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# Synthesis of Zinc Oxide in an Emulsion System and its Deposition on PES Nonwoven Fabrics

**Abstract**
*Zinc oxide precipitated from an emulsion system was characterised by a narrow particle size distribution and large specific surface area. The product was subjected to morphological and dispersive evaluations, and its application possibilities were indicated. The precipitated zinc oxide can be used as a surface modifier of different types of textile products mainly because it shows considerable barrier properties protecting against UV radiation and antibacterial activity. These new properties open possibilities for many new applications.*

**Key words:** *zinc oxide, textile materials, UV barrier properties, plasma treatment, antimicrobial effect.*

## Introduction

Recently, much attention has been paid to new technologies, called "added-value-technologies", permitting the production of functionalised and so-called "intelligent textiles". The dynamic development of functionalised textile products is directly related to progress in chemistry and polymer processing, and in particular to nanotechnology. The functionalisation of textile products by the introduction of chemical nanomolecules into them or on the surface of their fibres has recently become the main direction of the development of textile product engineering, permitting the obtaining of products which show properties unachievable with the use of conventional technology. Research work on the functionalisation of textile products has focused on the endowment of new properties or combinations of properties, e.g. antibacterial, self-cleaning, absorbing UV radiation or deodorising.

Extremely promising agents for functionalisation are nanoparticles of metal oxides, in particular titanium dioxide and zinc oxide [1]. Zinc oxide, showing high absorption of UV radiation [2, 3] and antibacterial properties [4], is a very interesting modifier used in the textile industry [5] due to its prospective use for the production of safety garments (health service) and all kinds of fabrics for the construction industry [6]. Zinc oxide can occur in a variety of structures, including one-dimensional (e.g. nanowires, nanorods, nanorings or nanohelices) or two-dimensional ones (nanoplates, nanosheets), and can have a rich gamut of applications [7]. Literature gives many methods for obtaining zinc oxide with particles of different sizes and structures: the sol-gel process [8, 9], precipitation [10 - 12], the emulsion route [13, 14], the hydrothermal process [15, 16], the organometallic method [17], the thermal decomposition of a precursor [18, 19] and many others. The sol-gel, solvothermal or hydrothermal methods have some considerable limitations with respect to e.g. the scale of production and economical aspects. The most promising solution from the point of view of the functionality of the product is the modification of conventional precipitation methods.

This paper is concerned with the analysis of the process of ZnO synthesis by precipitation using the emulsion method. The process of precipitation was performed with the use of new (alternative) substrates to make the emulsion, and a new way of producing it was applied. This method ensures the obtainment of products of defined dispersive-morphological and adsorptive properties because of the high homogeneity of particles and their high surface activity. Moreover, ZnO species obtained by this method can be successfully used as barrier and antibacterial materials.

## ■ Experimental part

### ZnO synthesis

The emulsion was composed of cyclohexane as the organic phase, zinc acetate as the water phase and appropriate emulsifiers. With a water solution of zinc acetate, a mixture of cyclohexane and surfactants was added to a beaker in portions. Precipitation was carried out in laboratory scale in a reactor equipped with a homogeniser. The emulsion, composed of zinc acetate of suitable concentration, cyclohexane and a mixture of emulsifiers, was placed in a reactor and a water solution of potassium hydroxide was introduced to it in doses with the help of a peristaltic pump. The system obtained was destabilised at the boiling point of cyclohexane. Then the organic phase was separated by distillation, and the mixture was filtered off under reduced pressure. At the next stage the sample was washed with water and methanol to get rid of residues of the emulsifiers. A sample of zinc oxide was obtained after drying at 120 °C.

