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2nd WORKSHOP ON MECHANICS OF NANOMATERIALS

Liberec 2019

Recenzent: Mgr. Čeněk Jirsák (Katedra aplikované matematiky, FP, TUL)

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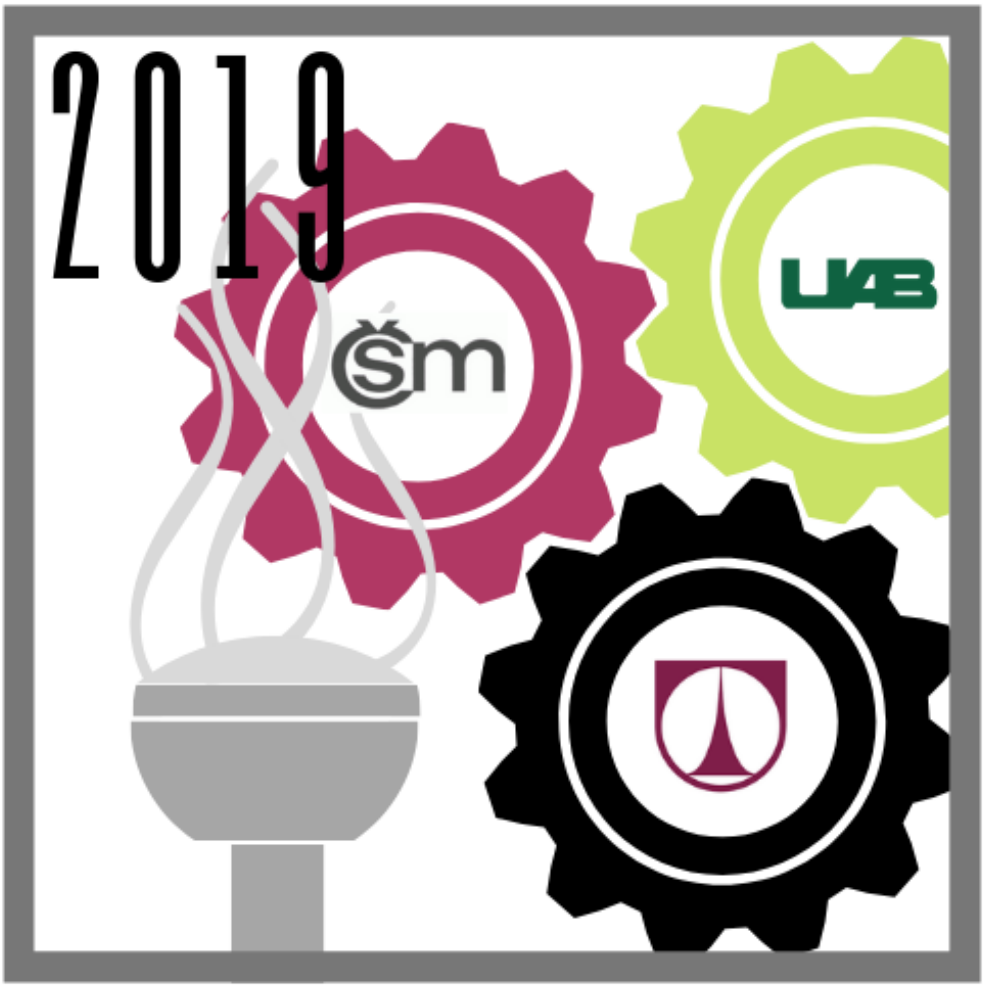
2nd Workshop on Mechanics of Nanomaterials

Characterization and Applications of Nanofibrous Materials and Electrospinning Processes



12th – 13th June 2019

Organizers: Courtney Severino, Hannah Lacy, David Lukáš

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Location and Details

Klášter Hejnice - vzdělávací, konferenční a poutní dům
č.p. 1
CZ 463 62 HEJNICE
Česko
telefon: +420 482 360 211
mobil: +420 603 459 684
http://www.klasterhejnice.cz
recepce@klasterhejnice.cz

Ceny ubytování (včetně bufetové snídaně)
Prices of accommodation (including buffet breakfast)

Klášter Hejnice nabízí 14 jednolůžkových pokojů, 21 dvojlůžkových pokojů, 2 třílůžkových pokojů
a 1 apartmán.
Všechny pokoje jsou vybaveny sociálním zařízením, chladničkou a televizí.

Prices of accommodation (including buffet breakfast)

Hejnice Monastery offers 14 single rooms, 21 double rooms, 2 triple rooms
and 1 apartment.
All rooms are equipped with bathroom, refrigerator and TV.

	1 osoba / noc	**2 osoby / noc**	**3 osoby / noc**
Jednolůžkový pokoj	820 Kč		
Dvoulůžkový pokoj	820 Kč	1 440 Kč	
Třílůžkový pokoj	820 Kč	1 440 Kč	2 160 Kč
Apartmán	2 720 Kč	2 740 Kč	

	1 guest / night	**2 guests / night**	**3 guests / night**
single room	820 Kč		
double room	820 Kč	1 440 Kč	
Three-beded room	820 Kč	1 440 Kč	2 160 Kč
Apartment	2 720 Kč	2 740 Kč	

Stravování

- Oběd - 150 Kč
- Večeře - 120 Kč
- Coffeebreak – cena v rozmezí od 65 Kč do 145 Kč
- Bezlepková a bezlaktózová dieta je za příplatek 50 Kč / den
- Vegetariánská kuchyně je součástí cenové nabídky

Catering

• Lunch - 150 CZK
• Dinner - 120 CZK
• Coffeebreak - price from 65 CZK to 145 CZK
• Gluten-free and lactose-free diet for an extra 50 CZK / day
• Vegetarian cuisine is part of the quotation

The monastery in Hejnice was founded in 1692 by a pilgrimage Gothic church built in the 14th century, where two angels were to appear to a poor craftsman in a dream. The first written mention of the local church dates back to 1364 and pilgrims from a wide area come to Hejnice at the same time.

In 1558, Emperor Ferdinand I sold the estate to the Protestant noble family Redern, who, under the imperial law of the Cuius regio, had all the churches turned into Protestant in his estate. However, the Hejnice church decided to close it due to numerous Catholic pilgrimages from the surrounding estate. The church then deteriorated and burned down, but the statue of the Virgin Mary escaped the ruin due to the fact that it was taken to the Liberec castle.
The church was reopened by the Duke Albrecht of Wallenstein, the generalissimo of the imperial army. When the Duke of Wallenstein was murdered in Cheb in 1634, Gallas acquired the local estate in 1636.

Foundation of the monastery:
It was Count Franz Ferdinand of Gallas who decided to initiate the founding of the monastery and invite the Franciscan Order brothers to his estate. Under the management of Gallas (later Clam-Gallas), Hejnic's renown reached its peak. Some pilgrims reportedly gathered up to 7,000 believers at that time. The yearly attendance of the place in the 17th century was around 80 000 pilgrims [1] [2] and the number of them increased, therefore in 1692 Count František Ferdinand of Gallas decided to found a monastery here, so that the monks could serve pilgrims as well as material.

The construction of the square monastery took place between 1692 and 1696 according to the design of a Czech-Italian architect named Marco Antonio Canevalle. When Count Franz Ferdinand died on 4 January 1697, his wife Johan Emerencian continued to expand the complex.

Under the apparition site, a three-part family tomb was built in 1698 under the supervision of Jan Václav of Gallas. One of the crypts belongs to the Gallas family, the second to Clam-Gallas' successors and the Franciscan third order brothers.

