

Vladimir S. Soldatov^{ac},
Zenon Polus^b,
Malgorzata Pawlowska^a,
Iwona Mączka^b,
Alexander Shunkevich^c,
Evgenij Kasandrovich^c,
Alexander Polikarpov^c

^aTechnical University of Lublin
ul. Nadbystrzycka 40, 20-618, Lublin, Poland

^bTextile Research Institute
ul. Brzezińska 5/15, 92-103 Łódź, Poland

^cInstitute of Physical Organic Chemistry
Surganova 13, Minsk 220072, Belarus

A Strong Acid Nonwoven Filtering Medium for Deep Air Purification

Abstract

The paper describes the basic chemical and mechanical properties of Fiban K-1 ion-exchanging fibres developed by the Belarus Institute of Physical Organic Chemistry. Since the virgin fibre loses its mechanical properties during different stages of its conversion into an ion exchanger, the most important problem is the ability to process ion-exchanging fibres into filtration fabric by means of textile processing. The results of technological trials of carding and needle-felting, as carried out by the Textile Research Institute in Łódź, are also described in this publication.

Key words: fibre, ion exchange, filtration, needle-felted fabric.

Introduction

The useful properties of chemically active fibres, mainly containing acid or base groups, have been described in a great number of publications, for example in reviews [1-7]. In these publications numerous advantages of these materials compared to granular sorbents of the same chemical nature, as well as their potential and real applications, have been discussed. Nevertheless, only a few such materials have been produced and are commercially available in the form of non-woven fabrics: Vion (Russia), Ionex (Japan) and Fiban (Belarus).

One of the most important practical problems at present is the development and organisation of mass production of non-woven fabrics, which can serve as filtering media for deep purification of air in the clean rooms of microelectronic production areas. The subject of this paper is a filtering medium for purifying air from basic impurities such as ammonia, amines, nitrogen containing heterocycles, etc. The most important problem at present is how to purify air of the ammonia present in the outdoor air, which has evolved from technological media in microelectronic production and from humans working in the workshops.

The specific requirements for this application are:

- extremely low concentrations of the impurities in the initial air (usually <100-200 µg/m³) and in the purified air (<10 µg/m³);
- a low relative humidity of the purified air, usually below 50%;
- large volumes of the purified air.

These requirements can be met if the filtering medium contains sufficiently strong acid groups, has high hydrophilicity, and low resistance to the airflow. The most suitable material with such properties is predicted to be a non-woven fabric made of filaments containing a high content of sulphonic acid groups. This is probably the strongest acidic group which can be incorporated into the polymer structure. For this reason it has a great affinity to water, which provides sufficiently high water sorption at a low relative humidity. These general ideas are quite obvious, and sulphonic-type fibrous ion exchangers long ago attracted the attention of several groups of researchers. However, it appeared that attempts to prepare such fibres encountered serious practical difficulties, and all the currently existing methods of synthesising them have proved to be rather complicated.

The polymers containing dissociated functional groups in high concentrations are unsuitable for the preparation of fibres, because their tensile strength and elasticity are too low. Therefore the fibres with ionised groups should have a special structure and contain some reinforcement which improves their mechanical properties and allows them to be processed in non-woven or woven fabrics. Several approaches to preparing such fibres are known. One of them is a mechanical combination of ion exchangers and fibre-forming polymers [2, 4, 5]. In spite of their apparent simplicity, such technologies have not found wide application; fibres obtained in this way had poor mechanical characteristics.

A special case is the Ionex fibres produced by Toray in Japan [4,5], which has the trade mark IONEX-TIN-100. The fil-

aments of this material were obtained by cross-linking with the following sulphonation of composite fibres with sixteen polyolefine parallel strings, and a mixture of polypropylene (60%) and polystyrene (40%) in the space between the strings. There is no information on the technological details of their production or the cost of the products. They are produced as short-cut fibres (<0.5 mm long). It has also been mentioned that they might be produced as a nonwoven material. It might be expected that this technology will be complicated, and the cost of non-woven fabrics of this type will be high.