**Table 1.** *Conditions of the zinc oxide precipitation process.*

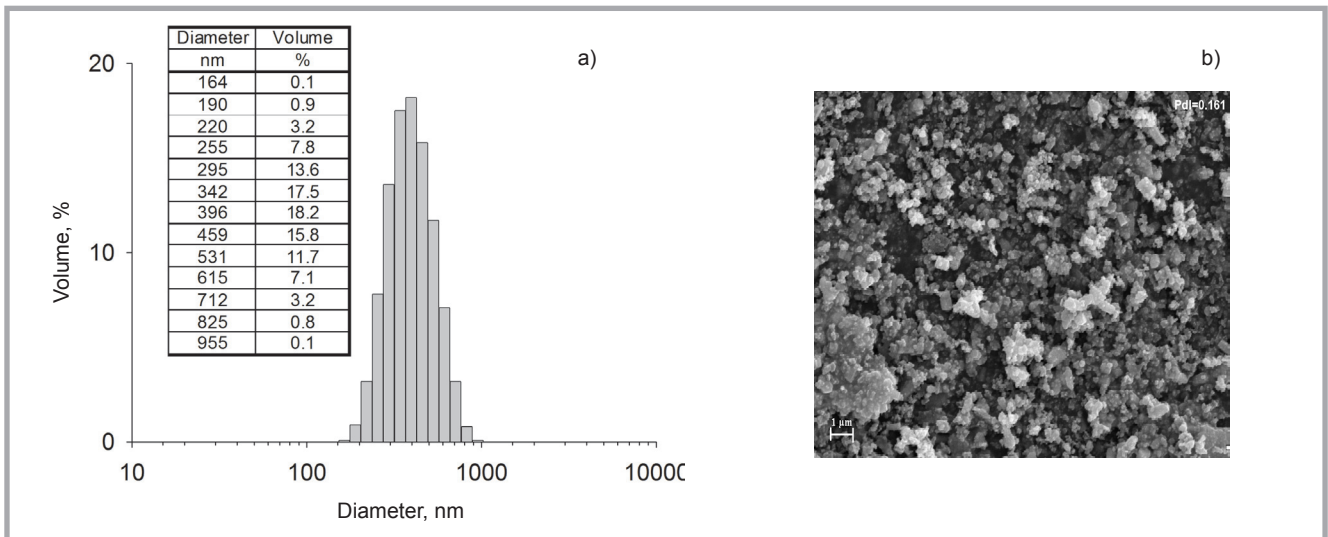| Sample no. | Zinc acetate concentration,% | Emulsifier mass ratio NP3/NP6 | KOH/cyclohexane volume ratio | Rate of dosing reagent, cm³/min |
|---|---|---|---|---|
| 1 (Z7) | 20 | 1/1.1 | 0.6/1 | 4 |
| 2 (Z8) | 10 | 1/1.1 | 0.6/1 | 30 |
| 3 (Z11) | 10 | 1/1.1 | 1/1 | 11 |
| 4 (Z14) | 15 | 1/1.1 | 0.9/1 | 4 |
| 5 (Z15) | 10 | 4.4/5.6 | 1/1 | 11 |

**Figure 1.** *a) Particle size distribution by volume and b) SEM microphotograph of zinc oxide – sample 3 (Z11).*

Basic details describing the precipitation conditions are presented in ***Table 1***.

**Physico-chemical characterisation**

The zinc oxide samples obtained using the emulsion precipitation method were subjected to the determination of their particle size distribution by the non-invasive backscattering technique (NIBS) using a Zetasizer Nano ZS, made by Malvern Instruments Ltd.

Cumulant analysis gives a width parameter know as the polydispersity, or the polydispersity index (PdI). Cumulant analysis is actually the fit of a polynomial to the log of the G1 correlation function:

$$Ln[G1] = a + bt + ct^2 + dt^2 + et^4 + \dots \quad (1)$$

The value of b is known as the second order cumulant, or the z-average diffusion coefficient. The coefficient of the squared term, c, when scaled as $2c/b^2$, is known as the polydispersity.