Agenda

June 12th

09:00 – 10:00	**Arrival and registration**
10:00-10:15	**Introduction** (Hannah Lacy and David Lukáš)
10:15-11:15	**First Session** [Věra Jenčová] – Not able to attend [Riley Yager, UAB] [Nikifor Asatiani, TUL] – Abstract not available [Sarah Nealy, UAB] [Manikandan Sivan, TUL] – Abstract not available
11:15-11:35	**Coffee Break**
11:35- 12:35	**Second Session** [Hannah York, UAB] [Micah Armstrong, UAB] [Rachid Bowles, TROY] [Abi Jernigan, USA]
12:35-13:45	**Lunch**
13:45- 18:30	**Small trip**
18:30- 19:30	**Dinner**

19:30-22:00	**Closing Discussion**

June 13th

09:00-10:00	**Breakfast**
10:00-11:15	**Third Session** [Micah Armstrong, UAB] [Eva Kuželová Košťáková, TUL] [Andrii Shynkarenko, TUL] [Dekel Azulay, TUL] [Amanda Duplain, UAB]
11:15-12:15	**Lunch**
12:15 -	**Trip**

Abstracts
First Session

Preparation and Properties of Electrospun Poly(ε-caprolactone)/Polyvinyl alcohol Hybrid Fibrous Materials

Věra Jenčová[1], *Jiří Oberreiter*[2], *Petr Mikeš*[1], *Šárka Hauzerová*[2], Jana Mullerova[1], Filip *Sanetrník*[2] *a Eva Kuželová Košťáková*[1]

[1]Department of Chemistry, Faculty of Humanities, Science and Education,
[2]Department of Nonwovens and Nanofibrous Materials, Faculty of Textile Engineering,
Technical University of Liberec

Introduction: The production of blended fiber materials from two kinds of polymer fibers via electrospinning is quite complicated in manufacturing technology. Typically, the need for a mixture of the two types of fibers in the resulting layer is bypassed using a method that allows each fiber type to be spun individually, then "stacking" the individual fiber layers in an alternating manner that creates the desired mixture of fiber types. The majority of published results concerning the combination of hydrophobic and hydrophilic electrospun fibrous materials is produced by needle electrospinning technologies (Maheshwari, Kumar, Nagiah, Uma 2013; Du, Xu, Li, Zhang, Zou 2016; Zahedi, Rezaeian, Jafari, Karami 2013). The main differences in these results are only in the arrangement of the needles (spinning electrodes) relative to the collector (usually in the form of a rotating cylinder). These technologies are referred to as Dual jet spinning, Multi-syringe electrospinning, and Co-electrospinning. In this study, the blend of polycaprolactone and polyvinyl alcohol fibers allows for interesting final properties of the layers produced. The blended layers are biocompatible and have optimum wetting characteristics. This paper presents the technology of producing such blended electrospun fiber layers, as well as their basic characterization with regard to their potential use as wound covers.

Materials and Methods: Polycaprolactone (PCL; Mn 45,000; Sigma Aldrich) was dissolved in mixture of chloroform/ethanol in ratio 8:2 by weight. Polyvinylalcohol (PVA, Mw 125,000; Kuraray) was dissolved in mixture of distilled water and ethanol in ration 8:2. Final concentrations were 16wt% PCL and 10wt% PVA. Dual jet DC spinning and needle-less electrospinning from two wires (modified spinning electrode in Nanospider ™) were used for production of pure PVA and PCL nanofibrous layers and PCL/PVA hybrid nanofibrous layers. Analysis of the nanofibrous layers was done using SEM to determine fiber diameter and morphological differences. Composition differences were determined using FTIR and contact angle measurements. The MTT viability assay on 3T3 mouse fibroblasts was also performed for selected samples.

Results: According to SEM images, electrospun materials are not beadless. Defects seen in the studied electrospun layers are typical for PCL and PVA within these types of solvents. Measurement of PCL fiber diameters showed the usual bimodal distribution, which was also seen in hybrid electrospun PCL/PVA fibrous layers. FTIR analysis revealed both types of base fibers within the composition, including both PCL and PVA within hybrid layers. For needle versus needle-less spinning this ratio is slightly different and must be explored further in future studies. MTT assay confirmed that the hybrid fibrous material is not cytotoxic and is suitable for cell growth. Contact angle measurements confirmed the hydrophobic behavior of pure PCL fibrous material, hydrophilic behavior of pure PVA fibrous electrospun layers, and the optimal wetting of hybrid PCL/PVA fibrous electrospun layers by distilled water.

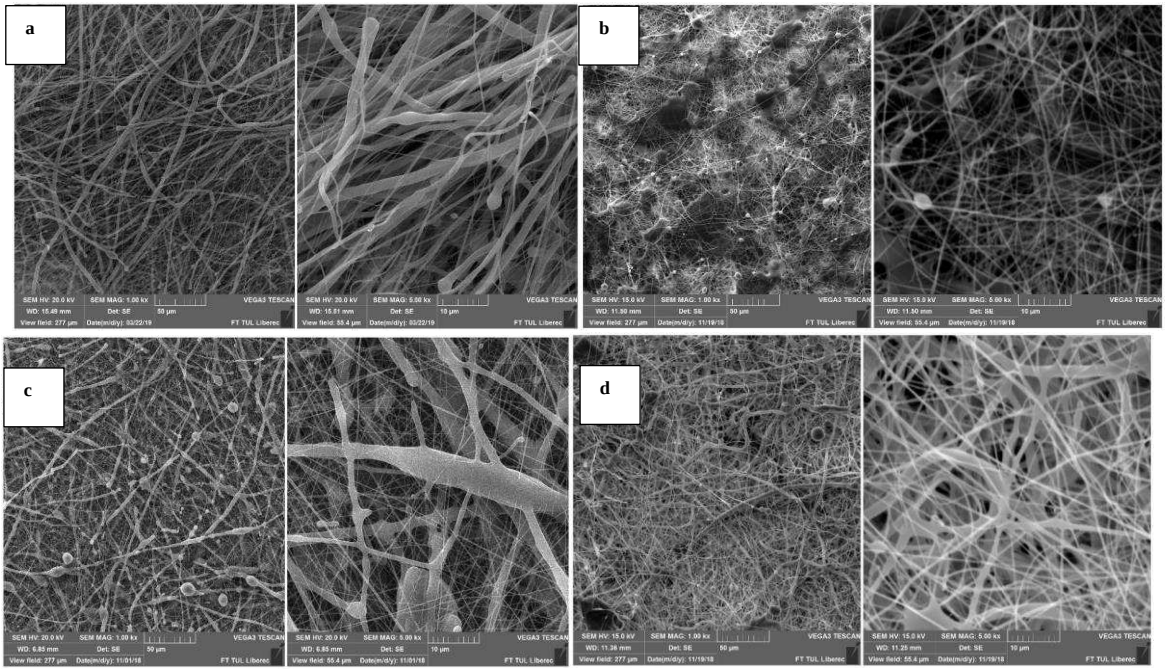

Figure 1: SEM image of pure PCL electrospun layer (a); pure PVA electrospun layer (b); PCL/PVA hybrid layer from dual jet spinning (c); PCL/PVA hybrid layer from needleless electrospinning (d).

Conclusion: A hybrid fibrous layer of PCL / PVA blend has been successfully produced by both needle and needle-less electrospinning. The material was homogenous and there was no separation of the layers. In-vitro testing confirmed that the addition of PVA fibers did not reduce cell viability on this mixture as compared to pure PCL. The material also retained a suitable wettability with distilled water.

Acknowledgements: The research was supported by grant of Ministry of Health of the Czech republic with number NV18-01-00332.

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DU, Lei, XU, Huaizhong, LI, Tao, ZHANG, Ying and ZOU, Fengyuan, 2016. Fabrication of silver nanoparticle/polyvinyl alcohol/polycaprolactone hybrid nanofibers nonwovens by two-nozzle electrospinning for wound dressing. Fibers and Polymers. December 2016. Vol. 17, no. 12, p. 1995–2005. DOI 10.1007/s12221-016-6813-0.

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Crystallization of Zirconia and Ceria Nanofibers Prepared from Polymer Precursors

R. Yager[1], C. Severino[1], W. Brayer[1], D. Lukas[2], A. Stanishevsky[2]
[1] Department of Physics, University of Alabama at Birmingham, USA, [2] Faculty of Textile Engineering at Technical University of Liberec, Czech Republic

Introduction: Highly porous advanced ceramics are important materials for catalysis, filtration, thermal insulation, biomedical scaffolds and many other applications. Fabrication of such ceramics with suitable microstructure and mechanical properties represent the major challenges in their technology. The use of AC-electrospinning in comparison to DC has the potential to lower these challenges and possibly increase the efficiency of nanofiber production. Nanofiber ceramics made using this method are expected to have properties greater than those in traditional ceramics.

