# Modification of the Cotton Textile Surfaces by the Depositing of Thin Coatings Using the Sol-gel Method

S. Vihodceva\*, S. Kukle†

Riga Technical University, Institute of Textile Materials Technology and Design, 14/24 Azenes street, LV-1048, Riga, Latvia

(Received 29 June 2012; published online 07 August 2012)

In the present research the commercial cotton textile was modified by the sol-gel method to evaluate sol-gel technology suitability for cotton textile modification. The nanosol was deposited on the cotton textile by dipping process. The evaluation of the dipping process for thin zinc oxide coating deposition was made. The sol-gel method for nanosol preparation used in this research to implement zinc oxide coating on the fabric surface is a simple process that can be easily transferred to the textile industry, nanosol can be also applied by conventional coatings techniques used in the textile industry – the application can be implemented by simple dipping process. The zinc oxide coatings were deposited on the textile surface by sol-gel method without deterioration of textile intrinsic properties, such as flexibility, softness and etc. The analysis of the coated textile surface was carried out by scanning electron microscope (SEM) and energy dispersive X-ray (EDX) spectroscopy. Data received by SEM and EDX analyses evince that the deposited coatings are evenly distributed, not only on surface of yarns but in the depth of textile material as well and resistant to the exploitation impact (laundry test).

Keywords: Sol-gel, Zinc oxide, Thin films, Cotton textile, Nanosol, Surface modification.

PACS numbers: 81.20.Fw, 68.47.Fg

## 1. INTRODUCTION

Since the appearance of the hole in the ozone layer and due to continuous decrease in the thickness of the ozone layer the UV intensity of solar radiation has increased. Nowadays the protection against skin cancer caused by UV radiation is of growing importance [1-3].

The ultraviolet radiation (UVR) is composed of three types: UV-A (320-400nm), UV-B (290-320nm), and UV-C (100-290nm). The UV-C radiation is absorbed by the ozone layer, however, the UV-A and UV-B reach the earth surface and cause serious health problems such as skin cancer, sunburn, and photo-aging [1, 3-9]. The UV-B radiation penetrates the upper skin layers (epidermis) while the UV-A radiation also reaches the deeper areas also (dermis) [1, 3, 4, 6, 10].

Therefore, special attention has been focused recently on the UV transmission of textile because of the growing demand in the marketplace for light-weight apparel that offers protection from UVR, while fostering comfort [3, 11]. When direct light shines onto a textile, a part of the radiation is reflected. The material will absorb a certain amount but the remainder can reach the skin [3, 4].

The absorption spectra of semiconductor such as zinc oxide show strong absorption in the UV region of the light spectrum but only very slight or no absorption of visible light [2, 3, 12, 13]. In comparison with the organic absorbers conventionally used in the textile industry, inorganic materials show no significant degradation and are therefore extremely stable and the oxides are classified as non-toxic materials [2, 3, 10-15]. Zinc oxide is harmless, that is why it is used in cosmetics such as sun creams. For the above-mentioned reasons, zinc oxide seems to be ideal for the preparation of highly UV-absorbing, nanosol based coatings [2, 3].

In addition to above mentioned textile materials

have intrinsic properties that make them extremely valuable - they are flexible, light weight, strong, soft, etc. Because of this, they are excellent objects for imparting additional functionalities.

The present paper describes the deposition of zinc oxide thin-coatings by the sol-gel method on cotton textile substrates.

## 2. MATERIALS AND METHODS

## 2.1 Sol-gel Method

The sol-gel dip process is almost exclusively applied for the fabrication of transparent layers, primarily for the deposition of oxide films on float glass as a transparent substrate with a high degree of planarity and surface quality [16].

This method is based on the preparation of colloidal suspensions - nanosols - from appropriately selected precursors, mostly metal oxides or organometallic compounds such as metal or semimetal silicon-containing alkoxides. These compounds, which are subjected to hydrolysis in an acidic medium, are converted into corresponding hydroxides that are unstable and susceptible to further condensation processes. Nanosols prepared in this way are deposited on fibres/fabrics and dried at an elevated temperature to condense them into cross-linked lyogels containing a considerable content of liquid phase. During further drying, the liquid phase is removed and a porous layer (xerogel) is formed on the fibre surface [2, 17].