The adsorptive properties were measured by ASAP 2020 apparatus, made by Micromeritics Instruments Co. Classical measurement of the adsorptive properties (using the BET equation) was carried out using low temperature nitrogen adsorption based on the evaluation of the amount of adsorbate covering the adsorbent with a monolayer (from the adsorption isotherm). Analysis of the adsorption and desorption isotherms was carried out in stationary conditions, allowing to determine parameters such as the surface area ($A_{BET}$), total volume ($V_p$), and the mean size ($S_p$) of pores by applying the BJH method. The adsorbate

most often used to determine the adsorption and desorption isotherms is nitrogen at a temperature of 77 K. Measurement of the amount of gas adsorbed on a pre-degassed porous material is made for subsequent predetermined relative pressures (adsorption isotherm), leading to

desorption, with a subsequent decrease in the relative pressure $p/p_0$ (desorption isotherm). The $p_0$ value is measured at intervals during the analysis.

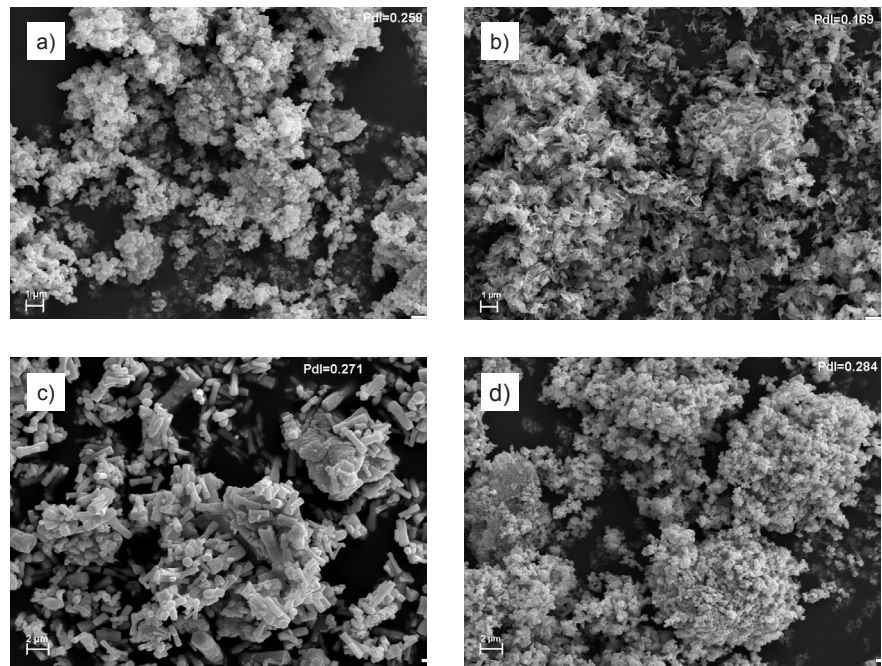The wettability was determined by a tensiometer K100, made by Krüss, with ap-