Materials and Methods: Various ratios of Zirconyl salt ($ZrOCl_2$) and Cerium Nitrate were added to PVB (mowital) precursor solutions with solvents including water and ethanol. Viscosity and conductivity tests were performed on the solutions to test precursor properties. The method of AC-electrospinning was utilized at voltages ranging from 20-40 kV to synthesize nanofibrous meshes from precursors. The precursor fibers are exposed to temperatures up to 1200 ^oC to result in continuous and flexible nanofibrous ceramic meshes with porosity up to 98%. Test such as X-Ray Diffraction (XRD) and Thermal Gravimetric Analysis (TGA) were performed in order to analyze the crystallization process of the ceramic nanofibers. Fiber diameters and grain sizes were observed using a Scanning Electron Microscope (SEM).

Results: XRD results revealed that, unlike pure zirconia nanofibers, no monoclinic crystalline phase was seen even at 1200 ^oC due to the presence of ceria. When CeO_2 is present, a solid solution with ZrO_2 forms and monoclinic phase does not form in all tested ZrO_2/CeO_2 compositions. The crystalline phase showed pure tetragonal at all temperatures and the XRD peaks were wider than those for pure zirconia which represented smaller crystalline structures. SEM images showed that the fibers with ratios containing more ceria than zirconia resulted in smaller grain sizes compared to that of pure zirconia**.** The grain size in pure ZrO_2 or CeO_2 fibers was always larger than that in composite fibers containing both salts.

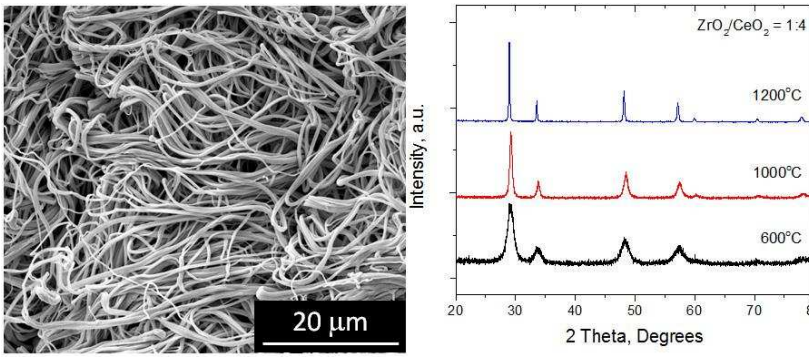

Figure 1. SEM image (left) and XRD patterns at different temperatures (right) of ZrO_2/CeO_2 composite nanofibers with 1:4 molar ratio.

Acknowledgements: R.Y. and C.S. have been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268). This study has also been supported NSF Grant Number 1708600.

AC Electrospinning of Titanium Oxide Nanofibers

S. Nealy[1], A. Jernigan[1], W. Maniukiewicz[2], M. Binczarski[2], I. Witonska[2], T. Kalous[3], P. Holec[3], D. Lukas[3], A .Stanishevsky[1]
[1]Department of Chemistry and Department of Physics, University of Alabama at Birmingham, USA, [2]Faculty of Chemistry, Lodz University of Technology, Poland, [3]Faculty of Textile Engineering at Technical University of Liberec, Czech Republic

Introduction: Traditional photovoltaic construction primarily consists of silicon- based subassemblies. However, these silicon-based monocrystalline solar cells have low durability, are vulnerable to impact, and expensive. Nanofibers can improve the light absorption and make the devices more flexible and mechanically stable [1]. In this study, nanofibers composed of titanium oxide (TiO_2) were spun using alternating current (AC) electrospinning. The high- yield of TiO2 nanofibers recovered after annealing has major implications into the photocatalytic capabilities of this nanomaterial. The TiO2 coated nanofibers have the potential to assume a relatively inexpensive alternative in addition to having a less brittle photovoltaic construction. When AC spun, the nanofibers also exhibit high viscoelastic properties, making it an ideal material for threading fabrics. The direction of future studies on this material will assume application into textile engineering, as well as continued research into catalytic applications.

Materials and Methods: TiO_2 fibers were prepared using a precursor based on polyvinylpyrrolidone (PVP) dissolved in ethanol. Titanium n-butoxide was hydrolyzed with acetic acid in the solution to yield the TiO_2component. Electrospinning of the fibers using AC was done in the 20 - 40 kV range, with an optimum production at 28 kV. The fibers were collected on a rotating cylinder with a rotation of 40 m/min. The production rate was observed to be about 1.5 - 2 mL per minute. Dynamic viscosity testing was performed on the optimized precursor solution. The resulting fibers were annealed at a range of temperatures between 500 - 1000 °C in air and annealed at 1000 and 1200 °C in argon atmosphere. As- prepared fibers were studied using SEM at up to 10,000X magnification. Further analysis was done using XRD and TGA.

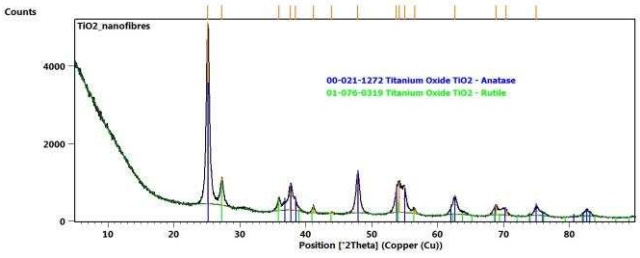

Figure 1: Depicts the XRD spectra of TiO_2 nanofibers

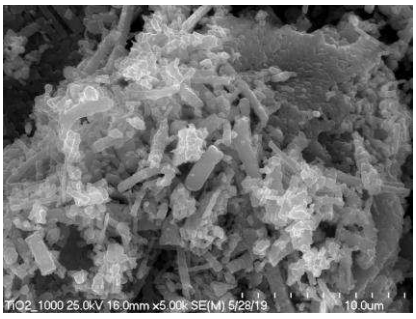

Figure 2: SEM analysis shows the crystalline structure of TiO_2 fibers annealed at 1000 °C in argon.

AC electrospun TiO_2 nanofibers were also annealed at 500 °C and submitted for photocatalysis studies.

Results: After annealing at 500 °C, the XRD data revealed that the TiO_2 nanofibers were mostly composed of the nanocrystalline anatase phase, with about 14% having rutile composition (Figure 1). Anatase is the metastable mineral form of TiO_2, with a high refractive

index [2]. This makes it an ideal material for refractory ceramics and catalysts. Shown in Figure 2, SEM imaging shows the crystalline structure of the annealed fibers in 1000 °C in argon, where that material is converted to pure rutile. Yet, the nanofiber structure is still somewhat visible, which indicates relatively low sinterability of this nanofibrous material at this temperature.

Conclusion: TiO_2 nanofibers were successfully created using AC electrospinning. This material shows promise as to its application in photocatalysis and catalytic abilities. Further studies will be pursued as to the catalytic capabilities of these TiO_2 nanofibers.

Acknowledgements: S.N and A.J. have been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268). This study has also been supported NSF Grant Number 1708600.

References:

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Second Session

Nanofiber Technology Applied to Absorbable Sutures

H. York[1], A. Shynkarenko[2], A. Stanishevsky[1]
[1]Department of Physics, University of Alabama at Birmingham, USA, [2]Department of Production Systems and Automation, Technical University of Liberec, Czech Republic

Introduction: Nanotechnology methods, in particular electrospinning, is both a promising technology and a vital scientific tool to be utilized for global benefits with a variety of applications. Over the past ten years, there has been a significant improvement of the techniques and methods used to design, assemble, and create nanomaterials by electrospinning. There are many techniques as our disposal for the creation of various nanomaterials, but electrospinning in particular has garnered significant interest over the years due to its ability to assemble nanostructures with unique properties. These properties encompass functions such as allowing for a high surface area, inter/intra fibrous porosity, and other specialized assets. In this study, cold water fish scale gelatin was electro spun to produce fibers to be made into strands. The work has been planned to use a braiding machine to form the functional structured from nanofibers, e.g., sutures. The work is in progress to explore these strands in combination with other biopolymers to create a high tensile strength, absorbable and bio-compatible suture. This braided suture could be infused with other ingredients (anesthesia, antimicrobials, etc) to be diffused into the body over time.

