Thin Coatings on the Raw Cotton Textile Deposited by the Sol-Gel Method

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Abstract **– In this research, pure raw cotton textile has been modified by the sol-gel method to implement zinc oxide coating on the fabric surface. Scanning electron microscopy (SEM) has been used to examine the nature of the surface modification after application of coating. Curing is an important step in the sol-gel coating process. Therefore, the present paper examines the application of different curing temperatures to the preparation of textile samples, as well as evaluates their suitability for the samples made of raw cotton textile.**

Keywords **– raw cotton textile, sol-gel method, zinc oxide, thin-coating finishing.**

I. INTRODUCTION

A. Textiles and UV-Protection

Since the appearance of the hole in the ozone layer and due to continuous decrease in the thickness of the ozone layer the ultraviolet (UV) intensity of solar radiation has increased [1-3]. The ultraviolet radiation (UVR) is composed of three types: UV-A (315-400nm), UV-B (290-315nm), 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 reaches the deeper areas (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 against UVR, while fostering comfort [3, 11]. Textiles are assembled to protect against environmental influence at all times. Textile garments will protect against the hazardous effects of the UV radiation to a certain extent, but light and light-shaded clothing textiles provide insufficient protection. When direct light shines onto a textile, part of the radiation is reflected. The material will absorb a certain amount, but the remainder can reach the skin [3, 4]. The fabric UV protective capacity varies significantly depending on many elements such as: fibre type, additives, fabric structure (weave construction, yarn number, weight, thickness, thread count, cover factor, etc.), colour and dyeing intensity, presence of optical brightening agents, pigments and finishing products (different UV absorbers), laundering and washing conditions, fabric extensibility, wetness, etc. [12]

The absorption spectra of a 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, 13, and 14]. 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, 10-16]. Zinc oxide is harmless, that is why it is used in cosmetics, for example, suncreams. For the above-mentioned reasons, zinc oxide seems to be ideal for the preparation of highly UV-absorbing, nanosol-based coatings [2, 3].

Besides, textile materials have intrinsic properties that make them extremely valuable – they are flexible, light weight, strong, soft, etc. As a result, 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 the raw cotton textile substrate.

B. Sol-Gel Method

The modification of textile materials is of major importance and an essential component in textile processing for imparting of additional properties. New materials can be developed, and new properties can be added to the existing materials by the use of special-treated fibres, coatings [17]. Therefore, besides all new functions, the trend towards even thinner fibre creates a need for thin nano-scaled or nano-structured finishing [18].

One of the various new approaches and surface modification methods of textile materials – and certainly not only of textile materials – is the sol-gel method, which offers far-reaching possibilities for creating new surface properties [1].

The sol-gel method has become an important tool for producing nanoparticles or for the preparation and application of thin layers or coatings based on either inorganic materials or inorganic-organic hybrids; it can be employed for modification of many textile materials and allows one to tailor certain properties and to combine different properties in a single coating step [2]. Nowadays sol-gel science allows the preparation and stabilization of a huge number of metal oxide sols [2].

Moreover, 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 [19].

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, 20].

One of the advantages of this method is the possibility of preparing thin layers on various materials, as well as the sol-gel layers can cover all fibres with high enough adhesion [21]. Major advantage of the method is the high degree of uniformity of obtained coatings; also, the advantage is that the size of substrate is not limited [19]. The thickness of the coatings applied to the fiber surface is mainly in the range of several nanometers to micrometres; besides, the flexibility of a coating is directly related to its thickness [2]. Thus, the use of sol-gel coating for the preparation of protective coatings seems to be appropriate. Also, nanosols can be applied to the textile surface by conventional coating techniques used in the textile industry. The application can be carried out by a simple dipping process, spraying and padding [3].

II. Materials and Methods

A. Materials

Raw woven plain weave 100% cotton fabric with the surface density of 147.36 g/m^2 from yarns of linear density 33.6 Tex has been used in the experiment. The thickness of the fabric is ~ 0.36 mm; the measurement was taken by the textile 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 its features, such as biodegradability, water absorbency, comfort and thermostatic capacity [22].