**Figure 2.** *SEM microphotographs of zinc oxide samples: a) 1 (Z7) b) 2 (Z8), c) 4 (Z14) and d) 5 (Z15).*

**Table 2.** *Dispersive characteristics of zinc oxide.*

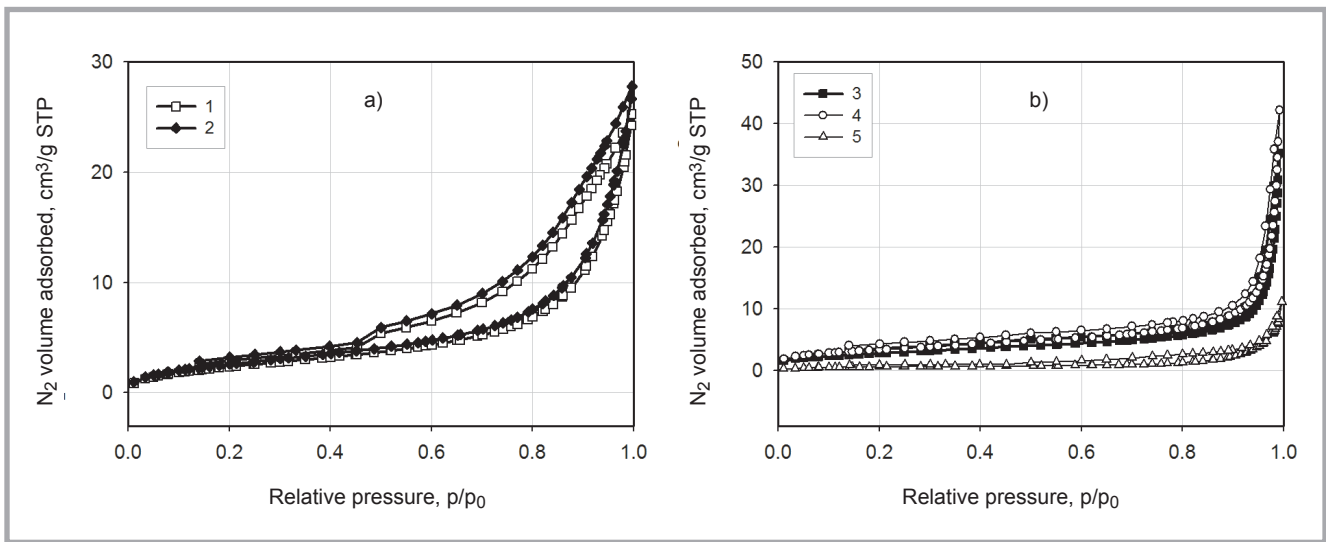| Sample no. | Range of diameter, nm | Max. volume / diameter,%/ nm | PdI |
|---|---|---|---|
| 1 | 342 – 955 | 27.4 / 531 | 0.258 |
| 2 | 295 – 825 | 27.4 / 531 | 0.169 |
| 3 | 164 – 955 | 18.2 / 396 | 0.161 |
| 4 | 396 – 825 | 32.2 / 615 | 0.271 |
| 5 | 825 – 6440 | 11.5 / 2670 | 0.284 |

***Figure 3.*** *$N_2$ adsorption/desorption isotherms of the zinc oxide samples examined; a) of sample 1 and 2, and b) of sample 3, 4 and 5.*
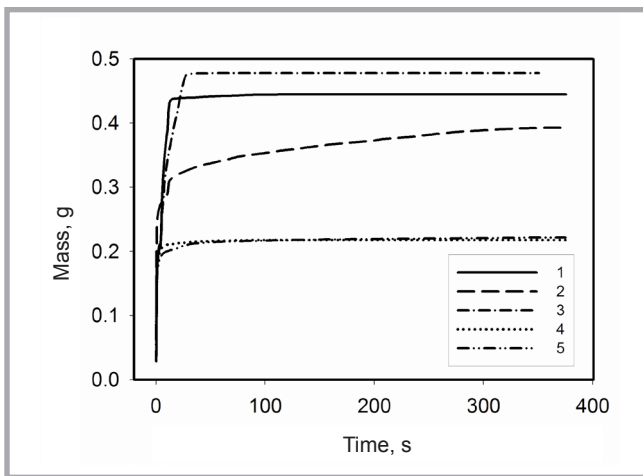


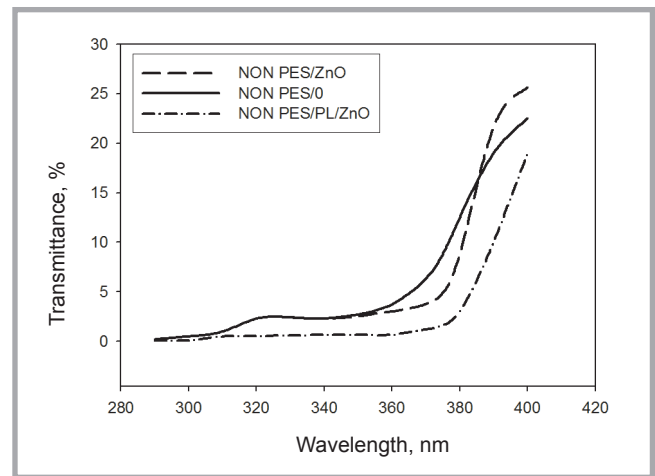***Figure 4.*** *Wettability curves of the ZnO samples.*