Materials and Methods: Cold water fish gelatin (GEL, Sigma Aldrich) of 45% weight percentage was blended with de-ionized water to make spinnable precursors. AC-electrospinning of the gelatin solution was performed at RMS voltages within 20-40 kV range. The fibers can be made into the strands to be braided on the machine built at TUL.

Results: The design of the nano-fiber strands to be braided has been proposed to be compatible with the machine designed at TUL. Similar studies were done on the exact same precursor (cold water fish scale gelatin with DI water - GES) and thickness, tensile strength, and elongation at break % were found based on amount of time (5,10,15,20, and 25 min on AC spinning) the precursors were electrospun. This confirms the personal studies done at UAB, which demonstrates the point at which the elongation at break % occurs becomes greater as the both the thickness of the fibers and the tensile strength increases. The compositions of the fibers are functional within the human body and are absorbable as they are a gelatin and water composition. With other materials added to accomplish antimicrobial properties or a localized anesthetic, the bio-compatibility of the sutures would not be compromised.

Table 1: Thickness and mechanical properties of GES products.

Sample	Thickness (μm)	Tensile strength (cN)	Elongation at break (%)
GES-5	25.5 ± 3.8	29.6 ± 11.6	11.6 ± 3.0
GES-10	34.8 ± 5.6	50.2 ± 22.5	11.3 ± 4.5
GES-15	46.1 ± 6.2	78.2 ± 35.0	14.5 ± 4.2
GES-20	72.4 ± 9.3	90.4 ± 24.5	13.3 ± 4.1
GES-25	76.4 ± 8.8	147.3 ± 33.7	15.8 ± 3.0

On the chart above are listed the values for a single strand of gelatin-based fibers. A rough estimate provides that three strands braided together is about three times stronger than one strand alone (allowing that there are no fractures or splits in the individual fibers). Thus, braided fibers would have a significantly larger tensile strength and a greater elongation at break %.

Conclusion: Gelatin is an incredibly versatile substance as it has gained great distinction for its versatility as a scaffold for tissue engineering due to its biodegradability, biocompatibility, ability to stimulate cell adhesion and proliferation, and resistance to immunogenicity. As such, a gelatin-based precursor is the perfect substance to create sutures with, in addition to their compatibility with other potential additives. The gelatin-based AC electrospun nanofibers can be made into strands to be braided together with the purpose of being utilized as a surgical suture to be infused with various compounds as needed. Thus, as the sutures are absorbed, the specified additive will be eluted into the body at a controlled rate.

Acknowledgements: H.Y. has been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268)

References: Gomes, S.R., Rodrigues, G., Martins, G.G., Henriques, C.M.R., Silva, J.C., In vitro evaluation of crosslinked electrospun fish gelatin scaffolds, Mater. Sci. Eng. C 33, 1219-1227, 2013. Lawson, C., Stanishevsky, A., Sivan, M., Pokorny, P., Lukas, D., Rapid fabrication of poly(□-caprolactone) nanofibers using needleless alternating current electrospinning, J. Appl. Polym. Sci. 2016.

Bending and Compression Strength of Nanofiber-based Glass-ceramic Porous Structures

C. Severino[1], M. Armstrong[1], M. Binczarski[2], I. Witońska[2], A. Stanishevsky[1]
[1]Department of Physics, University of Alabama at Birmingham, USA, [2]Institute of General and Ecological Chemistry, Faculty of Chemistry, Łódź University of Technology, Poland

Introduction: High corrosion resistance, hardness, strength, and low thermal conductivity make ceramic materials attractive for various applications. However, alongside these desirable properties come undesirable ones such as brittleness and poor impact strength. Recent developments of ceramic nanofibers formed from alternating current electrospinning (ACES) show promise for ceramic materials with competitive mechanical strength as well as high porosity compared to those made by traditional methods [1]. Incorporating these ceramic nanofibers into polymer or aerogel matrices can help to further create ceramic materials with unique combinations of mechanical properties. [2]. This study focuses on using nanofibers as the matrix and making three-dimensional (3-D) glass-ceramic fibrous structures. The results of structural analysis and mechanical tests of these 3D structures are also presented.

Materials and Methods: Figure 1 shows the process of creating 3D structures using nanofibers. Silica nanofibers were first prepared using needleless ACES (a1) and had diameters of 400 ±100 nm. The fibers were collected on a rotating drum (a2) to form a sheet that could be folded over onto itself and calcined at 600℃ to improve handleability for further processing (b1). The length and orientation of the fibers in the bulk sheet follow the black arrow in (b2). Next, the bulk sheet was chopped into small pieces creating short fibers (b3). Short fibers were then placed into a vial (c) and saturated with ethanol, salts (either a combination of $Mg(NO_3)_2(H_2O)_x$ + $Al(NO_3)_3{\cdot}9H_2O$ or just $Al(NO_3)_3{\cdot}9H_2O$ and polyvinyl butyral (PVB) to wet and coat them (d). Additional crushing using a spatula was done to ensure smaller and more uniform particle sizes and create a slurry (e) and molded into “green bodies” without applying pressure (f). Teflon tape (g) and a Teflon block (h) were placed over the samples to control evaporation and flatten the surface as much as possible. Still in the mold, the “green bodies” were placed on a hot plate for a minimum of 72 hours to allow ethanol to evaporate slowly (i) and then gently removed from the mold (j). Slow drying is critical to ensure that the “green bodies” do not bend or crack. The samples were then partially sintered at temperatures ranging from 800-1200℃ in air (k). 3D ceramic structures remained after the heat treatment (l1) and were lightly sanded to improve the overall surface appearance (l2).

