

The Technological Development of Testing Methodology for Textile Zinc Oxide Coating Durability Determination

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Abstract – This article reflects one development stage of the formation process of metal coated textile quality control methodology based on the analysis of sewage electrograms. The experiment has been carried out with the help of gas discharge visualization camera. To determine how the electrogram parameters reflect the presence of nanoparticles in water, the ZnO nanoparticles have been added to five distilled water samples in the following concentrations: 10%; 8%; 6%; 4%; 2%. According to the experiment results, the electrography method can be used for textile zinc oxide coating durability testing.

Keywords – GDV electrography, textile testing methodology, zinc oxide coatings on textile, zinc oxide nanoparticles.

I. INTRODUCTION

Nanotechnology has gained a great deal of public interest due to the needs and applications of nanomaterials in many areas of human activities such as industry, agriculture, business, medicine, public health and so forth. Zinc oxide (ZnO) is used in a variety of applications typically as an additive in a range of products including plastics, ceramics, glass, cement, rubber (e.g., car tyres), lubricants, paints, ointments, adhesives, sealants, pigments, food (source of Zn nutrient), batteries, ferrites, fire retardants, etc. Zinc oxide is also used in the textile industry with the aim of improving such characteristics of the fabric as antibacterial effect and blocking of UV radiation.

Among new features, which can be added to traditional products by means of modern technology, it is necessary to gain confidence that their entry into the manufacturing and household will not cause irreversible changes in living and working environment. The nanoscale particles as opposed to the substance, from which they come, are very active and released into the environment, including air and waste water, can cause serious problems, which should be taken into account in product design, manufacturing, use and removal. Despite positive effects of metal coatings, metal is required by living organisms in small quantities, but large quantities can cause toxicopathy and pollute the environment. Extensive research has been conducted on the metal nanoparticle effect on people and the environment; for example, the toxicity investigation of several metal oxide aqueous suspensions to Zebrafish early developmental stage has shown that ZnO is the most toxic substance to Zebrafish embryos and larvae. [1]. Comparing the various metal oxide nanoparticle effect on mammalian cells, it has been found that zinc oxide causes substantial cell mitochondrial changes and necrosis. The mitochondrial function (MTT) results have shown that ZnO

exhibits more toxicity than other metal oxide nanoparticles [2].

The main risks of the use of metal coated textile are related to the separation of metal nanoparticles. Nanoparticles can be detached from the product as a result of friction during manufacturing and usage processes, leading to pollution of environment, or inhaled by humans or animals. Nanoparticles can also be detached from the textile in the washing process, thereby contaminating the sewage and exterminating useful bacteria, causing harm to other inhabitants of water reservoirs. To avoid these risks, it is required to use a simple, fast and effective method for metal coated textile testing.

There are only a few methods employed for testing nanoparticles in water, they are as follows: Microscopy methods, Photon Correlation Spectroscopy and Nanoparticle Tracking Analysis [3]. The analysed tools vary in the required sample number for measurements, preparation techniques and the resulting parameter range. Microscopy methods require prior sample preparation; the necessary pre-treatment is problematic because the sample may react or decompose during the preparation. Photon Correlation Spectroscopy methods require additional measurements to calculate the parameters. Most of the devices cannot distinguish agglomerates from individual nanoparticles, which limits their use, because the toxicity of nanoparticles depends on their size – the size reduction increases toxicity. The toxicity of the nanoparticle agglomerates and particular individual nanoparticle is higher than the toxicity of the whole substance of the same size. Consequently, the creation of a new method will significantly facilitate the textile technologists' obligations, whose work is related to new material creation and testing.

The operating principle of gas discharge visualization (GDV) electrography is based on the Kirlian effect. High-frequency (1.1 kHz), high-voltage (5 kV) electric discharge affected radiation around a living or inanimate object is registered with a digital camera. GDV electrograms represent complex two-dimensional fractal shapes, whose area and spectral indicators provide information about the structure and properties of the object. The resulting images are analysed by specialised software. This approach to the image analysis has been implemented in software package "GDV Technique", on its base a series of programs/equipment has been created for the analysis of different origin objects.

GDV camera is commonly used for medical research – allergy diagnosis [4], diagnosis of autism [5], the detection of changes in the characteristics of blood, energetic preparations, homeopathic remedies under influence of different factors [6].

