Agnieszka Brochocka

Filtration Properties of Nonwoven Structures with Superabsorbents for Respiratory Protective Devices

Central Institute for Labour Protection
– National Research Institute,
Department of Respiratory Protective Devices,
Czerniakowska 16; 00-701 Warszawa,
Poland
E-mail: agbro@ciop.lodz.pl

Abstract
The paper presents a study of the protective and functional properties of nonwoven structures, such as penetration by solid and liquid aerosols, airflow resistance, and air moisture sorption capacity. Nonwovens were modified by the introduction of electrostatic charges in the structure of polymer fibers and by the addition of a superabsorbent polymer (SAP) directly to the fiber-forming area in a melt-blown process. The resulting materials (outer nonwovens containing SAP of different grain sizes and filtering nonwovens with and without electrostatic charges) had varied morphological structures. The materials developed were characterised by high filtering efficiency at low airflow resistance and good moisture sorption ability. The study proved the possibility to apply nonwoven structures containing SAP in respiratory protective equipment used in harsh work environments at high temperature and relative humidity. The materials fabricated were found to improve hygienic comfort.

Key words: *nonwoven structure, filtering efficiency, superabsorbent polymers, respiratory protective devices, melt-blown process.*

Introduction

The European textile market poses new challenges to manufacturers of polymeric nonwoven materials, prompting the development of novel technological solutions and the design of products with specialised structures, advanced properties, and new applications.

Numerous research efforts have been undertaken to explore the application potential of nonwoven structures as multi-purpose composite materials. Depending on the quantitative and qualitative composition of the raw material, its morphology, hydrophilicity and hydrophobicity, the techniques of material modification as well as the physical and chemical properties of the modifiers, composite nonwovens are increasingly used in various industrial sectors, such as the construction, automotive and textile industries, medicine, transportation, etc. [1-5]. The filtering materials applied in respiratory protective devices include multilayer nonwoven structures manufactured by means of different textile technologies.

The commercially available filtering respiratory protective devices for heavy-duty applications such as filtering half-masks and half-masks equipped with filters, may quickly lose their protective parameters in harsh working environments. Thus to improve protective properties, respiratory equipment manufacturers are obligated to undertake efforts to modify their products or develop new devices or materials. The basic criterion used for evaluation of such equipment is efficient filtering of harmful aerosol particles [3, 6, 7], which can be achieved by the use of highly porous nonwoven structures made of thin fibers which are electrostatically charged by corona discharge in the process of nonwoven manufacturing. The staple filtering materials for respiratory protective equipment are electret melt-blown nonwovens. Melt-blown technology enables the production of thin fibers with a diameter of 1-2 μm through appropriate selection of process parameters, which determine the morphological structure of the resulting material [8]. However, years of research on filtration modelling and evaluation have shown that such nonwovens do not provide efficient protection under conditions of high temperature and relative humidity in the workplace. Electromagnetic attraction forces, which constitute the basic mechanism of eliminating aerosol particles, become quickly neutralised, leading to an abrupt decline in protective performance, which also translates into decreased work comfort [9]. The most widespread cause of discomfort during the use of half-masks is difficulty with breathing during work as well as dermatological problems in the area where the half-mask makes contact with the face (the cheeks, nose, chin). An unfavorable microclimate quickly develops inside tightly fitting masks, with humid air making water molecules accumulate in the facial part of the device, which irritates the skin. As a result, workers may discontinue the use of half-masks, foregoing their crucial protective benefits. Recently literature has suggested the introduction of a superabsorbent (SAP) polymer into the structure of airlaid nonwovens designed for protective footwear [10]. Nonwovens with SAP additives have been shown to absorb air moisture more effectively than those without them or traditional felt materials. Thus the application of nonwovens with SAP in respiratory protective devices is expected to radically improve user comfort in heavy-duty applications at high temperatures and humidity.