One of the advantages of this method is the possibility of thin layers deposition on various materials, as well as the sol-gel layers can cover all fibres with high enough adhesion [18]. Major advantage is the high degree of uniformity obtained; also advantage is that the substrates size is not limited [16]. The thicknesses of

<sup>\*</sup> Svetlana.Vihodceva@rtu.lv

<sup>†</sup> skukle@latnet.lv

coatings applied to the fiber surface are mainly in the range of several nanometers to micrometers, besides the flexibility of a coating is directly related to its thickness [2]. Thus the use of sol-gel technology for the preparation of protective coatings seems to be appropriate.

## 2.2 Materials

In the present study sol-gel method was evaluated in terms of the deposition of zinc oxide thin films on the commercial cotton textile substrates. During the experiment woven 100% plain weave pure cotton fabric was subjected to processing by sol-gel method:

Commercial cotton fabric with surface density 50.91 g/m2 from yarns of linear density 8.4 Tex, the thickness of the fabric 0.21 mm, the measurement was taken by the textile fabric thickness tester "TH-25";

Cotton is a cellulosic fibre with a high ratio of hydroxyl groups that make it hydrophilic and that are available for polar interaction or potential surface reactions. In addition cotton is unique in features such as its biodegradability, water absorbency, comfort and thermostatic capacity [19].

## 2.3 Textile Surface Preparation

To provide good interfacial contact between the fibre surface and the deposited metal, cotton fabric samples were washed at temperature 90°C with detergent without optical brighteners.

Nevertheless, the washing did not remove all the oil. As a result about half of washed samples were immersed in 80% acetone solution at room temperature for 5 minutes [20, 21] and were washed twice with distilled water. Drying was carried out on a horizontal surface.

## 2.4 Nanosol Preparation and Textile Coating Process

Tetraethoxysilane (TEOS,  $C_8H_{20}O_4Si$ ), ethyl alcohol ( $C_2H_5OH$ ), hydrofluoric acid (HF), deionized water and  $Zn(CH_3COO)2$   $^22H_2O$  have been used for nanosol 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, after mixed with the zinc acetate with continuous stirring 10 minutes.

The process was performed at room temperature. The obtained nanosols were clear and homogeneous.

To evaluate the coating quality the commercial fabric samples were prepared with TEOS concentration 2% and zinc acetate concentration 2.5%.

The fabric samples were dipped into the prepared nanosols, immersed for 10, 20 and 30 minutes at room temperature. Subsequently, the samples were dried at 50 °C for 10 minutes and after cured in an oven at 120 °C for 10 minutes.

# 2.5 Testing the Resistance of the Coated Samples to Laundering

For evaluation of the exploitation impact on the zinc oxide coating laundry test was carried out, the samples from all groups were washed at temperature  $40\,^{\circ}\mathrm{C}$  with

detergent without optical brighteners. Drying was carried out on a horizontal surface.

## 2.6 SEM and EDX Analysis of the Coated Samples

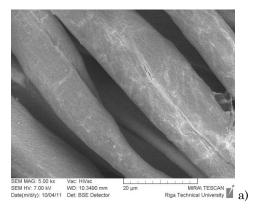
The morphological changes of the natural textile as a result of coating with zinc oxide and after its washing have been investigated using scanning electron microscope SEM (Tescan, Mira//LMU Schottky).

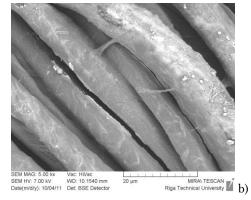
EDX spectroscopy was used for the analysis of the elemental composition of the coated textile samples before and after laundry test.

## 3. RESULSTS AND DISCUSSION

## 3.1 SEM Micrographs Analysis

Figure 1 (a, b, c) illustrates micrographs of the coated fabric samples depending on the time of dipping in nanosol 10, 20 and 30 minutes.





