The assessment of the release of microplastics from textile sources in the washing process of polyester fabric

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Original scientific paper**

Environmental issues, considered through the impact of textiles on the quality and health of people, have become increasingly interesting for researchers, professionals and regulators in recent decades. One of the topics that is attracting increasing interest from researchers and the public is microplastic pollution. The properties of the textile material in combination with the variables of the washing process have an influence on the release of fragments in the washing process. This study focuses on the analysis of a polyester fabric with a complex structure in a washing process with the reference detergent ECE-A and water according to procedure 2A of the EN ISO 6330 standard for the evaluation of the release of microplastics of textile origin. The fabric was analysed using FTIR and microscopy to gain insight into the structural properties and composition of the individual units. The effects of washing were assessed by analysing the wastewater and the filter cake. The drying effects were analysed by weighing the washed and dried fabric in relation to the unwashed. The physico-chemical parameters of the wastewaters were characterised by pH, conductivity, turbidity and chemical oxygen demand (COD). The filter cake was analysed by microscopy. The results show the importance of analysis three observed levels: fabric, wastewater and filter cake, in order to take into account the complexity of this system and assess its impact on the environment.*

Keywords: *polyester fabric; washing; drying; fragments; microplastics; wastewater*

Izvorni znanstveni rad**

Procjena otpuštene mikroplastike tekstilnog podrijetla u procesu pranja tkanine od poliestera

Problemi zaštite okoliša promatrani kroz utjecaj tekstila na kvalitetu i zdravlje ljudi u posljednjim desetljećima pobuđuju interes istraživača, stručnjaka i regulatornih tijela, a jedna od istaknutijih tema zagađenje je mikroplastikom iz različitih izvora. Svojstva sintetičkih tekstilija u kombinaciji s varijablama procesa pranja utječu na otpuštanje mikroplastike u procesima njege. Ovo istraživanje usmjereno je na analizu tkanine složene strukture u procesu pranja sa standardnim ECE-A deterdžentom i vodom prema postupku 2A norme HRN ISO EN 6330 s ciljem procjene otpuštene mikroplastike tekstilnog podrijetla. Tkanina je analizirana infracrvenom spektroskopijom s Fourierovom transformacijom u tehnici prigušene potpune refleksije (FTIR-ATR) i mikroskopijom radi uvida u strukturalna svojstva i sirovinski sastav pojedinih dijelova složene strukture. Učinci pranja procijenjeni su analizom otpadne vode i filtarskih kolača, a učinak sušenja gravimetrijskom analizom oprane i osušene tkanine u odnosu na neopranu. Fizikalno-kemijski parametri otpadnih voda procesa pranja karakterizirani su vrijednošću pH, vodljivošću, mutnoćom i kemijskom potrošnjom kisika (KPK), a filtarski kolač pregledan stereo mikroskopom. Rezultati su pokazali važnost analize tri promatrane razine: tkanine, otpadne vode i filtarskog kolača kako bi se sagledala složenost promatranog sustava s ciljem procjene utjecaja na okoliš*

Ključne riječi: *poliesterska tkanina; pranje;sušenje; fragmenti; mikroplastika; otpadna voda*

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1. Introduction

The properties of textiles change after several washing cycles depending upon numerous factors. The intensity of the changes depends on the properties of the textiles (composition, construction parameters, degree of polymerisation, swelling capacity) [1] and the design of the Sinner cycle the share of chemicals, temperature, time, mechanical agitation [2].

Fibrillation of cotton fibres can be a consequence of abrasion in the wet (washing bath), while cracks are generated by mechanical agitation and abrasion during the tumble-drying process [3]. Cracks that appear on cotton fibres after high temperature drying can reduce fabric strength and increase generation of fibrils/lint.

Polyester fibres are crystalline, hydrophobic and do not swell in water, and are not exposed to degradation and fibrillation in normal washing conditions [4]. However, excessive heat can cause shrinkage or damage to polyester materials if exposed to particular temperatures passing through distinct phase transition points (melting and glass transition temperature).

Different blends can meet the specific requirements on functional properties of textiles, e.g. feel, appearance, dimensional stability, easy maintenance and adequate comfort [4]. The advantages of cotton-polyester blends over pure cotton textiles, in addition to durability, include the ability to wash at lower temperatures, lower deposit content and less damage after multiple wash cycles.

