

ISOLATION OF MICROFIBERS IN THE PROCESSING OF POLYAMIDE FABRICS

IZOLACIJA MIKROVLAKEN PRI PROCESIRANJU POLIAMIDNIH TKANIN

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In recent years, the problem of the release of polymer microparticles (the so-called microplastics) from textile products has been extensively investigated. The main reason of the release of a large number of plastic microfibers is considered to be machine washing of synthetic clothing. However, approaches aimed at reducing the amounts of detached microfibers (MFs) differ significantly. This paper reviews the existing approaches, presents a method for processing polyamide fabrics with a chitosan solution, and describes the experiments that confirm the feasibility of the proposed method. The formation of a chitosan film on the surface of polyamide fibers was evidenced with the results of scanning electron microscopy. The authors have shown that fabric surface treatment with a 1 % chitosan solution reduces the amount of microfibers released during washing by ≈ 60 %. A fluorescent analysis demonstrated that mechanical processing of polyamide fabrics with a higher surface density is associated with a release of a smaller number of microfibers (358 ± 24) MF/g as compared to less dense fabrics (533 ± 16) MF/g. The results obtained in the present study can be used in the development of a standard method for quantifying the amount of synthetic microfibers shed from textile materials during their washing.

Keywords: microplastics, fiber, polyamide, chitosan, washing

V zadnjem času se raziskovalci zelo ukvarjajo s problemom sproščanja polimernih mikrodelcev oziroma mikroplastike. Ugotavljajo, da je glavni razlog za sproščanje velike količine mikrovlaknen strojno pranje sintetičnih oblačil oziroma tekstila. Vendar pa se pristopi za reševanje zmanjšanja količine izločenih mikrovlaknen (MF, angl.: microfibers) med seboj zelo razlikujejo. V tem članku avtorji opisujejo pregled obstoječih pristopov in novo metodo procesiranja poliamidnih vlaken v hitozanski raztopini ter opisujejo preizkuse, ki potrjujejo izvedljivost predlagane metode. S presevnim elektronskim mikroskopom so dokazali tvorbo hitozanskega filma na površini poliamidnih vlaken. Avtorji pričujočega članka so dokazali, da obdelava tekstila z 1 %-no hitozansko raztopino zmanjša vsebnost mikrovlaknen med pranjem za približno 60 %. S fluorescentno analizo so dokazali, da je mehansko procesiranje poliamidnih vlaken z večjo površinsko gostoto povezano s sproščanjem manjšega števila mikrovlaknen (358 ± 24) MF/g v primerjavi z manj gostimi tkaninami (533 ± 16) MF/g. Rezultati te študije se lahko uporabijo za razvoj oziroma izdelavo standardizirane metode za ovrednotenje količine izločenih sintetičnih vlaken iz tekstilnih materialov med njihovim pranjem.

Ključne besede: mikroplastika, vlakna, poliamid, hitozan, pranje

1 INTRODUCTION

1.1 General

The presence of microplastics (particles smaller than 5 mm) in almost all ecosystems presents a global problem.¹ Microplastic particles have been found not only in wastewater, but also in coastal areas as well as in the organisms of the species inhabiting rivers and seas.^{2,3} Recent studies have shown that 73 % of fish caught in the northwestern Atlantic Basin have microplastics in their stomachs.¹

As for the impact of microfibers (MFs) on human health, it will depend on the cumulative impact of diffuse terrestrial sources. There is evidence for the presence of microfibers in a wide range of foods and beverages such as seafood, drinking water, beer, salt and sugar.⁴ There is

no doubt that some degree of chronic exposure is now an integral part of human life.⁵

According to the Friends of the Earth International,⁶ about 64 % of all textile products are made of plastic. The polyester fiber (PEF) segment leads the synthetic fiber market, accounting for a share of about 84 %, followed by the nylon, acrylic and polypropylene segments.³ Synthetic fibers are considered to be cheap and versatile. They are comfortable and breathable for sportswear, warm and durable for winter apparel. However, microfibers are not as good in terms of biodegradability. It takes one month for 82 % of cotton to decompose, whereas polyester fibers do not decompose during this period at all.³

