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STUDIES OF THE POSSIBILITY OF ESTIMATING THE GROWTH OF THE CU_XSE LAYER FROM THE CHANGE IN THE MASS OF FIBRES

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ABSTRACT

In this presentation, the part of the results of the investigation of possibilities of the formation of the copper selenide layer on the surface of the different fibres is presented. For the modification of the researched fibres, the two-stage adsorption-diffusion process was used. Thread waste from Lithuanian companies was investigated in the work. The aim of these studies is to evaluate the possibility of evaluating the formation of the layer by observing the change in mass after each cycle of modification. The presence of a layer of Cu_xSe particles on the surface of the treated fibres was confirmed by SEM analysis. The mass gain of modified fibres can be taken as an indicator of the coating process; however, in the case of natural fibres (especially cellulosic), the possibility of fine fibres pieces falling off should be considered.

KEYWORDS

Different nature fibres, modification, mass gain, cupper selenide, SEM.

INTRODUCTION

Recently, researchers face the challenging task of finding a way to use industrial waste as secondary raw material for new applications. At this time, it has often been emphasized that textile waste is one of the largest sources of pollution in the world. Using industrial textile waste for the creation of new functional composites is a viable area for the development of sustainable technologies. New composite materials for special purposes with varying combinations of physical and chemical properties can be obtained by using polymers modified with thin semiconductive or electrically conductive layers of binary inorganic compounds. In the past decades, semiconductor nanomaterials have received broad attention because of their novel electronic, optical, photoelectric, and thermoelectric properties. As an important semiconductor, copper selenide (Cu_xSe) with nanostructure has potential applications in various fields, such as optical filters, highly efficient solar cells, superionic conductors, electro-optical devices, photo-thermal conversion, electro-conductive electrodes, microwave shielding coatings, etc. [1].

In this study, the part of the results of the investigation of possibilities of the copper selenide layer formation on the surface of the different fibres is presented. For the modification of the researched fibres, the two-stage adsorption-diffusion process was used. In this work, thread waste from Lithuanian companies was investigated in this work.

The SEM analysis of modified materials surface and the determining of the chemical composition of the crystal agglomerates are most commonly used to confirm the fact of the successful samples treatment process. However, in the case of the multicycle modification process, in search of optimal process conditions or in fast pilot experiments, it is appropriate to observe the weight gain of the modified samples. The textile mass gain can be taken as an indicator of the coating process. Successful modification of the semiconductor metal oxide cluster compound by a series of Ti:Si molar compositions of the fibre surface of the cotton textile was also confirmed with an increase in mass [2]. Scientists from England and Germany studied the possibilities of coating polyester cloth with Cu particles. In this work, the efficacy of different catalysts was determined by characterising electroless copper plated textiles in terms of mass gain (after plating), coating coverage, and deposit morphology using SEM [3]. A group of scientists from the Czech Republic proposed a simple way to prepare electrically conductive multifunctional fabrics made by imparting copper nanoparticles followed with copper electroplating. An increase in electrical properties was analysed with the weight gain rate of coated knitted fabric (Cotton:Nylon:Elastane / 80:15:5) was analysed [4]. Because it is known that chemical finishing on wool fabric generally increases the fabric weight, this indicator was used to evaluate and explore the new wool bleaching approach [5].

MATERIALS AND METHODS

The investigated samples were treated in a thermostatic vessel using a continuously stirred $K_2SeS_2O_6$ solution. The 0.1 mol×dm^{-3} solutions of $K_2SeS_2O_6$ in 0.1 mol×dm^{-3} HCl were used. Then, the samples were treated with a Cu(II/I) salt solution, and and the copper selenide layers on the samples formed. The Cu(II/I) salt solution was prepared using a crystalline $CuSO_4 \cdot 5H_2O$ and a reducing agent hydroquinone. It is a mixture of univalent and divalent copper salts consisting of 0.34 M Cu(II) and 0.06 M Cu(I) salt. A more detailed modification method is provided in a previously published article [6].