B. Material Pre-Treatment

To provide good interfacial contact between fibre surface and deposited coating, the surface of cotton fabric samples has been washed at a temperature of 90°C with a detergent without optical brighteners; nevertheless, during the washing procedure the oil has not been removed. Therefore, the samples were immersed in 80% acetone solution at room temperature for 5 minutes [23, 24], and then washed twice with distilled water. The drying step was carried out on a horizontal surface.

C. Chemicals

Tetraethoxysilane (TEOS, $C_8H_{20}O_4Si$), alcohol (C_2H_5OH), hydrofluoric acid (HF), deionized water, zinc acetate ($Zn(CH_3COO)_2 \cdot 2H_2O$) and zinc sulphate ($ZnSO_4$) have been used for nanosol preparation.

D. Zinc Oxide Nanosol Preparation and Textile Treatment

Nanosol was prepared by a typical controlled hydrolysis. Solution A was prepared by adding TEOS slowly into alcohol with continuous stirring.

Solution B was prepared by mixing deionized water and hydrofluoric acid.

Solution A was added slowly into solution B stirred for 30 minutes and after mixed with 2.5% of zinc acetate or zinc sulphate with continuous stirring for 10 minutes. The obtained nanosol was clear and homogeneous.

The cotton textile samples were dipped into the prepared nanosols, soaked for 10 minutes, then dried at 80°C for 10 minutes, cured at 100°C, 125°C and 150°C for 5 min.

III. Results and Discussion

A. Thermal Post-Treatment Influence on the Cotton Textile Samples after the Sol-Gel Method Modification

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 curing durations from 1 to 30 minutes as reported in the literature [1, 25-28]. In the present research, the cotton textile samples have been cured at 100°C, 125°C and 150°C for 5 minutes [3,5].

After thermal post-treatment at 150°C, the yellowness of the samples coated by two different nanosol solutions (prepared using zinc acetate and zinc sulphate) increased, whereas the samples coated by nanosol (prepared using zinc sulphate) also lost their strength, the textile fibres were damaged, and easily broke up in hands.

In order to explain the increase in yellowness, samples made of untreated pure cotton textile were prepared and cured at the same temperature as the samples treated by nanosol. The experiment showed that the yellowness of the untreated samples also increased. Thus, it can be concluded that the duration of curing being 5 minutes at a temperature of 150°C is too long for the samples made of pure cotton textile. For the next experiments it is necessary to reduce the curing duration.

In the case of samples treated by nanosol (prepared using zinc sulphate), decrease in strength and destruction of cotton textile can be explained by possible silicic acid origination on the fabric surface during the chemical reaction and the thermal post-treatment. The same level of destruction was observed for polyamide, polyester and flax fibre samples. The experiment results showed that flax, polyamide and polyester textiles treated by nanosol (prepared using zinc acetate) could be cured at a temperature of 150°C for more than 5 minutes (≤10 minutes). It can be concluded that for samples treated by nanosol (prepared using zinc sulphate) the curing temperature of 150°C is too high and unsuitable for sample preparation.

Experiment results show that a moderate thermal post-treatment at 125°C is appropriate for cotton samples coated by nanosol (prepared using zinc acetate), but is too severe for samples treated by nanosol (prepared using zinc sulphate). As a result of such post-treatment the samples treated by nanosol (prepared using zinc sulphate) change their color to black, it means that deep destruction of cotton fabric occur and post-treatment temperature has to be reduced at least till 100°C. It is considered that zinc sulphate monohydrate is a safe source of zinc for all animal species and the use of zinc-containing feed additives does not pose a direct concern for the agricultural soil compartment [29]. At the same time, our investigations show that some further refinement in the assessment of zinc sulphate-based nanosol application to the textile treatment needs to be considered, for which additional data would be required.