However, the presence of microplastic (MP) in natural and wastewater, sediment, soil, aquatic organisms, and the air is partly linked to textile sources. It is considered that approximately 35% of synthetic fibres in the environment originate from washing [5]. Microplastics are a generic term for very small fragments of plastic with an average length of 1 nm to 5 mm or for fibres 3 mm to 15 mm in length, where the ratio of length to diameter is > 3 [6].

At the fibre level, the formation of microplastics is associated with surface abrasion, damage due to fatigue, the development of the fibrillar structure and its degradation. At the fibre structure level, microplastics can be associated with the extraction of protruding (broken) fibres (associated with hairiness) [7].

The release of fibrous microplastics (FMPs) can be controlled by the choice of composition, structure and technology of preparation of the fibre structure [8]. The qualification and quantification of particulate matter emissions and other particles released during washing and drying is difficult due to the variability and complexity of secondary sources and the fact that particulate matter in a real system is a vector for the spread of other emissions with varying degrees of risk. Knowledge of the system in which microplastics from textile sources are generated determines the choice of one or more methods for their determination and/or separation in wet and dry environments.

In this paper, the influence of the composition of the washing bath and the drying procedure on microplastics of textile origin from the polyester fabric of a complex structure characterised by a three-colour pile on the front and back was investigated. Different methods were employed for analysis of fabric, wastewaters and filter cake. Complex polyester fabric was analysed by FTIR and microscopy; gravimetric analysis for the change in mass of polyester fabrics in washing and drying processes; laundry wastewaters by physico-chemical parameters and the filter cake by microscopic analysis.

2. Experimental

2.1. Material

The double-sided weft woven plush fabric with a unit area of 279 g/m^2 colour patterned in grey, red and yellow was purchased in the shop. The basic is a high-density structure (plain, twill), while the low density fleur floats on the face and back side surface [9].

The individual structural elements of selected fabric were analysed by FTIR-ATR. To better visualise structural units, the surface of the fabric was observed using a digital microscope, Dino-Lite AM7013MZT, from AnMo Electronics Corporation, Taiwan.

2.2. Washing and drying process

Samples of fabric were prepared using an ultrasonic cutter, model TTS 400, Sonowave S.r.l., Italy, to prevent defragmentation of the edges of these fabric samples during the washing and drying process. The prepared fabric samples were weighed and subjected to a washing process by two protocols: in a detergent solution (1) and in tap water (2).

The samples were subjected to a regular washing process with ECE-A+ detergent at a concentration of 1.25 g/L according to protocol 2A of the EN ISO 6330 standard [10] and according to modified protocol with tap water. Both washing processes were carried out in a bath ratio of 1:7 under 5 cycles at 60 °C in a Rotawash laboratory device, SDL Atlas, Rock Hill, USA, for 40 min.

The four rinsing cycles were carried out with tap water (hardness 21.0 °dH) at a bath ratio of 1:8.

After washing with detergent and water, the samples were dried at ambient conditions and in a household tumble dryer T5130-LAB, Electrolux, Sweden (drying programme Low Lab, drying rate 20 mL/min).

The drying of polyester samples at room conditions through 24 hours was done on a flat surface while tumble drying was performed in two variants. One part of the samples before drying was placed in a polyester net, size 25 x 40 cm (dm-drogerie markt GmbH + Co, KG, Austria), and the rest was tumble dried in a free state.

The procedures for washing and drying, the labels and the description of the procedures are specified in Table 1.

Tab.1 Designation of the fabric samples

Sample	Composition of washing bath	Drying	Drying net
1	ECE-A* detergent	tumble	-
2	water		-
3	ECE-A* detergent		+
4	water		+
5	ECE-A* detergent	air	-
6	water		-

The single-stage vacuum filtration of the wastewater was carried out using FBM047PES022 PES membrane filter, diameter 47 mm, pore size 0.22 μm, FilterBio, China. The result of a filtration procedure was a filter cake containing suspended particles and microplastics of textile origin from wastewater.

Based on the designations in Table 1, the wastewater from the washing process with ECE-A* detergent is labelled WW-1 and the wastewater from the washing process in water is labelled WW-2. The corresponding filter cakes are labelled FC-1 and FC-2 in the same way.

2.3. Methods

All parts of the tested sample were analyzed with an FTIR spectroscope using the ATR technique (Perkin Elmer, Spectrum 100, USA). For each sample, 4 scans were recorded at a resolution of 4 cm^{-1} between 4000 cm^{-1} and 380 cm^{-1}.

The samples were weighed before and after 5 drying cycles so that the gravimetric method [11] was used to determine the change in weight. A Kern balance, model ALJ 220-5DNM, Germany, was used to determine the weight of the polyester fabric before (m_0) and after 5 wash cycles (m_1) to determine the change in mass (Δm) of the fabric and the degree of defragmentation.

The wastewater (WW) collected after 5 washing and 20 rinsing cycles was characterised using physico-chemical methods: pH value, conductivity (κ), turbidity (NTU) and chemical oxygen demand (COD). A detailed description of the methods and conditions is available in the previous publication [12]. The SevenCompact™ Duo S213 multimeter from Mettler Toledo was used for determining the pH [13] and conductivity [14]. The turbidity was measured nephelometrically [15] using the TU5200 turbidimeter from Hach and the COD [16] using the WTW Photolab S6 spectrophotometer, Xylem Analytics, Germany.

Filter cakes (FC-1 and FC-2) obtained after filtration of WW-1 and WW-2 were observed with Meiji Techno EMZ series stereomicroscope, Japan under 45x magnification.

3. Results

The results were presented into four sections: (i) the identification of structural elements of complex fabric by microscopic and spectroscopic evaluation; (ii) gravimetric parameters of fabric before and after five cycles washing/drying process; (iii) the composition of wash wastewaters and (iv) appearance of the filter cakes.

3.1. Surface observation of the fabric

The double-sided weft woven plush fabric consists different structural elements, patterned in grey, red and yellow, so microscopic examination of particular elements (Fig.1) as well as FTIR analysis (Fig.2) were employed for identification of surface and chemical composition.

Fig.1 visualises a structural units: grey, red and yellowed patterned fleur on the face side of the fabric, observed using a digital microscope.

The spectral curves of all parts of the examined sample have peaks that match the peaks of poly(ethylene-terephthalate) fabric [17]. In addition, the characteristic peaks for all recorded parts of the examined samples are listed and shown in Fig.2. Peaks at wavenumbers 3050 cm^{-1}, 2923 cm^{-1}, and 2901 cm^{-1} occur due to symmetric stretching in the CH group. The sharpest peaks are observed at a wavenumber of 1710 cm^{-1}, which arise from the stretching of the carboxyl group C=O. Peaks at wavenumbers 1577 cm^{-1} and 1505 cm^{-1} indicate stretching vibrations within the C=C bond of the aromatic ring. Peaks at wavenumbers 1471 cm^{-1},

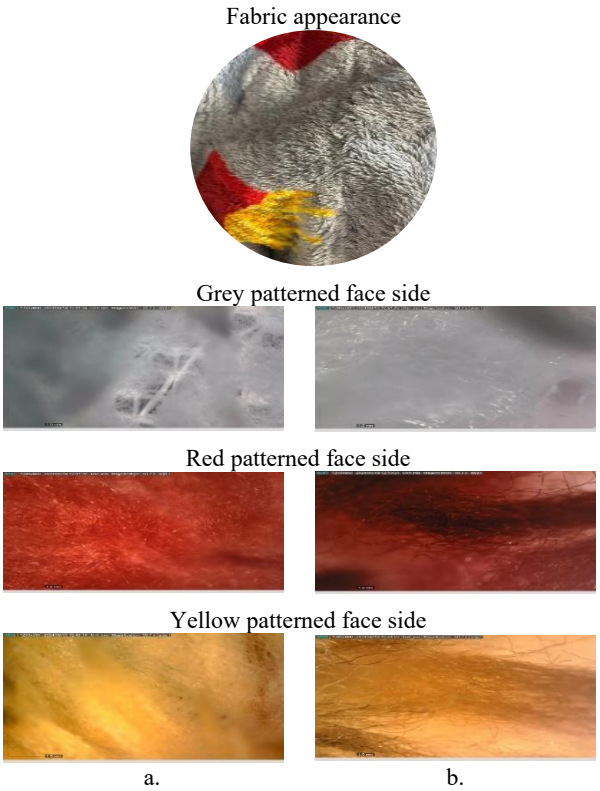


Fig.1 Digital micrographs of fabric structural units: a. magnification 50x, b. magnification 200x

1408 cm^{-1}, and 1340 cm^{-1} indicate stretching of the C-O group, as well as deformation within the O-H group, and bending and vibrating within ethylene glycol groups. Vibration within the terephthalate group are recorded at wavenumbers 1243 cm^{-1} and 1124 cm^{-1} ($OOCC_6H_4$-COO).

A sharp peak at 1094 cm^{-1} occurs due to vibrations within methylene groups. Vibration within aromatic rings are observed through the appearance of sharp peaks at wavenumbers 970 cm^{-1}, 871 cm^{-1}, and 847 cm^{-1}. The peak at 795 cm^{-1} arises from vibrations in aromatic bonds. A sharp peak is recorded at a wavenumber of 712 cm^{-1}, resulting from interactions between polar ester groups and benzene rings [18]. From all the above, it can be concluded that all components of the examined samples have the same raw material composition, i.e., the sample is made of poly(ethylene-terephthalate) material.

3.2. Gravimetric analysis

In view of the fact that the standards for monitoring the release and analysis of microplastics from textile sources were published in 2023 [11,19,20], the gravimetric method was used in the investigation. Table 2 shows the changes in the mass of washed and dried polyester fabric samples according to the labels in Table 1.

Table 2 shows the results of the gravimetric analysis of the polyester fabric samples before and after 5 washing and drying cycles. Samples 1, 2, 5 and 6 showed a loss in mass during the washing and drying process, while samples 3 and 4 showed an increase in mass. The increase in mass can be attributed to deposits of calcite from hard water (hardness 21.0 °dH) at 60 °C, which was used for the washing process.

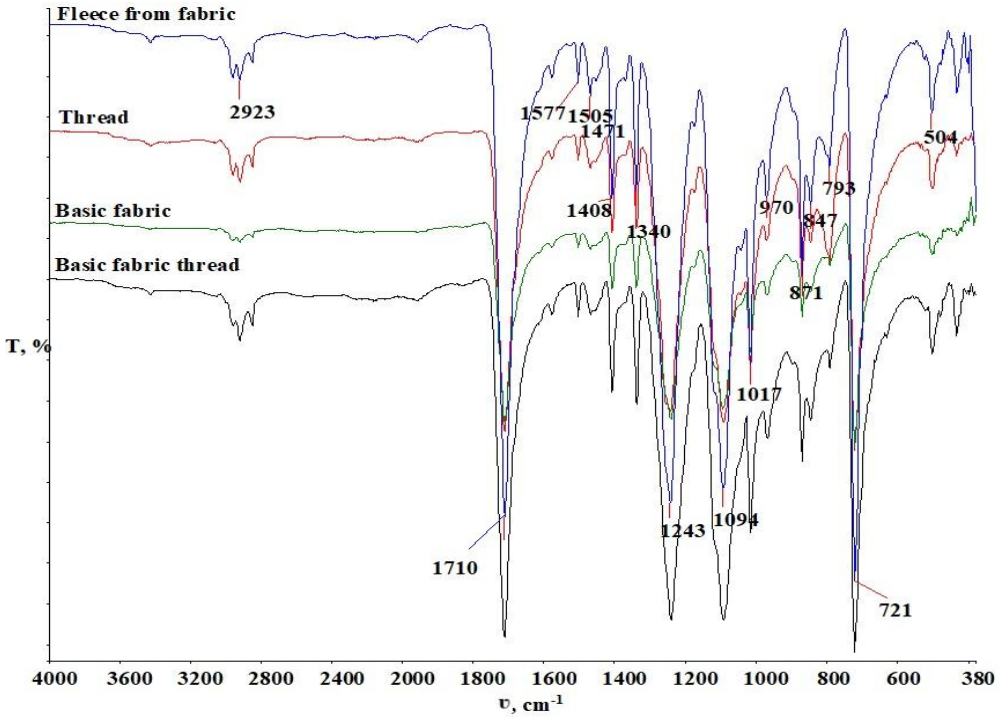


Fig.2 FTIR-ATR of the structural units of double-sided weft plush fabric

Tab.2 Weight of polyester fabric samples before and after drying process

Sample	m_0 (g)	m_1 (g)	Δm (g)	Δm (%)
1	4.58222	4.53254	0.049680	1.08
2	4.13405	4.12524	0.008810	0.21
3	4.54308	4.57191	-0.028830	-0.63
4	4.08123	4.15481	-0.073580	-1.80
5	4.70023	4.67012	0.030110	0.65
6	4.31043	4.27013	0.040300	0.93

The mass loss of samples 1, 2, 5 and 6 indicates that the release of microplastics from the textile source is due to the processes of washing and drying, which are more dominant in terms of deposition of residues. Based on the value of the differences in the mass of the samples washed and stored under different conditions (drying net) and dried under the same conditions (tumble dryer), it is clear that the use of the drying net protects the surface of the polyester fabric samples (samples 3 and 4) and reduces the release of FMP during these processes.

After drying samples 1, 2, 3 and 4 in the tumble dryer, the filter of the dryer was observed, Fig.3. The collected mass of microplastic released from polyester samples was 0.023 g.

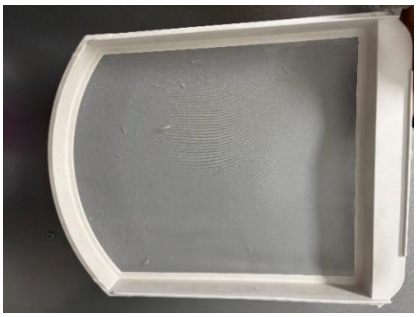


Fig.3 Appearance of the filter after 5 drying cycles

3.3. Laundry wastewaters analysis

The results of analysing the composition of the wastewater from 5 wash cycles of the polyester fabric with ECE-A* detergent and water and the subsequent 20 rinsing cycles in hard water based on the values of pH, conductivity, turbidity and COD are shown in Table 3. The analysis of the individual pH and conductivity parameters confirms the significant influence of the multiple rinsing cycles (20 in total), which dilute the detergent bath suspension collected from the washing process.

Tab.3 Physico-chemical parameters of wastewaters (WW)

WW	pH	κ (μS/cm)	Turbidity (NTU)	COD (mg O_2/L)
-1	8.04	730.3	37.6	93
-2	7.93	741.5	4.6	144

The turbidity of the wastewater from the washing process with detergent is eight times higher compared to water, which is to be expected given the composition of the ECE-A* detergent. This detergent contains surfactants and an aluminosilicate builder, zeolite A, which is not soluble in water, and this conductivity is to be expected.

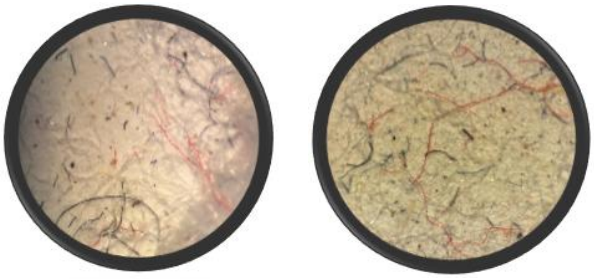
The turbidity of the wastewater from the washing process with water is 4.6 NTU, which may be the effect of residuals and suspended particles of calcite.

The COD values confirm the presence of some chemical loading, with WW-1 having a lower COD value compared to WW-2. The wastewater, WW-1 contains suspended ECE-A* detergent, so the contamination was expected. The level of chemical contamination in sample WW-2 may be superimposed by microplastics released from the polyester fabric during the washing process.

3.4. Filter cakes

The microscopic images of the filter cakes shown in Figure 4 are the result of a single-stage filtration of wastewaters collected from washing process with ECE-A* detergent (FC-1) and with water (FC-2) using a polyethersulfone filter.

Both filter cakes are covered by microplastic released from polyester fabric as a textile source. Fibrous fragments of all colours of fleur are visible on both filter cakes. However, the degree of coverage of the FC-1 filter with suspended matter is higher compared to FC-2.



a. b.

Fig.4 Microscopic images of filter cakes, magnification 45x: a. FC-1, b. FC-2

4. Conclusions

The results obtained show that the composition of the washing bath and the drying method influence the release of microplastics of textile origin from the polyester fabric with a complex structure characterised by a tricolour pile on the front and back. The gravimetric analysis showed that washing and drying

in the free state lead to weight loss and defragmentation. Drying polyester fabric in a net prevented defragmentation of the polyester fabric.

The composition of the laundry wastewaters collected and analysed using physico-chemical parameters showed a similar pH and conductivity, while the turbidity and chemical oxygen demand were different.

Both filter cakes are covered with microplastics, which are released from the polyester fabric as a textile source. Fibrous fragments in all colours of the fleur can be seen on both filter cakes. However, the degree of coverage of the FC-1 filter with suspended particles is higher than that of the FC-2.

All methods used are indicative for the evaluation of the release of microplastics from polyester fabric as a textile source.

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