SILICON OXIDE NANOFIBERS

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Abstract: In addition to organic polymers**,** inorganic polymers can also be used for electrospinning. The research objective was the preparation of inorganic nanofibers from pure silicon oxide using a sol-gel method and electrospinning. Diameters and characters of nanofibers prepared under different experimental conditions were assessed by a Scanning electron microscope.

1. Introduction

Nanofibers are generally described as fibers with their diameter in a submicron area, which means up to 1000 nm. These very fine fibers have some specific characteristics such as a large surface of fiber, large porosity of nanofiber web and small sizes of the pores between fibers. Electrospinning is one way of preparing very fine fibers from a polymeric solution or a polymeric melt using electrostatic power. There were various kinds of natural and synthetic organic polymers electrospun by this method [1,2].

In addition to organic polymers inorganic polymers can also be used for electrospinning, e.g. those prepared by sol-gel technology. By the term "sol-gel technology" we understand a group of methods of silicate and similar material preparation. The methods transform initial components (solution) to sol and then to gel by controlled hydrolysis and polycondensation as well. Homogeneity is preserved during this process [3].

The main components for preparation of special materials by the sol-gel method are alkoxides derived from alcohols. The hydrogen in the group C-O-H is substituted by the atom of metal (Si,Ti,Al,Zr,Na, ...) and eventually non-metal (B, P, …). In most cases the initial material is tetraethoxysilane (Figure 1). The generic scheme of material preparation by the sol-gel method based on alkoxide is shown in Figure 2.

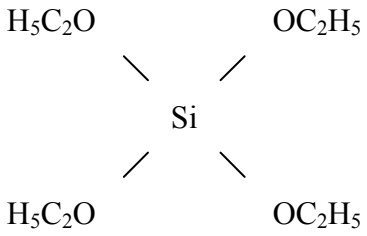

Figure 1:*Tetraethoxysilane $C_8H_{20}O_4Si$ (TEOS)*

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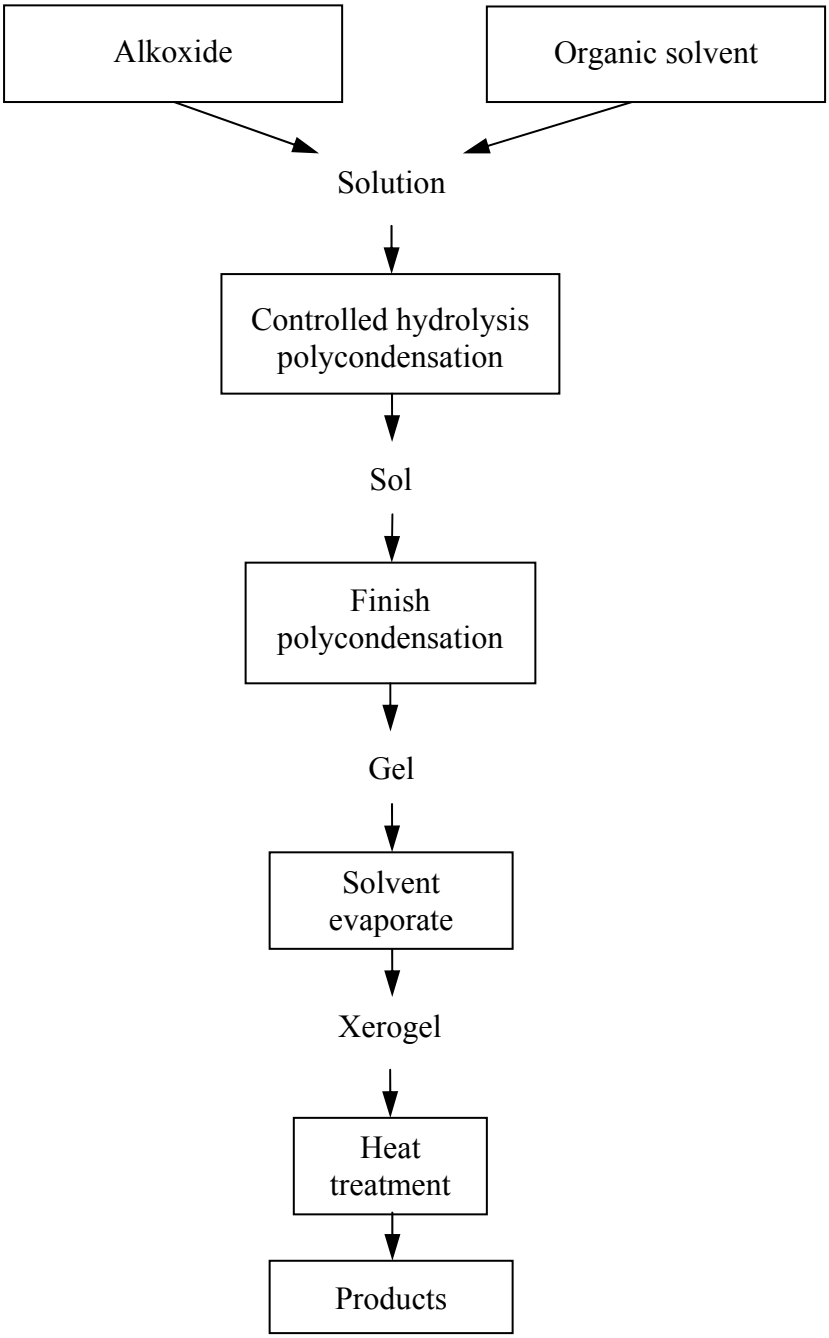