The other sulphonic acid fibrous ion exchanger is based on chemically modified polyvinylalcohol fibres [5]. This was developed by Nichibi (Japan) and had the trademark IEF SC3050. At present it is used for producing chemical filters for air purification.

The other approach to this problem is to graft the polyelectrolyte or polyelectrolyte-forming polymer into the chains of the main polymer of the fibre. The grafting can be induced by ionising radiation. A number of Fiban materials have been obtained in this way. Systematic research on syntheses, properties and applications of sulphonic ion exchangers on the basis of a graft copolymer of styrene-polypropylene started in Belarus in 1972 [8]. The grafting was initiated by γ radiation. These researches were described in a number of original papers and reviews [3, 6, 7, 9-14].

In [15] it was noted that sulphonic fibrous ion exchangers were obtained by sulphonation of a graft copolymer of polypropylene and styrene prepared in a similar way. The grafting was initiated by fast electrons.

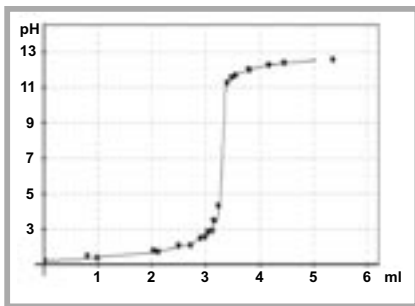


Figure 1. Potentiometric titration curve of Fiban K-1 fibre. Conditions of the titration: one sample titration, mass of fibre 0.6 g, initial solution volume 30 ml, forward titration (RH+KOH) with 2 N KOH, backward titration (RK+HCl) 2 N HCl. The supporting electrolyte in the solution and titrants was 1 N KCl, and the interval between additions of the titrant portions 20 min. The points are experimental, the line theoretical, computed from the model in [16] with the following parameters: $E_1=3.0$ m-equiv/g, $pK_1=-0.4$, $\Delta pK_1=0.5$; $E_2=0.07$ m-equiv/g, $pK_2=7$, $\Delta pK_2=0$.

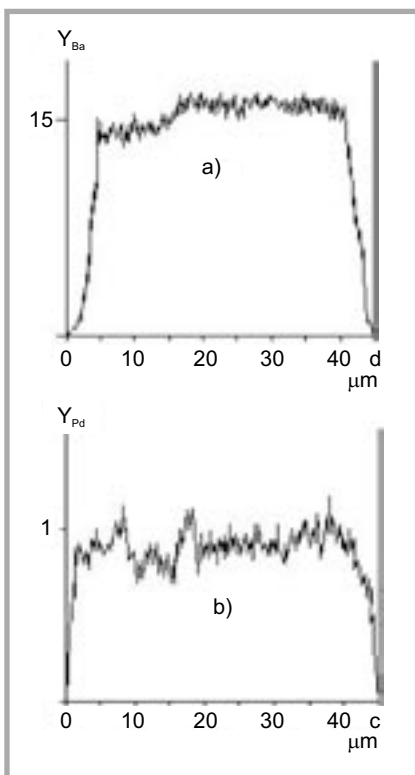


Figure 3. X-ray microanalysis of Fiban K-1x2 fibre in Ba^{2+} and $Pd(NH_3)_4^{2+}$ ionic forms. Concentration of the metal (conventional units) versus cross-section of the fibre. The fibre diameter is 45 μm .

It is the aim of this paper to summarise some of the properties of the Fiban K-1 sulphonic fibrous ion exchanger, which are important for its textile reprocessing into a nonwoven fabric used as a chemical filtering medium, as well as to describe some properties of the new filtering medium.

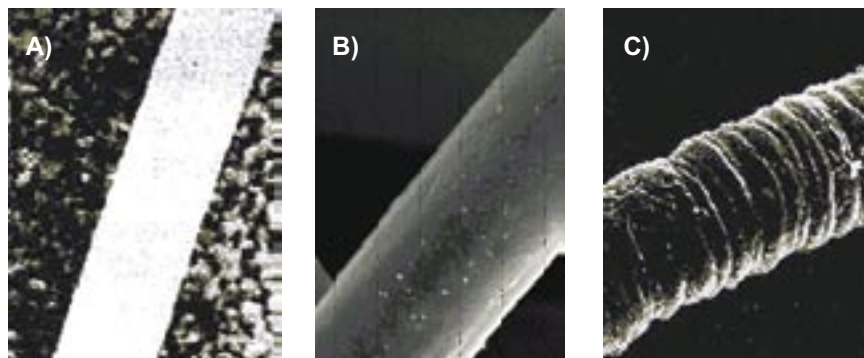


Figure 2. SEM view of a single fibre: A - initial PP fibre; B - Fiban K-1x1%DVB; C - Fiban K-1x2%DVB.

