SPECTROSCOPIC ANALYSIS OF MICROPLASTIC FIBERS RELEASED DURING LAUNDRY WASHING CYCLE

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Abstract:
Modern life and contemporary materials have brought ubiquitous microplastic particles into our surroundings. Many researchers have focused their research towards investigating sources, pathways, toxic effects and mitigation possibilities of microplastic pollution. One of the most abundant types of microplastics is microplastic fibers, mainly released from synthetic clothes. This study investigates the possibility of identifying microplastic fibers released from textiles during the laundry washing cycle by Fourier-transform infrared spectroscopy (FTIR). Microplastic fibers released from some types of synthetic clothes during the laundry washing cycle at 40°C were collected and analyzed. Thanks to FTIR spectroscopy, it has been proven that a certain amount of microplastics is separated every time synthetics are washed.

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1. INTRODUCTION

Versatile physic-mechanical properties and relatively low prices have established polymers as irreplaceable industrial materials for various applications, such as in construction, the textile industry, packaging, the hi-tech industry, toys etc. In fact, it is hard to find an industry that does not utilize plastic in some form during its production process, highlighting the indispensability of plastics in our lives. However, due to the relatively low hardness of polymers, mechanical forces applied to polymer materials can generate microplastics, which are small particles that separate from the source material. The mounting concerns about the potential impact of microplastics on human health [1-17] and the well-being of other living organisms [6, 18] have spurred scientists worldwide to take a keen interest in the study of microplastics. Consequently, numerous investigations have been conducted to examine their health effects, identify their sources [12, 19-28], explore their pathways [9, 17, 19, 23, 29-33], and develop possible mitigation strategies [21, 34-37]. Microplastic particles can manifest in various forms, including pellets, spheres, fragments, fibers, foams, films, granules, and more [38]. Commonly encountered types of microplastics found in nature encompass polypropylene (PP) from plastic caps, polyethylene (PE) from single-use plastic bags, polyethylene terephthalate (PET) from plastic bottles, polyester (PES) from synthetic clothing, polyamide (nylon) from fishing nets and synthetic clothing, polyvinyl chloride (PVC) from plastic pipes, polystyrene (PS) from styrofoam, and others [39]. These microplastic particles can be broadly classified into two categories based on their entry into the environment: primary and secondary microplastics. While primary microplastics are directly released into the environment in the form of small particles, secondary microplastics are generated through the degradation of larger

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plastic items [27]. The three primary categories of fibers in the textile industry are natural fibers, polymers derived from natural fibers, and synthetic fibers [40]. Polyester (PES) and polyamide (nylon) are the most frequently employed polymers for clothing manufacturing [41]. During the household washing cycle, microplastic fibers shed from synthetic clothing can enter rivers and other waterways through the wastewater system, as conventional wastewater treatment processes do not effectively capture them. Notably, microplastic waste from textiles contains over 170% more synthetic fibers than natural fibers, heightening concerns about their environmental impact [42]. Synthetic fibers, particularly microfibers, are known to enter aquatic environments mainly through the laundry processes [43]. Studies have demonstrated that approximately 35% of the identified microplastics in aquatic settings originate from microfibers present in textile wastewater [44]. This time-consuming work aims to examine the release of microplastics originating from synthetic fibers during a single garment washing using optical microscopy and the FTIR method.

2. MATERIALS AND METHODS

In order to analyze microplastics from the aquatic environment, it is necessary first to isolate them. Unfortunately, there is still no standardized method for isolating and identifying microplastics. Different isolation approaches exist for different sample matrices from solid or aqueous media. Three sampling divisions can be applied to all environments: selective, extensive, or volume-reduced sampling [45]. Four main steps are applied during sample processing: volume separation, filtration, sieving and visual sorting [45].

For research into the identification of microplastics, a spectroscopic method was used, i.e. infrared spectroscopy with Fourier transform - FTIR spectroscopy.

An ATR-FTIR Shimadzu Infiniti device and Shimadzu Labsolutions software, with the Shimadzu Standard Library database, were used to determine the spectrum of infrared spectroscopy with the Fourier transformation.