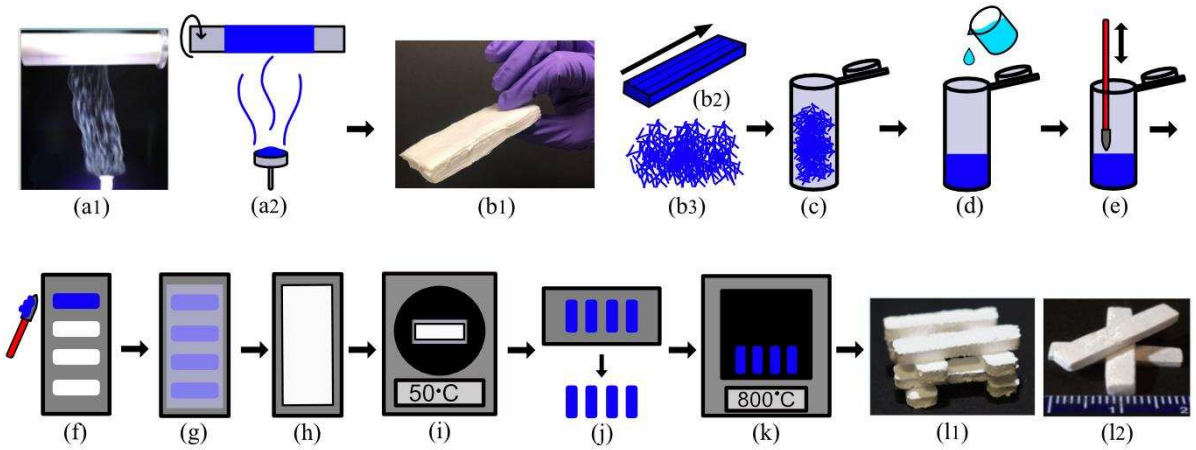


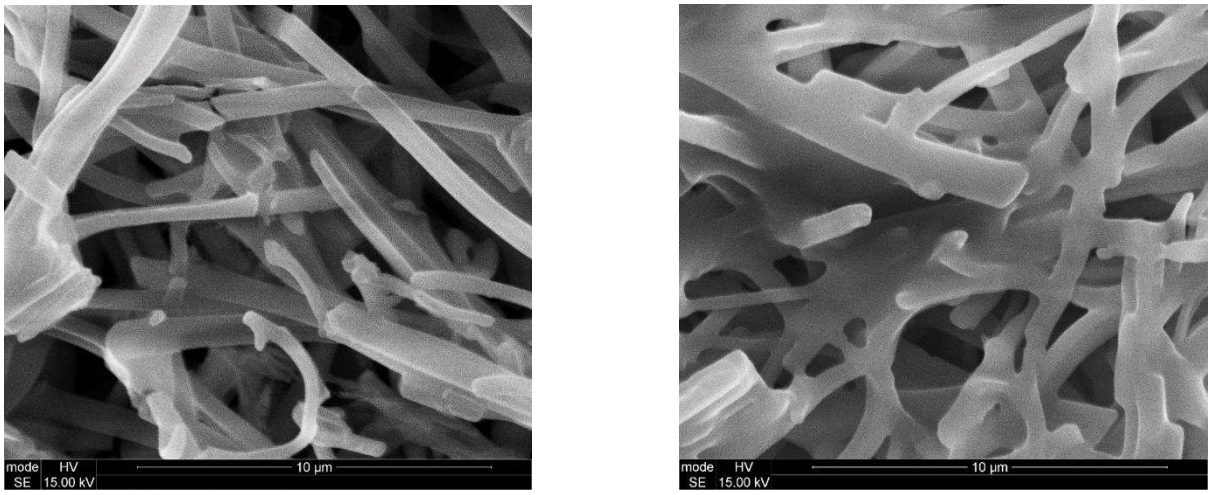
Figure 1: Process of making (3-D) glass-ceramic fibrous structures.

The 3D structures were analyzed by XRD, SEM/EDS, bending and compression tests. The roles of Mg- and Al-additives on stability of the base fibers during the calcination and sintering have been explored.

Results: After partial sintering at 1200ºC, the mechanical properties of the structures were measured and shown below in Table 1 and SEM images are shown in Figure 2.

Table 1: Mechanical properties of 3D structures after partially sintering at 1200℃ in air.

Composition	Apparent Porosity (%)	Bending Strength (MPa)	Compressive Strength (MPa)
Silica / Alumina	88.5 - 91.8	0.6 – 2.7	1.0 – 2.5
Silica / Spinel	88.5 – 91.8	1.1 – 1.8	1.5 – 3.5



(a) (b)

Figure 2 : SEM images of silica/alumina partially sintered at 1200 (a) and silica/spinel partially sintered at 1200 (b)

Conclusion: Nanofibrous silica-based glass ceramic constructs with good mechanical integrity have been prepared from ACES silica precursor fibers by using a high-temperature binder approach. Solid glass-ceramic structures with low shrinkage and 88–94 % porosity were obtained by heating at temperatures up to 1200℃ in air. Bending and compressive strengths of such nanofibrous constructs exceed those of reported so far microfiber- and xerogel-based silica and alumina structures with comparable porosities. The internal microarchitecture and fiber-fiber interactions in nanofibrous ceramic constructs seem to play important roles and need to be further studied.

Acknowledgements: C.S and M.A have been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268). This study has also been supported NSF Grant Number 1708600.

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[1] A. Stanishevsky, W.A. Brayer, P. Pokorny, T. Kalous, and D. Lukáš, Ceram. Int., 42, 17154 (2016).
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Alternating Current Electrospinning of PVA from Aqueous Solutions

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[1]Department of Physics, Troy University, USA, [2]Department of Physics, University of Alabama at Birmingham, USA, [3]Faculty of Textile Engineering, Technical University of Liberec, Czech Republic

Introduction: Polyvinyl alcohol (PVA) is a polymer used in many applications and currently is one of very few synthetic polymers that can be spun from an aqueous precursor solution by using Alternating Current (AC) electrospinning. The goal of this study is to investigate the AC spinnabilty of PVA from its aqueous solutions with different concentrations and additives (e.g., salts). Its spinnability is dependent on three factors - viscosity, surface tension, and electrical conductivity. Several tests are being conducted to understand the effect of those parameters on the spinnablity of PVA. Also, the mechanical properties will be explored as they can be affected by the fiber diameter, morphology, and packing in the fibrous mat. Although the mechanical properties of fibrous PVA mats have been explored using DC electrospinning, which showed that tensile strength was higher when prepared from more concentrated solutions, it is hard to say if similar results are expected for AC spun PVA.

Materials and Methods: Using PVA Mowiol 20-98 (Sigma Aldrich) with a Molecular weight 125,000 and hydrolysis ratio of 98%, and deionized water, 100-gram quantities of PVA solutions were prepared. The solutions had to be heated to 80°C during the mixing process. Several concentrations, 8, 10, 12, and 14 wt.%, were prepared. The viscosity and electrical conductivity of the precursor solutions have been measured. The fibers were spun using the AC electrospinning method. To find the trends in fiber morphology, SEM images were taken. ImageJ has been used to determine the fiber diameter and their size distribution depending on the process and process parameters. Mechanical test were conducted to find tensile strength and how different concentrations affected the mechanical properties of the fibers.

Results: It has been found that there is a relatively narrow range of concentrations of PVA aqueous solution where the AC electrospinning can take place. SEM image in Figure 1 shows typical appearance of PVA nanofibers. The AC generated fibrous flow was found to be the most stable at the precursor concentration of ~12 wt.%, although it was somewhat dependent on the temperature and humidity in the laboratory. Still, there was some bead fraction present in all tested samples that can be attributed, in part, to a high surface tension of the solution. The spinnability of the precursors reduced quickly outside the 8 – 14 wt.% concentration range. SEM image analysis showed that the average fiber diameter increased along with the increase of PVA concentration. For example, when a 10 wt% PVA solution was used, the average fiber diameter was 790 nm. It increased to 850 nm with 12 wt% solution. The resulting fibrous PVA mats were uniform, dense, and showed good mechanical integrity. The tensile properties of these mats have been discussed both theoretically and experimentally.

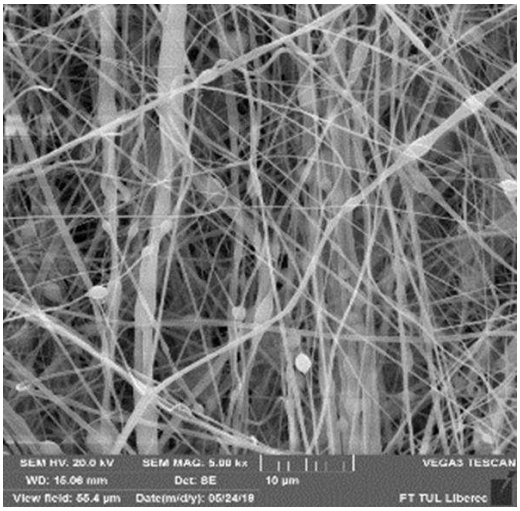

Figure 1: A sample of PVA nanofibers prepared from 12 wt.% precursor solution.

Acknowledgements: R.B has been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268) and by LSAMP at the Troy University.

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TiO_2 Catalysts: Micropowders, Nanopowders, and Nanofibers

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Introduction: Catalytic technologies are vital in the advancement and development of many industries, including chemical, biological, environmental, and energy. In the constant struggle for the optimization of the surface area to volume ratio, the focus of research has turned toward the possibility of catalysts on a smaller scale that will enhance the efficiency of any process. Amid this push for smaller technologies, attention has been recently drawn to the potential of nanotechnology as an alternative to micromaterials in catalysis. While studies have been performed to test the potential of nanopowders, there are many challenges associated both with the inherent nature of the material as well as the current infrastructure of industry. Nanofibers are gaining significant interest as an alternative to both micropowders and nanopowders that have advantages of both with significantly less of the disadvantages. In this work, TiO_2 nanofibers synthesized using needleless AC electrospinning were prepared under different conditions in order to create a catalyst that can been compared to TiO_2 micropowders through their efficiency in various photocatalytic reactions. These experiments were used in order to better understand the nature of the nanofibers used as compared to that of the micropowders which are commonly used across catalytic processes today.