Physical Chemical Properties and Structure of the Fibre

The *Fiban K-1 ion exchange fibre* is a sulphonic-type monofunctional ion exchanger. It is obtained by grafting a styrene-divinylbenzene (ST-DVB) monomer mixture into industrial polypropylene (PP) fibres (staple with the filament diameter 22 μm and average length 6 cm) with its following sulphonation by chlorosulphonic acid. The grafting was carried out in the field of ^{60}Co radiation. The exposure dose was 6.5 Mrad, at a dose rate of 0.9 Mrad/h. The active process time was 7 hours. After irradiation, the fibre was washed out from the homopolymer, dried and taken for sulphonation.

Two variants of the ion exchanger with 1 or 2% of DVB in the monomer mixture were tested. The total amount of the grafted ST-DVB copolymer was 100% and 120% of the mass of the initial PP fibre respectively. These are named Fiban K-1x1 and K-1x2. The main part of the research described in the paper is related to the latter.

After sulphonation, we obtained the sulphonic ion exchanger with an exchange capacity of 3.0 m-equiv/g of hydrogen form. This value is close to the theoretical one, 3.1 m-equiv/g, computed on the assumption that each phenyl group in the graft fibre contains one sulphonic group. Computer analysis of the potentiometric titration curve (Figure 1), performed according to the model in [16], has shown that the Fiban K-1 fibre is practically monofunctional, and contains no more than 0.07 m-equiv/g of weak acid (probably carboxylic acid) groups. Such a value is typical of the best resinous ion exchangers of the same type.

The quantities in the caption to Figure 1 have the following significance: E_i - exchange capacity according to group

type i ; K is the constant of ion exchange equilibrium H^+K^+ , ΔpK accounts for the variation of the ion exchange equilibrium coefficient [17] with the degree of ion exchange from 0 to 1.

The macroscopic structure of the fibrous ion exchanger's filaments is rather peculiar, and substantially different from the structure of the initial filament. Figure 2 shows that the ion exchange fibre with a higher degree of grafting and cross-linkage has a pleated structure, with periodic widening at a distance of 3-5 μm .

The filaments are regular in one and the same sample. Figure 2 represents a typical example of their view. We anticipate that such a periodic structure has resulted from mechanical strength in the fibre in the course of grafting when the volume of the fibre more than doubles, because the ST-DVB copolymer has entered into the fibre. The narrowest parts of the filament are internal cracks, or parts of the structure which have a weakened polymer matrix formed at the relaxation of the fibre filled with the grafted copolymer. The fibre with a lower percentage of DVB has a smooth surface in spite of the same degree of grafting (100% ST-DVB). The periodic structure starts with a low DVB percentage at higher degrees of grafting.

The volume distribution of the grafted copolymer in the fibre was confirmed by X-ray analysis [18,19]. It has been shown that the fibre consists of interpenetrating networks of crystallites of polypropylene and domains of sulphonated ST-DVB copolymer with the size of 7-15 nm. At the same time, the fibre is macroscopically homogenous as seen from the results of electron probe microanalysis (Figure 3). There is no indication of uneven distribution of the ion exchange constituent of the fibre across the cut of the fibre. On the basis of this data, we can imagine the

internal molecular structure of the fibrous ion exchanger as shown in Figure 4.

The polypropylene fibre loses its mechanical properties during different stages of its conversion into the ion exchanger. This is illustrated by Table 1, which shows that the final fibre nevertheless has a set of properties which are sufficient for it to be reprocessed into a non-woven fabric.