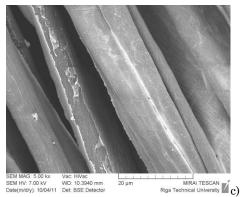


Fig. 1 – SEM micrographs of the coated textile samples, dipping time a) 10 min; b) 20 min; c) 30 min

On the surfaces of the samples dipped in nanosol for 20 and 30 minutes (Fig. 1 b, c) were formed agglomerated particles and bridges between fibres.

The formation of small bridges can be tolerated to a certain extent – these will break during use, leaving small fins (Fig. 1 b). Large bridges (Fig. 1 c) may cause problems, since during stress an exfoliation of the coating material can be observed, leaving the bare fiber surface. Such defects are critical for barrier coatings that are applied to protect fiber material in aggressive atmospheres [2].

After reducing of the dipping time till 10 minutes qualitative, distributed evenly, not only on surface of yarns but in the depth of textile material as well, zinc oxide coating was received (Fig. 1 a and Fig. 2 a, b).

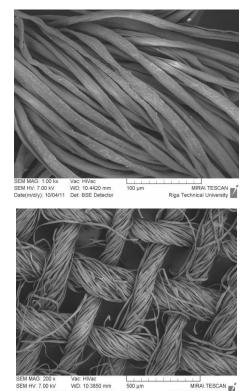


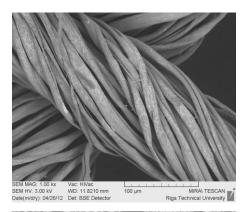
Fig. 2 – SEM micrographs of the coated textile samples, dipping time 10 minutes

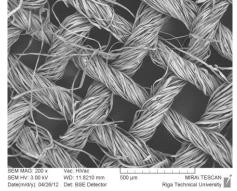
From the micrographs observations, follows that zinc oxide nanosols can be deposited on cotton textiles in the form of thin elastic coating, which completely and uniformly cover the fibres surfaces, as it is clearly seen in the SEM image (Fig. 2 a).

SEM micrograph (Fig. 3 b) reveals that received zinc oxide thin-coatings are resistant to exploitation impact (laundry test), as the coating on the cotton sample is still distributed evenly and without significant defects on the fibres surface.

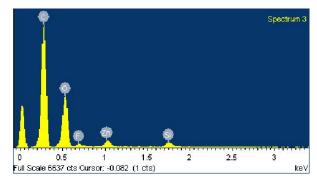
## 3.2 EDX analysis

For the analysis of the elemental compositions of samples after the samples coating was used EDX analysis. From EDX analysis, of the samples coated with nanosol are visible that the main elements of the surface species of treated cotton textile samples are C, Zn, O, F and Si (Fig. 4).





 ${\bf Fig.\,3}-{\bf SEM}$  micrographs of the coated textile samples after laundry test



 ${f Fig.~4}-{f EDX}$  spectrum after sol-gel treatment

The EDX analysis (Tab. 2) of the coated samples after laundry test evince that the percentage weight of zinc on the surface of the samples coated by nanosol with dipping time 10 minutes remained almost the same as before laundry test.

Table 2 - EDX analysis - elemental weight in %

Samples	C	О	F	Si	Zn
Uncoated	46.67	53.31	-	-	-
Coated (dipping time 10 min)	62.62	29.96	2.81	1.26	3.35
Coated (dipping time 10 min) after laundry test	43.83	46.10	0.06	6.71	3.30

## 4. CONCLUSIONS

The zinc oxide coatings were deposited on the textile surface by sol-gel method without deterioration of textile intrinsic properties, such as flexibility and softness etc. SEM micrographs evince that the zinc oxide coating is not a flat film on the cotton textile surface, but deposited on fibres without changing the textile surface structure and trim.

