all four solvents used together in total concentration of 400 ppm, is longer and with different character from that presented above. In a first step the reaction is immediate, during 10 s the resistance of submicrofibres decreases by about 10%. Then the drop of resistance is slower and during 150 s decreases by about 13%. After this long time, near 200 s, the resistance is constant what testifies to the saturation, *Figure 12*.

Such a long response time probably results from the diversity of vapours particles in the mixture.

The concentration of vapours is less important. The results obtained for acetone vapours showed that irrespective of the concentration of vapours the reaction of activated carbon submicrofibres is similar, i.e. for concentrations of 100, 200 and 1000 ppm values of the sensory coefficient are about 16, 19 & 19, respectively, and the response time in all cases is lower than 20 s.

The activated carbon submicrofibres studied can be used as a solvent vapour-sensitive material more than once. The sample after the experiment with acetone vapours at a concentration of 100 ppm, *Figure 13*, and then exposed to air for a few minutes showed again sensitivity to vapours. The saturation of fibres used for the first time usually occurs very quickly, in less than 20 s, but if the fibres are used a second time, saturation occurs for a longer time, for example in the case of acetone vapours - for longer than 80 s, *Figure 14*. In air atmosphere fibres return to their initial state in a manner dependent on the type of vapour. In the case of acetone vapours the purification process of the activated carbon submicrofibres studied takes about 150 s, *Figure 14*.

▪ Conclusions

The activated carbon submicrofibres developed are promising solvent vapour-sensitive material. These submicrofibres can be produced from electrospun polyacrylonitrile fibre mat in the process of stabilisation and oxidation, pyrolysis and chemical activation. If precursor fibres are used as a nonwoven/mat, activated carbon fibres are also obtained in form of nonwoven. In contact with vapours of methanol, acetone, benzene and toluene at a concentration of 200 ppm, the activated carbon submicrofibres show an electric response. Both in experiments where the sample studied is in direct contact with vapours and in those where the sample is in a prototype structure between polypropylene meltblown layers, the submicrofibres are characterised by sensitivity, a very short response time not longer than 20 s, stability, and by selectivity in relation to vapours of polar and non-polar solvents. The sensitivity to vapours of polar solvents, which are characterized by a considerably higher dielectric constant, is higher than to those of non-polar solvents. The activated carbon submicrofibres developed can be

used as a solvent vapour-sensitive material also in combination with other materials. This prototype structure, being sensitive to gas, could be used in filters and half-masks for individual protection of the human respiratory system.

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6th International Technical Textiles Congress
14-16.10.2015, Izmir, Turkey

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Congress topics:
- functional fibers
- nano technical textiles
- medical textiles
- protective clothes
- military textiles
- filtration textiles
- composite materials
- smart and interactive textiles
- packaging textiles
- automotive textiles
- sportive textiles
- agricultural textiles
- civil engineering and constructive textiles, geotextiles
- technical constuctions
- ecological aspects for technical textiles
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