In spite of its rather poor mechanical properties, Fiban K-1 appeared very resistant to severe treatment with different aggressive chemical agents, and significantly loses its usefulness for filtering media properties only after prolonged treatment with hot nitric acid (Table 2). The elasticity of the fibre is most sensitive to the chemical treatment.

It is seen from Table 2 that values characterising elasticity are especially sensitive to chemical treatment, namely their elongation at break L and the elasticity modulus E. The fibre becomes more elastic after a short time of treatment with NaOH or HCl than the non-treated fibre, probably due to the strong swelling of the ion exchange component of the fibre and its structure subsequently loosening (the values in Table 2 were measured in an air-dry state). The prolonged treatment with the same chemical agents may cause depolymerising of the grafted copolymer, as well as some destruction of the main polymer chains, which should result in a loss of elasticity compared to the initial fibre.

The opposite is observed for nitric acid, which acts not only as a protolytic agent but also as an oxidant destroying the polymer matrix.

Textile Processing and Properties of Nonwoven Fabrics

The fibre processing was carried out at the Textile Research Institute, Łódź, Poland [20]. These experiments were aimed at answering the following questions:

- Is it possible to obtain a needle-felted fabric from the samples of Fiban K-1x2 staple fibre with the properties described above?
- What is the effect of different softening agents on the fibres in the textile processing?
- What are the basic characteristics of the nonwoven fabrics produced?

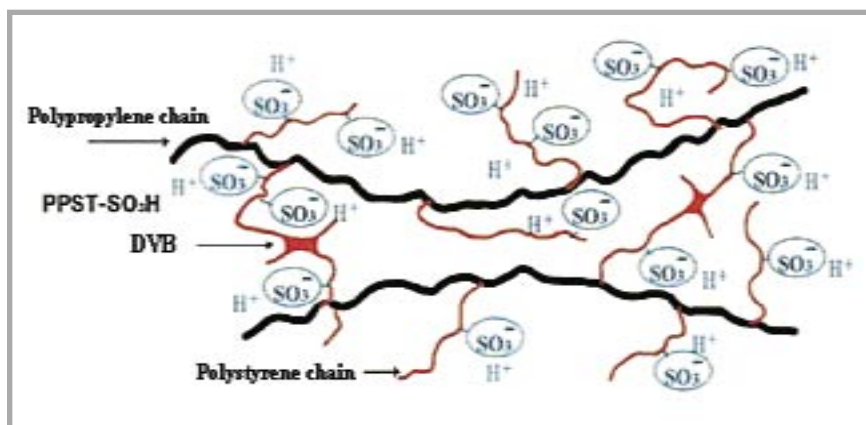


Figure 4. Imaginable molecular structure of Fiban K-1 fibre.

Table 1. Mechanical properties of the polypropylene fibre at different stages of their conversion into the sulphonic acid ion exchanger.

Fibre	Fibre diameter, μm	Breaking force, cN	Tensile strength R, N/mm^2	Elongation at break L, %	Elasticity modulus E, N/mm
Initial PP	25	21.7	36.0×10^7	62	246×10^4
PP washed by benzene	26	13.3	24.0×10^7	105	176×10^4
PP-120%ST-2%DVB	35	14.1	14.5×10^7	52	175×10^4
Fiban K-1x2 (H^+ form) in air dry state (20% H_2O)	39	8.1	6.7×10^7	11	282×10^4

Table 2. Effect of chemical treatment on the properties of Fiban K-1x2 fibres (*water uptake in H^+ form).

Property	Initial	0.5 h in 5 M NaOH 100°C	5 h in 5 M NaOH 100°C	0.5 h in 5 M HCl 100°C	5 h in 5 M HCl 100°C	5 h in 5 M HNO_3 100°C	48 h in 10% H_2O_2 20°C
E, m-equiv/g	3.63	3.69	3.37	3.58	3.58	3.43	3.90
W,* g $\text{H}_2\text{O}/\text{m-equiv.}$	0.34	0.34	0.28	0.32	0.29	0.29	0.35
r, % of initial	100	102	102	105	98	77	107
R, % of initial	100	99	99	99	97	94	102
L, % of initial	100	157	52	166	43	28	138
F, % of initial	100	82	94	84	95	107	82

Table 3. Carding results of Fiban K-1x2 fibres.

