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Optimization of the textile wastewater pretreatment process in terms of organics removal and microplastic detection

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ABSTRACT

The textile industry, which offers innovative and diversified products to meet the rising demand, comes to the fore with the damage it causes to the environment. In the modern world, where synthetic fibers made from petroleum derivatives dominate the textile sector, it is evident that plastic wastes at macro, micro, and nano scales put the health of all living things at risk. The release of microplastics into the environment is significantly affected, especially from wastewater treatment plants. The primary goal of this study is to identify the most effective pretreatment approach for reducing organic matters in textile wastewater to in order to better detect microplastics and microfibers. For this, synthetic microfibers containing acrylic, polyester, and polyamide were put through various procedures using H_2O_2, Fenton's reagent, HCl, KOH, and NaOH under two different process conditions (25 °C for 5 days and 60 °C for 6 h). The outcomes revealed that H_2O_2 was the most efficient chemical for separating organic from textile wastewater, while KOH and NaOH resulted in physical and chemical damages for all polymer types. Although it was noted that HCl and Fenton's reagent did not affect other microfibers, they had been found to alter the physical structure of polyamide, making it more challenging to extract from wastewater. In addition, the use of heat had no further effect on the separation and merely served to speed up the procedure.

1. Introduction

In almost every industry, plastics, a material with relatively recent history, have managed to become indispensable for modern living (Stuchtey et al., 2019). Plastics have surpassed other materials in prominence because of their many benefits, and they are now used as the primary raw material throughout the whole world (Li et al., 2018; Halden, 2010). Plastic materials have found their way into a wide range of products, from packaging to clothes, from equipment parts to building materials (Shen et al., 2020). Globally, more than 20 different types of plastic are widely used today (North and Halden, 2013). The plastic production of the global industry in 2019 was recorded as 368 million metric tons, and there is no doubt that this amount will increase exponentially in the coming years (Ian Tiseo, 2021). Considering these increased productions, concerns over the environmental impact of plastics, especially microplastics (MPs) and nanoplastics (NPs), are also increasing. This worry has grown to include the effects of MPs and NPs on ecosystems and human health (Shen et al., 2020). MPs and NPs are found in sea surface water, sediments, beaches, deep oceans, arctic ice, freshwater systems, soil, and other terrestrial niches, and even in our food and drinking water (Hasan Anik et al., 2021). Due to their smaller size compared to macroplastics, microplastics are more easily ingested by a variety of species (Law and Thompson, 2014). They have a negative impact on about 700 aquatic animals worldwide (including sea turtles, penguins, and various crustaceans) (Issac and Kandasubramanian, 2021). Several studies have shown that microplastics have profound effects, such as growth inhibition, causing oxidative damage, and stress on the immune system (Zhang et al., 2020). The air we breathe contains microplastics and synthetic fibers, exposing us to an estimated 272 microplastics daily via inhalation (Vianello et al., 2019). Textiles, including synthetic fibers, significantly contribute to microplastic pollution (European Environment Agency, 2022). ISO/TR 21960 (2020) defines MPs as particles less than 1 mm. However, many studies classify plastics in the environment into: >100 mm (megaplastics), 20–100 mm (macroplastics), 5–20 mm (mesoplastics), 1–5 mm (microplastics), and <1 mm (small microplastics, or nanoplastics) (Thushari and

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Senevirathna, 2020; Ribeiro-Brasil et al., 2022; Bancin et al., 2019; Lee et al., 2013; Bianco and Passananti, 2020). Microplastics released from textiles are often in fiber form and are referred to as microfibers (MFs) (European Environment Agency, 2022). According to recent research, fibers and fragments are the most frequent kinds of MPs in the oceans, accounting for up to 80% of total MPs, much higher than pellets and granules (Wu et al., 2019). There are two types of sources for microplastics: primary and secondary sources. The difference is dependent on whether the particles are made to be small (primary) or whether they come from the fragmentation of bigger objects (secondary) (Kershaw and Rochman, 2016). MFs may also enter the environment via both primary sources (fibers <5 mm in size released during textile manufacture and usage) and secondary sources (fragmentation of larger objects such as discarded clothes). It is estimated that around 0.19 million tons of MFs originating from the production and use of synthetic textiles are mixed into the sea. This amount will increase as consumption increases (Henry et al., 2019). In addition, 8% of MPs in Europe and 16–35% of MPs worldwide are thought to come from synthetic textiles, and each year the world's seas are confronted with between 200,000 and 500,000 tons of microplastic waste, which is assumed to be emitted from textiles (European Environment Agency, 2022). According to the Global Synthetic Fibers Market Report, the market for synthetic fibers is expected to grow at a compound annual growth rate of 7.98% between 2022 and 2026 (Research and Markets - Market Research Reports, 2022). In addition, potentially harmful microorganisms that can adhere to the surface of MPs can infect humans. For example, MPs in the environment have been proven to contain Vibrio spp., a type of bacteria often associated with foodborne illness (Kirstein et al., 2016; Letchumanan et al., 2019).