Currently the chemical industry offers a wide range of SAPs in different forms and with constantly improving properties. In addition to granules, SAPs are now made as coarse and fine powders, hydrogels, and fibers. At the same time, the fast development of these materials provides an incentive to explore their application potential. So far, such materials have been widely used in personal hygiene products manufactured by traditional nonwoven methods, such as hydrodynamic technology with crystal clear water and airlaid needle punching (with or without thermal or chemical bonding). Modern disposable personal hygiene and health care products have been found to exhibit excellent absorption properties, ensuring very good comfort of use [11]. However, it may be expected that an improvement in one parameter may lead to deterioration of other functions of the product. High efficiency and multi-purpose design require the separation of functional elements and their incorporation in fibers in a specific way to gain

better control over liquid flow and to direct those elements in a suitable manner. However, it is difficult and non-economical to apply traditional textile methods in the manufacture of nonwoven with SAP additive composites that could isolate, distribute and retain liquids. An interesting alternative is melt-blown technology, in which nonwovens are fabricated directly from a polymer melt. Indeed melt-blown nonwovens offer excellent filtering, thermal insulating, and sorptive properties, mostly due to the small diameter of fibers. The melt-blown process has also been selected due to the fact that it enables effective modelling of nonwoven structure in terms of the mass per unit area and porosity. In the work presented, of particular note is the first direct use of superabsorbents in conjunction with melt-blown technology. Currently in the European market there is no multi-purpose filtering melt-blown nonwoven that would exhibit high filtering efficiency at low airflow resistance and good moisture absorption capacity. The solution proposed is innovative in that it enables the fabrication, in one fiber-forming and modification process, of a multi-purpose nonwoven composite for respiratory protective equipment used in harsh work environments.

Materials and methods

The raw material for melt-blown nonwoven production was Borealis HL 508J atactic polypropylene granulate with a melt-flow index of 800 g/10 min and density of 50 g/cm² (from NEXEO Solutions Poland Sp. z o.o, Poland).

The modifier was EK-X EN52 superabsorbent (SAP) polymer with grain sizes of 250 μm and 30 μm, added to the polymer matrix in the amount of 3% and 7%

v/v (from Toruńskie Zakłady Materiałów Opatrunkowych S.A., Poland).

The melt-blown apparatus was equipped with an electrostatic activation device and die assembly enabling the introduction of SAP into semi-liquid polypropylene fibers (Extruder from AXON ABB, SWEDEN, Fibre-forming head, air heater, take-up device – from CIOP-PIB, Poland). This setup was used to produce a total of 6 variants of nonwovens, characterised in *Table 1*.

Outer nonwovens which are commonly used in respiratory protective devices such as filtering half-masks and filters typically stop large airborne particles and protect the filtering layer from mechanical damage.

In turn, filtering nonwovens are responsible for the main process of air filtration. It was assumed that outer nonwovens used in the composite material should fulfill two roles:
- nonwoven with coarse SAP (250 μm) should dehumidify the air flowing through it and eliminate large particulate matter,
- nonwoven with fine SAP (30 μm) should improve user comfort in terms of contact between the mask and face in harsh work environments.

The nonwovens were used to design three nonwoven systems for respiratory protective devices. These systems consisted of the following layers (from the air inlet side):

System I
1. outer nonwoven with SAP additives with a grain size of 250 μm,
2. electret filtering nonwoven with a mass per unit area of 90 g/m²,

3. outer nonwoven with SAP additives with a grain size of 30 μm.

System II
1. outer electret nonwoven with SAP additives with a grain size of 250 μm,
2. electret filtering nonwoven with a mass per unit area of 90 g/m²,
3. outer electret nonwoven with SAP additives with a grain size of 30 μm.

System III
1. outer electret nonwoven with SAP additives with a grain size of 250 μm,
2. electret filtering nonwoven with a mass per unit area of 90 g/m²,
3. outer nonwoven with SAP additives with a grain size of 30 μm.

Testing methods
In order to evaluate the basic protective and functional properties of the systems developed, the following tests were conducted:

Sodium chloride aerosol and paraffin oil mist penetration coefficients were determined pursuant to the standards EN 149:2001+A1:2009 and EN 13274-7:2008 concerning the testing methodology and requirements for respiratory protective devices [12, 13]. This coefficient expresses the ratio of aerosol concentration upstream and downstream of the sample. The mass median diameter of sodium chloride particles was 0.6 μm. The distribution of particle sizes for oil mist was log-normal with a median Stokes diameter of 0.4 μm. The test period corresponded to initial filtration (3 min).

Airflow resistance testing was conducted according to the European standards EN 149:2001+A1:2009 and EN 13274-3:2008, specifying the testing method-

Table 1. Characteristics of outer melt-blown nonwovens.