Sample No	Percent of softener, %	Fibre weight before carding, g	Web weight after 1 st carding, g	Web weight after 2 nd carding, g	Total collected waste, g
1	0	300	161	78	210
2	1.1% glycerol	300	195	130	155
3	1.1% polyethylene glycol 400	300	178	96	185
4	1.1% polyethylene glycol 400 + 0.02% polyvinyl alcohol	300	168	92	195
5	1.1% sorbitol	300	184	108	174

Table 4. Carding results of Fiban K-1x2/ PP fibre blends (20% PP).

Sample No	Percent of softener, %	Fibre before carding weight, g	Web width, cm	Web weight after 1 st carding, g	Web weight after 2 nd carding, g	Total collected waste, g
6	1.1% glycerol	300	50	237	175	104
7	1.1% polyethylene glycol 400	300	50	245	190	93
8	1.1% sorbitol	300	50	240	184	95

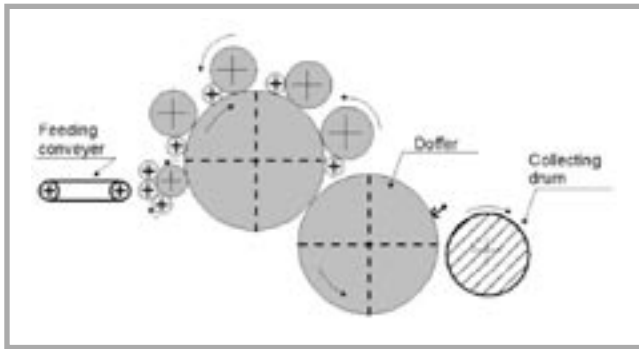


Figure 5. Laboratory carding machine.

Experiments on processing Fiban K-1 ion exchange staple fibres were carried out on two laboratory machines: a carding machine (Figure 5) and a Heuer needle felting machine. In order to improve the elasticity of the fibres and to suppress dusting during the textile processing, we treated the fibres with different softeners, glycerol, polyethyleneglycol and sorbitol.

The following sequence of operations was used in the web formation:

- fibre opening - by hand-feeding the conveyer of the card,
- web forming - on collecting the drum of the card,
- web width setting - 50 cm,
- resulting fibre orientation - machine direction,
- number of passages - 2.

Five different Fiban K-1x2 fibres were processed: sample No. 1, Fiban K-1x2 with no softening agent, and samples No. 2-5 treated with different softening agents described in Table 3, which also shows the carding results. During carding a part of the fibre was broken; some parts could be collected and others not. In Table 3, only the total collected wastes are shown.

Since processing of 100% Fiban K-1 fibre is rather difficult, additional trials with the blends were carried out. The results of the next step (processing the blends of 80% Fiban K-1x2/20% polypropylene fibres 4 dtex/60 mm) are shown in Table 4.

Needling

The webs produced were subjected to the needling process on the Heuer needling machine. Due to the high fibre breakage of samples 1-5, two web-layers were used for nonwoven production. For

samples 6-8 (listed in Table 4), due to lower fibre breakage, one layer of web was used. Foster-Gauge type needles 15 × 18 × 40 × 3.5" RB (3-edge needle, two barbs on one edge) were used. Two passages of 95 needles/cm² in each case were made, with depths of penetration of 7 and 10 mm respectively.

A low yield of the nonwoven material is caused by the poor mechanical properties of Fiban K-1x2 fibres, reflected in Figure 2B. Therefore we modified the preparation conditions of the sulphonated fibres by lowering the degree of grafting and the DVB percentage. This led to a fibre with a more even surface (Figure 2C) and higher elasticity. Carding such fibres allowed a sharp rise in the yield of the nonwoven fabric and a reduced percentage of wastes (compare samples 1 and 9 in the Tables). Further improvement of the textile processing was obtained by adding 20-50% of polypropylene to the

Table 5. Carding results of Fiban K-1x1 / PP fibre blends.

