METHOD FOR PRODUCING NANOFIBRE LAYERS INCORPORATING PARTICLES

Jiří CHVOJKA[a, b], Petr MIKEŠ[a], Jiří CHALOUPEK[a], Filip SANETRNÍK[a], Eva KUŽELOVÁ KOŠŤÁKOVÁ[a], Julie SOUKUPOVÁ[a], Lenka BLAŽKOVÁ[a], Radek JIRKOVEC[a], Ivan ULMAN[a], David LUKÁŠ[a]

[a] *Technical University of Liberec, Faculty of Textile Engineering, Department of Nonwovens and Nanofibrous Materials, Liberec, Czech Republic, EU*

[b] *Technical University of Liberec, Centre for Nanomaterials, Advanced Technologies and Innovation, Liberec, The Czech Republic, EU*

jiri.chvojka@tul.cz

Abstract:

A new technology for production of unique nanofibrous composite materials is presented here. This article aims with the continual incorporation of nano and micro particles by ultrasonic dispergation in-situ into a nanofibrous matrix produced by electrospinning. The technology of a nanocomposite material production based on usage of needleless electrospinning method in combination with ultrasound enhanced sputtering of nano or micro particles in between nanofibers depositing onto support material. Also the scanning electron images of the final nanocomposite materials are presented. These materials can be used for drug delivery, controlled scaffold degradation for tissue engineering and cell proliferation or wound covers as well as for many special technical applications in filtration or sorption.

Keywords: Nanofiber layer, incorporated particles, electrospinning, composite material

1. INTRODUCTION

Composite materials are roughly defined as heterogenous system consisted of at least two phases with different properties. The combination of these phases represents material with new properties or provides enhanced properties by so called synergic effect. [1] In this article we introduce technology for production of unique composite nanofibrous material. The key of technology and material production is needle less electrospinning technique [2] in combination with micro particles. Needle less electrospinning machine called Nanospider [3] is widely used for production nanofibrous mats or nanofibrous layers. These layers are consisted of sub micron fibers which can be randomly or parallel deposited. [4] Thanks to small fiber diameter and huge surface area are nanofibrous mats used as a filter media in medical application or automotive. Improve the filtration properties could be achieved by incorporated particles as an active carbon. The method for incorporating particles is described in [5-8]. The composite material consists of nanofibers and micro particles which are in-between fibres. Ultrasonic vibrations are applied on particles which are then captured in-between deposited nanofibers. Advantage of using of ultrasound is the dispergation of agglomerated particles. This process is continual and multilayered material is created. Described technology could be in the future transferred to the industrial manufacture. The main advantage of this technology is variability.

2. EXPERIMENT

Polymer for electrospinning process was used the poly(vinylbutyral) (PVB 60H, Kuraray). The PVB is usualy used for applications as a binder and for adhesion to many surfaces. Chemical properties of PVB are good optical clarity, toughness and flexibility. Preparation process PVB from polyvinyl alcohol by the reaction with butyral aldehyde. The polymer powder was dissolved in the solvent ethyl alcohol. The final concentration of polymer solution was 10wt% PVB and it was prepared 100g of this solution. The process parameters, are voltage, distance between spinner and collector, air humidity and temperature. These parameters are described below. The temperature during the experiment was 21 $\pm$2°C and the air relative humidity was 65$\pm$5%. As a sources of positive and negative high voltage, the 300 Watt High Voltage DC Power Supplies with regulators; model number SL 150; manufactured by Spellman High Voltage, INC. with output parameters: 0-50 kV, 6mA, were used. Positive voltage of 32 kV was applied on the roller (spinning electrode) and negative 12 kV on the belt collector. The distance between the spinner and collector was kept on 18 cm. The rotational speed of the Interroll zone control was 0,01m/s and the belt was 80 cm length and 20 cm wide. The duralumin sonotrode with length 18 cm and wide 1 cm, produced by Ultratech Company and 20 kHz Sonic Digital ULC generator produced by Weber Ulrasonics with power 400W.