The mass of the investigated fibre samples was measured with AB104-S Analytical Balance (Mettler Toledo) featuring a measurement range of 110 g ± 0.1 mg, a scale interval of 0.1 mg and an error (0 ± 0.1) mg. The percentage change in the mass of the samples Δm was calculated according to the formula:

$$\Delta m = ((m_n - m_0)/m_0) \times 100\% \qquad [1]$$

where, m_0 is the mass of an untreated sample (mg); m_n is the mass of the sample after treatment (mg); n is the number of the treatment cycle.

Nine types of waste from different fibres (cotton, flax, bamboo, ramie, wool, PES, PA6, PA66, and PAN) were used for this experiment.

RESULTS AND DISCUSSION

This work used a two-step adsorption/diffusion method for the formation of a copper selenide coating on the surface of different nature fibres threads waste has been successfully applied to the modification of wool fibres in previous work [6]. In order to reduce emissions from industrial textile waste, unused residues of various fibres can be adapted for the development of new functional composites. The possibilities of modifying the samples under analogous conditions of other different polymeric nature fibres were investigated.

A successful course of the treatment process is the visible change in the colour of the investigated fibres. The change in colour of the investigated samples from white to grey indicates the formation of copper selenide crystals on the surface of the thread microfibers. By repeating the modification cycles, the coating layer of Cu_xSe thickens and the fibre samples become completely black (Fig. 1)

Figure 1. Example of modified fibres (from left to right): before treatment, after the first cycle of treatment, after the second cycle of treatment.

In the case of the modification of the wool fibres, the formation of a Cu_xSe crystal coating was visibly confirmed by an increase in the mass of the investigated wool fibres after each treatment cycle (Fig. 2).

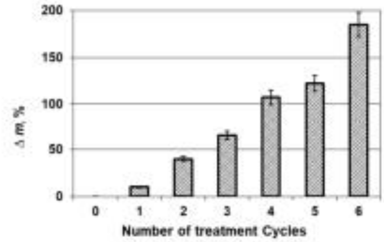


Figure 2. Influence of the number of treatment cycles on the fibres mass change [6].

As can be seen in Figure 3, a similar tendency of results was obtained for synthetic fibres. It can be seen that the mass growth of different nature synthetic fibres differs and the mass growth of PES fibres clearly increases less, but in all cases, we can see the nature of the tendentious process.

Meanwhile, in experiments with some natural fibres, it has been observed that the mass of the test specimens often decreases after the first treatment cycles. Although the SEM examination showed that the first crystal agglomerates were already formed on the surface of all fibres after the first treatment cycle (Fig. 4), the split-off of short parts of the fibres during the treatment process resulted in an overall decrease in sample weight.

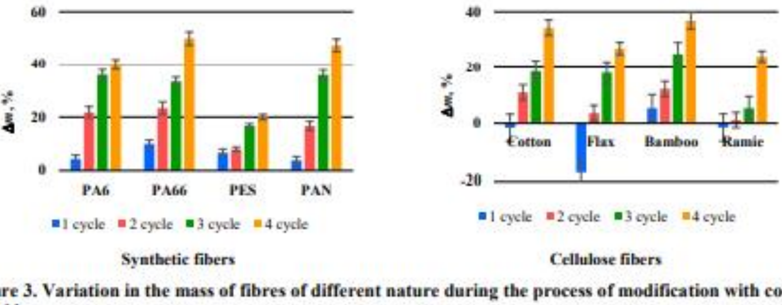
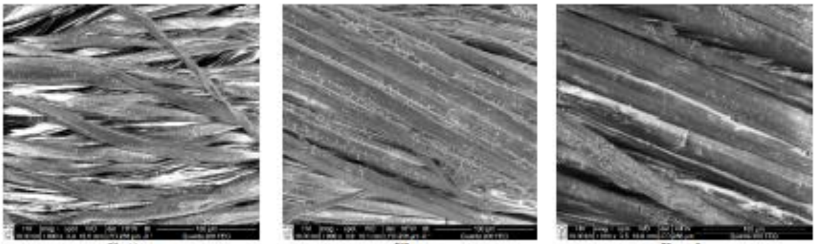


Figure 3. Variation in the mass of fibres of different nature during the process of modification with copper selenide.