B. Scanning Electron Microscopy

With respect to the textile structure and since the applied coatings are extremely thin, commonly used peelings tests appear to be unsuitable. Tests with adhesive tapes may give certain information, but since the coatings are mostly colourless, a simple visual evaluation will fail; it is therefore necessary to employ at least optical or SEM. In this research, morphological changes as a result of sol-gel treatment of cotton textile have been investigated using SEM.

Fig. 1 illustrates SEM micrograph of the uncoated cotton textile. Fig. 2 and Fig. 3 represent micrographs of cotton textile samples treated by two different nanosol solutions – prepared using zinc acetate with thermal post-treatment at 125°C and prepared using zinc sulphate with thermal post-treatment at 100°C.

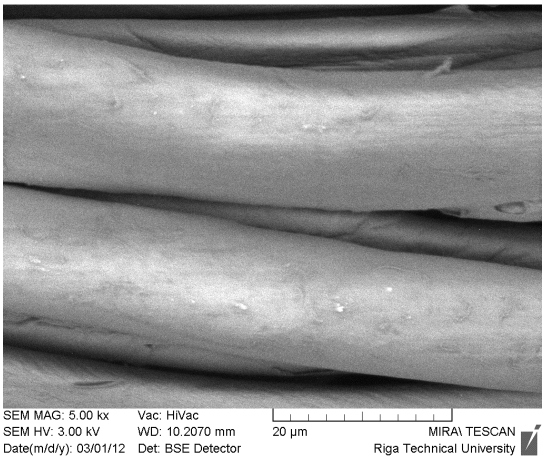

Fig. 1. Uncoated cotton textile

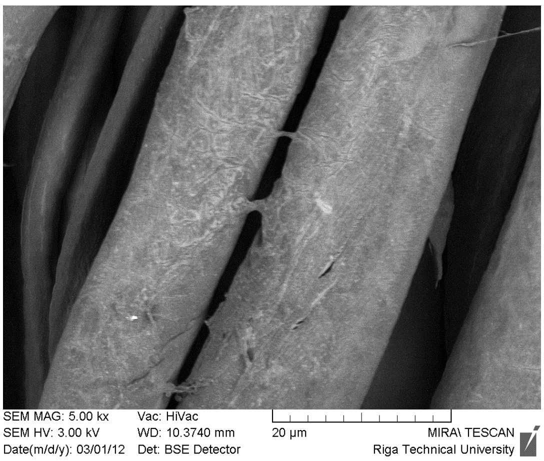

Fig. 2. Coated cotton textile by nanosol prepared using zinc acetate, thermal post-treatment temperature of 125°C.

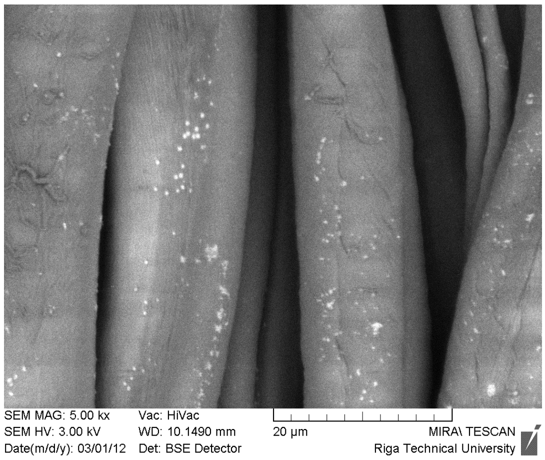

Fig. 3. Coated cotton textile by nanosol prepared using zinc sulphate, thermal post-treatment temperature of 100°C.

Figure 2 indicates that samples treated by nanosol (prepared using zinc acetate) have sufficiently even coating. SEM micrograph in Fig. 3 shows that the cotton samples treated by nanosol (prepared using zinc sulphate) have sufficiently even coating, without visible defects; though, in some places one can notice the presence of silica particles (Table 1 and Fig. 4).