Particles are stored in the reservoir and continually falling into the grooves and then transported to the edge of ultrasound sonotrode where they are homogenously dispersed (deagglomerated) by the mechanical movement of the ultrasound vibrations. The dosage of particles can be controlled by the rotational speed of the grooved cylinder. The particles are subsequently covered by nanofibers. The set-up of machine used for the composite material combining nanofibers and particles is shown in **Fig. 1**. Technology is patented as A method of forming a functional nanofiber layer and the device for implementing the method [9]

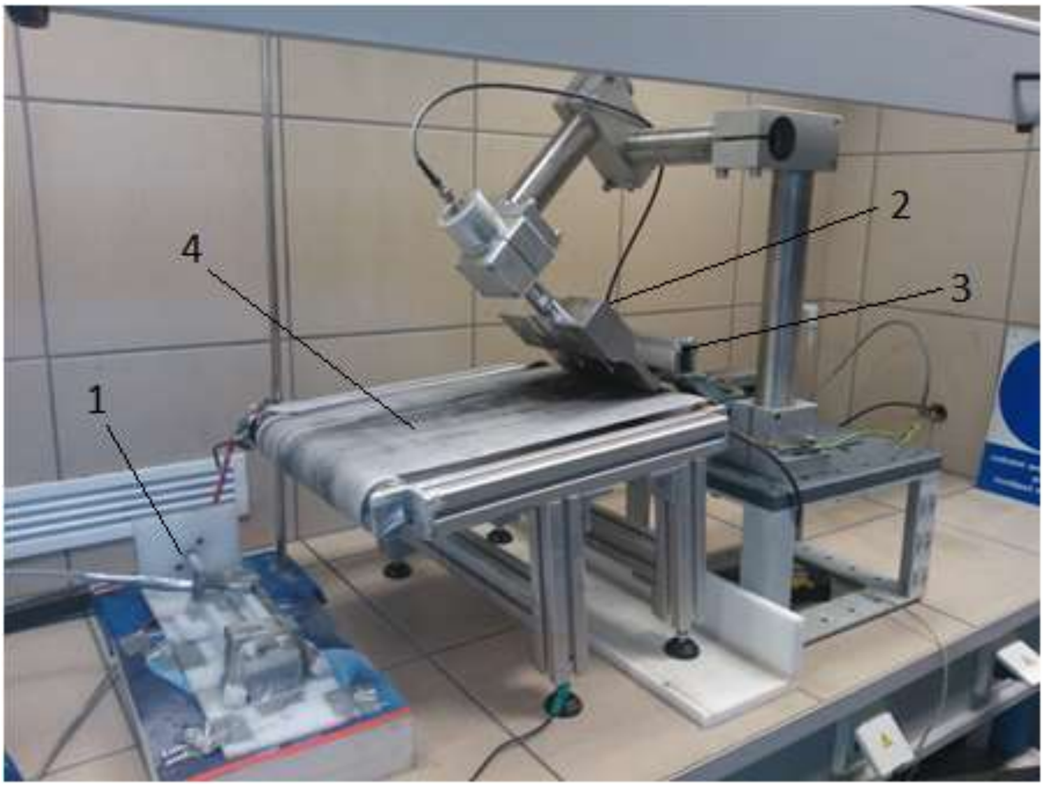

Fig. 1 The production of electrospun layers with incorporated particles set up: 1) cylinder needle-less eelctrospinning (roller rotating inside polymer solution bath as spinning electrode); 2) sonotrode disintegrating particles of active coal; 3) active coal dosing equipment; 4) collector and conveyor

3. RESULTS AND DISCUSSION

Scanning electron microscope (SEM) images of the produced materials were used for subsequent morphology analysis. The analysis monitored the impact of changes in surface density of the material on the fiber diameter and the influence of sampling site on layers of fiber diameter (edges of the specimen or center of the sample). According results no significant difference in between these measurements was measured see **Fig. 3**. Produced electrospun composite materials were laminated in between two spunbond nonwoven materials. The lamination was done by machine Meyer RPS mini with continuous downforce 10 Ncm^{-2}, rate 2 ms^{-1} and temperature 135 °C. These materials should be used as a filters and the amount of particles loose during loading. Thus measuring the final material weight loss due to the air flow was carried out by the filter plant for flue gas filters. The materials were tested here at 22 °C, air flow rate 5 ms^{-1}, time duration 30 minutes and sample diameter 15 cm. The laminated materials images are shown in **Fig. 4**. The final weight loss after the air flow loading was: *1* – 1.10 %; *2* – 1.02 %; *3* – 1.37 %.