Based on the SEM and EDX analyses it is concluded that with concentration of the TEOS 2% in the nanosol and dipping time 10 minutes, the received textile coating:

- is distributed evenly, not only on surface of yarns but in the depth of textile material as well;
- is resistant to exploitation process (laundry test), after process it is still without significant defects, besides has not changed the elemental percentage weight of the samples
- a functionalized coating is gained in a very cost effective way.

The sol-gel method has to be adapted for the treatment of textiles because of textile low heat resistance; a thermal after-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 and to the applied nanosol. Experiment results show that a moderate thermal after-treatment at 120°C after deposition is appropriate for the cotton textile samples coated by sol-gel method.

The sol-gel method used in this research to implement zinc oxide coating on the cotton fabric surface is a simple process that can be easily transferred to the textile industry, nanosol can be also applied by conventional coatings techniques used in the textile industry – the application can be implemented by simple dipping process.

## AKNOWLEDGEMENTS





This work has been supported by the European Social Fund within the project «Support for the implementation of doctoral studies at Riga Technical University».

#### REFERENCES

- B. Mahlting, T. Textor, Nanosols and Textiles (Singapore: World Scientific Publishing: 2008).
- Surface modification of textiles (Ed. Q. Wei) (United Kingdom: CRC Press: 2009).
- 3. S. Vihodceva, S. Kukle, J. of Riga Techol. Un. Mat. Sci. and Clothing Tech. 6, 19 (2011).
- A. Farouk, T. Textor, E. Shollmever, AUTEX Rsrch J. 10 No3, 58 (2010).
- N. Abidi, E. Hequet, S. Tarimala, J. of Appl. Polym. And Sci. 104, 111 (2007).
- Textiles for protection (Ed. R.A. Scott) (Lincoln: CRC Press: 2005).
- 7. D. Saravan, AUTEX Rsrch J. 7 No1, 53 (2007).
- 8. A.M. Grancarić, A. Tarbuk, I. Dumitrescu, J. Bišćan, *AATCC Rew.* **6**, 44 (2006).
- 9. A.M. Grancarić, Ž. Penava, A. Tarbuk, Proc. of the VI Symp. Contenporary Technol. And Economic Devel. (2005).
- A. Putnina, S. Kukle, D. Turigina, J. of Riga Techol. Un. Mat. Sci. and Clothing Tech. 5, 157 (2010).
- S. Kathirvelu, L. D'Souza, B. Dhurai, J. of Fibre & Tex. Rsrch. 34, 267 (2009).
- 12. R.S. Karmali, A. Bartakke, V.P. Borker, K.S. Rane, J. Bio-

- interface Rsrch. in Appl. Chem. 1 No2, 057 (2011).
- 13. Н.С. Николаева, В.В. Иванов, А.А. Шубин, *J. of Siberian Federal Univ. Chem.* **2**, 153 (2010).
- Y.W.H. Wong, C.W.M. Yuen, M.Y.S. Ku, H.L.I. Lam, *AUTEX Rsrch J.* 6 No1, 1 (2006).
- Chi-ming Lai, Keh-moh Lin, S. Rosmaidah, J. Sol-Gel Sci. Technol. 61, 249 (2011).
- Sol-gel technology for thin films, fibers, performs, electronics, and specialty shapes (Ed. L.C. Klein) (U.S.A.: Noyes publications: 1988).
- S. Brzeziński, D. Kowalczyk, B. Borak, M. Jasiorski, A. Tracz, J. Fibres & Tex. In Easterns Eur. 19 No6, 83 (2011).
- 18. A.Chládová, J.Wiener, J.M. Luthuli, V. Zajícová, *AUTEX Rsrch J.* 11 No1, 18 (2011).
- Industrial Applications of Natural Fibres. Structure, Properties and Technical Applications (Ed. J. Müssig) (United Kingdom: John Wiley & Sons, Ltd: 2010).
- S. Kukle, S. Vihodceva, Proc. of the 41st Symp. on Novelties in Text. 413 (2010).
- S. Vihodceva, S. Kukle, J. of Riga Techol. Un. Mat. Sci. and Clothing Tech. 5, 123 (2010).