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# Improvement of UV Protection Properties of the Textile from Natural Fibres by the Sol-gel Method

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**Abstract.** In this research pure cotton textile was successfully modified by zinc oxide nanosol prepared by the sol-gel method. The cotton fabric was dipped in the nanosol solution for 10 minutes, dried at 90 °C for 10 minutes with further thermal post-treatment at 120 °C, 140 °C or 160 °C for 2 minutes. Comparison of coating of samples prepared using different thermal post-treatments was made. Before and after laundering tests ultraviolet protective properties of the textile samples were determined according to the standards, results show that textiles after treatment with nanosol have excellent ultraviolet protection properties, as well treated samples after laundering tests (50 washing-drying cycles) still provide excellent ultraviolet protection. Analyses based on the scanning electron microscopy and spectrophotometer measurements show that obtained textile coatings are distributed evenly, not only on the surface of yarns but in the depth of textile material as well, and are resistant to exploitation process that indicates about very good adhesion between the coating and the fabric surface.

## 1. Introduction

Natural fibres cannot be replaced by synthetic fibers due to their intrinsic biodegradable and wear comfort properties [1]. Nowadays, an advance in the knowledge of the fine structure and chemical composition of natural fibers has stimulated research and development into methods of improving their chemical and physical properties [1]. Special attention has been focused recently on the ultraviolet (UV) transmission of textile because of the growing demand in the marketplace for light-weight apparel that offers to human's protection from UV radiation (UVR). Zinc oxide is wide direct band gap (3.2 - 3.3 eV) semiconductor, which are transparent in the visible and absorb in the UV range [2-4]. Zinc oxide absorbs UVR via process of electron excitation called band-gap absorption. In comparison with the organic absorbers conventionally used in the textile industry, inorganic materials show no degradation under UV exposure and are therefore extremely stable and the oxides are classified as non-toxic materials [2-4]. For the abovementioned reasons, zinc oxide seems to be ideal for the preparation of highly UV-blocking, nanosol based coatings [2]. The present paper describes the deposition of zinc oxide thin-coatings by the sol-gel method on cotton textile substrates.

## 2. Materials and Methods

In the present study sol-gel method was evaluated in terms of the deposition of zinc oxide thin films on the commercial plain weave 100% cotton textile substrate.



### 2.1. Nanosol Preparation and Fabric Treatment

Tetraethoxysilane (TEOS,  $C_8H_{20}O_4Si$ ), ethanol ( $C_2H_5OH$ ), hydrofluoric acid (HF), deionized water and  $Zn(CH_3COO)_2 \cdot 2H_2O$  have been used for nanosols preparation. Nanosols were prepared by a controlled hydrolysis, by adding ethyl alcohol slowly into TEOS with continuous stirring, after adding deionized water and hydrofluoric acid, stirred for 30 minutes at 50 °C temperature, after mixed with the zinc acetate with continuous stirring 10 minutes. Samples were prepared zinc acetate concentration 5%. The fabric samples were dipped into the prepared nanosol, immersed for 10 minutes at room temperature. Subsequently, the samples were dried at 90 °C for 10 minutes with further thermal post-treatment at 120 °C, 140 °C or 160 °C for 2 minutes.

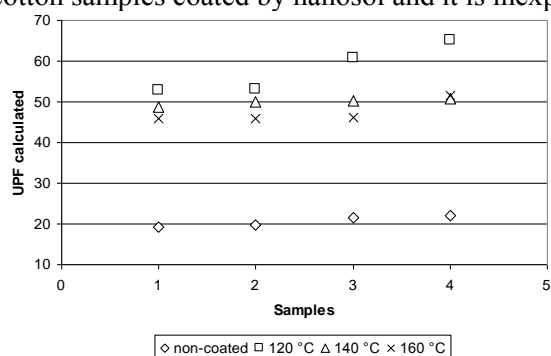
## 3. Results and Discussion

The nanosol process has to be adapted for the treatment of textiles because of textile low heat resistance; a thermal post-treatment at higher temperatures should be avoided to reduce degradation and deformation of the textile materials, and has to be adapted with regard to the type of textile coated and to the applied nanosol. Curing is an important step in the sol-gel coating process and curing temperatures of between 100 - 170 °C and different time from 1 to 30 minutes reported in the literature [5-8].