Wastewater treatment plant (WWTPs) effluents, which can contain both primary and secondary MPs, are a significant pathway for microplastics to infiltrate the aquatic environment (Iyare et al., 2020). MPs have been discovered at all stages of WWTPs, with varying clearance percentages. Plastic debris was found in influent samples ranging from 1 to 7000 particles and effluent samples ranging from 0.0009 to 81 particles. Given the vast volumes of wastewater released by WWTPs around the world, even modest quantities of MPs in effluents could contribute significantly to the MPs load in the environment (Syranidou and Kalogerakis, 2022). While WWTPs are not currently intended or optimized for removal of microplastics, some research suggests that enhanced treatment methods can help (Iyare et al., 2020). In general, the following processes are used in order to identify microplastics in WWTPs: sample pretreatment, microplastic separation, and characterization/quantification. However, there is currently no standardization of the procedures used at each level. Since samples from WWTPs may include a significant amount of organic matter or inorganic solids, a variety of procedures are used to separate the microplastics from the matrix in which they are originally found. Quantification and identification of microplastics are anticipated to be facilitated by these procedures. For microplastics' chemical identification, removing organic materials is crucial (Sun et al., 2019). As seen in Table 1, chemical and enzymatic digestion are the two primary divisions of the purification process (Li et al., 2018).

The majority of oxidative digesting procedures described in the literature use hydrogen peroxide (H_2O_2). It is used for several different environments such as wastewater (Magni et al., 2019; Akarsu et al., 2020; Gies et al., 2018), sludge (Li et al., 2018; Sujathan et al., 2017), soil (Lusher et al., 2018), aquatic sediments (Yao et al., 2019; Zhang et al., 2019) and biota (Markic et al., 2018; Avio et al., 2020). In the literature, there are studies on variable concentration values (15–35%), temperature ranges (room temperature up to 70 °C), and reaction times (from a few hours to a week) for hydrogen peroxide (Al-azzawi et al., 2020). Ambrosini et al. (2019) reported the first evidence of microplastic contamination in a mountain glacier. Within the scope of their studies, samples were treated with H_2O_2 solution to remove organic matters before microplastic determination. The results showed that natural organic debris could be removed by filters by overnight treatment with hydrogen peroxide solution (15%).

In addition to hydrogen peroxide, the use of catalysts such as Fenton's reagent is also emphasized in the literature (Bretas Alvim et al., 2020). Fenton's reagent represents a mixture of hydrogen-peroxide and ferrous salts, an effective oxidant of a wide variety of organic substrates (Goldstein et al., 1993).

Acidic digestion, such as HCl, is commonly applied to sediment (Nuelle et al., 2014; Zobkov et al., 2020) and marine organisms (Cole et al., 2014; Wang et al., 2020). Studies have revealed that acids may destroy or damage polymers by chemical digestion (He et al., 2018). In order to avoid endangering the effectiveness of the digestive process, low concentrations of acidic solutions should be utilized. In a study by Nuelle et al. (2014), HCl was used to dissolve biogenic substances of animal and plant origin, such as leaf fragments and chitin barks, among other types of solvents. HCl has been declared to be used at a concentration of 20%, as many polymers are known to be damaged at high concentrations.

Other digestion approaches include the use of alkalis such as potassium hydroxide (KOH) and sodium hydroxide (NaOH) (Athey and Erdle, 2021). KOH has often been employed to break down the tissues of freshwater and marine biota (Cole et al., 2014; Karami et al., 2017) while NaOH is generally applied to water (Chen et al., 2020; Liu et al., 2019), sediment (Nuelle et al., 2014; Herrera et al., 2018), and marine biota (Catarino et al., 2016; Cole et al., 2014). On the other hand, alkaline solutions may also harm the MPs' morphology. Similar to acid treatment, sample loss after alkaline treatment may result in an underestimation of particle size, leading to inaccurate findings (Bretas Alvim et al., 2020). However, Dehaut et al. (2016) prove that KOH has no detrimental effects on polymers other than cellulose acetate, which is damaged in shape and size. When the studies incorporating KOH solution to extract microplastics were examined, it was discovered that 10% solution was used in the majority of them (Herrera et al., 2018; Thiele et al., 2019; Rochman et al., 2015; Dehaut et al., 2016).

Some studies show that alkaline digestion, especially NaOH, may change the color or damage the microplastics (Al-azzawi et al., 2020). For this reason, NaOH concentrations have been tried to be optimized by working in wide ranges (20–50%) (Nuelle et al., 2014). Although different concentrations have come to the fore at different ambient temperatures and times exposed during the application, it has been declared that 20% NaOH concentration gives positive results in many studies (Al-Azzawi et al., 2020).