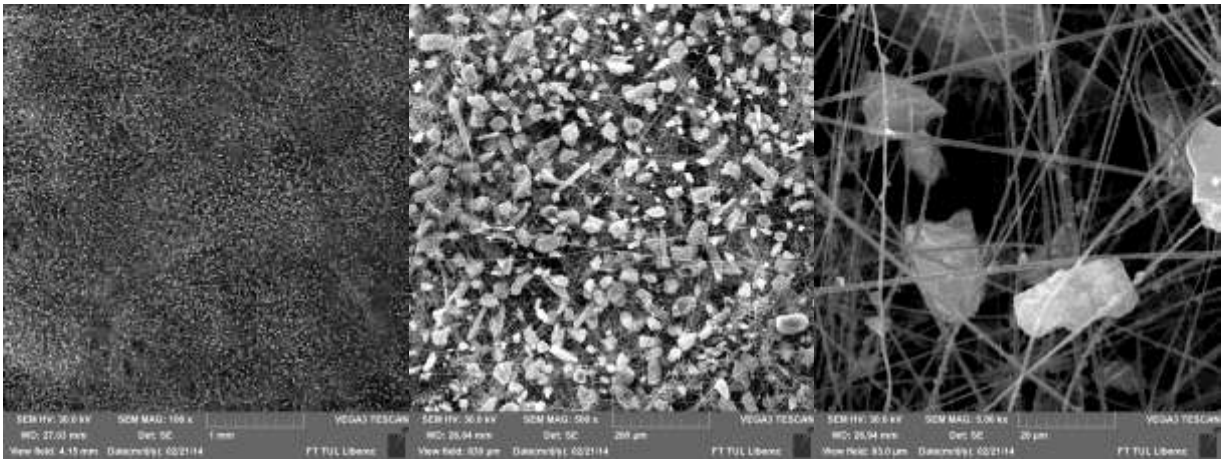

Fig. 2 SEM images at different magnification (scale bar from left 1mm, 200μm and 20μm) presenting structure of the produced materials

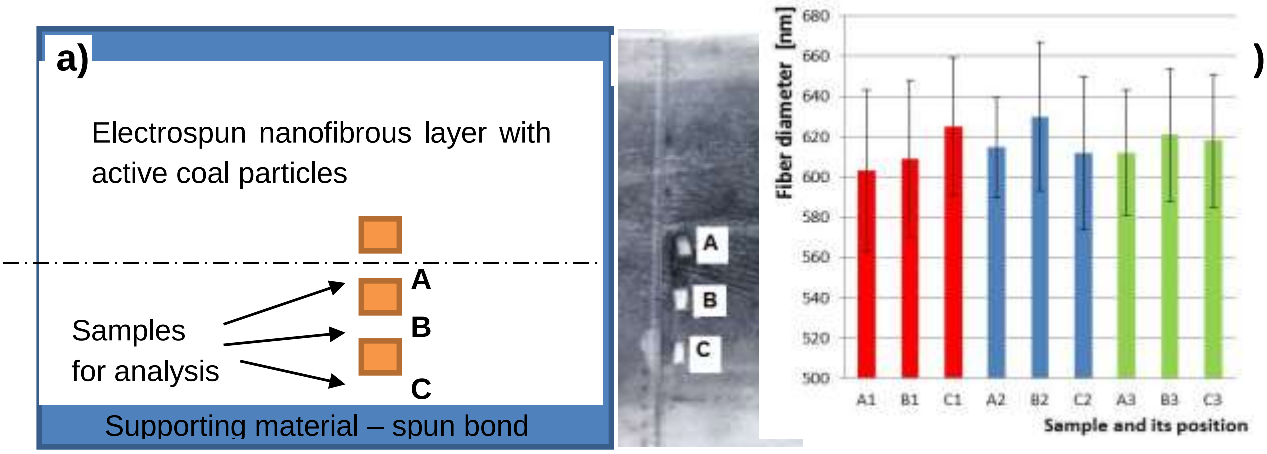

Fig. 3 Scheme of sampling (a); a photo of sampling (b) and graph introducing influence of sampling and surface density of produced materials onto average fiber diameter (1 – 5,85gm^{-2}; 2 – 6,31gm^{-2};3- 7,43gm^{-2})