Type of outer nonwoven	Mean actual mass per unit area, g/m²	Standard deviation, g/m²	Mean fiber thickness, μm	Standard deviation, μm	Mean nonwoven thickness, mm	Standard deviation, mm
Outer nonwoven with SAP grain size 250 μm, without electrostatic charge	48.00	1.048	–	–	0.81	0.42
Outer nonwoven with SAP grain size 250 μm, with electrostatic charge	49.64	1.49	4.11	2.54	0.74	0.02
Outer nonwoven with SAP grain size 30 μm, without electrostatic charge	40.91	0,79	–	–	0.73	0.02
Outer nonwoven with SAP grain size 30 μm, with electrostatic charge	39.46	1,72	4.48	2.87	0.74	0.02
Filtering nonwoven made of PP fibers without electrostatic charge	96.69	1.52	1.25	1.52	2.53	0.047
Filtering nonwoven made of PP fibers with electrostatic charge (Q)	96.92	1.26	1.19	1.26	2.51	0.065

Table 2. *Mean NaCl penetration for the nonwovens tested (descriptive statistics).*

Type of nonwoven structure	Mean value of NaCl penetration, %	Standard deviation, %	Median, %	Maximal value, %	Minimal value, %
Nonwoven with fine SAP (30 μm)	72.94	1.18	73.22	74.71	70.53
Corona charged nonwoven with fine SAP (30 μm)	58.17	1.33	58.03	60.23	56.05
Nonwoven with coarse SAP (250 μm)	73.37	0.91	73.43	74.89	72.07
Corona charged nonwoven with coarse SAP (250 μm)	54.76	0.92	54.51	56.99	53.25
Filtering nonwoven	8.43	0.60	8.47	9.49	7.49
Corona charged filtering nonwoven	0.61	0.10	0.63	0.77	0.41

Table 3. *Mean paraffin oil mist penetration for the nonwovens tested (descriptive statistics).*

Type of nonwoven structure	Mean value of paraffin oil mist penetration, %	Standard deviation, %	Median, %	Maximal value, %	Minimal value, %
Nonwoven with fine SAP (30 μm)	92.20	1.11	92.00	94.00	90.00
Corona charged nonwoven with fine SAP (30 μm)	87.00	1.75	87.00	90.00	84.00
Nonwoven with coarse SAP (250 μm)	92.80	1.40	93.00	95.00	90.00
Corona charged nonwoven with coarse SAP (250 μm)	86.20	1.82	86.00	92.00	84.00
Filtering nonwoven	10.08	0.45	10.01	10.90	9.07
Corona charged filtering nonwoven	1.48	0.14	1.50	1.70	1.20

Table 4. *Mean airflow resistance for the nonwoven variants tested – descriptive statistics.*

Nonwoven type	Mean airflow resistance, Pa	Standard deviation, Pa	Median, Pa	Maximal value, Pa	Minimal value, Pa
Nonwoven with fine SAP (30 μm)	12.1	1.5	12.0	14.6	9.2
Corona charged nonwoven with fine SAP (30 μm)	12.6	1.0	12.8	14.6	11.0
Nonwoven with coarse SAP (250 μm)	13.0	1.1	13.2	14.6	11.0
Corona charged nonwoven with coarse SAP (250 μm)	13.4	1.1	13.3	14.6	11.4
Filtering nonwoven	198.98	7.7	199.5	209.7	184.9
Corona charged filtering nonwoven	260.12	4.8	259.0	269.0	251.0

Table 5. *Mean paraffin oil mist penetration through the nonwoven systems developed – basic statistics.*

Nonwoven system	Mean penetration by paraffin oil mist, %	Standard deviation, %	Median %	Maximum value, %	Minimum value, %
I	1.21	0.06	1.20	1.33	1.10
II	1.16	0.03	1.17	1.19	1.12
III	0.66	0.24	0.70	0.96	0.29

Table 6. *Mean sodium chloride aerosol penetration through the nonwoven systems developed – basic statistics.*

Nonwoven system	Mean penetration by sodium chloride aerosol, %	Standard deviation, %	Median, %	Maximum value, %	Minimum value, %
I	0.39	0.03	0.40	0.45	0.33
II	0.26	0.05	0.27	0.33	0.17
III	0.22	0.04	0.22	0.29	0.17

ology and requirements for respiratory protective devices [13, 14]. Measurement involved passing air through the filtering material at a constant airflow rate and with a negative pressure differential downstream of the material with respect to atmospheric pressure. The tests were conducted at an airflow rate of 95 L/min, corresponding to a minute respiratory volume during light and strenuous work. Pressure values were read from a CMR-10 A digital differential micromanometer.