***Figure 5.*** *Transmittance spectra of polyester nonwoven samples containing zinc oxide – sample 3 (Z11).*

propriate equipment on the basis of the capillary penetration of water into a sample of ZnO.

## Modification of the nonwoven fabric

An important stage in the production of the modified textile products was the preparation of the nonwoven fabric support. In order to improve the adhesive properties of textile products towards ZnO, the surface of the textile products selected was treated in low-temperature plasma (cold plasma). The treatment was performed in a reactor - Tetra 30 (made by Diener Electronic) during RF discharge (13.56 MHz), and plasma was generated in carbon dioxide. The treatment lasted for 5 seconds.

## Coating of polyester nonwoven fabric

Firstly pastes of homogeneous dispersion and appropriate viscosity were prepared based on acryl resins (10% wt.), containing micronised zinc oxide (3% wt.), a densifying agent (3% wt.), wetting agent (10% wt.) and water to make 100%. The paste was deposited onto the nonwoven support with the use of a special kit for the deposition of nanostructural coatings made by MATHIS, type KTF-350S. The thickness of the coating was controlled by the width of the supply gap; a width of 0.05 mm was used. The samples were dried and heated at 120 °C for 6 minutes.

## Estimation of UV radiation barrier properties

The UV barrier properties of the modified textile products, expressed in terms of the Ultraviolet Protection Factor (UPF), were evaluated on the basis of UV transmittance measurements by the spectrophotometric method using a double beam a V-550 type Jasco UV-Vis spectrophotometer equipped with an integrating head. The UPF value was calculated

***Table. 3.*** *Adsorptive properties of the zinc oxide obtained.*

| Sample no. | Secific surface area $A_{BET}$, m²/g | Total volume of pores $V_p$, cm³/g | Mean size of pores $S_p$, nm |
|---|---|---|---|
| 1 | 7.5 | 0.006 | 2.7 |
| 2 | 7.8 | 0.006 | 2.7 |
| 3 | 8.6 | 0.006 | 2.9 |
| 4 | 10.8 | 0.008 | 3.1 |
| 5 | 3.7 | 0.005 | 2.4 |

***Table 4.*** *Comparison of the amount of zinc oxide deposited and the UPF value for the samples of coated polyester non-woven fabric; W0 – polyester nonwoven - raw sample; W – polyester nonwoven; PL – after plasma pre-treatment.*

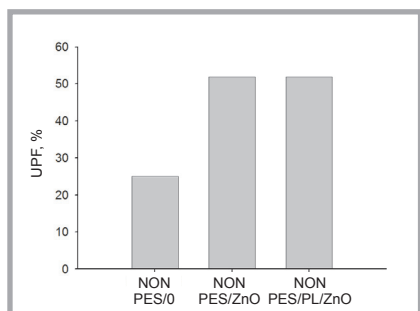| Sample type | Amount of zinc oxide, g/m² | UPF |
|---|---|---|
| W0 | 0.00 | 25 |
| W/Z11 | 5.61 | 50 |
| W/PL/Z11 | 9.03 | 50 |

***Figure 6.*** *UPF values of polyester nonwoven fabric: samples with zinc oxide incorporated.*

according to PN-EN 13758-1:2005 from the formula (1):

$$UPF = \frac{\int_{\lambda=290}^{\lambda=400} E(\lambda)\varepsilon(\lambda)d(\lambda)}{\int_{\lambda=290}^{\lambda=400} E(\lambda)\varepsilon(\lambda)T(\lambda)d(\lambda)} \quad (1)$$

where: $E(\lambda)$ – the solar irradiance, $\varepsilon(\lambda)$ – the erythema action spectrum (measure of the harmfulness of UV radiation for human skin, connected with the wavelength; the erythemal effectiveness is highest at about 290–310 nm), $\Delta\lambda$ – the wavelength interval of the measurements, and $T(\lambda)$ – the spectral transmittance at wavelength $\lambda$.