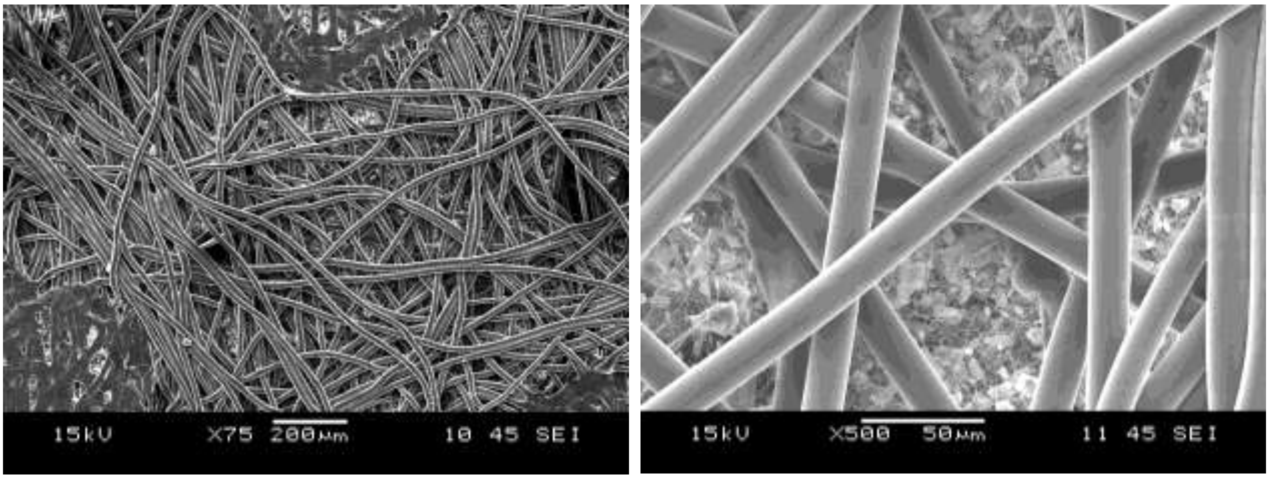

Fig. 4 Electrospun nanofibrous composite with integrated active coal particles laminated into two spunbond nonwoven materials

4. CONCLUSIONS

The produced materials are prepared for transformation into industrial production. The morphology analysis demonstrated uniform distribution of fibres diameter over one sample and in between different surface density samples. The measurement of weight loss during the air flow loading showed the sufficient stability of laminated samples for standard applications.

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LITERATURA

[1] HUSSAIN F, HOJJATI M. Polymer-matrix Nanocomposites, Processing Manufacturing, and Application: An Overview, Journal of composite materials, Vol. 40, No.17/2006, 6-13pp

[2] LUKAS D, SARKAR A, POKORNY P. Journal of Applied Physics 2008; 103; 1-7pp.

[3] JIRSAK O, SANETRNIK F, LUKAS D, KOTEK V, MARTINOVA L, CHALOUPEK J. US Patent 2005, WO2005024101.

[4] ANTONY L, ANDRADY, Science and Technology of Polymer Nanofibres, Wiley, September 2008, ISBN: 978-0-471-79059-4, 266-274pp

[5] MOGHE A K, GUPTA B S. *Co-axial electrospinning for nanofiber structures: Preparation and applications, Polymer Reviews 48, 353-377* (*2008*).

[6] MICKOVA A, BUZGO M, BENADA O, RAMPICHOVA M, FISAR Z, FILOVA E. et al. Core/shell nanofibers with embedded liposomes as a drug delivery system, Biomacromolecules 2012; 13; 4; 952-962pp.

[7] YANG P, ZHAN S, et al. The fabrication of PPV/C60 composite nanofibers with highly optoelectric response by optimization solvents and electrospinning technology, Materials Letters, 2011; 65:3:537-539pp

[8] KIM G M, MICHLER G H, PÖTSCHKE P. Fabrication of Bio-nanocomposite Nanofibers Mimicking the mineralized Hard Tissues via Electrospinning Process, Polymer 2005;46:7346-51pp

[9] POKORNY P, LUKAS D, a kol. Způsob vytváření funkční nanovlákenné vrstvy a zařízení k provádění způsobu, patent CZ 302901, 2011-328