Given the growing demand for synthetic textiles in various industries, the growth of the world population and the rise in living standards, the market for synthetic fibers is constantly expanding. As estimated by analysts,

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the global production of synthetic materials increases by more than 9 million tons each year and will reach 80 million tons by 2025.³

As of 2017, the volume of synthetic fibers produced in Russia was 377.5 thousand tons, of which the production of synthetic textile accounted for 53.6 thousand tons. The global trend is also observed in the Russian market, as the production of polyester fibers predominates over the production of other synthetic fibers.⁷

1.2 Literature review

Scientists assume that MF shedding occurs at all stages of the synthetic fabric production, from the raw material processing to MF disposal.^{4,7–11} The International Union for Conservation of Nature reports that about 35 % of all primary microplastics are generated from washing synthetic materials,¹² since washing machine filters and wastewater treatment plants do not remove such fibers.¹³ Thus, freshwater ecosystems are at considerable risk, since they receive not only domestic, but also industrial wastewater, including the wastes dumped by textile factories.¹⁴

Given the growing environmental concern and the continuous growth of the synthetic textile market, active research is being carried out in the area of MF shedding. It is dedicated to the development of measures intended to reduce the amount of MFs generated during machine washing and subsequently released into the environment.^{4,11,14–18} While developing sufficiently effective methods aimed at the prevention of harmful effects associated with this source of pollution, many researchers focus on the optimization of washing parameters, including the temperature and cycle time, the presence of detergents and conditioners, the amount of water used, the number of revolutions during spinning, etc.^{13,19–22}

There are contradictory findings concerning the exposure to detergents. Notably, a group of investigators^{2,18,23} showed that the use of detergents promotes the release of microfibers during washing. Other authors concluded that the use of detergents decreases the number of microfibers released from synthetic clothing during machine washing.¹⁴ A team of researchers confirmed the selective action of biodetergents (detergents containing enzymes): in some cases, their use promotes the fiber loss, while under other circumstances it may decrease the number of fibers released or have no significant effect at all.²⁴

Similar discrepancies have been identified in studies investigating the effects of fabric softeners or, as they are also called, fabric conditioners.^{13,23–25} The authors of one paper note that the use of fabric softeners can enhance the release of microfibers under certain conditions.²³ However, these findings contradict the results obtained by other researchers,²⁴ who concluded that these products can reduce the release of microfibers by more than 35 %.

Some researchers have shown that clothes shed more microfibers in the first wash than in subsequent washing cycles.^{10,13,20} However, there are authors who state that the number of released microfibers does not depend on the number of washing cycles.²³

Summing up the literature review, one promising, relatively inexpensive and simple method of chemical treatment of polyamide fabrics should be mentioned, which is their pre-treatment with an accessible and natural reagent – chitosan.^{26,27} This method enables increasing the strength of synthetic fibers and, consequently, reduces the number of MFs released.

Thus, the purpose of the present paper is to elucidate the effects of chitosan treatment of polyamide fabrics on the release of microfibers during the washing of fabrics of different densities. To achieve this purpose, the researchers should accomplish several objectives. Firstly, they are to develop a procedure for the treatment of fabrics with chitosan. Secondly, the researchers intend to identify the effects of treatment on the number and characteristics of microfibers released before and after washing, and to analyze the results obtained in comparison with the data presented in the literature.

2 EXPERIMENTAL PART

2.1 Materials

Samples of two polyamide fabrics, used for tailoring, where chosen for the experiment. According to the manufacturer's specification, the surface densities of the samples were 92 MF/g and 200 MF/g. Polyamide fabric is a synthetic textile made of petroleum-based plastic polymers.

To prepare the treatment solution, the investigators used chitosan with a molecular weight of 200 kDa and a degree of deacetylation of 82 %, and acetic acid of chemically pure grade. Distilled water was used in all experiments.