TABLE I

ENERGY DISPERSIVE X-RAY (EDX) ANALYSIS – ELEMENTAL WEIGHT IN %

Spectrum	C	O	F	Si	Zn
1	67.47	20.64	6.02	-	5.86
2	62.40	23.82	5.58	-	8.19
3	74.70	17.08	5.65	-	4.57
4	47.19	23.35	4.54	13.45	11.47
5	33.03	17.60	-	49.37	-

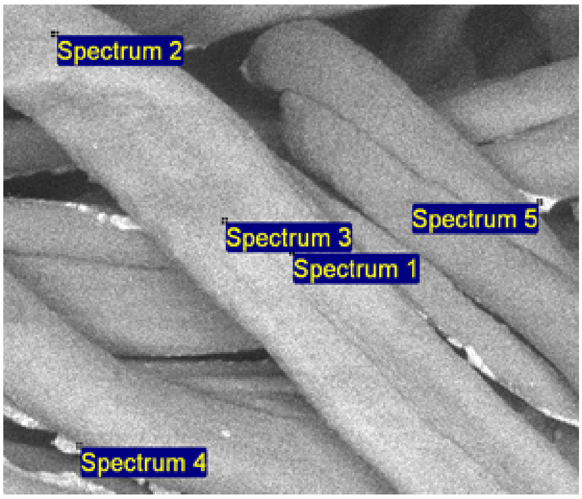

Fig. 4. EDX analysis spectra of sample treated by nanosol prepared with zinc sulphate concentration of 2.5%, thermal post-treatment temperature of 100°C

EDX analysis is an analytical technique commonly used for the analysis of chemical compositions. EDX has been performed, using SEM. Figure 4 illustrates, where the surface chemical composition of treated cotton is measured. Data in Table 1 confirms that zinc oxide coating is formed at the cotton textile surface, and confirms the presence of silica particles (spectrum 4 and spectrum 5).

IV. Conclusions

After thermal post-treatment at 150°C, the yellowness of the samples coated by nanosol (prepared using zinc acetate) increased, whereas the samples coated by nanosol (prepared using zinc sulphate) also lost their strength and easily broke up in hands. Taking into account that both the cotton fabric and the fibre structure were damaged, post-treatment temperature has to be reduced at least till 100°C. As zinc sulphate is considered to be a safe source of zinc for human beings and animals, further refinement in the assessment of zinc sulphate-based nanosol application to the textile treatment needs to be considered, for which additional data would be required.

Experiment results show that a moderate thermal post-treatment at 125°C is appropriate for samples coated by nanosol (prepared using zinc acetate), but is too severe for samples treated by nanosol (prepared using zinc sulphate). As a result of such post-treatment, samples treated by nanosol (prepared using zinc sulphate) change their color to black, it means that deep destruction of cotton fabric occur and post-treatment temperature has to be reduced at least till 100°C.

The sol-gel approach used within the framework of the research in order to prepare the coating materials guarantees a simple processing procedure that can be easily transferred to the textile industry, as well as nanosol can be applied by conventional coating techniques used in the textile industry – the application can be carried out by a simple dipping process, spraying or padding.