Moisture sorption testing during dynamic airflow was conducted on an experimental station simulating the conditions of use of air-purifying respiratory protective devices pursuant to the standard EN 149:2001+A1:2009 [13]. The station is equipped with artificial lungs set to 25 cycles/min and 2.0 L/cycle, simulating the phases of inhalation and exhalation, a Sheffield head model, and an air humidifier installed between the artificial lungs and head model. In the mouth region of the model, the air is humidified at (37±2) °C. The ambient temperature was 23.6 °C and relative air humidity 44.0%. Nonwoven samples were mounted inside a connector placed in the orifice (mouth) of the model head for 3 h 20 min. Subsequently the samples were weighed using an analytical balance immediately after their removal and after 5 min, 30 min, and 60 min.

■ **Result and discussion**

The results of penetration of the fabricated nonwoven structures by the test aerosols: sodium chloride and paraffin oil mist are presented in *Tables 2* and *3*.

The introduction of electrostatic charges in the structure of polymeric fibers improved the filtering efficiency, as expressed by the aerosol penetration coefficient, with the charged nonwoven structures being characterised by lower values. In the case of non-charged outer nonwovens, the penetration coefficient for sodium chloride was by approx. 21% lower than that for paraffin oil mist, while in the case of charged outer nonwovens, the coefficient was lower by approx. 30%. In the case of charged outer nonwovens with 30 μm SAP additives, the percentage change in penetration relative to sodium chloride was approx. 20%, and in the case of charged outer nonwovens with 250 μm SAP additives, the change was approx. 25%. In non-charged outer nonwovens with SAP additives with dif-

ferent grain sizes, the relative change in NaCl penetration was 0.57%, and in the charged ones − 5.9%. The results show that while SAP grain size does not influence NaCl aerosol penetration through non-activated nonwovens, its effect becomes prominent for nonwovens activated with an electrostatic charge.

In the case of electrostatic modification, the percentage change in penetration with respect to paraffin oil mist was approx. 5.6% for outer nonwovens with 30 μm SAP additives and approx. 7% for those with 250 μm SAP. For non-activated nonwovens with SAP additives of different grain sizes, the relative change in penetration with respect to paraffin oil mist amounted to 0.65%, while the corresponding value for activated outer nonwovens was 0.9%. These results show that the SAP grain size has no influence on penetration by paraffin oil mist through non-activated nonwovens. However, as in the case of sodium chloride, this parameter exerts a significant effect for electrostatically charged nonwovens.

The relative change in aerosol penetration through the electrostatically activated filtering nonwoven made of thin fibers (approx. 1 μm) was 93% and 85% for sodium chloride and paraffin oil mist, respectively. The large relative differences in aerosol penetration are attributable to modification with atmospheric-pressure plasma. Electrostatic activation led to physical changes on the surface of elementary fibers, which are the basic units comprising nonwoven materials. These results are similar to those obtained in studies examining the effects of low-temperature plasma on polypropylene and polycarbonate materials [15, 16]. Thus there is a substantial body of evidence

Figure 1. Mean penetration of the nonwoven systems developed by the two test aerosols.
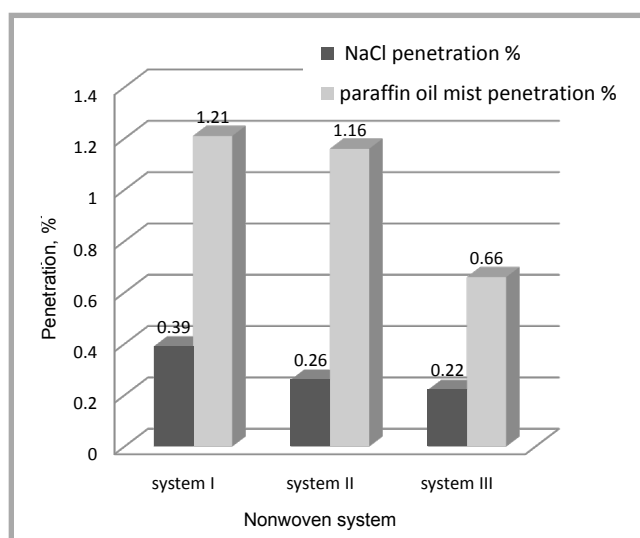
showing that corona discharges afford wide possibilities of improving the filtering properties of nonwovens.

In the case of outer nonwovens with a nominal mass per unit area of 40 g/m² and 50 g/m², only a small increase in airflow resistance was observed for the variants tested. The highest increment in that parameter (23%) was found between non-activated and activated filtering nonwovens with a nominal mass per unit area of 90 g/m² (*Table 4*).