Sample No	Polypropylene, %	Fibre weight, g	Web width, cm	Web weight after 1 st carding, g	Web weight after 2 nd carding, g	Waste, g
9	0	300	50	246	194	106
10	20	300	50	270	232	68
11	50	300	50	284	268	32

Table 6. Basic characteristics of Fiban K-1x2 nonwovens.

Sample No	Area weight, g/m ²	Thickness, mm	Breaking strength, N Machine direction/ Cross direction	Elongation at break, % Machine direction/ Cross direction	Air permeability, mm/s at pressure drop, 100 / 200 Pa
1	150	2.53	14.2 / 3.0	61.5 / 93.6	3260 / 5407
2	184	3.29	53.5 / 15.4	57.9 / 114.5	2864 / 4550
3	168	3.10	37.6 / 13.6	54.0 / 131.4	3009 / 4990
4	147	2.84	15.5 / 6.0	57.3 / 114.8	3728 / 5858
5	159	3.04	28.1 / 7.3	55.0 / 106.5	3289 / 5362
6	145	3.05	141.5 / 20.6	62.5 / 151.5	2818 / 4427
7	168	3.22	141.3 / 30.4	61.8 / 152.8	2509 / 3999
8	157	3.16	134.2 / 25.5	62.5 / 162.2	2663 / 4234
Testing method	PN-EN 29073-1:1994	PN-EN 29073-2:1994 Pressure 0.5kPa	PN-EN 29073-3:1994	PN-EN 29073-3:1994	PN-EN ISO 9237:1998

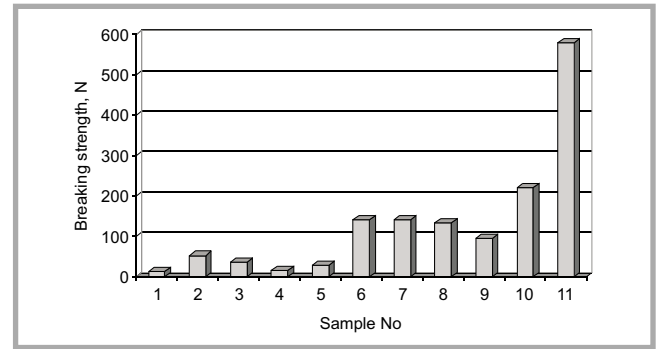


Figure 6. Machine direction breaking strength of samples tested.

Fiban K-1 fibres, as illustrated by Tables 4 and 5.

Properties of Fiban K-1 Nonwoven Fabric

The mechanical properties of Fiban K-1 fabric are presented in Tables 6 and 7. In spite of the rather poor mechanical properties of staple fibres, the mechanical properties of the nonwoven fabrics are acceptable as filtering media. A micrograph obtained with a scanning electron microscope shows its internal structure (Figure 7).

From the point of view of fibre loss, the most important problem during processing is the fibre's breaking strength. The nonwoven machine direction breaking strength (presented in Figure 6) shows that the 20% blend of Fiban K-1 and polypropylene fibre allows much better results to be achieved. The presented graph

Table 7. Basic characteristics of Fiban K-1x1 nonwovens.

Sample No	Area weight, g/m ²	Thickness, mm	Breaking strength, N Machine direction/ Cross direction	Elongation at break, % Machine direction/ Cross direction	Air permeability, mm/s Pressure drop, Pa at 100/200
9	177	2.86	95.2/33.7	70.5/124.0	2711/4314
10	177	3.0	221.1/64.2	65.0/132.5	2054/3284
11	223	3.42	577.3/126.9	58.5/149.0	1191/1999