Figure 2: *Generic scheme of material preparation by the sol-gel method based on alkoxide*

2. Experiment description

2.1. Polymeric solution preparation

The research objective was the preparation of inorganic nanofibers from pure silicon oxide by electrospinning. The initial solution was prepared by the sol-gel method under acid

catalysis [4]. The initial raw material for preparation of the polymeric solution was tetraethoxysilane (Figure 1) and the solvent was isopropyl alcohol (the polymeric solution marked J11) or ethanol (the polymeric solution marked J9). The polymeric solution was prepared following the scheme in Figure 3.

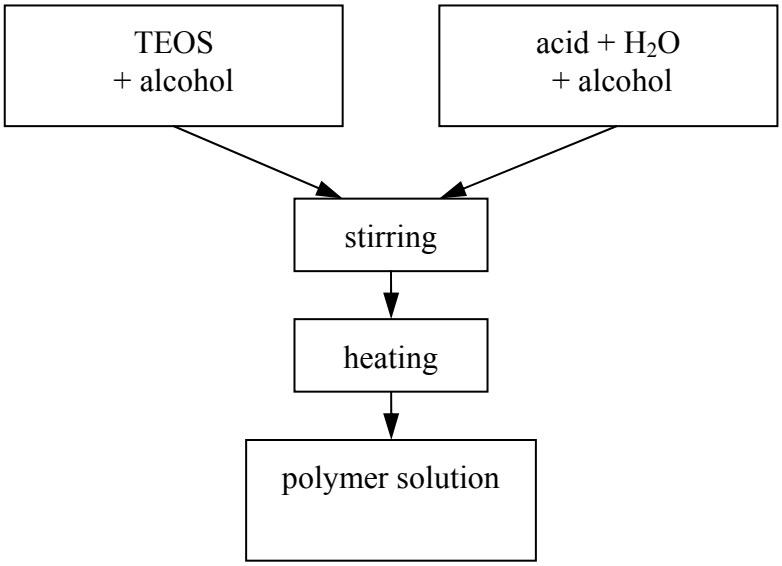

Figure 3: *Polymer solution preparation scheme*

Optimal concentration of the prepared polymer solution used for electrospinning was achieved by its gradual thickening.

2.2. Nanofiber web creation

The spinning was done on a laboratory device in the pilot plant of the Department of Nonwovens. The scheme is shown in Figure 4. The support material, poly(propylene), was coated with the nanofiber web (Figure 5), and then separated again (Figure 6).