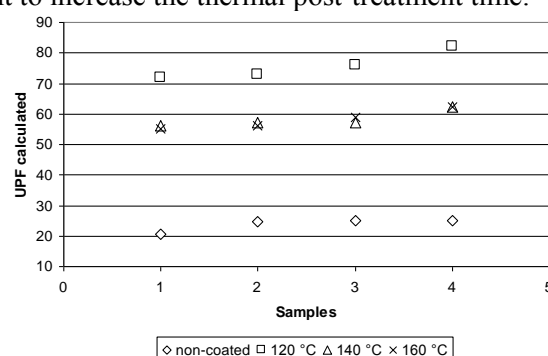
### 3.1. UV Protection Measurements

The UV protection of prepared samples was measured using the Varian Cary50 Solascreen spectrophotometer (Australia) according to the Australian/New Zealand AS/NZS 4399:1996 [9] and European EN 13758-1:2001+A1:2006 standards[10]. From each sample group 4 samples were scanned for UV transmission, 4 scans (one scan totally consist of 6 measurements, 3 measurements from each of 2 different points of the sample surface) per group were acquired.

Graphs in figure 1 and figure 2, and data table 1 indicate that thermal post-treatment time has influence on the calculated UV protection factor (UPF), UPF is the ration of the average effective UVR irradiance calculated for unprotected skin to the average effective UVR irradiance calculated for skin protected by the test fabric [9]. Data indicates that the best results of UPF of samples after treatment with nanosol were obtained with the thermal post-treatment at 120 °C for 2 minutes, achieved excellent UPF results as well as after 50 washing-drying cycles. According to the experiment data can be concluded that moderate thermal post-treatment at 120 °C for 2 minutes is appropriate for cotton samples coated by nanosol and it is inexpedient to increase the thermal post-treatment time.



**Figure 1.** Comparison of calculated UPF of samples with different thermal post-treatments.



**Figure 2.** Comparison of calculated UPF of samples with different thermal post-treatments after 50 washing-drying cycles.

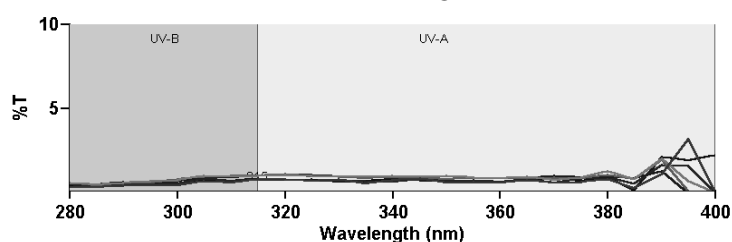
The UPF of cotton textile coated with zinc oxide nanosol with thermal post-treatment at 120 °C for 2 minutes increased approximately by 2.9 times in comparison with UPF values of uncoated textile (table 1) and the UPF range corresponds to the standards [9, 10] rating “50+ UPF” that mean excellent

protection (table 1). According to the requirements of standard [10] for all materials used for clothing assembly, the lowest UPF value shall be not less than 40 UPF.