Tests conducted on the basic protective and functional parameters showed that different nonwoven structures designed for filtering half-masks can be fabricated in one technological process.

Basic statistics for the mean aerosol penetration of the nonwoven systems developed are given in *Table 5* for paraffin oil mist and in *Table 6* for sodium chloride aerosol, as well as in *Figure 1*.

The combination of individual modified nonwoven structures into systems led to lower aerosol penetration, with the lowest values for both sodium chloride aerosol (0.22%) and paraffin oil mist (0.66%) found for system III, consisting of the following layers:
1. outer electret nonwoven containing 250 μm SAP,
2. electret filtering nonwoven with a mass per unit area of 90 g/m²,
3. outer nonwoven containing 30 μm SAP.

Descriptive statistics for mean airflow resistance are given in *Table 7* and *Figure 2*.

In terms of breathing comfort, the nonwoven systems developed are characterised by airflow resistance between 330 Pa and 370 Pa. The highest airflow resistance (364 Pa) was obtained for system III, and the lowest (approx. 336 Pa) for system I. In terms of the filtering pa-
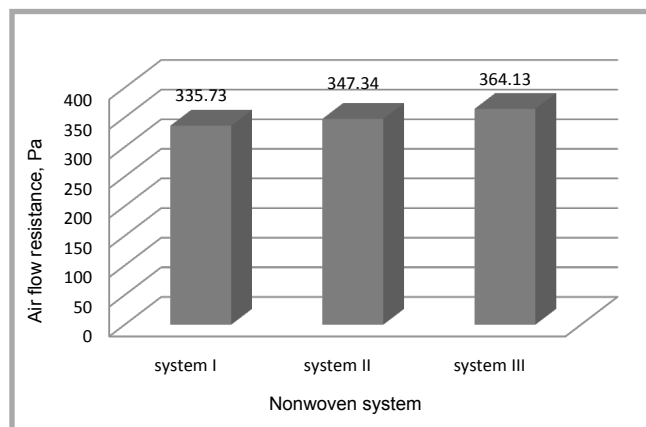

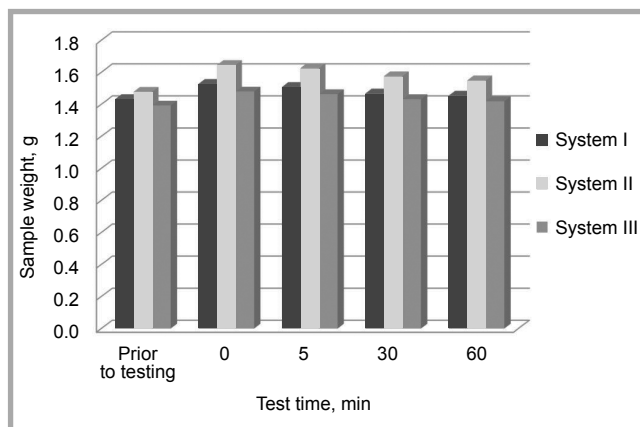*Figure 2. Mean airflow resistance values for the nonwoven systems.*

Figure 3. Sample weight over time in dynamic flow tests of moisture sorption.

Table 7. *Mean airflow resistance for the nonwoven composites developed.*

Nonwoven system	Mean airflow resistance, Pa	Standard deviation, Pa	Median Pa	Maximum value, Pa	Maximum value, Pa
I	335.73	14.33	337.90	358.20	310.30
II	347.34	6.51	346.90	358.60	338.20
III	364.13	7.23	364.55	377.30	352.90

Table 8. *Dynamic flow tests of moisture sorption.*

Parameter	Time, min	Nonwoven system		
		System I	System II	System III
Mean, g	Before test	1.43	1.48	1.39
	0	1.53	1.65	1.48
	5	1.51	1.63	1.46
	30	1.47	1.58	1.43
	60	1.45	1.55	1.42
Standard deviation, g	Before test	0.02	0.05	0.03
	0	0.09	0.14	0.10
	5	0.09	0.13	0.10
	30	0.08	0.10901	0.08
	60	0.05	0.08	0.06
Median, g	Before test	1.44	1.49	1.39
	0	1.49	1.58	1.45
	5	1.47	1.56	1.44
	30	1.45	1.54	1.42
	60	1.46	1.53	1.40
Max/Min, g/g	Before test	1.45/1.39	1.53/1.40	1.44/1.36
	0	1.73/1.47	1.87/1.50	1.67/1.39
	5	1.71/1.45	1.84/1.49	1.65/1.38
	30	1.64/1.39	1.76/1.45	1.58/1.36
	60	1.57/1.40	1.70/1.45	1.53/1.36

rameters specified in the standards concerning filtering respiratory protective devices, the systems developed exhibited very high performance levels.