GDV camera is widely used in sports medicine – for athletes' fitness and health determination [7]. In the light of the extensive use of GDV electrography, it is necessary to adapt the method for detection of nano-level metal particles in water.

II. METHODOLOGY

For liquid testing with GDV Camera, it is required to use accessories from the package "GDV Mini-Lab". GDV electrogram acquisition is made by GDV Capture software. GDV Scientific Laboratory software provides static and dynamic electrogram parameter processing, ensures a range of solutions: the calculation of numerical value for entire picture and a single sector; the calculation of each sample characteristics using the selected parameters; trend calculation; entropy and fractal analysis of dynamic characteristics; visual analysis and comparisons with the initial GDV electrograms; calculation of numeric data blocks [8].

GDV Scientific Laboratory software calculates 12 parameters for each electrogram. Important parameters are as follows: area, intensity, form coefficient and entropy. The most important parameter is the area – the number of pixels with intensity, which is higher than that in filtering set noise level [10], but the intensity is brightness of the pixels. Form coefficient characterizes the size of image edge indentations (the larger the form coefficient, the higher indentations are), but entropy is the measurement of informativity and deviation from balance – it diminishes gaining the equilibrium. The article focuses on electrogram area and intensity. In the previous study [11] it was concluded that the form coefficient and entropy of GDV electrogram did not represent the zinc oxide concentration in water.

To determine how the presence of zinc oxide nanoparticles in the water is reflected in the electrogram parameters, different concentrations of ZnO nanoparticle powder have been added to the five samples of distilled water (Table 1). For comparison pure distilled water (Control) has been used.

TABLE I
CHARACTERISTICS OF EXPERIMENTAL SAMPLES

Sample designation	ZnO concentration (w_{cmpd}), %	Size of ZnO nanoparticles, nm
Zn2A	10	48 – 58
Zn2B	8	48 – 58
Zn2C	6	48 – 58
Zn2D	4	48 – 58
Zn2E	2	48 – 58
Control	0	-

ZnO nanoparticle concentration is expressed in the parts of mass (1):

$$w_{cmpd} = \frac{m_{cmpd}}{m_{cmpd} + m_{slvt}}, \quad (1)$$

where m_{cmpd} – the mass of the substance and m_{slvt} – the mass of the solvent.

Before GDV electrography session, a bowl with water has been shaken thoroughly to disperse the sludge. Prepared water (0.2 ml) has been drawn into a syringe, and a syringe has been fixed on a stand above the GDV camera lens. From the same sample 5 GDV static electrograms have been obtained. The experiment has been repeated eight times, resulting in the 40 files with the same time interval (5 seconds) between electrogram fixing moments. In GDV camera settings it is possible to enter the time interval starting from 3 seconds; whereas in the previous experiments [11] it was found that the time interval did not have any significant effect on the results, and the decision was made to use a 5-second interval between electrogram fixations in further experiments. Equipment test results [8] have shown that for sufficiently reliable data it is necessary to obtain at least 40 measurements per experimental subject.

During the previous experiments it was found that the amplitude of the results increased [11], [12]. The following possible causes of increased fluctuations were mentioned: electromagnetic radiation from the computer and other equipment located in the venue of experiment, as well as the static electricity resulting from the operator's clothing friction. In order to reduce them, in the present experiment an antistatic wristband has been used, and water samples have been stored in metal containers, thereby isolating them from electromagnetic radiation.

GDV electrograms have been fixed with gas discharge visualization camera "GDV Camera Pro", using accessories from the GDV Mini-Lab for the liquid analysis. The data has been recorded in the GDV Capture software and processed in GDV Scientific Laboratory, which automatically calculates 12 parameters for each electrogram. The parameter analysis has been performed in Microsoft Office Excel software. To create the testing methodology, mathematical statistical methods have been used in order to reduce the variations of the main parameters and enable the distribution of the dependent variables that react to metal nanoparticles in the liquid. In the process of electrogram analysis, for each parameter the following statistical indicators have been calculated: the arithmetical mean, variance, standard deviation, range and the average relative standard error.

III. RESULTS

Electrogram area and intensity acquired in the experiment are summarized in Table 2.