The UPF value of the textile products was determined as the arithmetic mean of the $UPF_i$ values, measured for all samples and reduced by a statistical value dependent on the number of measurements made at a confidence level of 95%.

**SEM analysis of the surface of the textile products**
The surfaces of PES nonwoven fabric after the deposition of paste with ZnO were analysed using SEM images taken with a scanning electron microscope made by FEI, model Quanta 200F. Because of the insulating properties of the fabrics studied, the measurements were made under a steam pressure of 100 Pa to avoid the necessity of covering the surface with a thin film of a conductor. SEM micro-

photographs provided information on the dispersion and morphology of particles and on tendencies towards agglomeration (SEM VO40 made by Zeiss).

To estimate the amount of zinc oxide deposited on the textile product, quantitative analysis of a selected fragment of the material was performed with the help of a microscope attachment - Quanta 200F (EDS type, made by Oxford Instruments, equipped with a detector - X-Max 50). The results are given as the number of zinc atoms per 100 carbon atoms.

**Evaluation of the antimicrobial properties of polyester nonwoven fabric modified with ZnO**
The antimicrobial properties of the modified polyester nonwoven fabric were evaluated by the screening method, according to PN-EN ISO 20645 (diffusion test on a plate with agar), on the basis of the presence and size of the zone inhibiting the growth of a given bacteria around the fabric sample tested and the growth of bacteria or its absence in the contact zone between the sample and medium. Tests were performed for two strains of bacteria: gram positive *Staphylococcus aureus* (ATCC 6538) and gram negative *Escherichia coli* (ATCC 11229).

## ■ Results and discussion

Results of the dispersive–morphological study of zinc oxide samples obtained by precipitation using the emulsion method are given in ***Figures 1*** & ***2,*** and ***Table 2***. Analysing the characteristics of zinc oxide (***Table 2***), it was noted that the properties of ZnO significantly depend on the parameters of the precipitation process. The samples obtained were characterised by particles with diameters ranging from 164 to 6440 nm and a low polydispersity index.

Zinc oxide – sample 3 (Z11) has particles of the smallest size. The particle size distribution according to the volume contribution (***Figure 1.a***) shows a single band covering a diameter range of 164–955 nm, and the maximum volume of 18.2% is taken by particles of 396 nm in diameter. Because of a low polydispersity index of 0.161, one band in the particle size distribution of this sample has good dispersive properties, which is confirmed by the SEM image shown in ***Figure 1.b***.

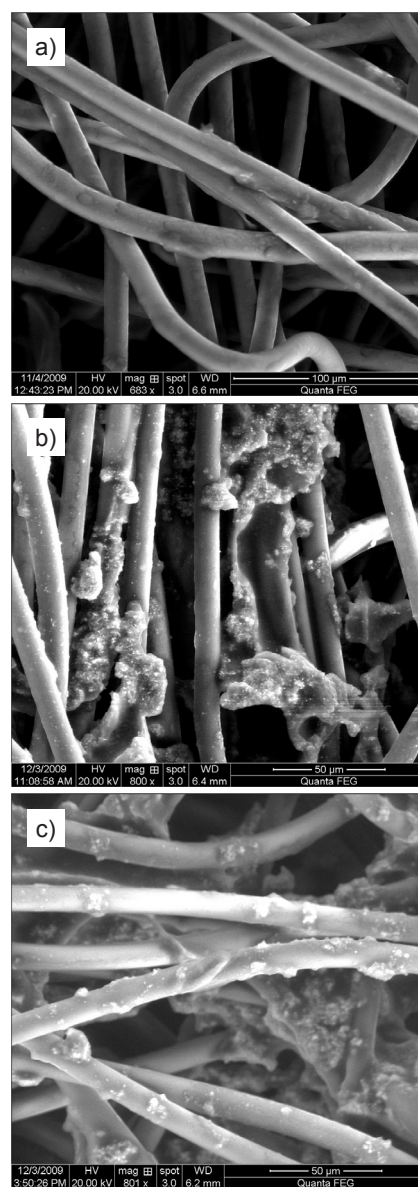Other samples are characterised by much larger particles, respectively: 1 – 531 nm,