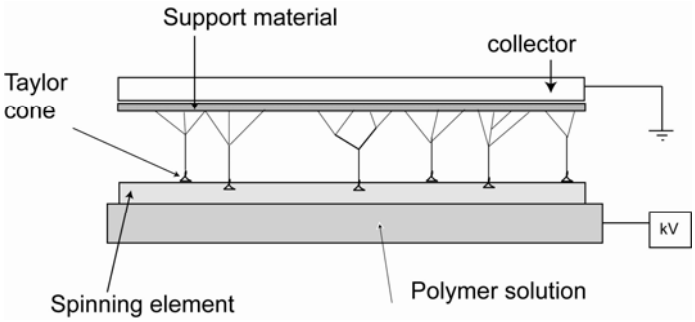

Figure 4: *Generic schema of nanofiber web creation*

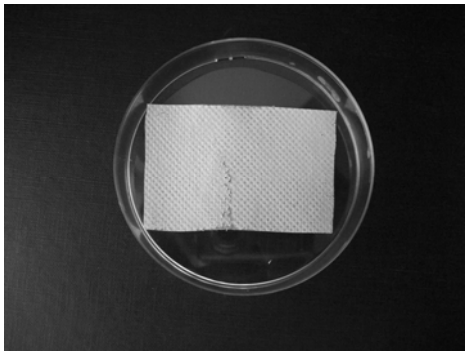

Figure 5: *Nanofiber web on support material*

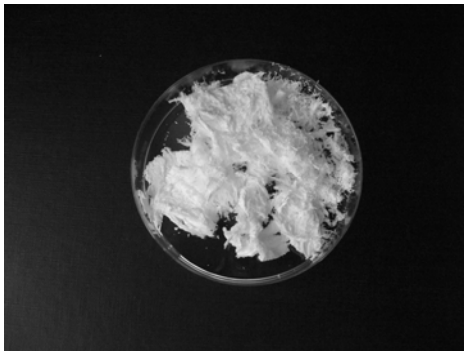

Figure 6: *Nanofiber web removed from support material*

3. Experiments results

The durability (time to transition to gel) and ability of the polymeric solution to electrospin based on the dependency of the concentration degree and used alcohol was observed. The polymeric solution based on isopropyl alcohol (marked J11) demonstrated a higher suitability for electrospinning than the one based on ethanol (polymeric solution marked J9). That is why the concentrated polymeric solution based on isopropyl alcohol was then used for nanofiber preparation. Under various parameters, the nanofibers were produced. Diameter and character of the nanofibers prepared under different experimental conditions were assessed by a VEGA Scanning electron microscope (SEM) at the Department of Textile Materials, Technical University Liberec . The good quality nanofibers with the smallest diameter are shown in Figure 7 ,8, 9 and Table 1. Histograms of nanofiber diameter were created based on LUCIA photo image analysis for various experimental conditions. A typical histogram is presented in Figure 10.

A part of the nanofiber web with mean diameter (snapshot 2, Table 1) removed from the support material was heat-stabilized for 2 hours at 180 °C (Figure 11 and 12) and sent for additional tests to the Institute of Chemical Technology in Prague (ICT).