Descriptive statistics concerning sorption testing in dynamic flow are given in *Table 8* and *Figure 3*.

Dynamic flow testing of moisture sorption showed a dramatic increase in the weight of all systems immediately following the trials. The highest increase (10%) was found for system II at 0 min. The corresponding values for systems I and III were 6.5% and 6.08%, respectively. Following storage under normal conditions, all the systems studied exhibited a gradual loss of weight attributable to fast evaporation of the water that had precipitated on the fibers and the water vapour that had been trapped in interfiber spaces.

In terms of moisture sorption, it has been shown that the composite nonwoven systems containing SAP absorb air moisture much better than those without SAP. This has been corroborated by works examining hygienic products designed for fast absorption and storage of body fluids (urine, blood) as well as inserts for protective footwear developed to improve user comfort and hygiene [17-19]. These results show new potential applications of nonwoven materials fabricated using different technologies and containing superabsorbent polymers in personal protective devices with a view to improved comfort of use.

The present study on the basic protective and functional parameters revealed that a range of different nonwoven structures designed for respiratory protective devices can be produced in one technological process.

Conclusions

The study presents innovative nonwoven with SAP additive materials fabricated in the course of one technological process. The individual nonwoven layers with varied morphological structures produced are designed to fulfill specific functions in multilayer systems. The modification of the nonwoven structure by the introduction of SAP directly in the fiber-forming area and by electrostatic activation may be successfully used for the production of filtering materials for air-purifying half-masks protecting the user's respiratory system in harsh working environments. The method of introducing SAP into the thermoplastic fiber stream developed makes it possible to partially embed grains of the modifier in fibers without hindering the functionality of the modifier or causing major changes to the fiber structure, while enabling an exact dosage of the modifier rather than its dispersion throughout the polymer matrix. Electrostatic activation by corona charge led to the physical modification of elementary fibers, which constitute the elementary material for nonwoven structures. The multi-purpose filtering material developed, obtained in the melt-blown process, is characterised by high filtering efficiency with low airflow resistance and good moisture sorption capacity.

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INSTITUTE OF BIOPOLYMERS AND CHEMICAL FIBRES

LABORATORY OF BIODEGRADATION

The Laboratory of Biodegradation operates within the structure of the Institute of Biopolymers and Chemical Fibres. It is a modern laboratory with a certificate of accreditation according to Standard PN-EN/ISO/IEC-17025: 2005 (a quality system) bestowed by the Polish Accreditation Centre (PCA). The laboratory works at a global level and can cooperate with many institutions that produce, process and investigate polymeric materials. Thanks to its modern equipment, the Laboratory of Biodegradation can maintain cooperation with Polish and foreign research centers as well as manufacturers and be helpful in assessing the biodegradability of polymeric materials and textiles.

The Laboratory of Biodegradation assesses the susceptibility of polymeric and textile materials to biological degradation caused by microorganisms occurring in the natural environment (soil, compost and water medium). The testing of biodegradation is carried out in oxygen using innovative methods like respirometric testing with the continuous reading of the CO_2 delivered. The laboratory's modern MICRO-OXYMAX RESPIROMETER is used for carrying out tests in accordance with International Standards.

The methodology of biodegradability testing has been prepared on the basis of the following standards:

■ **testing in aqueous medium:** 'Determination of the ultimate aerobic biodegrability of plastic materials and textiles in an aqueous medium. A method of analysing the carbon dioxide evolved' (PN-EN ISO 14 852: 2007, and PN-EN ISO 8192: 2007)

■ **testing in compost medium:** 'Determination of the degree of disintergation of plastic materials and textiles under simulated composting conditions in a laboratory-scale test. A method of determining the weight loss' (PN-EN ISO 20 200: 2007, PN-EN ISO 14 045: 2005, and PN-EN ISO 14 806: 2010)

■ **testing in soil medium:** 'Determination of the degree of disintergation of plastic materials and textiles under simulated soil conditions in a laboratory-scale test. A method of determining the weight loss" (PN-EN ISO 11 266: 1997, PN-EN ISO 11 721-1: 2002, and PN-EN ISO 11 721-2: 2002).

The following methods are applied in the assessment of biodegradation: gel chromatography (GPC), infrared spectroscopy (IR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM).