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Separation and identification of microfibers in the wastewater of textile finishing process

Tekstil terbiye işlemi atıksularından mikroliflerin ayrıştırılması ve tanımlanması

***Yazar(lar) (Author(s))**: Sinem Hazal AKYILDIZ[1], Rossana BELLOPEDE[2], Silvia FIORE[3], Hande SEZGİN[4], Bahattin YALCİN[5], İpek YALCİN ENİS[6]*

ORCID[1]: 0000-0002-8346-5895

ORCID[2]: 0000-0002-9569-0678

ORCID[3]: 0000-0001-5949-0559

ORCID[4]: 0000-0002-2671-2175

ORCID[5]: 0000-0003-4448-1101

ORCID[6]: 0000-0002-7215-3546

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Separation and Identification of Microfibers in the Wastewater of Textile Finishing Process

Highlights

- *Significance of how much microfibers get into the wastewater from just one textile finishing machine.*
- *Characterization of microfibers with micro-FTIR and EDX/SEM analysis of released microfibers.*
- *Macro- and micro- images of microfibers and their length range.*

Graphical Abstract

The focus of this study is the determination and separation of microfibers released from a textile finishing machine used in a textile factory to give the fabric a soft touch. In this context, textile wastewater was pre-treated, then filtered, and MFs were analyzed.

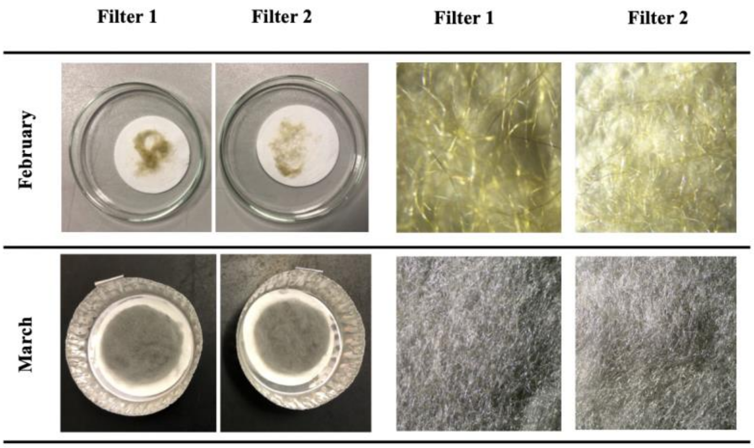

Figure. Macro and micro images of filters composed of microfibers.

Aim

The aim of this study is to measure the quantity of microfibers released from the textile finishing machine used to give fabric a velvety feel and to separate these microfibers from wastewater.

Design & Methodology

Wastewater samples were pretreated and then filtered. Microscopic, physical, and chemical analyses of the microfibers were made.

Originality

This study contains novel findings to identify textile finishing processes, specifically the Biancalani machine, as a significant source of microfiber pollution in industrial wastewater.

Findings

A total of 0.058 grams per liter and 0.251 grams per liter of microfibers, acrylic and cotton, were detected in the wastewater obtained on different dates.

Conclusion

Microfibers released from such finishing processes should be considered and prevented in the production process.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Separation and Identification of Microfibers in the Wastewater of Textile Finishing Process

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Araştırma Makalesi / Research Article

Sinem Hazal AKYILDIZ[123*], Rossana BELLOPEDE[2], Silvia FIORE[2], Hande SEZGİN[3], Bahattin YALCİN[4], İpek YALCİN ENİS[3]

[1]Department of Textile Engineering, Marmara University, Istanbul, Turkey
[2] Department of Engineering for Environment, Land and Infrastructures, Politecnico di Torino, Torino, Italy
[3] Department of Textile Engineering, Istanbul Technical University, Istanbul, Turkey
[4] Department of Inorganic Chemistry, Marmara University, Istanbul, Turkey

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ABSTRACT

Microplastic pollution is an important global problem caused by the textile industry, which accounts for 35% of microplastics emitted as microfibers (MFs). Microplastics interact with a variety of organisms due to their small dimensions, leading to chromosomal mutations. The goal of this research is to identify and separate microfibers discharged by textile finishing machinery, which is used to give textiles a soft touch. Within the scope of the study, wastewater samples were taken separately in February and March 2022, right after the device exit, before being discharged to the factory effluent and pre-treated for 5 days at 25 °C with 15% H_2O_2. Then, the microfibers were separated from the wastewater with the help of a filter. The accumulated microfibers on the filters were examined using a light microscope, and their chemical composition was determined using micro-FTIR analysis. Furthermore, energy dispersive X-ray scanning electron microscopy (EDX/SEM) was used for analyzing the structure of microfibers. The findings revealed the presence of acrylic and cotton microfibers in the wastewater samples, with varying concentrations observed on different dates (0.058 g/L and 0.251 g/L), emphasizing the severity of the microplastic issue we currently face.

Keywords: Microplastic pollution, microfibers, textile wastewater, filtration, micro-FTIR

Tekstil Terbiye İşlemi Atıksularından Mikroliflerin Ayrıştırılması ve Tanımlanması

ÖZ

Mikroplastik kirliliği, mikrolif olarak salınan mikroplastiklerin %35'inden sorumlu olan tekstil endüstrisinin neden olduğu önemli bir küresel sorundur. Mikroplastikler, küçük boyutları nedeniyle çeşitli organizmalarla etkileşime girerek kromozomal mutasyonlara yol açar. Bu araştırmanın amacı, tekstil ürünlerine yumuşak bir tuşe vermek için kullanılan tekstil terbiye makinelerinden çıkan mikrolifleri belirlemek ve ayırmaktır. Çalışma kapsamında, atıksu numuneleri fabrika çıkış suyuna verilmeden önce cihaz çıkışından hemen sonra Şubat ve Mart 2022 tarihlerinde ayrı ayrı alınmış olup, %15 H_2O_2 ile 25 °C'de 5 gün ön işleme tabi tutulmuştur. Ardından mikrolifler filtre yardımı ile atıksudan ayrıştırılmıştır. Filtreler üzerinde biriken mikrolifler ışık mikroskobu ile incelenmiş ve mikro-FTIR analizi ile kimyasal kompozisyonları belirlenmiştir. Ayrıca, mikroliflerin morfolojisi enerji dağılımlı X-ışını taramalı elektron mikroskobu ile incelenmiştir. Bulgular, farklı tarihlerde gözlemlenen farklı konsantrasyonlarda mikrolif içeren (0,058 g/L ve 0,251 g/L) atık su numunelerinde akrilik ve pamuk mikroliflerinin varlığını ortaya çıkarmış ve şu anda karşı karşıya olduğumuz mikroplastik sorunun ciddiyetini vurgulamıştır.

Anahtar Kelimeler: Mikroplastik kirliliği, mikrolifler, tekstil atıksuları, filtrasyon, mikro-FTIR.

1. INTRODUCTION

The most common type of marine litter, comprising 60% to 80% of all, is plastic particles [1]. The use of plastic for diverse purposes began in the 1930s, and when industrial plastic manufacturing rose in the 1940s, a large amount of plastic waste entered marine and freshwater habitats [1, 2]. Global production of plastics derived from petrochemicals with high molecular masses and plasticity reached 368 million metric tons in 2019 [3,4]. The worldwide output of thermoplastics is predicted to reach 445,250 million metric tons in 2025. Over the next few decades, annual production is expected to keep going up, reaching about 590 million metric tons by 2050 [5]. There are numerous industries that use plastic, with the textile industry being among the most prevalent. Worldwide textile fiber production reached 108 million metric tons in 2020, with synthetic fibers accounting for about 62% of the total [6]. While 60% of the synthetic fibers produced are buried or disposed of as waste after use, it can take up to 100 years for these fibers to decompose and disappear in nature [7, 8].

**Sorumlu Yazar (Corresponding Author)*
e-posta : sinem.akyildiz@polito.it

Plastics pose a risk to human health at every stage of their life cycle, from the processing of petroleum and petroleum derivatives to consumer use and disposal [9]. Microplastics (MPs) are among these dangers that are most crucial. In accordance with ISO/TR 21960, they are particles smaller than 1 mm in size and up to 5 mm in size according to scientific literature [10-12]. MPs sources can be categorized as primary and secondary. The distinction is based on the fact that the particles are purposefully small (primary) or fragments of larger ones (secondary) [13]. The environment may get contaminated with microplastics from textile products as a result of both primary (fibers generated during manufacturing and consumption) and secondary sources (fragmentation of bigger objects for example textile wastes) [14]. According to Gies et al., in Vancouver, Canada, 1.76 ± 0.31 trillion MPs enter the wastewater treatment plant (WWTP) annually, whereas 0.03 ± 0.01 trillion MPs are discharged into the environment [15]. The majority of MPs end up in nature due to human error, wastewater treatment plants, or the textile industry [9]. Annually, between 200,000 and 500,000 tons of MPs are released from synthetic fibers, the vast majority of which end up in the ocean. Approximately 8% of microplastics originating from synthetic textiles are released into European oceans, whereas on a global scale, this proportion is predicted to range from 16% to 35%. [7, 16]. According to studies, up to 1 million microplastic fibers can be released from textiles after a single wash of cloths [17]. Regarding the Ellen MacArthur Foundation, more than 160 million tons of textiles will be sold by 2050, and 22 million tons of microfibers will be released into the oceans between 2015 and 2050 [18]. In a study conducted by Özkan et al. (2020), MPs levels were detected in the inflow and outflow waters of Seyhan and Yüreğir WWTPs in Turkey for six days in August 2017. Besides visual inspection, Raman spectroscopy was used for the detection of microplastics. The results indicate that 1–6.5 million MPs in the influent and 220,000–1.5 million MPs in the effluent per day were observed. The most common kind of polymer detected during the examination was polyester [19].

MPs released into the environment may collect in aquatic creatures and be transported up the food chain to higher trophic levels, such as humans. Due to the prevalent presence of microplastics in human foods and places such as milk, honey, seafood, beer, table salt, drinking water, and the air, the potential health risks posed by microplastics have gotten a lot of attention nowadays [20].

Although 85-99% of MFs can be got rid of during the wastewater treatment plants, the amount of MFs released into the environment by textile wastewater is significantly greater than that of municipal wastewater treatment facilities [21]. Although it is known that almost all machinery in the textile production process cause MF release, some of them result in more MFs in line with their working principles and usage purposes. To enhance the ability to handle and bulkiness of textiles, mechanical finishes such as singeing, calendaring, raising (napping), and brushing are commonly used. With different finishes, the shedding behavior may be altered. The surface of the cloth is brushed throughout the raising process to promote bulkiness and smoothness. Consequently, fibers will protrude from the surface, allowing them to readily glide away, perhaps increasing shedding. However, projecting fibers are removed during singeing and calendaring, which might result in less shedding [22]. Biancalani is a textile finishing machine which contains a raising procedure that involves removing a fiber layer from the fabric's surface to give it a hairy surface or generate a pile. This finishing procedure gives the fabrics a warm, velvety texture [23]. The fibers that come out of this finishing machine, however, mix with the wastewater and cause a high concentration of MFs to form in the effluent. Comprising an in-line vacuum system that can be used to remove loose fibers via air filtration and exhaustion after any brushing, sanding, or raising processes frequently used in the textile industry to improve comfort [24].

The following techniques are used to detect microplastics in WWTPs: sample collection, sample pretreatment, microplastic separation, and characterization/quantification. However, there has been no standardization of techniques [25]. The extraction of the microplastics from the matrix in which they were located involves a multitude of techniques since WWTP samples may contain significant amounts of organic matter or inorganic solids. These approaches should make it easier to quantify and identify microplastics. For the chemical identification of microplastics, the separation of organic components is essential [26]. The two fundamental divisions of the purifying process are chemical and enzymatic digestion [27]. Acidic, alkaline, or oxidative methods can be used for chemical digestion [28-32]. In our previous study, H_2O_2, Fenton's Reagent, HCl, KOH, and NaOH were employed to eliminate organic compounds from wastewater and 15% H_2O_2 at 25 °C for 5 days was found to be the best pretreatment for removing organics from wastewater to correctly analyze it [33]. Moreover, the second step is the capture of microfiber from wastewater, and different separation techniques such as density separation, centrifugation and filtration were tried, and filtration gave the best result [34].

Due to the significant quantity of MFs it contains, wastewater, especially from the textile sector, has drawn the attention of researchers in the literature. This study specifically calls attention to the quantity of MF discharged from the Biancalani machine, which is employed in textile factories to give fabric a pleasant touch. To identify and categorize MFs in wastewater, this research entails undertaking physical, chemical, and microscopic investigations. In this situation, samples of industrial effluent were pre-treated with 15% H_2O_2 at 25 °C for five days prior to filtering. The microfiber length ranges gathered on the filter were identified, and EDX/SEM and micro-FTIR were used to examine their morphologies and chemical compositions. The total release of microfiber was calculated by weight. The findings of this research offer a particular viewpoint on the microfiber contamination that the textile industry is responsible for and emphasize the significance of inspecting each textile process individually.

2. MATERIAL and METHOD

2.1. Materials

The wastewater examined in the study was obtained from the exit of a finishing machine (Biancalani) belonging to a Turkish textile company. In this factory, the fibers emerging from the surface of the fabric in the Biancalani process are conveyed to the wastewater with the help of water. Wastewater from all wet processes in the factory passes through separate tanks, first reaches the inflow wastewater for pre-treatment, and then is discharged as effluent wastewater. The wastewater to be analyzed within the scope of the study was taken from the tanks located at the exit of the raising process before they reach the inflow wastewater. In the factory, one kind of fabric is processed every day in the raising process. Therefore, the microfiber coming out of the wastewater must belong to the fabric processed that day. However, there is also the possibility that the tank where the wastewater is collected may contain microfibers released from the previously processed fabric. The wastewater sample was taken over two different time intervals (February and March in 2022) at the exit of the finishing process for cotton and acrylic fabric treatments before mixing with the effluent. Totally, 1 L of wastewater was collected in both months. The equipment, including the bottles, was sanitized with ethanol and distilled water before being stored in a fridge to prevent contamination.

2.2. Methods

2.2.1. Pretreatment and filtration of wastewater

The 15% H_2O_2 (Sigma-Aldrich) was used to pretreat the 1 L samples for 5 days at 25 °C. Following pretreatment, filtration was carried out using a 47 mm diameter filter (Whatman) that contained glass fiber with a pore size of 0.7 µm and a weight of 0.092 g, followed by an overnight drying at 40 °C (Figure 1).

Figure 1. Filtration process for separation of MFs.

2.2.2. Analysis of microfibers

2.2.2.1. Physical analysis

After filtration and drying, each filter was weighed with a precision balance (± 0.001 g).

2.2.2.2. Microscopic analysis

An ORTHOLUX II POL-MK optical microscope was used to examine the GF filters and the length of microfibers were measured from at least 50 MFs under UV light for fluorescent fibers. The morphology of the microfibers was observed by energy dispersive X-ray scanning electron microscopy (EDX/SEM) (FEI Inspect S).

2.2.2.3. Chemical analysis

The microfibers were chemically characterized using a Shimadzu AIM-9000 Micro-FTIR at 700–4000 cm^{-1} and the library of spectra used was Shimadzu-T-Polymer2. Micro-Fourier Transform Infrared (micro-FTIR) spectroscopy is a sophisticated analytical method that allows for microscopic sample examination. It employs Fourier Transform Infrared (FTIR) spectroscopic techniques to gather precise information on a sample's chemical composition and molecular structure [35]. Both a microscope and an FTIR lens are used in micro-FTIR. Micro-sized particles on the filter are first identified during the measurement with the microscope lens, and their image is captured on the computer screen. Then, the FTIR lens is attached to the apparatus and with the aid of the computer image, sample is subjected to FTIR analysis.

3. RESULTS

3.1. Physical analysis

When the MFs filtered from the February and March wastewater samples were weighed, it was found that the samples contained 0.058 g/L and 0.251 g/L microfiber, respectively. The main reason for the different amounts of microfibers obtained from wastewater samples can be explained by the amount of fabric processed that day. On the other hand, it is thought that the fiber density can also affect the microfiber weight obtained regardless of the fiber amount. The results of the Micro-FTIR characterization in this context confirmed the findings; the cotton fiber with a higher density compared to acrylic fibers had been detected in greater proportions in the March sample.

3.2. Microscopic analysis

Macro- and micro-images of the MFs can be seen in Figure 2. The resulting images prove that the Biancalani process releases a high amount of MFs. This can be explained by the fact that, as was previously noted, a fiber layer is removed from the fabric's surface during the finishing procedure used by the Biancalani textile finishing machine [23].

Due to a large number of fibers, MFs' analysis could not be performed by counting but rather done by weight measurement.

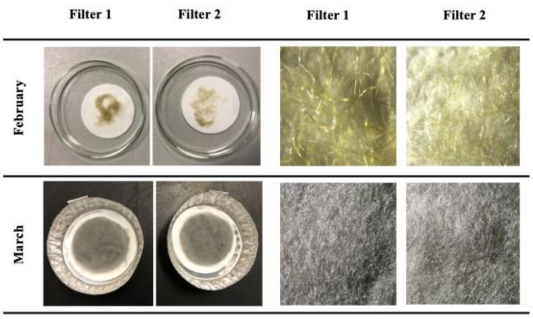


Figure 2. Microfibers captured in filters

EDX/SEM images of microfibers are given in Figure 3. Due to the characteristic cross-sections of fibers, both acrylic and cotton microfibers can be noticed on EDX/SEM images. A cotton fiber appears under a microscope to be a twisted ribbon or a collapsed and twisted tube [36]. On the other hand, it is possible to obtain synthetic fiber with a rounded cross-section and smoother surface with the spinneret geometry used [37]. These EDX/SEM images also support the Micro-FTIR results in which the raw material of the processed fabric is predominantly in the wastewater as of the date of sample collection.

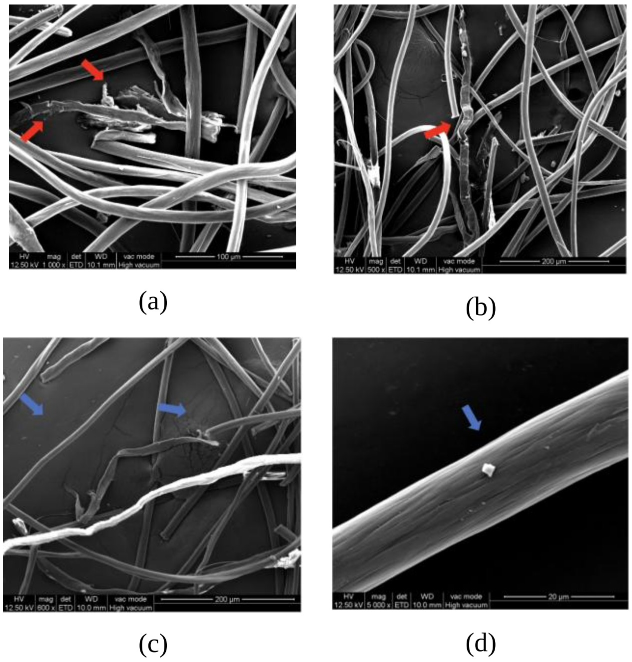


Figure 3. EDX/SEM analysis. a-b: cotton fibers are highlighted; remained fibers are acrylic (February sample), c-d: acrylic fiber are highlighted; remained fibers are cotton (March sample).

Due to the huge amount of microfibers and their entanglement, the lengths of all fibers could not be measured. However, for the determination of the fiber length range, at least 50 fiber length measurements were carried out. The results obtained showed that the fiber length distribution was in the range of 30 µm-6 mm as seen in the Figure 4. The fluorescent microscope images of some fibers whose length was measured are given in Figure 5.