2.1 Preparation of clothing samples

Eight clothing items of different sizes and colors were randomly selected, on the declarations of which it was written that they were made of synthetics or were assumed to be made of synthetics. Each garment was subjected to FTIR analysis to confirm the validity of its synthetic origin. The name, color, name of the polymer listed on the declaration and the weight of each piece of clothing are shown in Table 1. For the research, a selection of previously analyzed (weight, FTIR) different clothes was put in a mesh bag, with the purpose to trap all released fibers during a laundry washing cycle.

Table 1. The data for clothing samples - color, polymer type listed on the declaration and weight of each piece

Serial number	clothing sample	Color	The polymer listed on the declaration	Weight [g]
1.	mask	black	/	10.573
2.	sweatshirt	black	100% PES	436.001
3.	shirt	blue	/	137.970
4.	swim trunk	blue	100% PES	170.340
5.	tights	yellow	/	25.895
6.	tights	red	polyamide and lycra	21.874
7.	bag	black	/	102.691
8.	shorts	black	/	161.266

Before washing, the clothes were placed in the GUPPYFRIEND Washing Bag. This washing bag was chosen because it is considered to have a high degree of microplastic collection during washing. The analyzed clothes were washed in a “Gorenje” washing machine, type PS15/36140, model WEI943, on the program for synthetic clothes at 40°C at 1,000 revolutions per minute. Powdered detergent was used for washing purposes. After washing, the laundry was removed from the bag, where all microfibers were carefully collected from the laundry bag and the clothes. Then, the bag and clothes dried naturally in the wind. Microplastics were collected from clothes that had separated during washing and from the dried bag; all visible microplastics were carefully transferred with tweezers into a petri dish (Fig. 1). In order to obtain a representative sample, fibers of different sizes and colors were chosen. The separated particles were weighed on an analytical balance and analyzed under a “Motic DM111” optical microscope.

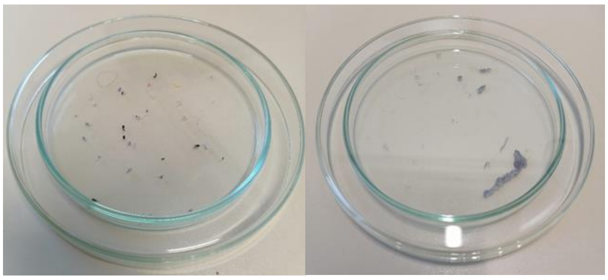


Fig. 1. Collected sample from washed clothes

2.2 FTIR method

ATR-FTIR Shimadzu Infinity 1s device was used in the range from 400 to 4000 cm^{-1} and a resolution of 4 cm^{-1}. Software Shimadzu Lab solutions with the Shimadzu Standard Library database were used to identify microplastic samples. Identification results with a matching score greater than 850 were considered valid.

3. RESULTS AND DISCUSSION

3.1 Imaging samples under an optical microscope

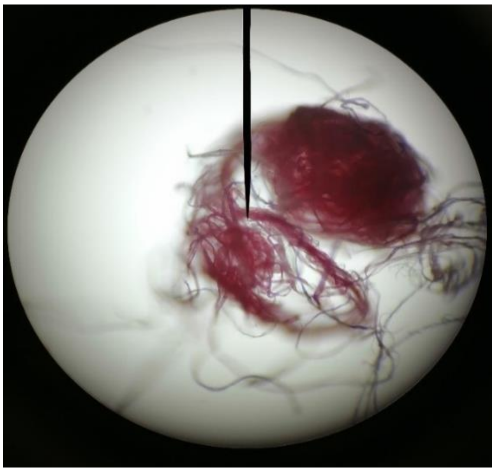
Three selected samples were chosen and examined under a camera and an optical microscope with different magnifications for this examination. It can be clearly seen that each of the particles consists of several fibers.

Fig. 2 shows an image of red fiber, (Fig. 2a shows an image made with a camera with no magnification, while Fig. 2b shows an image under the optical microscope with a magnification of 10x). The fiber shown in Fig. 2 is red and has a mesh shape gathered into a sphere. Little melting was observed, indicating the beginning of thermal degradation.

Fig. 3 shows an image of yellow fiber, (Fig. 3a shows an image of yellow fiber made with a camera with no magnification, while Fig. 3b shows an image under the optical microscope with a magnification of 40x). The fiber shown in Fig. 3 is yellow in color and irregular in shape. Some melting is observed, indicating the beginning of thermal degradation.

Fig. 4 shows an image of torn fiber, (Fig. 4a shows an image of torn fiber made with a camera, while Fig. 4b shows an image under the optical microscope with a magnification of 10x. The fiber shown in Fig. 4a is black and appears as if it was torn from a larger part at one end of Fig. 4b. No melting points were recorded on the observed sample.

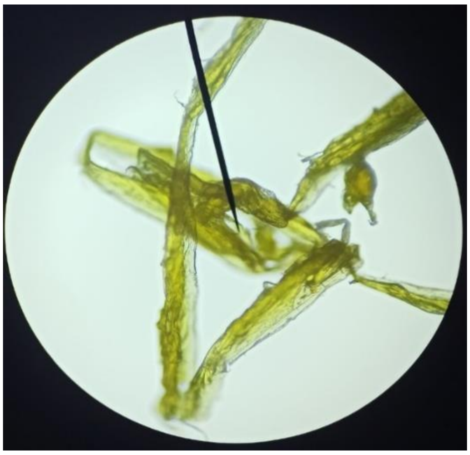
a)



b)

Fig. 2. a) Image of red fiber made with a camera and b) Magnified view of the red fiber (10X)

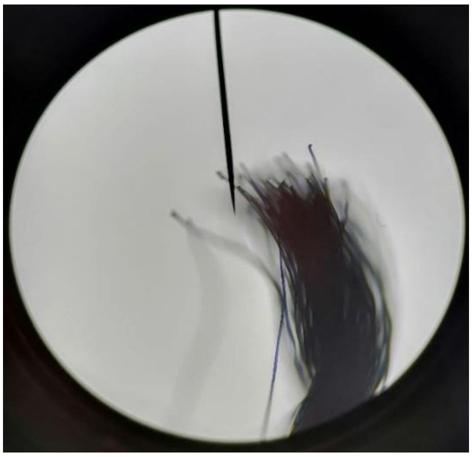
a)



b)

Fig. 3. a) Image of yellow fiber made with a camera and b) Magnified view of the yellow fiber (40X)

a)



b)

Fig. 4. a) Image of torn fiber made with a camera and b) Magnified view of the cut fiber (10x)

3.2 Analysis of clothing by FTIR spectra

The results of the chemical composition of clothing obtained by the analysis of FTIR spectra are shown in Table 2.

It was noticed that the synthetic composition of the clothing that was listed on the declaration matches the detected polymers. This confirms the synthetic composition of the clothes, consisting of polyester (PES) and polyamide (nylon) polymers, which is expected considering that the textile industry uses mostly these materials for clothing production. It was observed in several cases that the obtained spectra of PES match with the spectrum of PET with great accuracy (Fig. 5 and 6), where Fig. 5a and 6a are analyzed FTIR spectra, and Fig. 5b and Fig. 6b show matching FTIR spectrums from the database PES and PET. This is explained by the similar spectra of these two polymers. However, it is also possible to interpret it as a mixture of these two materials. This phenomenon is common in the textile industry because most of the plastic used to make clothes is recycled and, therefore, not clean.

Table 2. Presentation of the FTIR results for clothing samples

Serial number	Clothing sample	The polymer listed on the declaration	Detected polymer	Overlap accuracy	Overlap with declaration
1.	mask	/	PES	925	/
2.	sweatshirt	100% PES	PES	912	yes
3.	shirt	/	PES	929	/
4.	swim trunk	100% PES	PES	915	yes
5.	tights	/	nylon6/6	935	/
6.	tights	polyamide and lycra	nylon 6/6	924	yes
7.	bag	/	nylon6	917	/
8.	shorts	/	PES	928	/

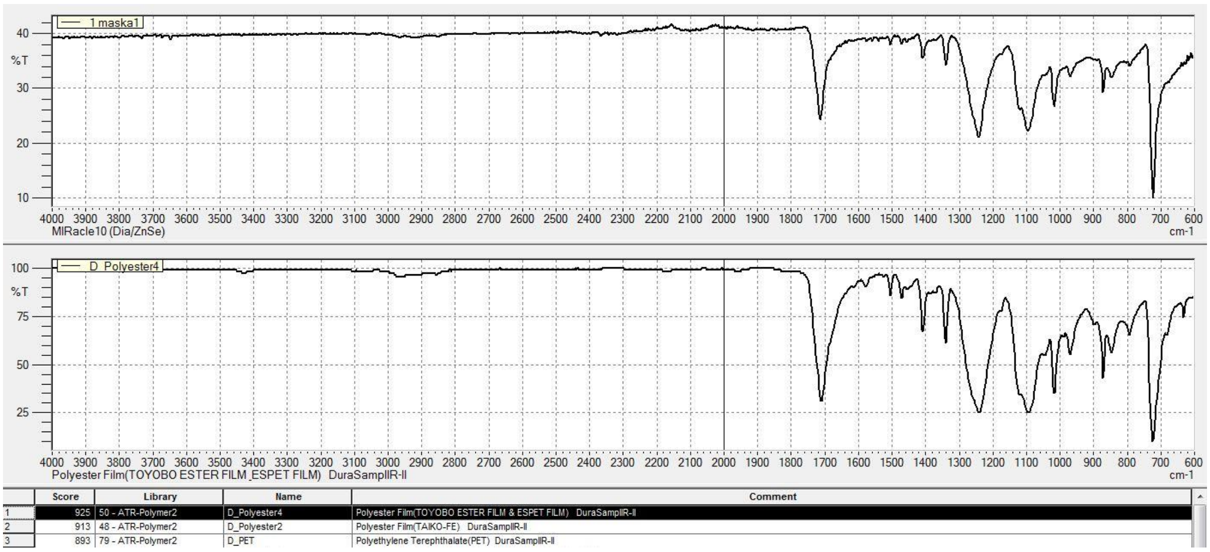


Fig. 5. a) FTIR spectrum of the black mask b) Matching FTIR spectrum from the base PES

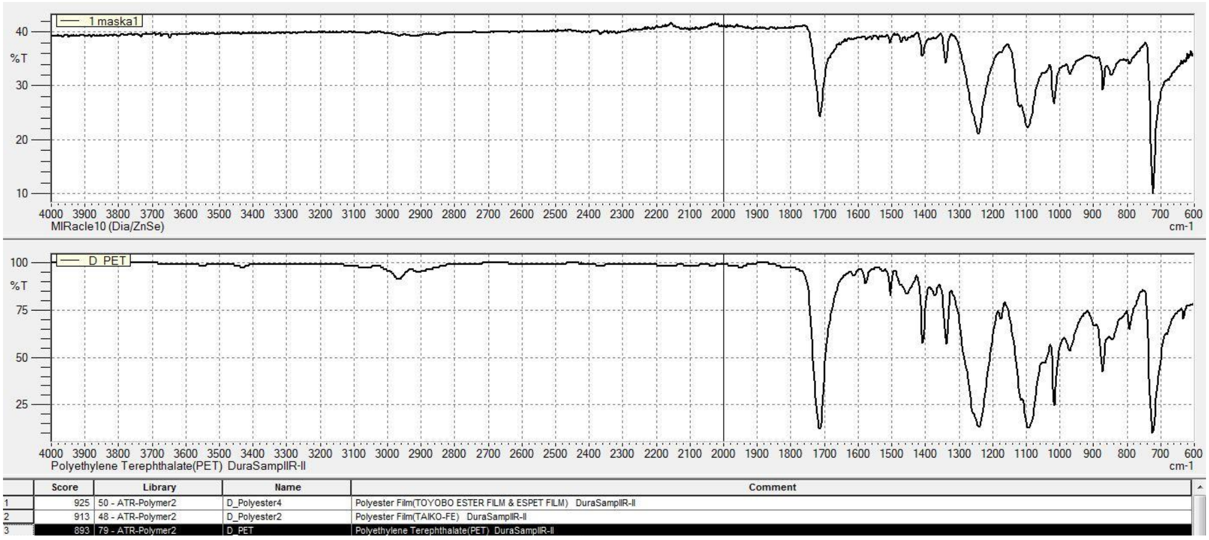


Fig. 6. a) FTIR spectrum of the black mask b) Matching FTIR spectrum from the base PET

The analyzed FTIR spectra of fibers (three selected samples) after the washing cycle are shown in Figs. 7a, 8a and 9a, while Figs. 7b, 8b and 9b show smatching FTIR spectrums from the database (cellulose, PES and PET).