Materials and Methods: Titania nanofibers were created using needleless AC electrospinning and then annealed in air at 500°C and 750°C, and then portions of these were reduced at 500°C in H_2. These fibers as well as titania P25 micropowder are to be used in the photocatalytic reduction of CO_2 in order to compare their efficiencies as photocatalysts. Analysis of these samples were performed using XRD, TGA, MS, SEM and EDS for characterization of the morphological and chemical character of the fibers.

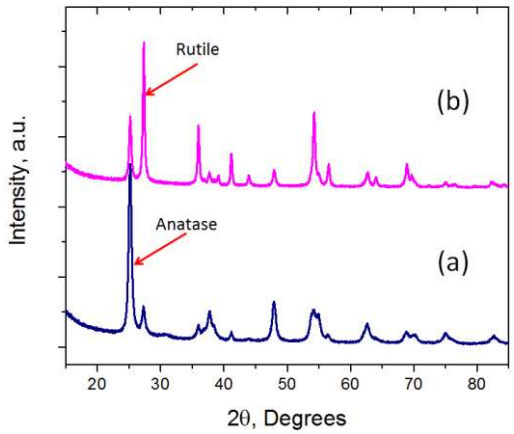

Figure 1. XRD patterns of (a) anatase-rich and (b) rutile-rich titania nanofibers prepared by AC-electrospinning and heat treated at *500°C*.

Expected Results:
It was found that the ratio of anatase to rutile crystalline phases in the annealed titania fibers can be adjusted by exposing the original precursor to moist air to facilitate the hydrolysis and nucleation of titania already in the precursor solution. Figure 1 shows the XRD patterns of titania fibers prepared from fresh and air-exposed precursor.

It is expected that the titania nanofibers will have a significantly higher efficiency in photocatalytic reactions than their micropowder counterparts.

Acknowledgements: A.J, S.N, and C.S have been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268). This study has also been supported by NSF Grant Number 1708600.

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Third Session

Synthesis and Characterization of Ceramic Nanofiber/GMB Composite Materials

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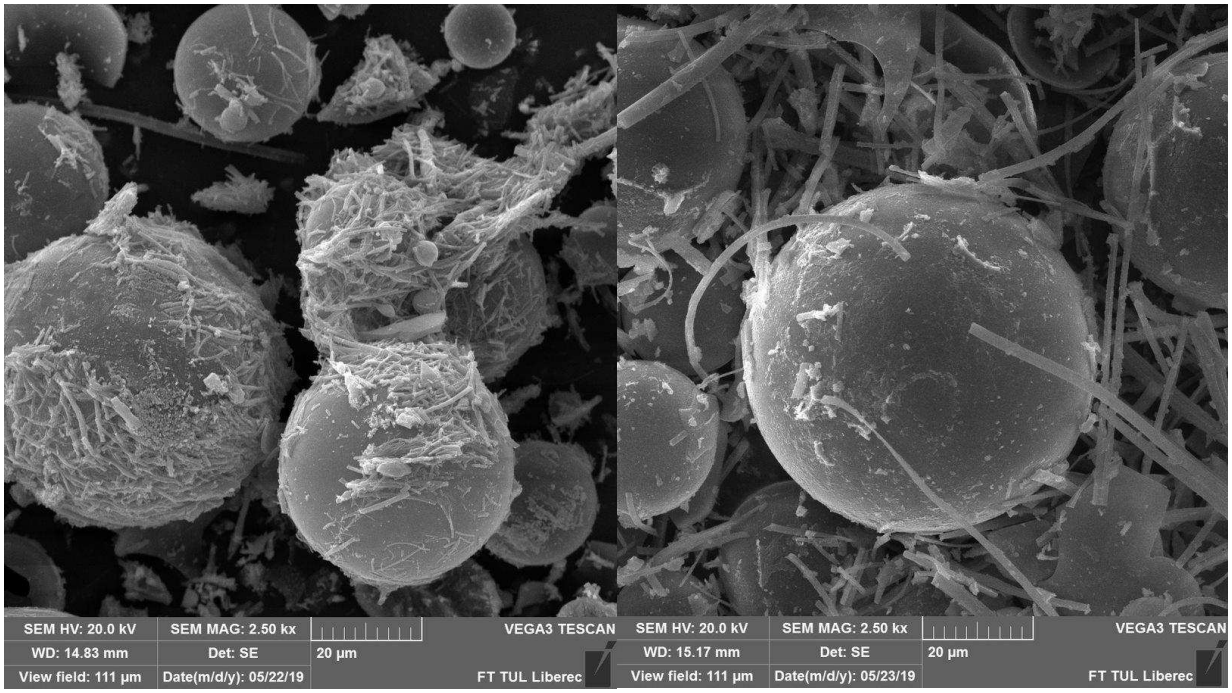
Introduction: Organic pollutants, such as dyes used by the pharmaceutical industry or natural organic matter, are harmful to the environment and humans due to their high toxicity and chemical complexity. The complexity of the molecules of these pollutants makes them difficult to remove through traditional water purification methods, so the use of heterogeneous photocatalysts, such as titania (TiO_2), has been observed as a possible method for the degradation of these pollutants in wastewater. In order for titania to be suitable for this application, it must be constructed into a suitable form. In this project, syntactic foams were made from a combination of ceramic nanofibers and glass microballoons (GMBs) through the deposition of a thin titania coating that served as both a binder and a catalyst. These foams utilized either silica or titania nanofibers, and were heat-treated at multiple temperatures to determine the effect of processing temperature on the phase evolution of the material. After synthesis of these foams was complete, they were characterized using SEM and XRD to determine the morphology of the foam as well as the composition.



(a) (b)

Figure 1: (a) a sample of titania nanofiber/GMB composite heat-*treated at 500°C (b) a sample* of silica nanofiber/GMB composite heat-*treated at 500°C*

Materials and Methods: Ceramic nanofibers were synthesized through the use of needleless AC electrospinning, then they were cut and combined with glass microballoons in various ratios. Each nanofiber/GMB mixture was added to a solution of titanium(IV) butoxide, then dried and annealed at either 500°C or 700°C to determine the effect of temperature on the

phase of the titania component and on the morphology of the syntactic foam. Samples were then analyzed under SEM, and images were taken to determine the interaction between nanofibers and GMBs, the effectiveness of the titania coating, and the overall morphology of the final syntactic foam. XRD was performed on each sample to determine the effect of temperature on the composition of the material with respect to the crystalline phases of titania.

Results: SEM imaging showed that fiber diameter has an effect on the interaction between the nanofibers and the GMBs (Fig.1). The titania fibers shown in Figure 1a wrap much more completely around the GMBs, while the silica fibers shown in Figure 1b only wrap around the GMBs a small amount. In all samples, a uniform titania coating was formed, although the coating began to delaminate at higher annealing temperatures due to compressive stresses on the surface of the GMBs. Overall, all samples had some interaction between the fibers and GMBs which allowed for binding of the materials into a syntactic foam. This phenomenon was most prominent in the samples that used titania nanofibers, likely due to the smaller diameter of the fibers in comparison to that of the silica fibers.

Acknowledgements: M.A., C.S. and S.N. have been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268).This study has also been supported NSF Grant Number 1708600.