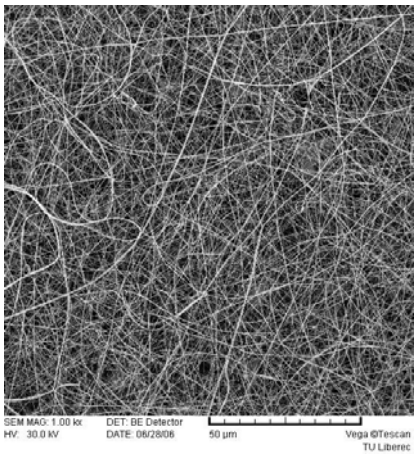

Figure 7: *SEM photo of nanofibers from polymeric solution J11, snapshot 1*

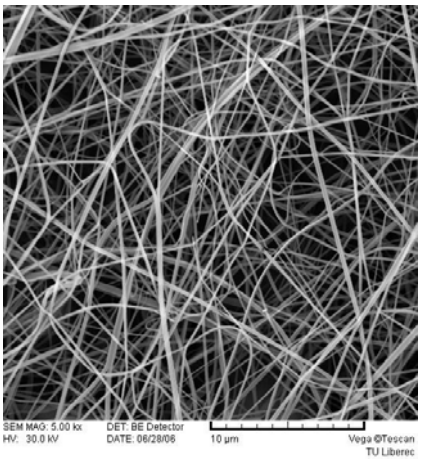

Figure 8: *SEM photo of nanofibers from polymeric solution J11, snapshot 1*

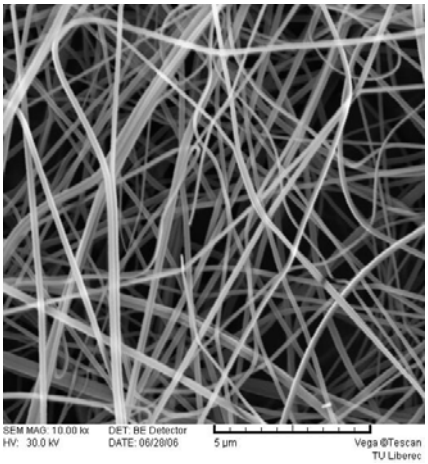

Figure 9: *SEM photo of nanofibers from polymeric solution J11, snapshot 1*

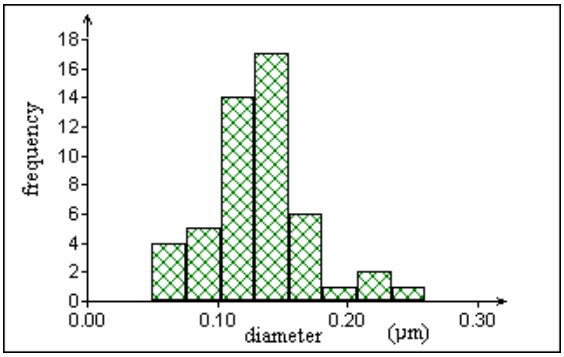

Figure 10: *Nanofibers diameter histogram (polymeric solution J11, snapshot 1)*

Table 1: *Nanofiber diameter (polymeric solution J11)*

Snapshot	Diameter			
	Mean value [nm]	Standard deviation [nm]	95% conf. interval	
			L_D	L_H
1	131	40	112	143
2	239	80	214	264

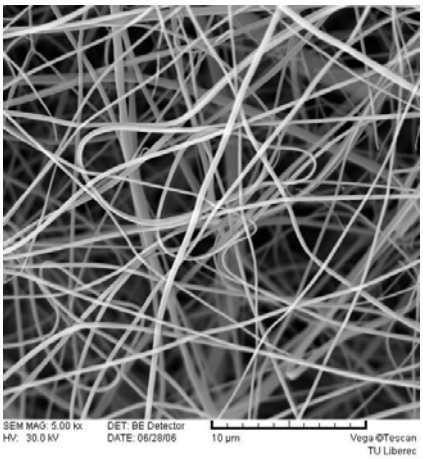

Figure 11: *SEM photo of nanofibers from polymeric solution J11, snapshot 2*

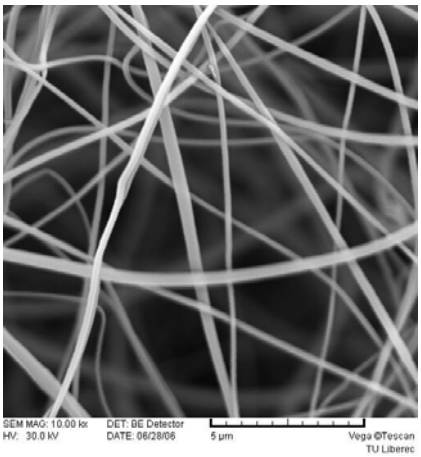

Figure 12*: SEM photo of nanofibers from polymeric solution J11, snapshot 2*

Under non-optimal conditions (snapshot 3) the nanofibers with particles were produced, they are shown in Figure 13 and 14.

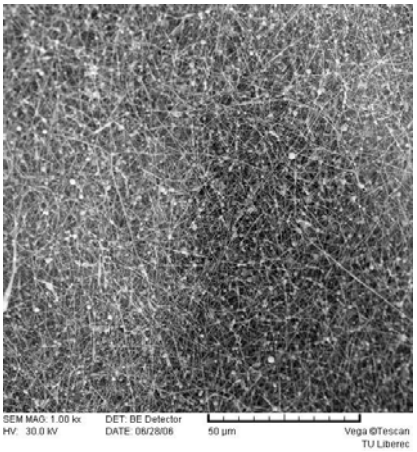

***Figure 13**: SEM photo of nanofibers from polymeric solution J11, snapshot 3*

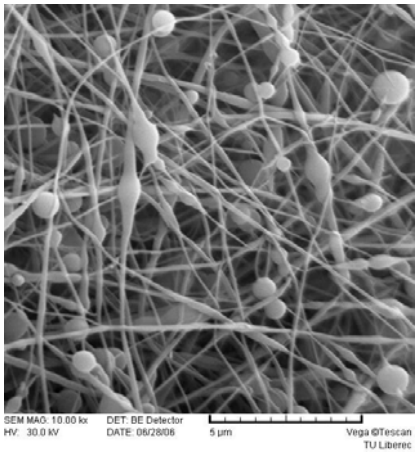

***Figure 14:** SEM photo of nanofibers from polymeric solution J11, snapshot 3*

4. Conclusion

Nanofibers have a circular profile as seen in the Scanning electron microscope photos. The produced nanofibers have properties comparable to a silicagel, e.g. resistant to temperatures of at least 180 °C without characteristic change, have large specific surface of fiber (approx. hundreds m^2/g) and other features. Further works with nanofibers have been suspended because tests about their potential carcinogenity at ICT Prague are still in progress. In simulated body fluid there is gradual degradation and disolution of nanofibers**.** That is the basic hygienic requirement for particles which could be breathed in. These preliminary results indicate that nanofibers could potentially be used, but complete official results are not available yet. They will be published separately.

Acknowledgments

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References

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