The results showed the presence of particles of cellulose, PES and PET. The particles detected from PES and PET are “torn fiber” and “yellow fiber”, making them microplastic particles obtained from the wardrobe. The FTIR spectrum of the "red fiber" shows the presence of cellulose, which can be explained in several ways. The first explanation is the probable presence of cotton in the wardrobe obtained during earlier washing and mixing with cotton clothes. Another explanation for the appearance of mismatched spectra is the occurrence of thermal degradation (observed in Figs. 2b and 3b). The temperature of 40°C at which the laundry was washed is insufficient to degrade microplastic particles. However, this laundry was previously used and washed at higher temperatures, where the structure in some parts of the polymer chains probably changed. Due to this melting, the polymer particles change their appearance and structure, making them much more difficult to detect on FTIR. The problem of such detection is observed in two out of three samples where after washing, the matching result is very unreliable (<800).

Thanks to FTIR spectroscopy, it has been proven that a certain amount of microplastics is separated every time synthetic clothes are washed. If more particles were scanned and Raman spectroscopy was performed, more accurate results would be obtained regarding the appearance and origin of released microplastics.

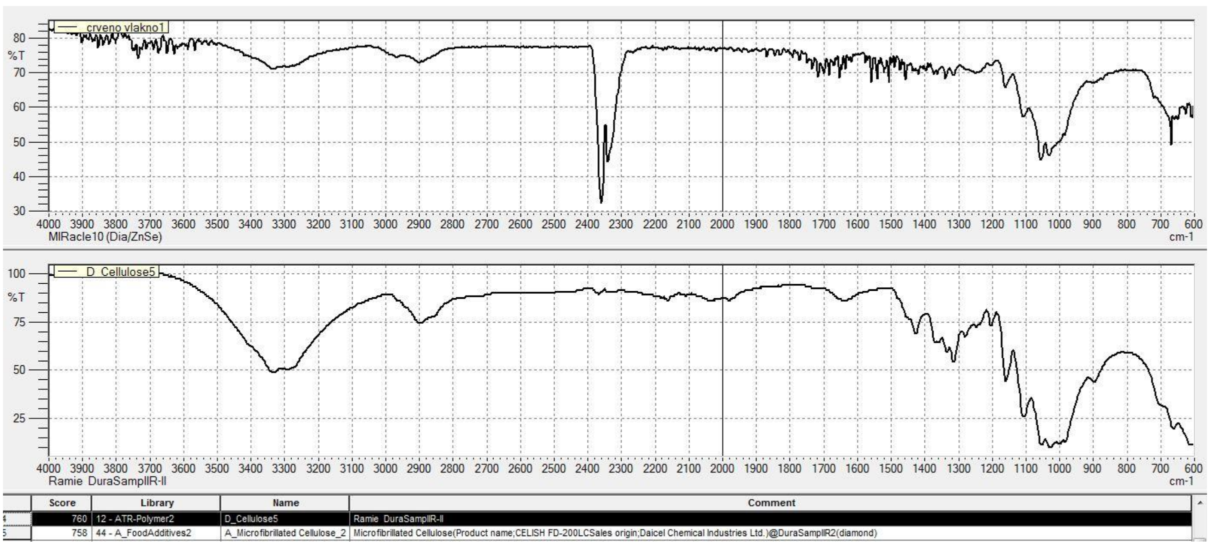


Fig.7. a) FTIR spectrum of the red fiber b) Corresponding FTIR spectrum from the base (cellulose)