TABLE II
IMPORTANT INDICES OF GDV ELECTROGRAM AREA AND INTENSITY

Sample designation	ZnO concentration, %	Average relative standard error of area, %	Average relative standard error of intensity, %
Zn2A	10	1.02	0.51
Zn2B	8	0.94	0.53
Zn2C	6	0.66	0.44
Zn2D	4	1.23	0.32
Zn2E	2	1.23	0.47
Control	0	1.87	0.42

Average relative standard error for any data package does not reach 2%, which means that the representativity of the all data is considered to be excellent. In general, for the intensity a lower relative standard error level is less than 1%.

By performing the data analysis of selection, it has been concluded that a straight line best describes the datasets "Area-concentration" (Fig. 1) and "Intensity-concentration" (Fig. 2).

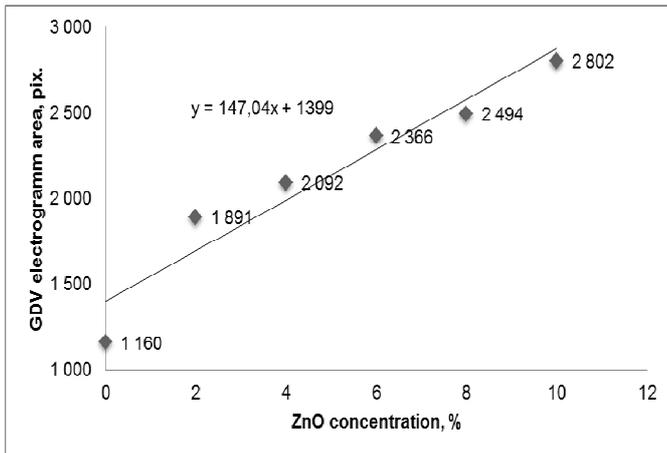


Fig. 1. Arithmetic mean of GDV electrograms and the descriptive lines of relationship "Area-concentration"

Based on the values of the coefficient of determination, it can be concluded that linear regression model describes 92.3% changes in the area and 95.6% of changes in the intensity. The F-test *p-value* is $0.002 < 0.05$ for the area and $0.0007 < 0.05$ for the intensity; it means that with 95% probability it confirms the hypothesis that between features "Area-concentration" and "Intensity-concentration" there is a linear relationship. Whereas the hypothetical test value $\beta_i = 0$ does not belong to the interval $205.85 \geq \beta_i \geq 88.23$ (area) and $1.02 \geq \beta_i \geq 0.56$ (intensity), then with probability of 95% the null hypothesis can be rejected and assumed that the changes in concentration are an important factor that affects both the area and intensity.

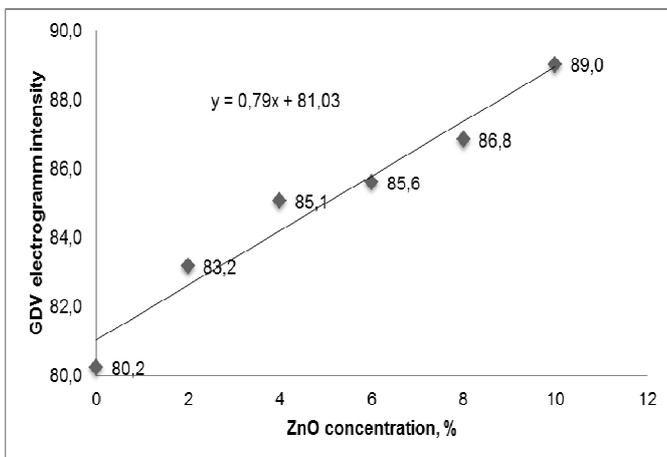


Fig. 2. Arithmetic mean of GDV electrograms and the descriptive lines of relationship "Intensity-concentration"

The average arithmetic results (Fig. 1, Fig. 2) show that with the increase in the zinc oxide concentration in distilled water, the GDV electrogram area and intensity also increase. By adding the smallest amount of zinc oxide (2%), the electrogram area increases rapidly, but starting with the 4% threshold, the area increases practically linearly. Similar results are also for the intensity. The area growth is shown in Fig. 3 – by adding zinc oxide nanoparticles to distilled water, the electrogram area is increased by 63% in relation to the Control water. Among the other samples there are not so pronounced differences (5–13%). It means that the GDV camera reacts very sensitively to the presence of zinc oxide nanoparticles in distilled water, which in turn opens up a wide range of possibilities for metal coated textile testing methodology development, because it is important that in the measurement results directly reflect the lower concentrations, thereby identifying the smallest amount of ZnO separated from the fabric.