***Figure 7.*** *SEM microphotographs of polyester nonwoven fabric: a) unmodified, b) grafted with zinc oxide, and c) subjected to pre-treatment with plasma and coated with zinc oxide Z11.*

2 – 531 nm, 4 – 615 nm and 5 – 2670 nm, as well as by a lower polydispersity (1 – 0.258, 2 – 0.169, 3 – 0.271 and 5 – 0.284), as can be seen in the SEM images (***Figure 2***).

At the subsequent stage of the study, the surface areas of the ZnO samples produced were determined by recording the nitrogen adsorption/desorption isotherms, see ***Figure 3***.

The shape of the isotherms testifies to the mesoporous structure of ZnO. For all samples the amount of nitrogen adsorbed smoothly increases up to $p/p_0 = 0.8$. Above this value of relative pressure
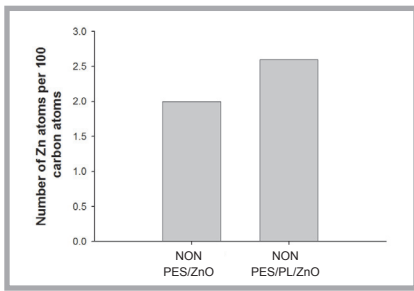
***Figure 8.*** *Content of zinc on the surface of polyester fabric coated with paste containing ZnO with or without plasma pre-treatment.*

the amount of nitrogen rapidly increases; for samples 3 and 4 it reaches a maximum of 35 – 40 $cm^3$/g at $p/p_0 = 1$. For ZnO – sample 5 the amount of nitrogen at $p/p_0 = 1$ is the lowest, equal to only 10 $cm^3$/g, whereas for samples 1 and 2 it is close to 30 $cm^3$/g.

The largest surface areas (BET) - 8.6 $m^2$/g and 10.8 $m^2$/g are characterised by samples 3 and 4, respectively, whereas the smallest surface area - 3.7 $m^2$/g is represented by sample 5, which also has the largest particles from among all the samples precipitated. The resulting high surface area of the ZnO prepared has a better bond with the nonwoven. The ***Table 3*** presents the following parameters: surface area ($A_{BET}$), total volume ($V_p$) and mean diameter ($S_p$) of pores.

As far as the wettability of the ZnO samples obtained is concerned, the most hydrophobic sample was the one precipitated in sample 1 (***Figure 4***). The most hydrophilic proved to be those precipitated in samples 4 and 5.

The criteria of choosing samples for further study were the particle size and surface area. The samples of zinc oxide containing small particles and characterised by a large surface area can be used for the modification of nonwoven fabric. Applying these criteria, sample 3 was selected and applied for fabric modification. Furthermore, sample 3 has the most hydrophobic character, which also gives it a higher affinity to nonwovens.

***Table 4*** presents the characteristic of the polyester nonwoven fabric samples coated with the paste containing zinc oxide Z11 (samples 3), expressed as the amount of ZnO deposited and as a UPF value. The amount of ZnO deposited on polyester nonwoven fabric after preliminary treatment with low-temperature plasma is considerably greater than in the case of unmodified samples.