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Electrospun Composite Nanofibers with Integrated Metal Oxides Nanoparticles

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Introduction: Composite nanofibrous materials produced by electrospinning have been presented in literature for many years with various modifications. These can include both the use of needles, such as spinning electrodes (Huang, Zhang, Kotaki, Ramakrishna 2003), and needleless spinning technology (Kostakova, Meszaros, Gregr 2009). The polymer nanofibers created by these means are "reinforced" by various particularly inorganic particles. They can be carbon nanomaterials such as fullerenes, carbon nanotubes or carbon nanofibers, or even metal oxide particles. Composite electrospun nanofibers are, in most cases, spun from polymeric solutions. These solutions must be prepared by first mixing the additive particles with the solvent or solvent system. Therefore, it is necessary to homogenize the solution, for example, by using ultrasonic homogenizers, then add the polymer to complete the solution. Electrospinning into the form of nanofibers requires particularly homogeneous polymer solutions. Aggregates of additive or microparticles would have a significant negative impact on the spinning process.The number of additives in the form of micro or nanoparticles is discussed in the field of production and testing of composite electrospun materials with respect to the possibility of a slowed production process not only due to the increasing density of the spinning solutions, but also as a result of the increasing viscosity and consequential inability to form fine fibers. The large number of inorganic micro and nanoparticles (e.g., up to three times the amount of polymer in solution) significantly alters fiber morphology. Electrospun nanofibers consisting of polyvinyl butyral and various metal oxides are studied in this work by several different analytical methods. The study presents the effect of the amount and type of metal oxide particles on the resulting fiber diameter, the uniformity of the metal oxide distribution in the resulting fiber layer, and the actual weight ratio of metal oxides in the final electrospun fibers. The aim of this study is to achieve a maximum ratio of particles to polymer dry matter in fibers without affecting the electrospinning process.

Materials and Methods: Polyvinyl butyral (PVB; Mowital B 60 H) manufactured by Kuraray was chosen as the polymeric material. The particles inserted into the polymer solutions were selected from the following inorganic materials: bismuth oxide Bi_2O_3; iron oxide Fe_2O_3; magnesium oxide MgO; their mixtures in were created in different proportions. All particles were obtained from Sigma Aldrich. The solvent for the spinning solutions was ethanol and the polymer concentration in the final solution was 10wt%.
Electrospinning was performed on needle-free electrospinning equipment manufactured by Elmarco NanospiderTM type NS 4S1000U. The spinning electrode was positively charged with a DC high voltage source and the collector was negatively charged.
A scanning electron microscope was used for sample analysis to visualize the resulting fibrous structures. Furthermore, analysis of the chemical composition of the surface SEM-EDS analysis and TGA analysis allowed the verification of the final amount of inorganic particles in spun nanofibers. This was done with temperatures up to 650°C and samples weighing 10mg.

Results: The spinning in all the mentioned cases was done with needleless electrospinning. Initial control of all samples was carried out under a scanning electron microscope. Since the fibers are their diameters above the limit value of the visible light wavelength, it is possible to observe changes in the color of the fiber layers containing individual inorganic particles or their mixtures in different weight ratios. Furthermore, SEM images show a uniform distribution of particles in individual fibers. The analysis of samples under SEM-EDS analysis showed uniform distribution of particles in samples in larger areas in all spinning cases.
The thermogravimetric analysis then showed the actual particle ratio that contained the spun submicron polymer composite fibers. Notably, there has been a minimal decrease in these values compared to the weighted ratios of the spinning solution, which is considered a great

success. The particles were not trapped or deposited/settled anywhere throughout the process but instead were able to pass through without causing major defects in the fibers. From the TGA analysis of the PVB, PVB + MgO, PVB + Fe_2O_3 and PVB + Bi_2O_3 samples, the following results showed in the mass balances after analysis as residue: PVB (blank) 2.3%; PVB + MgO (1: 1) 44.3 ± 4.2%, PVB + Fe2O3 (1: 1) 50.7 ± 2.7%, PVB + Bi_2O_3 (1: 1) 47.8 ± 3.4% and PVB + Bi_2O_3 (1: 3) 76.5 ± 1.9%. Therefore, it can be assumed that the density of the particles does not significantly affect the electrospinning process.

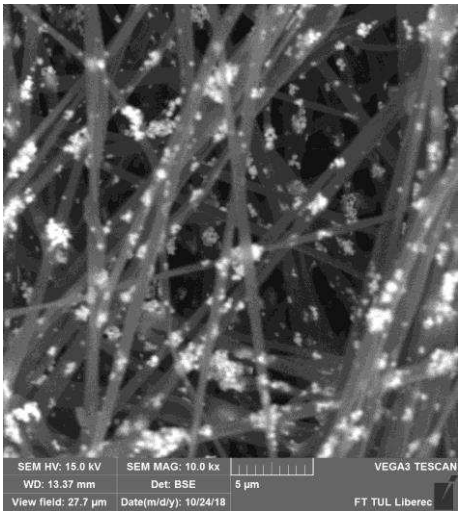

Figure 1: SEM image of electrospun PVB/ Bi_2O_3 in ratio 1:1 (material contrast).

Conclusion: The experiments presented demonstrate the possibility of inserting inorganic particles in micro- and nano- dimensions up to a weight ratio of polymer solids of 1: 5 in the spinning solution. Despite these high particle weights, the electrospinning process was not deteriorated. The diameters of the fibers have been shown to differ after insertion of the particles. It depends not only on the size of the original particles, but also on their disposition to create loops in the solutions. TGA analyses showed that all samples also contained in the resulting fibers an almost identical mass ratio between fiber dry matter and inorganic particles. SEM-EDS analyses showed a uniform distribution of particles in the area of the studied samples in all samples; this quality is very important in the final applications of the samples.

Acknowledgements: The result was obtained through the financial support of the European Union in the frames of the project “Modular platform for autonomous chassis of specialized electric vehicles for freight and equipment transportation”, Reg. No. CZ.02.1.01/0.0/0.0/16_025/0007293 and by grant "Intelligent textiles for protection against CBRN substances" of ministry of interior of the Czech republic; VI20172020059.

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Braiding technique for preparing tissue scaffolds

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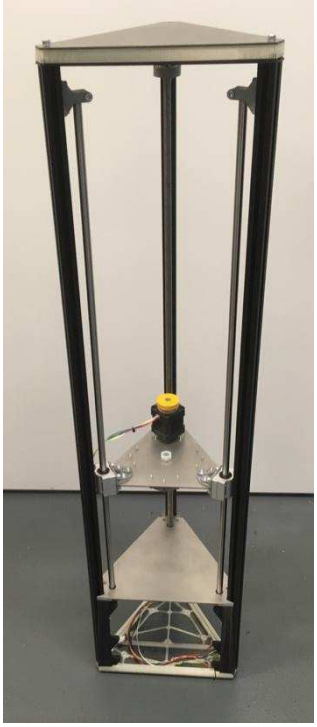
Introduction: Tissue engineering along with regenerative medicine can be used to create "Scaffolds" in the human body. These scaffolds are used to support organs and organ systems that may have been damaged after injury or disease. Tissue engineering is the use of a combination of cells, engineering and materials methods, and suitable biochemical and physico-chemical factors to improve or replace biological functions. Scaffolds are of great importance in clinical medicine. [1]

There are different ways to produce scaffolds. For example, it could be electrostatic fiber spinning [2], drawing method [3] or even additive manufacturing, also called 3D printing, is an effective method for preparing scaffolds with defined structure and porosity [4].

Oriented fibres and materials based on orientated fibres have great potential for use in tissue engineering for tissues, where the arrangement of extracellular matrix is fundamental [3].

Our idea is to develop a tissue by braiding threads covered by nanofiber layer [5]. After braiding it would have strong and oriented structure.



Technical characteristics of braiding machine (fig. 1): The main part of this device is braiding element. The braiding platform could move up and down. Movement distance is 800mm. These movements operated by two stepper motors. We can adjust height and speed of braiding process as an experiment requires. Number of spinners could be from 3 to 12. Speed of braiding process is adjustable and will be discovered experimentally. On the button part there is enough space to fix braided yarns. Also they could be placed in a container with medium.

Experiment will be divided into the following phases:

1) manufacture of yarns coated with nanofibers [5]. The nanofiber layer on the yarns should serve as a scaffold for the cells that we are going to cultivate;
2) cultivate cells on a yarns using special holder made from biocompatible material;
3) further yarns are planned to be woven using the braiding machine;
4) put the sample back into the medium for the further development of the cellular structure.

Figure 2. Prototype of braiding machine

Acknowledgments: This publication was written at the Technical University of Liberec as part of the project SGS 21293 with the support of the Specific University Research Grant, as provided by the Ministry of Education, Youth and Sports of the Czech Republic in the year 2019.