Chitosan is a natural cationic polysaccharide composed of randomly distributed β (1–4)-linked N-acetylglucosamine and glucosamine. This polymer has been used as a textile finishing substance, conferring antibacterial properties, as an adsorbent of anionic dyes or a shrink-resist agent in wool, among other applications.²⁸

2.2 Experiment

A sample of chitosan was dissolved in a 2 % acetic acid solution during 24 h at room temperature. The concentration of the resulting chitosan solution was 1 %. Tissue samples, which had been previously cut into squares of (500 × 500) mm, were placed in the prepared solution for one hour and completely dried at a temperature of 125 °C.²⁹

Samsung WF8590NLW9 was chosen for the study. The washing mode was chosen according to the recommendations of the polyamide-fabric manufacturers (deli-

cate wash, 30 °C, spinning with a minimum number of revolutions). The amount of the bleach-free laundry detergent used corresponded to the amount indicated by the manufacturer for washing machines of the given class. No fabric softener was used. Each experiment lasted 30 min and was performed in triplicate. After the end of the washing cycle, the drainage water was filtered through a laboratory sieve with a pore size of 100 µm. The fibers captured by the filter were washed with distilled water into a beaker, dried in a desiccator, and tested.

2.3 Testing methods

To assess the effectiveness of the developed process we used scanning electron and fluorescence microscopy.

In the course of scanning electron microscopy, the fiber surface morphology was analyzed. The tests were performed before and after the fabric treatment with chitosan using a Philips XL30 scanning electron microscope (a 2.0 nm resolution at a voltage of 30 kV and a 1000× magnification).

Fluorescence microscopy was undertaken to evaluate the amount of generated MFs. A 10 mg/L solution of Nile red in n-hexane was used. The test samples were prepared according to the prescribed procedure.³⁰ The samples were analyzed in the green part of the visible spectrum since a wavelength of 510–560 nm is the most illustrative for the visual examination of polyamide fibers stained with Nile red.³⁰

3 RESULTS

The results of the surface morphology analysis are shown in **Figure 1**.

The fibers of the sample that was pretreated with a 1 % chitosan solution (**Figure 1b**) are ‘glued’ together as compared to the control sample (untreated fabric, **Figure 1a**). The chitosan solution is likely to bind individual fibers.

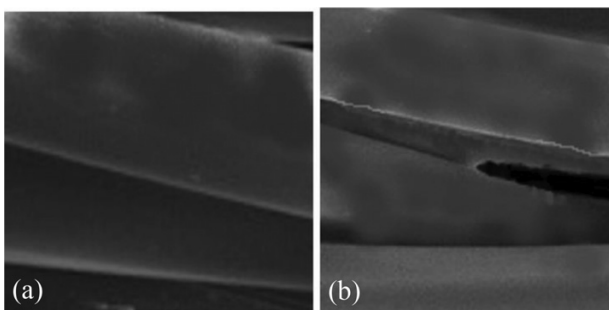


Figure 1: Surfaces of the polyamide fabrics with a surface density of 200 g/m² visualized during a SEM analysis (a magnification of 1000×): a) untreated surface, b) chitosan-treated surface

The results obtained during a fluorescence analysis after washing the untreated control sample (**Figure 2a**) and the sample that had been treated with the 1 % chitosan solution (**Figure 2b**) confirm the above assumption.

The researchers calculated that the amount of microfibers in the control sample was (893 ± 41) MF/g, and the amount of microfibers in the pre-treated sample was (358 ± 24) MF/g. The values are presented as arithmetic means, calculated based on the number of fibers released after each of the three experiments, plus or minus standard deviation.

When examining the samples under a microscope, no agglomeration of fibers was observed. These results are consistent with the assertion that household washing machines cannot trap microfibers as they are not equipped with special devices.¹³

To determine how the surface density of a polyamide fabric affects the process of MF release, two samples were compared. As mentioned in Section 2.1 and provided in the manufacturer’s specification, the surface density of the first sample (O1) was 92 g/m² and the surface density of the second sample (O2) was 200 g/m². Control (untreated) samples were labeled as KO1 and KO2, respectively. The results of the experiments are presented in **Table 1**.