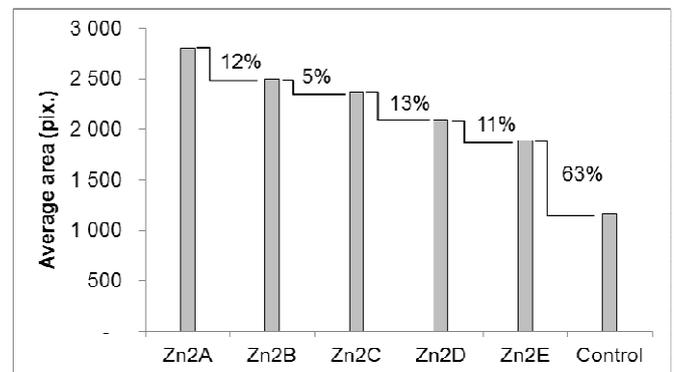


Fig. 3. Percentage changes in the area related to the concentrations of the samples.

Less pronounced differences have been observed in the intensity indicators (Fig. 4), the intensity difference between the Control and the lower of the concentrations of ZnO is 3.7%; among the other samples there is even smaller difference (0.6–2.5%). The intensity is less susceptible GDV characteristic to zinc oxide concentration changes.

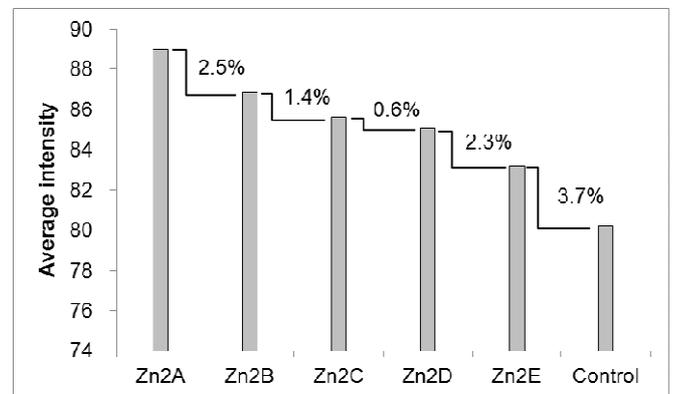


Fig. 4. Percentage changes in the intensity related to the concentrations of the samples.

Fig. 5 shows the percentage of interdecile range in relation to the arithmetic mean. Obviously, the greatest fluctuations are observed in Control water (31%) and water samples with the lowest ZnO concentration. Also in the previous experiments, increased fluctuations of measurements were observed [11]. To prevent them, in this experiment an antistatic wristband and a metal container for water storage have been used. The description of the experimental samples is summarized in Table 3.

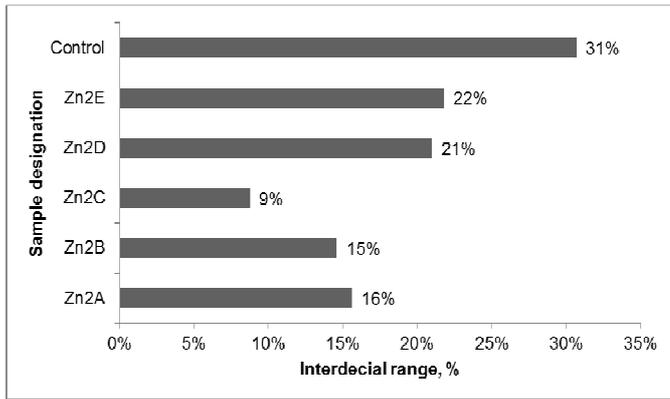


Fig. 5. Percentage of interdecile range (I_{80}) for GDV electrogram area: experiment with ZnO concentration of 2-10%.

By comparing the experiment analysed in this article (Fig. 5) and the previous experiment (Fig. 6), it can be seen that the dispersion diminished: in this experiment interdecile amplitude ranges from 9% to 31%, but the previous experiment it was 26-49%.

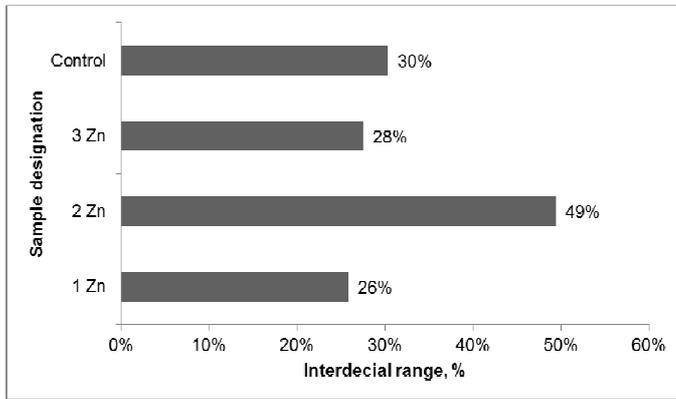


Fig. 6. Percentage of interdecile range (I_{80}) for GDV electrogram area: experiment with ZnO concentration of 0.5-1.5%.

According to the interdecile range, the average relative standard error was 1.75–2.30% in the previous experiment (Table 3), but in this experiment it is just 0.66–1.87% (Table 2).

TABLE III
CHARACTERISTICS OF EXPERIMENTAL SAMPLES AND STATISTICAL INDICATORS [11]

Sample designation	1 Zn	2 Zn	3 Zn	Control
ZnO concentration, %	1.5	1.0	0.5	0.0
Size of ZnO nanoparticles, nm	30 - 60	30 - 60	30 - 60	-
Average relative standard error of area, %	2.29	2.30	1.77	1.75
Average relative standard error of intensity, %	0.92	0.81	0.91	0.61

By comparing both experiments, not only the difference in amplitude is observed, but also different types of descriptive functions – the polynomial model of non-linear regression suits best the previous experiment (Fig. 7, Fig. 8).

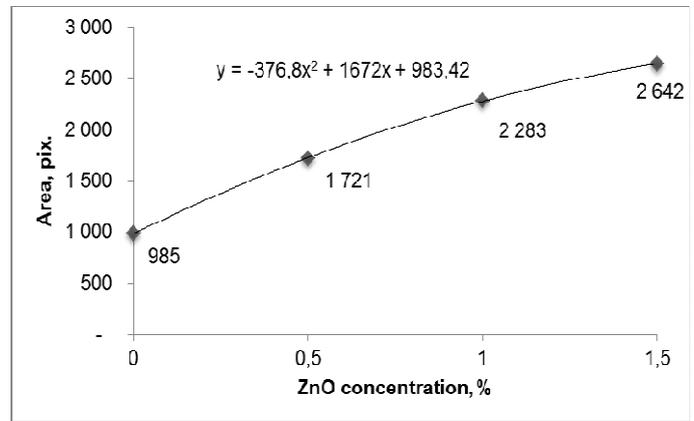


Fig. 7. Arithmetic mean of area and the descriptive polynomials for experiment with ZnO concentration of 0.5–1.5%.

To estimate the regression equation, a new variable x^2 has been introduced. Based on the values of the determination coefficient, it can be concluded that the polynomial regression model describes 100% the changes of area and intensity. The F-test p -value is $0.005 < 0.05$ for the area and $0.02 < 0.05$ for the intensity, it means that the regression equation statistically significantly explains the changes in the performance indication. Whereas, the limits of the confidence interval ($1931.28 \geq \beta_1 \geq 1412.74$ and $-211.15 \geq \beta_2 \geq -542.44$ (area); $24.39 \geq \beta_1 \geq 10.89$ and $-3.00 \geq \beta_2 \geq -11.63$ (intensity)) do not include hypothetical values $\beta_1=0$ and $\beta_2=0$, and the corresponding p -values are less than 0.05 (0.01 and 0.02 (area); 0.02 and 0.03 (intensity)), then the factor x and x^2 is an important with probability of 0.95.