The transmittance spectra of polyester nonwoven sample containing zinc oxide (sample 4) are presented in ***Figure 5***.

***Figure 6*** presents the values of UPF (*Ultraviolet Protection Factor*) for the unmodified and modified samples of nonwoven fabric. The UPF value for the unmodified polyester fabric was 25, but after modification with ZnO its value increased above 50 for PES nonwoven fabric with and without plasma pre-treatment.

SEM microphotographs of polyester nonwoven fabric coated with Z11 with and without preliminary treatment with plasma are shown in ***Figure 7***. The surface of unmodified polyester nonwoven fabric is smooth, see ***Figure 7.a***, but has small particles in some places, probably impurities. Single fibres of nonwoven polyester fabric modified with Z11 (***Figure 7.b***) have the same parameters as those of the unmodified sample; their smooth surface is covered with dispersed particles of ZnO. The diameter of these particle aggregates reaches about 350 nm.

The sample of nonwoven polyester fabric coated with a paste containing zinc oxide was subjected to the determination of its elemental surface composition, expressing the content of ZnO as the number of Zn atoms per 100 carbon atoms. For PES nonwoven fabric coated with ZnO without plasma pre-treatment, the content of Zn was 2.0, while for PES coated with zinc oxide and pre-treated with plasma it was 2.6, see ***Figure 8***.

Activation with low-temperature plasma increases the amount of carboxylic groups on the surface of polyester fibres, resulting in the improvement of adhesive properties and, hence, in the attachment of a greater amount of ZnO particles to the fabric surface, see ***Table 4*** and ***Figure 8***.

The antibacterial properties of polyester nonwoven fabric modified with zinc oxide of particles of a range 160–955 nm were tested against *Staphylococcus aureus* (ATCC 6538) and *Escherichia coli* (ATCC 11229). After pre-treatment with plasma, the samples revealed high antibacterial activity against *Escherichia coli* (ATCC 11229) on the coated and uncoated side.

The antibacterial activity of the samples tested is presented in ***Table 5***,

## ■ Conclusions

The proposed method of ZnO precipitation in an emulsion environment permits the obtainment of products of defined shape (particle spheres, wires). Such products are built of particle aggregates whose diameters vary in the range 160–955 nm. The surface area of the zinc oxide obtained varies from 3.7 to 10.8 $m^2$/g. As follows from the results obtained, (SEM and EDS analyses) the preliminary treatment of nonwoven fabric with low-temperature plasma resulted in the attachment of a considerably greater amount of micronised zinc oxide particles to the fabric. Products from fabric preliminary treated with cold plasma and coated with paste containing ZnO can be classified as protecting against UV radiation. Apart from the improvement of UV barrier properties, such products also show antibacterial activity. New textile products characterised by UV barrier and antibacterial properties can find a wide range of applications in the construction

***Table 5.*** *Antibacterial activity; A – uncoated side; B – coated side; W0 – polyester nonwoven - raw sample; W – polyester nonwoven; PL – after plasma pre-treatment.*

| Bacteria | Sample type | | Bacteria growth inhibition zone – H, mm - growth assessment | Assessment |
|---|---|---|---|---|
| *Escherichia coli* (ATCC 11229) gram negative | W0 | A, B | 0 heavy | |
| | W/Z11 | A | 0 moderate | insufficient effect |
| | | B | 1 none | good effect |
| | W/PL /Z11 | A | 0 slight | limit of efficacy |
| | | B | 0.1 none | good effect |
| *Staphylococcus aureus* (ATCC 6538) gram positive | W0 | A, B | 0 heavy | |
| | W/Z11 | A | 0 moderate | insufficient effect |
| | | B | 0.8 none | good effect |
| | W/PL /Z11 | A | 0.5 none | good effect |
| | | B | 0.8 none | good effect |

industry, furnishings and protective garments (medical personnel, catering establishments).

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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).

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

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