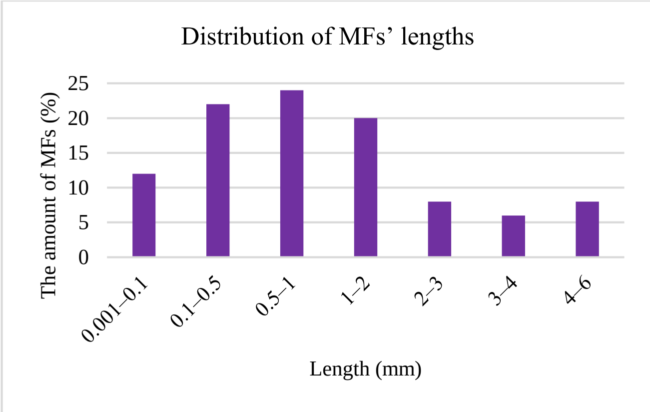


Figure 4. Distribution of MFs' lengths

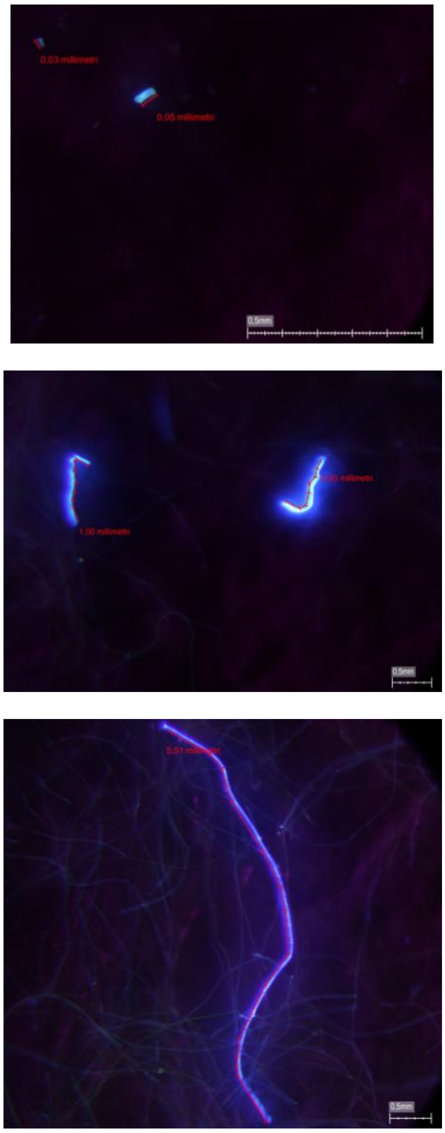
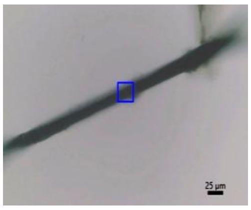
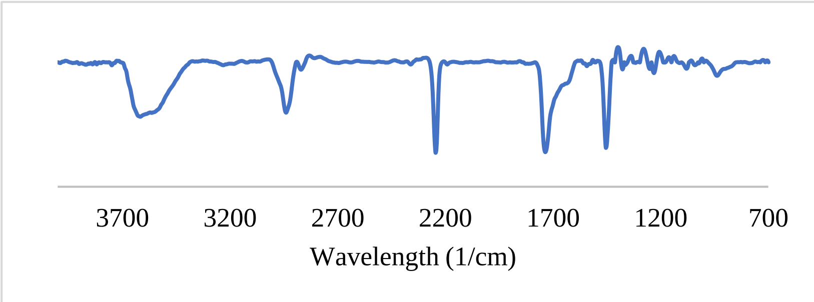
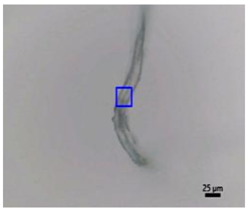
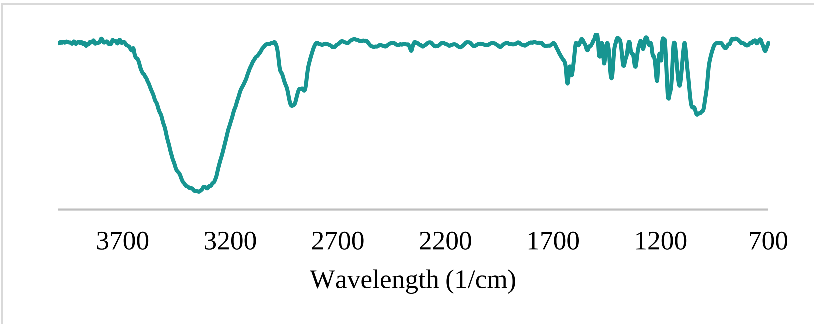


Figure 5. Fluorescent microscope images indicating length measurements of the microfibers

3.3. Chemical analysis

According to the Micro-FTIR results (Table 1), the characteristic peaks of acrylic (C=H stretching at 2924–2853 cm^{-1}, CN stretching at 2242 cm^{-1}, C=O stretching at 1734 cm^{-1}, and C-C stretching in-ring at 1452 cm^{-1}) [38] and cotton fibers (O-H stretching at 3300 cm^{-1}, C-H stretching at 2896 cm^{-1}, C=O stretching at 1730 cm^{-1}, C-H bending at 1428 cm^{-1}, C-C, C-O, and C-O-C stretching at 1030 cm^{-1}) [39] were clearly detected in the spectra. These results prove that both cotton and acrylic fibers are present in the wastewater taken in both months.

Table 1. Micro-FTIR Analysis

Fiber Types		**Micro-FTIR Results**
Acrylic		
Cotton		

As mentioned earlier, it is known that a single type of fabric is processed every day in the Biancalani machine in the factory. However, it is thought that there may be residues of previously processed fabrics in the tank where the wastewater is supplied. On the other hand, while approximately 80% acrylic and 20% cotton fibers were detected in the wastewater sample in February in the Micro-FTIR analysis, it was determined that this ratio was the opposite (90% cotton and 10% acrylic fiber) in March. These results prove that the raw material of the fabric processed in the production line is predominant. The weight of fibers deposited in the filter is directly related to two factors: the density of the fibers and the amount or type of fabric processed that day. Fabrics composed of cotton fibers have a higher density, which contributes to weight, compared to fabrics composed of acrylic fibers. This explains why the March sample, where more cotton fibers were present, had a higher microfiber weight (0.251 g/L) compared to the February sample (0.058 g/L), which contained more acrylic fibers. This can be supported by the higher density of cotton fiber (1.54 g/cm^3 [40]) than acrylic fiber (1.17 g/cm^3 [41]).

4. CONCLUSION

This study attempts to separate, characterize, and detect MFs in wastewater from a finishing procedure used to give fabric a soft touch. In this context, wastewater samples obtained from the same tank in two different months were treated with 15% H_2O_2 at 25 °C for 5 days, then the treated wastewater was filtered. An EDX/SEM and a micro-FTIR were used to investigate the MFs that had collected in the filter. In addition, their weights were measured and the fiber length range was determined. The prominent findings are as follows:

- After the organics in the wastewater were removed by the oxidative method, microfibers were successfully captured in the filter by the filtration method.
- It was not possible to count MFs one by one because there were too many, and it was concluded that weight measurement would be more appropriate for quantitative microfiber determination.
- The length range of fibers found in wastewater was measured between 30 μm and 6 mm.
- Both cotton and acrylic microfibers were detected in the both wastewater samples.

Significant amounts of microfibers were detected in 1 L of wastewater supplied at different time periods. While this situation reveals the microfiber waste that a single device is responsible for, it also reveals the inevitable microfiber pollution that the textile industry is responsible for. This preliminary study on MF identification and separation will provide guidance for planning and improving the process of separating MFs from the wastewater of textile companies in the future.

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DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Sinem Hazal AKYILDIZ: Writing – original draft, Conceptualization, Methodology, Investigation, Visualization, Writing – review & editing.

Rossana BELLOPEDE: Methodology, Conceptualization, Supervision, Investigation, Writing – review & editing.

Silvia FIORE: Conceptualization, Supervision, Writing – review & editing.

Hande SEZGİN: Conceptualization, Methodology, Investigation, Visualization, Writing – review & editing.

Bahattin YALCİN: Conceptualization, Supervision, Writing – review & editing.

İpek YALCİN ENİS: Methodology, Conceptualization, Supervision, Investigation, Writing – review & editing.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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