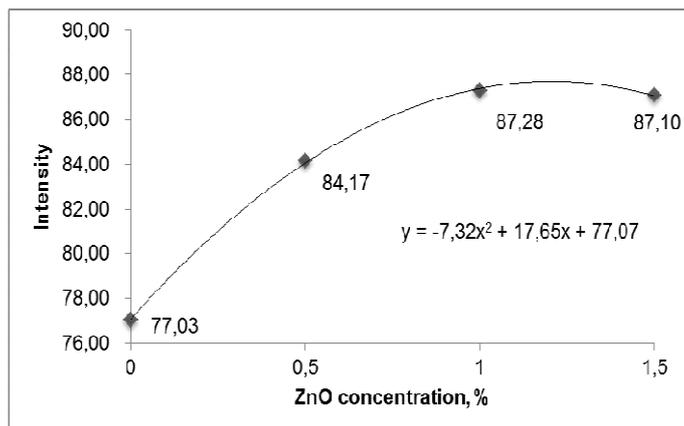


Fig. 8. Arithmetic mean of intensity and the descriptive polynomials for experiment with ZnO concentration of 0.5–1.5%.

Comparison of both experiments has shown that the average values of the area do not form a single graphic representation – for ZnO concentration of 1.5% the average area is 2642 pixels (Fig. 7), but for the ZnO concentration of 2.0% the average area is smaller (1891 pixels – Fig. 1), but it should be larger. This is explained by the fact that in each experiment zinc oxide nanoparticles of different origin and size have been used (Table 1 and Table 3).

IV. CONCLUSIONS

This article partially describes the development process of the metal coated textile testing methodology. In this article main attention has been devoted to the detection of zinc oxide nanoparticle concentration through GDV electrogram parameters and the way of stabilising the results in order to reduce their amplitude. In previous studies carried out by the authors, textiles coated with copper nanoparticles were investigated [3], [12] and results showed significant changes in GDV electrogram parameters in comparison with the control indicator. It can be concluded that the GDV camera can be used for textile metal coating durability testing. Future studies should focus on zinc oxide film resistance to the textile fabric through detection of particles in water separated from the fabric during the washing process, as well as attention should be devoted to the effect on electrogram parameters that will lead to the necessity to adjust the results. However, in view of the fact that the electrogram parameters have changed by adding the powder of nanoparticles to water, it can be concluded that the GDV camera is able to capture the presence of nanoparticles in distilled water.

Previous studies showed that the GDV Camera Pro electrograms reacted very sensitively to metal nanoparticle impurities in distilled water [3], [10], [11], [12]. It proves the experiment analysed in this article – the rapid growth of the electrogram area, after adding zinc oxide nanoparticles to distilled water (63% in relation to the Control water), confirms the potential of GDV electrography in metal coated textile testing. For textile metal coating testing it is especially important that the lower concentrations are reflected in the measurement results because nanometal amount that separates

from the textile in the washing process is usually small. Changes in the intensity affected by zinc oxide are smaller than the changes in the area (0.6–3.7%).

In the previous experiments, one of the main problems was the large amplitude fluctuations of measurements. To prevent these fluctuations, in this experiment an antistatic wristband for static electricity grounding and metal containers for the storage of water samples have been used to protect samples from the electromagnetic radiation generated by electrical devices. The comparison of the results of the present experiment with the results of the previous experiments has shown that the total range of amplitude fluctuations has decreased. Consequently, the aforementioned practice can be used to stabilize the results.

The results of both experiments have shown that the character of the area and intensity relations differs in terms of particle concentration range – if the concentration is small (up to 2%), connection between corresponding values of concentration and area/intensity is non-linear by nature; if the increase in the nanoparticle concentration is above 2%, the relationship approaches the linear character. It is possible that the certain effect is also caused by such nanoparticle characteristics as different origin and dimensions; therefore, it should be taken into consideration when creating nomographs for assessment of nanoparticle concentration.

ACKNOWLEDGEMENTS

This research has been supported by the European Social Fund within the project “Establishment of Interdisciplinary Research Groups for New Functional Properties of Smart Textiles Development and Integration in Innovative Products”, No. 2009/0198/1DP/1.1.1.2.0./09/APIA/VIAA/148.