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Versatile Robotic Liquid Handling system - Based on “Uarm Swift Pro” robotic arm

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Introduction: For various laboratory application, a precise dosing is required with many kinds of liquids. Inspired by “**OpenLH**” Project, in which, a system for creative experimentation with biology was created, we started to design a liquid handling system that will fit a wider range of laboratory applications.
The base for the system is “**Uarm swift pro**”, an affordable, 4DOF, open sourced robotic manipulator.
in addition it is based on 3D printed parts, technical supplies and parts from an existing manual positive displacement pipette that can easily be bought online. It makes the hole system very accessible and affordable for any laboratory, compared to professional pipetting solutions in the market. The use of positive displacement pipette, allows wide range of liquids to be handled. Unlike manual pipettes where you need to use different pipette for different volumes, we designed the system to use different sizes of pipette tips so it will cover the whole range of traditional pipettes.

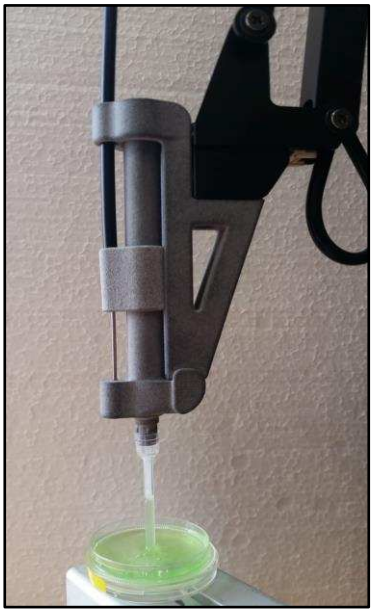

Figure 1. The pipette end effector

Operation Mechanism: The pipette end effector is operated using a linear actuator that pulls a bicycle brake cable. The linear movement is used to operate a clamp that catches a small piston inside the disposable pipette tip, acting as a miniature syringe (fig. 1). Using different pipette tips it is possible to determine the volume range for the specific application (1-1000 ul).

User Interface (fig. 2): In order to make it possible for every user to program the system to his needs, we are also working on GUI that will be based on the “Blocky” interface supplied by Ufactory, producer of the Uarm. This interface allows the user to drag and drop operations “Blocks” instead of writing direct code.

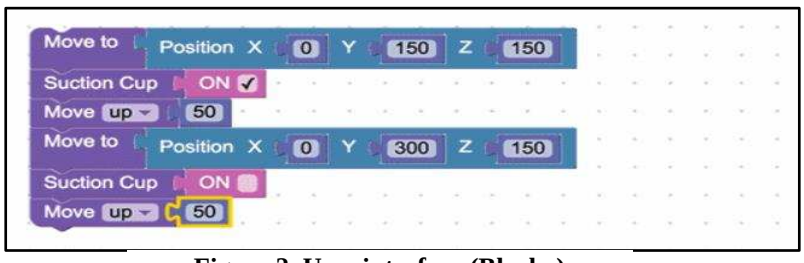

Figure 2. User interface (Blocky)

Possible Applications: The system can be used in many laboratory pipetting operations, currently done only manually. It can assist in performing a pipetting routine during night time or on holidays.In addition, due to its accurate dosing of liquids it could also enable new research techniques in the fields of nanomaterials and biology. It is expected to enable <0.01 [ul] dosing increments.

Acknowledgments: This publication was written at the Technical University of Liberec as part of the project SGS 21293 with the support of the Specific University Research Grant, as provided by the Ministry of Education, Youth and Sports of the Czech Republic in the year 2019.

References: Gilad Gome, Julian Waksberg, Andrey Grishko, Iddo Yehoshua Wald, Oren Zuckerman - OpenLH project http://milab.idc.ac.il/teaching/projects/openlh/

Structure and Catalytic Performance of Silica-Supported Vanadium Oxide Nanofibers

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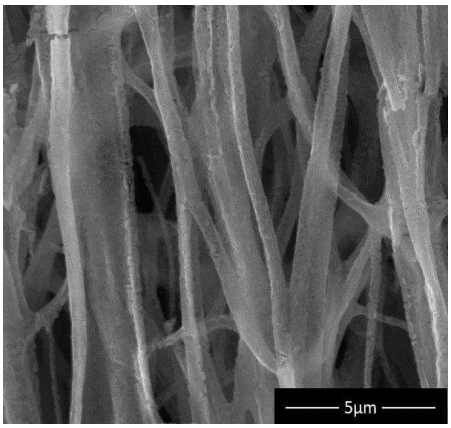
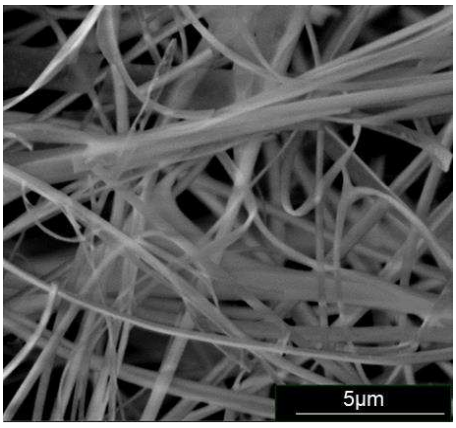
Introduction: Vanadium oxides are brittle transition metal oxides that have electronic properties suitable for applications such as selective oxidative reactions and gas sensing [1]. Vanadia is often supported by materials that have good mechanical integrity, flexibility, and thermal stability like amorphous silica, alumina, or titania [2]. There were several attempts to produce the vanadia nanofibers by electrospinning to prepare the materials with high surface area. It was found that such fibers collapse and break easily during either annealing of precursor fibers or mechanical loading after the calcination. The goal of this study was to prepare the vanadia-based composite nanofibers with silica as an additive to improve the fiber processability and mechanical properties. Catalytic performance of the vanadia/silica nanofibers is subject to future testing. Potential processes may include oxidation reactions of volatile organic compounds (VOC), water splitting, and reduction reactions of unsaturated VOCs.

Materials and Methods: Silica-supported vanadia nanofibers were created with varying amounts of silica through alternating current (AC) electrospinning. Product precursors were ethanolic solutions that contained silica and vanadia sources as well as an AC electrospinning compatible polymer. Effects of fiber calcination in a reducing atmosphere were explored. Fourier Transform Infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction (XRD) were used to analyze the nanofibers' chemical and phase properties. Vanadyl acetylacetonate, the source of vanadium, was mixed with dry ethanol in a 30% concentration and completely dissolved with 5N hydrochloric acid (HCl). Ethanolic tetraethyl orthosilicate (TEOS) combined with polyvinylbutryal (PVB) was added to the vanadium precursor solutions. The TEOS solution and ethanol were added to solutions to allow for optimal electrospinning given ambient humidity. Vanadium precursors were combined in various molar ratios with the TEOS precursor (V2O5/SiO2 = ~1:1 and ~1:2). Nanofibers were produced from the solutions using an AC electrospinning method up to 36 kV RMS in ambient atmosphere. Fibers were collected on inert collectors. The fibers were dried in vacuum before being annealed for 3 hours at 450°C and 600°C, respectively, in ambient atmosphere to crystallize the vanadium oxide and remove PVB. Fibers were then calcinated under a hydrogen atmosphere to reduce fibers at 500°C for an hour.

Results:

(a) 

(b) 

Silica-vanadia composite nanofiber morphology gradually improved as silica content increased from 0 mol.% (Figure 1a) to 66 mol.% (Figure 1b) annealed at 450°C. Annealing of the vanadia precursor fibers with no silica led to complete degradation of the fibers and the formation of a powdered vanadia. Nanofibers at the 66 mol.% silica concentration were generally smooth after annealing at 450°C, and formed bulky, mechanically stable flexible mats with bright orange color. Nanofibers between the concentrations 33 mol% and 50 mol.% displayed various morphologies of the vanadia crystalline phase in silica matrix depending on the vanadia/silica ratio. The preliminary data on the reduction of such composite fibers are further discussed.

Acknowledgements: A.D. has been supported by NSF International Research Experience for Students (IRES) award to UAB (Grant Number 1558268). This study has also been supported by NSF Grant Number 1708600.

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