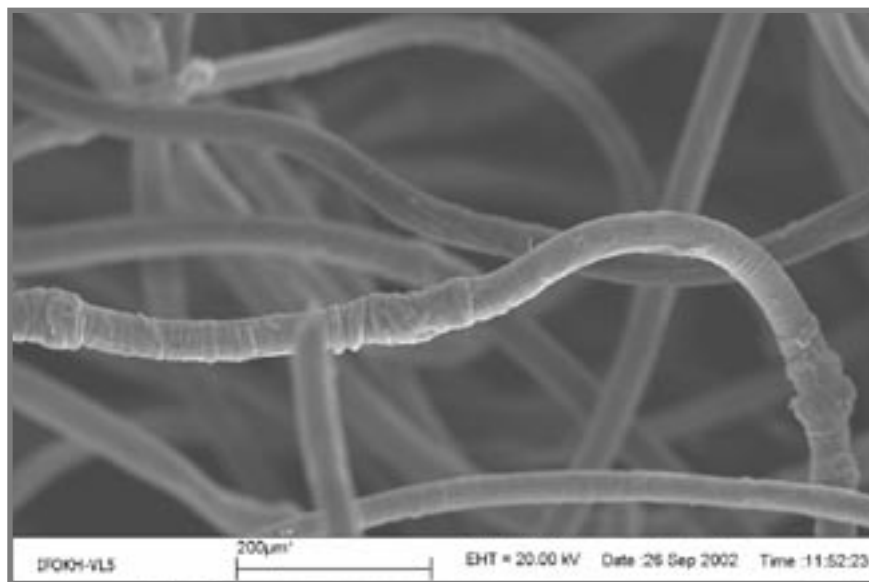


Figure 7. A view of the structure of nonwoven manufactured from a blend of polypropylene and Fiban K-1.

also confirms the results from Table 3, that the best results are obtained for 1.1% glycerol as an additive to all materials made of Fiban K-1x1.

It is seen that the filaments of Fiban K-1x2 have a pleated surface and plentiful fractures or pre-fracture points. The filaments are brittle because the fraction of the ion exchange component in their structure is high, and can hardly be increased without a dramatic loss of the mechanical

properties of the material. The value of the exchange capacity of this type of ion exchange fabric (3-3.5 m-equiv/g) may be accepted as the highest possible. This is one of the most important characteristics of chemically active filtering media, because its operation time is proportional to the exchange capacity. The other two basic characteristics are the acid strength of the functional groups in the fibres and the lowest relative humidity at which the fibrous ion exchanger can utilise its full

capacity. As mentioned, the sulphonic group is the strongest acidic group possible in the polymer fibres. It has the same acid strength as simple strong mineral acids ($pK < 0$). Our measurements of its efficiency in air purification from ammonia have shown that it is practically independent of the relative air humidity if the latter is higher than 7% (no measurements below this value were performed). The breakthrough curve of NH_3 at the lowest relative humidity under conditions similar to real technological processes is presented in Figure 8a.

This figure illustrates the excellent kinetic properties of this filtering material. The curve is steep and close to symmetric. At the point of the breakthrough $C/C_0=0.5$, the working capacity of the filter, calculated from the air flow-rate, the NH_3 concentration in the inlet and outlet air and the mass of the filter, was 2.5 m-equiv/g, which is equal to an efficiency of 82%. Such an efficiency for a thin filtering material should be regarded as extremely high. From the end point of view, considering the filter material's usability, sorption of toxic substances is the most important factor. Sorption of NH_3 in the case of 1, 2, and 3 layers of filtering medium is shown in Figure 8b. The conditions of testing remained the same as during the experiment presented in Figure 8a.

Summary

Strong acid fibres obtained by radiation graft polymerisation of styrene and divinylbenzene onto polypropylene fibres with their subsequent sulphonation can be used for preparing highly efficient