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Eva Trumsina, Silvija Kukle un Gunta Zommere. Tekstilmateriālu cinka oksīda pārklājuma noturības testēšanas metodikas tehnoloģiskā izstrāde

Nanotehnoloģijas ir ieguvušas lielu publikas interesi, pateicoties to pielietojumam dažādās nozarēs, kā, piemēram, rūpniecība, lauksaimniecība, medicīna, sabiedrības veselība u.c. Cinka oksīds tiek lietots arī tekstila industrijā, lai piešķirtu izstrādājumiem tādas īpašības kā antibakteriālā aizsardzība un ultravioleto staru bloķēšana. Šis raksts ataino vienu posmu no kopējās metālpārklātu tekstilmateriālu kvalitātes kontroles metodikas izstrādes procesa, kas balstīta uz tekstilmateriālu mazgāšanas procesā radušos notekūdeņu elektrogrammu analīzi. Eksperiments veikts, pielietojot gāzizlādes vizualizācijas kameru un specializētu programmu paketi, kā arī matemātiskās statistikas metodes. Lai noteiktu, kā elektrogrammu parametros atspoguļojas cinka oksīda nanodaļiņu klātbūtne ūdenī, pieciem destilētam ūdenim tika pievienots ZnO nanodaļiņu pulveris sekojošā koncentrācijā: 10%; 8%; 6%; 4%; 2%. Eksperimenta rezultāti pierāda, ka ar cinka oksīda nanodaļiņām piesārņota destilēta ūdens elektrogrammu parametri ir nozīmīgi palielinājušies salīdzinājumā ar kontroles ūdeni. Straujais laukuma pieaugums, pievienojot destilētam ūdenim cinka oksīda nanodaļiņas (par 63% attiecībā pret kontroles ūdeni), apliecina GDV elektrogrāfijas potenciālu metālpārklātu tekstilmateriālu testēšanā. Tekstilmateriālu metālu pārklājumu testēšanā īpaši svarīgi, lai mērījumu rezultātos atspoguļotos tieši zemākās koncentrācijas, jo nano metāla daudzums, kas atdalās no izstrādājuma mazgāšanas procesā, parasti ir neliels. Ņemot vērā eksperimenta rezultātus, var secināt, ka elektrogrāfijas metode ir pielietojama tekstilmateriālu cinka oksīda pārklājuma noturības testēšanai. Turpmākos pētījumos jākoncentrējas uz cinka oksīda pārklājuma noturības testēšanu, analizējot ar cinka oksīdu pārklātu tekstilmateriālu mazgāšanas procesā radušos notekūdeņu elektrogrammas, kas, iespējams, radīs nepieciešamību koriģēt rezultātus.

Эва Трумсина, Силвия Кукле, Гунта Зоммере. Технологическое развитие методологии тестирования для определения устойчивости покрытия оксида цинка на текстиле

Нанотехнология приобрела большой общественный интерес из-за применения наноматериалов во многих областях человеческой деятельности, таких, как промышленность, сельское хозяйство, бизнес, медицина, общественное здравоохранения и так далее. В текстильной промышленности оксид цинка используется с целью улучшения такие характеристики ткани как антибактериальная защита и блокирование ультрафиолетового света. В настоящей статье отражена одна стадия развития общего процесса формирования методологии по контролю качества текстиля с металлическим покрытием, который основан на анализе электрограмм сточных вод. Эксперимент был проведен с использованием камеры газоразрядной визуализации (ГРВ), используя пакет специализированного программного обеспечения и методы математической статистики для разработки методологии тестирования. Чтобы определить, как параметры электрограмм отражают присутствие наночастиц в воде, наночастицы оксида цинка были добавлены в пять образцов дистиллированной воды в следующих концентрациях: 10%; 8%; 6%; 4%; 2%. Результаты эксперимента показывают, что параметры электрограмм дистиллированной воды, загрязненной наночастицами оксида цинка, значительно увеличиваются по сравнению с контрольной водой. Быстрый рост площади электрограмм (63% по отношению к контрольной воде) после добавления наночастиц оксида цинка в дистиллированной воде подтверждает потенциал ГРВ электрографии в тестирование металлопокрытого текстиля. В тестирование текстиля с металлическим покрытием особенно важно чтобы в результаты измерения были отражены особенно более низкие концентрации, потому что количество nano металла, который отделяется от текстиля в процессе промывки, обычно мал. По данным результатов эксперимента можно сделать вывод, что метод ГРВ электрографии может использоваться для тестирования устойчивости покрытия оксида цинка в текстильной промышленности. Будущие исследования должны быть сосредоточены на тестировании устойчивости покрытия оксида цинка, анализируя электрограммы сточных вод, полученных в процессе мытья текстиля, что может вызвать необходимость уточнить результаты.