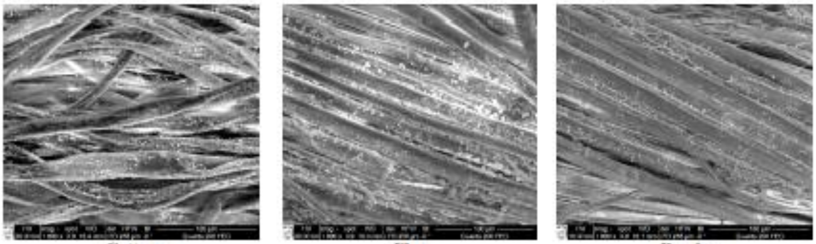
Both the photos of the treated thread in Figure 1 and the SEM images in Figure 4 show that after the first cycle of modification, the surface of the cellulosic filaments is not yet covered with a continuous layer of copper selenide crystals.



Cotton **Flax** **Ramie**

Figure 4. SEM images of copper selenide coated fibres after the first treatment cycle (mag. 1000×).

Greater weight loss after the first cycle was observed by treating the threads with higher hairiness such as flax and ramie, but a similar decrease of mass was also observed for cotton.



Cotton **Flax** **Ramie**

Figure 5. SEM images of copper selenide coated cellulose fibres after the second treatment cycle (mag. 1000×).

Although the copper selenide coating layer formed on the surface of the fibres after the second treatment cycle visibly thickens (Fig. 5), the loss of mass of the high-hairiness fibres due to short part breakdown is the factor that decides the peculiarity of fibres mass gain. In all cases, the situation changes only after the third or even fourth cycle of treatment. It can be assumed that the growing and thickening layer of copper selenides strengthens and splices the fine filaments of the treated fibres, which causes the mass of the samples to increase visibly.

Therefore, the experiment showed that the weight gain dependence of the investigated samples after the first few treatment cycles may be inaccurate when working with natural cellulosic fibres.

CONCLUSION

The experiment showed that monitoring the weight gain of the samples after each treatment cycle is a suitable way to evaluate the growth of the cupper selenide crystal layer when the yarns of synthetic polymer fibres and wool are modified.

In the case of natural cellulose, monitoring of fibre mass growth after the first treatment cycles does not provide accurate data due to the separation and fall of fine particles of modified fibres.

REFERENCES

[1] Hussain R.A., Hussain I., *Copper selenide thin films from growth to applications*, Solid State Sciences 2020, vol. 100, no 106101, doi:10.1016/j.solidstatesciences.2019.106101.

[2] Rilda Y., Syukri F., Alif A., Aziz H., Chandren S., Nur H., *Self-cleaning TiO2-SiO2 clusters on cotton textile prepared by dip-spin coating process*, Jurnal Technology (Sciences & Engineering 2016, vol. 78, no 7, pp. 113-120.

[3] Azar G., T., P., Fox D., Fedutik Y., Krishnan L., Cobley A., J., *Functionalized copper nanoparticle catalysts for electroless copper plating on textiles*, Surface & Coatings Technology 2020, vol. 396, no 125971. https://doi.org/10.1016/j.surfcoat.2020.125971

[4] Ali A., Baheti V., Javaid M., U., Militky J., *Enhancement in ageing and functional properties of copper-coated fabrics by subsequent electroplating*, Applied Physics A 2018, no 124, p. 651. https://doi.org/10.1007/s00339-018-2071-x

[5] Morshedi S., Montazer M., *Photo bleaching of wool using nano TiO2 under daylight irradiation*, Journal of Industrial and Engineering Chemistry 2014, vol. 20, no 1, pp. 83-90. https://doi.org/10.1016/j.jiec.2013.04.023

[6] Belukhina O., Milasiene D., Ivanauskas R., *Investigation of the Possibilities of Wool Fiber Surface Modification with Copper Selenide*, Materials 2021, vol. 14, no 7, p. 1648. https://doi.org/10.3390/ma14071648