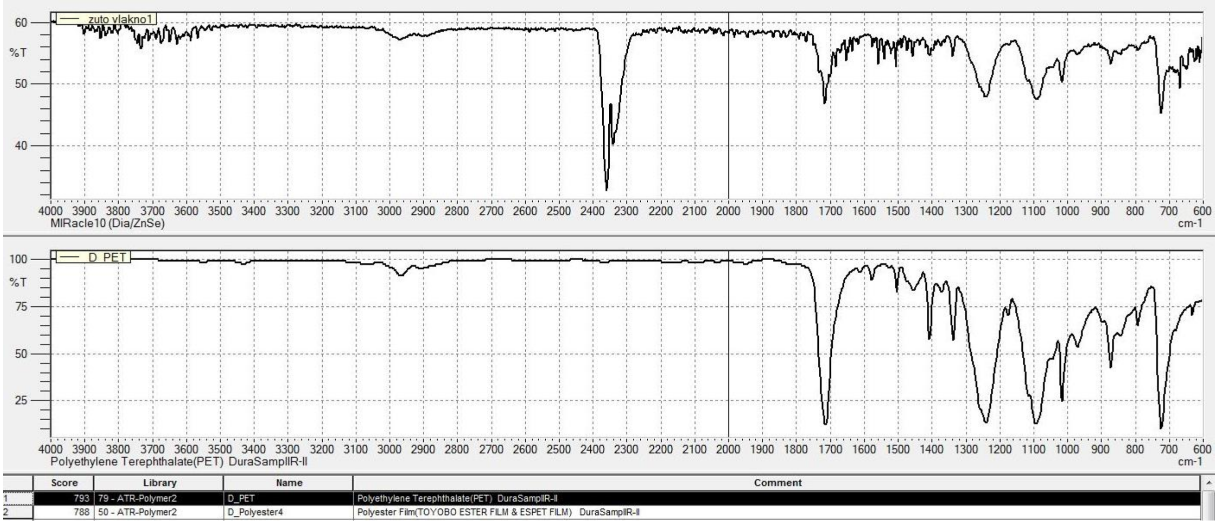


Fig. 8. a) FTIR spectrum of yellow fiber b) Corresponding FTIR spectrum from base (PET)

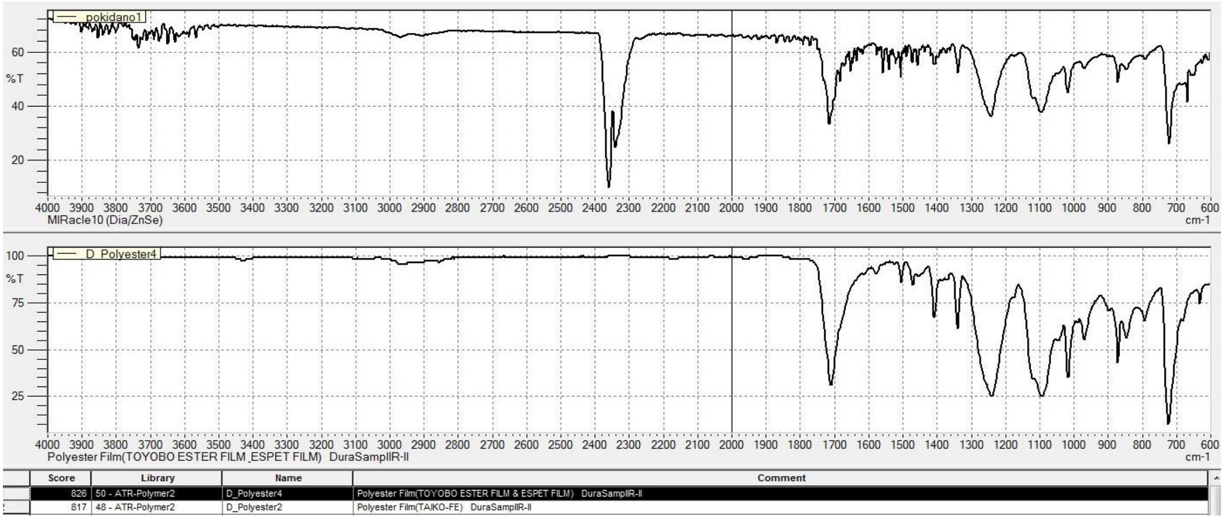


Fig. 9. a) FTIR spectrum of the torn fiber b) Corresponding FTIR spectrum from the base (PES)

4. CONCLUSION

Due to its accumulation, microplastics are attracting more and more attention in the scientific world. If this accumulation continues, which is already damaging the environment; it may start to have a direct negative effect on humans. The phenomenon of chemical degradation of polymers was observed when washing clothes at higher temperatures and changes in the synthetic material, as a result of which polymer chains often break, and a large amount of microplastic particles are released into the environment. Therefore, textile materials should be improved to undergo minor changes during washing. The possibility of spectroscopic methods of identifying microplastics during the decomposition of synthetic clothing during washing was demonstrated in this work. Thanks to FTIR spectroscopy, it has been proven that a certain amount of microplastics is separated every time synthetics are washed.

A potential obscuration of the sample by external impurities was observed, which may represent a flaw that should be overcome in further FTIR research. A good start to reach the desired goal is to use mesh washing bags during the laundry washing cycle and raise awareness on a global level about the danger of accumulating microplastics or installing filters on the discharge of washing machines, which would collect microfibers.

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