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Svetlana Vihodceva, Silvija Kukle. Plānie parklājumi uz kokvilnas jēlauduma, izmantojot sol-gēla metodi
Rakstā aprakstīta no dabīgām šķiedrām iegūto drānu modificēšana, izmantojot sol-gēla metodi, lai piešķirtu tekstilmateriāliem ultravioleto (UV) starojumu bloķējošas īpašības, uznesot plānu cinka oksīda pārklājumu. Tā kā sol-gēla metodei ir svarīga termiskā pēcapstrāde un tās temperatūra, rakstā tika novērtēta dažādu termiskās apstrādes temperatūru ietekme uz kokvilnas jēlauduma paraugiem.
Kopš tā laika, kad ozona slānī parādījies caurums un tiek novērota nepārtraukta ozona slāņa biezuma samazināšanās, palielinās arī UV starojuma intensitāte, kas savukārt rada nepieciešamību pēc UV starojumu bloķējošiem izstrādājumiem. Rakstā aprakstītajos pētījumos kā UV starojuma absorbētājs tika izmantots cinka oksīds, jo tam piemīt laba UV staru absorbcijas spēja. Cinka oksīds nav kaitīgs cilvēka veselībai, to plaši pielieto kosmētikā, piem., sauļošanās krēmos. Balstoties uz augstākminētajiem faktiem, var secināt, ka cinka oksīds ir piemērota viela UV starojumu absorbējošu pārklājumu izgatavošanai. Nanokoloīdu šķīdumu var uzklāt uz drānu un pavedienu virsmas ar tradicionālām, tekstila industrijā pielietotām tehnoloģijām. Pārklājumu uz tekstilmateriālu virsmas var iegūt ar vienkāršu mērcēšanu, uzputināšanu vai drukāšanu.
Paraugu virsmas izmaiņu analīze pēc to pārklāšanas ar cinka oksīdu tika veikta ar skenējošā elektronmikroskopa palīdzību. Eksperimentu gaitā tika konstatēts, ka kokvilnas jēlauduma paraugu, kas apstrādāts ar nano koloīdo šķīdumu, izmantojot cinka acetātu, optimāla termiskā pēcapstrādes temperatūra ir 125°C. Pie lielākas temperatūras kokvilnas jēlaudums paliek dzeltens. Paraugiem, kas apstrādāti ar nanokoloīdo šķīdumu, izmantojot cinka sulfātu, optimālā temperatūra ir 100°C; pie 125°C temperatūras kokvilnas jēlaudums vietām paliek melns, bet pie 150°C temperatūras zaudē savu stiprību un viegli sabrūk.

Светлана Выходцева, Сильвия Кукле. Тонкие покрытия необработанного хлопка, используя золь-гель метод
В статье описано использование золь-гель технологии для модификации полотен из натуральных волокон, чтобы придать им защитные свойства, против ультрафиолетового излучения, нанося тонкое покрытие оксида цинка. Так как в использовании золь-гель метода важна температура последующей термической обработки, в статье описано воздействие различных температур на обработанные образцы.
С того времени, как появилась дыра в озоновом слое, так и из-за непрерывного уменьшения толщины озонового слоя, увеличилась интенсивность ультрафиолетового излучения, что привело к увеличению необходимости защиты против ультрафиолетового излучения.
В описанных в статье экспериментах в качестве поглотителя ультрафиолетового излучения был использован оксид цинка, которому свойственна хорошая поглощаемость ультрафиолетового излучения. Оксид цинка безвреден для здоровья людей, поэтому он используется, например, в кремах для загара. Исходя из выше сказанного, оксид цинка кажется идеальным, чтобы создать покрытие, высоко поглощающее УВ излучение, основанное на золь-гель методе. Нанораствор может быть нанесён на поверхность текстильных материалов при помощи традиционных, используемых в текстильной индустрии технологий. Покрытие на поверхности материалов можно получить при помощи обычного замачивания, напыления или набивки.
При помощи электронного сканирующего микроскопа был произведён анализ изменений поверхности образцов после их покрытия оксидом цинка. Путём экспериментов констатировано, что покрытие при помощи золь-гель технологии на поверхности натуральных полотен можно получить при помощи обычного замачивания. А так же путём экспериментов констатировано, что для образцов обработанных наноколлоидным раствором, полученным используя ацетат цинка, оптимальной температурой для термической обработки является 125°С, при использовании большей температуры образцы желтеют, а для образцов обработанных наноколлоидным раствором, полученным, используя сульфат цинка, оптимальной температурой для термической обработки является 100°С, при температуре 125°С образцы местами чёрнеют, а при воздействии температуры 150°С теряют прочность и легко разрушаются.