Table 1: Number of MFs shed from the untreated and chitosan-treated polyamide fabric samples

Sample	Fabric surface density (g/m ²)	Number of MFs (MF/g)	Reduction in MF shedding (%)
KO1	92	1 346 ± 34	60.4
O1	92	533 ± 16	
KO2	200	893 ± 41	60.1
O2	200	358 ± 24	

As expected, a smaller amount of MFs was released from denser fabric samples (1 346 ± 34) MF/g for KO1 and (893 ± 41) MF/g for KO2. The tendency did not

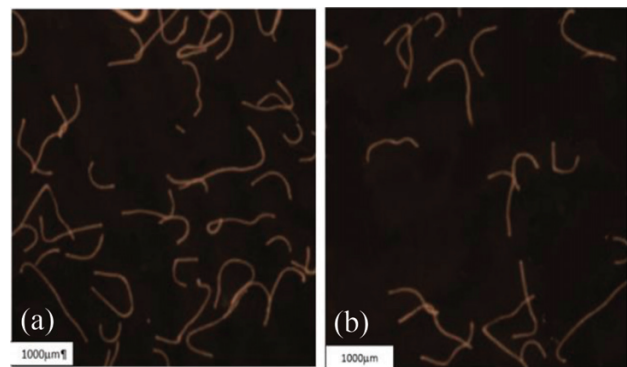


Figure 2: Fibers detached from the polyamide fabrics with a surface density of 200 g/m²: a) untreated, b) chitosan-treated

change after the fabric treatment with chitosan (533 ± 16) MF/g for O1 and (358 ± 24) MF/g for O2.

4 DISCUSSION

4.1 Rationale for the choice of study material

Some authors focused their attention on testing fabric samples and non-woven materials (e.g., microfleece),² new finished products and those that had been artificially aged.²³ The choice of textile for the present study is justified by the fact that polyamide is characterized by high strength, high impact and wear resistance.

The authors of this paper assumed that the same type of fabric with different surface densities releases different amounts of MFs. The experimental data presented in **Table 1** confirm this assumption. The higher the fabric density, the lower is the amount of microfibers released (358 ± 24) MF/g and (533 ± 16) MF/g for O2 and O1, respectively.

4.2 Chemical treatment of polyamide fabrics

The formation of chitosan films is known to be significantly affected by the polymer molecular weight and structure, the origin of chitosan, the degree of chitosan deacetylation, the presence of free amino groups, and the type of solvent.³² In most cases, acetic acid is used as the standard solvent to form chitosan films. The use of other acids (formic, lactic, citric, glycolic ones) results in a deterioration of the mechanical properties of chitosan films as compared to the films obtained with the use of acetate solutions.³³ Therefore, 2 % acetic acid was chosen as the solvent by the researchers.

A 1 % chitosan solution was used in the process of developing the described method. This concentration of chitosan was chosen based on the results published by other investigators.^{34,35}

It is a well-known fact that the rigidity and stability of the highly ordered structure of chitosan are supported by a system of hydrogen bonds. Hydroxyl groups, oxygen atoms of the pyranose ring and glycosidic bond, and amino groups are involved in their formation.³⁴ If the degree of protonation is low, the unprotonated amino groups remain hydrogen bonded, maintaining the rigidity of the chitosan molecule. In case of a critical increase in protonation, a change in the macromolecule configuration occurs caused by the destruction of hydrogen bonds. Such a change leads to an increase in the macromolecule flexibility.³⁵

5 CONCLUSIONS

In order to reduce the formation of plastic microfibers (the so-called microplastics), a method for treating polyamide fabrics with a 1 % chitosan solution was developed. The formation of a chitosan film on the fabric surface was confirmed with spectral scanning micros-

copy. The researchers have suggested that the film forms due to hydrogen bonds, which form spatial structures. The amount of microfibers shed from textiles during washing can be reduced by more than two times in case of a textile pretreatment with chitosan. To illustrate, the amount of microfibers in the untreated sample was (893 ± 41) MF/g, and the amount of microfibers in the sample pre-treated with chitosan was (358 ± 24) MF/g. The results of the experiment confirm that the surface density of a fabric has a significant impact on the amount of MFs generated. Fabrics with a higher surface density (200 g/m^2) released (358 ± 24) MF/g, while fabrics with a two-times lower surface density (92 g/m^2) released (533 ± 16) MF/g. The findings described in this paper were compared with the data provided by other authors. Based on the results of this comparison, the developed method is considered to be promising. Further detailed studies are required in this area. However, the data available today may already be used for the development of a standard method for quantifying the microfibers shed from synthetic fibers during washing.

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