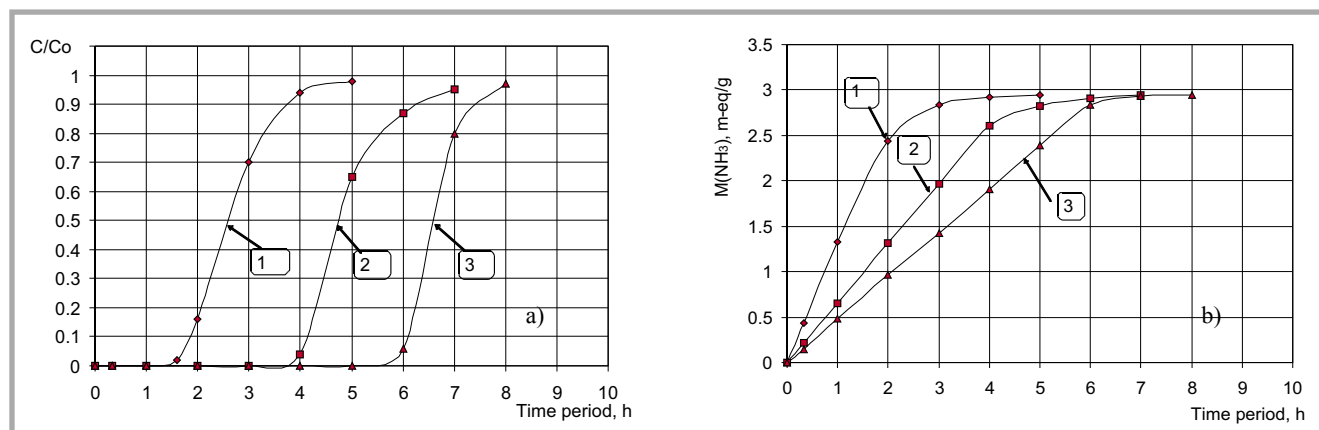


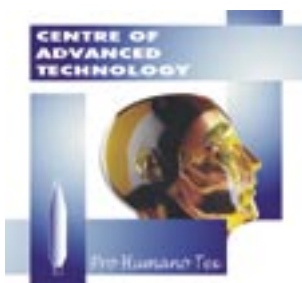
Figure 8. a) Breakthrough curve of ammonia through the Fiban K-1 non-woven filter with different numbers of layers of the filtering medium. C and C_0 are the concentrations of ammonia in the air at the outlet and inlet of the measuring cell respectively; 1, 2, 3 - indicate the number of layers; and b) Sorption curves of NH_3 . The conditions of both experiments are given below: $C_0=13.4$ mg/m³, the thickness of one layer is 3 mm, mass 0.3 g, the air flow rate 0.095 m/s, air relative humidity 50%, $t=20^\circ C$.

filtering media for deep purification of the air from ammonia and other basic impurities. They contain only sulphonic groups in amounts equivalent to that of the phenyl rings in the grafted copolymer. The ion exchange groups are evenly distributed in the volume of the fibres. The fibres are stable in aggressive media such as strong acids, alkalis and oxidants, and have mechanical properties which allow them to be reprocessed into nonwoven fabrics. The fibres with 100% of the graft copolymer containing 1% DVB and their blends with polypropylene fibres provide satisfactory textile processing characteristics, and allow nonwoven fabrics with good mechanical properties and air permeability to be obtained. The nonwoven fabrics obtained appear to be highly efficient filtering media for deep purification of the air from ammonia.

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For manufacturing textiles friendly for human, not only textile technique processes will be used, but also advanced electronic, informatics, chemical, and nano-technologies.

Assortments, which belong to the following fields will be manufactured: general-use clothing for disabled, protective clothing and other individual protective means, furnishing & automotive textiles, and technical textiles.

At present, the Centre associates 2 universities (University of Łódź, Technical University of Łódź) 10 R&D centres (Central Institute of Labour Protection, Warsaw; Institute of Teratechnology, Radom, R&D Centre for Textile Machines POLMATEX-CENARO, Łódź; Textile Research Institute, Łódź; Institute of Chemical Fibres, Łódź; Institute of Dyes and Organical Products, Zgierz; Institute of Textile Material Engineering, Łódź; Institute of Textile Architecture, Łódź; Institute of Knitting Technique and Technologies TRICOTEXTIL, Łódź), and many enterprises (e.g. FILTER-SERVICE, Zgierz; POLONTEX Co, Poraj; TEOFILÓW Co, Łódź; WOLA, Zduńska Wola; MIRANDA, Turek; ALGA-BIS, Łódź; ASTILTEX CONSULTING, Łódź).

The co-ordinator of the Centre of Advanced Technologies PRO HUMANO TEX is the Faculty of Textile Engineering and Marketing, Technical University of Łódź.

The proxy of the co-ordinator is Professor Izabella Krucińska Ph.D., D.Sc.

For more information please contact: Monika Malinowska-Olszowy, tel.: 508297171, e-mail: monika10@mail.p.lodz.pl