**Table 1.** UV Protection Measurements

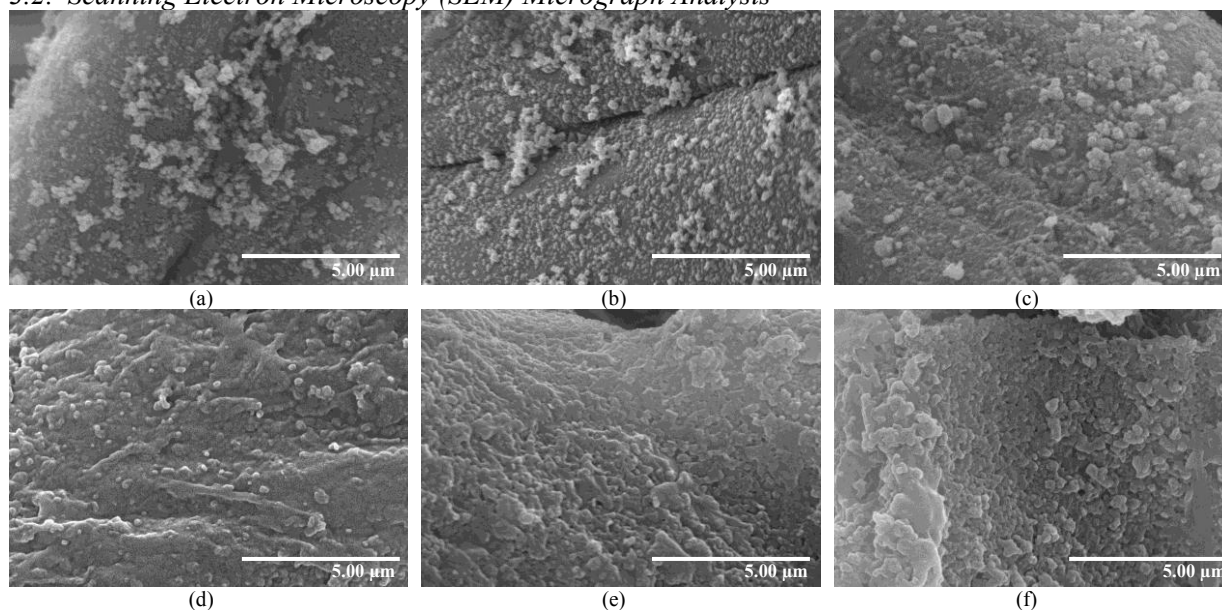
Samples	Calculated UPF	UVA transmission, %	UVB transmission, %	UPF rating
<i>After treatment with zinc oxide nanosol</i>				
Non-coated	19.929	3.874	4.058	15/20
Post-thermal treatment 120 °C	57.901	1.405	1.238	50+
Post-thermal treatment 140 °C	50.030	1.834	1.734	45/50+
Post-thermal treatment 160 °C	47.398	1.703	1.290	45/50+
<i>After 50 drying-washing cycles</i>				
Non-coated	22.961	3.407	3.438	20/25
Post-thermal treatment 120 °C	75.680	1.698	1.081	50+
Post-thermal treatment 140 °C	58.107	1.531	1.412	50+
Post-thermal treatment 160 °C	58.304	1.704	1.373	50+

The durability and stability of the treatment to multiple home laundering it indicates that is achieved very good adhesion between the coating and the fabric surface, as well treatment imparted excellent UVR protection to the cotton textile in both regions of UVR – UVA and UVB (figure 3).



**Figure 3.** UVA and UVB transmission after sample treatment

### 3.2. Scanning Electron Microscopy (SEM) Micrograph Analysis



**Figure 4.** SEM micrographs of samples with different post-thermal treatment: coated - a) 120 °C, b) 140 °C, c) 160 °C; after 50 washing-drying cycles - d) 120 °C, e) 140 °C, f) 160 °C.

Morphological changes as a result of sol-gel treatment of cotton fabric were investigated using scanning electron microscopy (SEM) Hitachi S-3400N (Japan). Figure 4 (a, b, c) shows the

morphological changes of the cotton textile surface after treatment by nanosol with different thermal post-treatment time. At high magnification, the formation of a thin layer is observed on the fiber surface (figure 4 a,b,c ); surface of untreated cotton fabric has grooves and cracks in contrast, such characteristics completely disappeared on the surface of the treated cotton fabric. Figure 4 (a,b,c) indicates that samples treated by nanosol have sufficiently evenly distributed coating, on fibres can notice the presence of silica particles that was proofed by energy dispersive x-ray (EDX) analysis of treated samples in previous experiment [11].

SEM micrographs figure 4 (d, e, f) reveals that received zinc oxide thin-coatings are resistant to exploitation impact (50 washing-drying cycles) the coatings on the cotton samples still are distributed evenly on the fibres surface without significant defects, coating became thicker, as well increased the roughness of coating, it can explain the increasing of UPF after washing-drying cycles.

#### 4. Conclusions

Spectrophotometer and SEM analyses show that a drying at 90 °C for 10 minutes and a moderate thermal post-treatment at 120 °C for 2 minutes after the nanosol with zinc acetate concentration 5% applied to the cotton textile is appropriate for the cotton textile sample coating by nanosol, the deposited coating on textile is distributed evenly and is resistant to exploitation process (laundry tests – 50 washing-drying cycles), show UPF rating 50+ after coating as well as after multiple washing processes. If compared to the standards, it is an excellent fabric protection towards UV radiation.

Results show that the UPF increase after washing cycles the possible reasons of increasing are: that the process of washing led to compaction due to shrinkage presumably decreasing porosity, decreasing of silica element in coating composition during washing-cycles, because during the washing cycles from textile fibers rewashed silica particles that were formed during the coating formation, as well thermal treatment below 200°C form xerogel and xerogels are amorphous networks and received coating is amorphous it is not so stable like crystalline coating, because of that during washing was occurred coating changes - coating became thicker and increased the roughness of coating.

#### 5. Acknowledgements

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