Many studies in the literature have focused on optimizing pretreatments that can be used to remove organics from a wide variety of matrices containing microplastics such as wastewater, sludge, sediment, biota and marine organisms. Just like in all other media, there are organic compounds in textile wastewater that must be removed in order to detect microplastics. Dyes, starch and cellulose are the main organic compounds found in textile wastewater (Bhattacharjee, 2017). In addition, various surfactants, soaps, dispersing agents, oils, and antistatic agents are included as organic substances in dyeing wastewater, depending on the fiber type which is dyed (Correia et al., 1994). Within the scope of this study, textile dyeing wastewater, which was predicted to contain a significant amount of MPs in the form of MFs, had been studied and it was aimed to determine the most appropriate pretreatment for the removal of organic substances in order to separate MFs

Table 1
Various methods used to remove organics (Wu et al., 2019; Ding et al., 2020; Miller et al., 2017; Gatidou et al., 2019; He et al., 2018).

Chemical Digestion			
Acidic	Alkaline	Oxidative	
HCl	KOH	H_2O_2	**Enzymatic Digestion**
HNO_3	NaOH	Fenton's reagent	
H_2SO_4		NaClO	

from textile dyeing wastewater without damaging their chemical and physical structure.

In this study, artificial wastewater containing MFs obtained by dyeing different synthetic yarns (acrylic, polyester, and polyamide) was treated with various chemicals (Fenton's reagent, H_2O_2, HCl, KOH, and NaOH) under different process times and temperatures, and the most efficient method for removing organics from wastewater was determined.

2. Materials & methods

2.1. Materials

During the preparation of artificial wastewater, 100% acrylic, 100% polyester, and 100% polyamide yarns (yarn counts of Nm 15, Nm 25 and Nm 50, respectively), distilled water, dyestuffs (a cationic dye (Maxilon blue, CIBA-GEIGY), disperse dye (Dianix, Red K-3G), metal complex dye (Lanaset, Yellow 2R)) and various auxiliary chemicals (Na_2SO_4 (Kent Kimya), dispersing agent (Kent Kimya) were used. The removal of organic substances from wastewater was investigated using H_2O_2, Fenton's reagent (pH value < 3 (adjusted with H_2SO_4), 5 mL 30% H_2O_2 and 0.1 g $FeSO_4$), HCl, KOH, and NaOH supplied by Sigma-Aldrich.

2.2. Methods

The steps followed in the experimental process of the study are given in Fig. 1.

2.2.1. Preparation of artificial wastewater

The wastewater used within the scope of the study was obtained by dyeing polyester, acrylic, and polyamide yarns using a laboratory-scale high-temperature dyeing machine (Roaches) in a controlled laboratory environment in order to contain industrial organic components. In addition, the yarns were exposed to the rubbing, sanding, and cutting effect, and the microfiber ratio in the wastewater was increased. Dyeing baths were prepared separately for each yarn type as follows:

i 5 g of acrylic yarn was dyed with 0.2 L of distilled water, a cationic dye was dosed to get 1% color intensity, and 10% wt. Na_2SO_4 so that the dye can get into the yarns well.
ii 5 g of polyester yarn was dyed with 0.2 L of distilled water, a disperse dye was dosed to get 1% color intensity, and a 0.2 g dispersing agent improved dye solubility.
iii 5 g of polyamide yarn was dyed with 0.2 L of distilled water, and a metal complex dye was dosed to get 1% color intensity.

The dyeing process was carried out twice to obtain sufficient wastewater.

In addition to the individual dyeing waters of these three fibers (acrylic, polyester, polyamide), dyeing waters of binary (acrylic-polyester, acrylic-polyamide, polyester-polyamide) and triple fiber combination (acrylic-polyester-polyamide) were also prepared, in total 7 pieces of 50 mL aliquots were obtained.

2.2.2. Wastewater pretreatment

To remove the organic substances from wastewater, several chemicals at various concentrations (wt.) (15% H_2O_2, Fenton's Reagent, 20% HCl, 10% KOH, and 20% NaOH) were used. The wastewater samples were treated at either 25 °C for 5 days or 60 °C for 6 h.

The reactions of the five chemicals used in this study with organic substances are as follows:

1 ***Hydrogen Peroxide:*** H_2O_2 reduces carbonyls to hydroxyl and oxy by providing CO_2 output from the environment (see Fig. 2) (Guarino et al., 2019).
2 ***Fenton's Reagent:*** In this process, organic compounds react with hydrogen peroxide (H_2O_2) in the presence of ferrous sulfate ($FeSO_4$), resulting in the breakdown of organic substances into smaller molecules and carbon dioxide. Numerous studies have shown that the oxidation process is the result of the reactive hydroxyl radical produced by the catalytic breakdown of H_2O_2. Following is a description of the procedure (Bai et al., 2019):

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^- + \cdot OH \quad (1)$$

$$RH + \cdot OH \rightarrow R\cdot + H_2O \quad (2)$$

$$R\cdot + Fe^{3+} \rightarrow R^+ + Fe^{2} \quad (3)$$

$$Fe^{2+} + \cdot OH \rightarrow Fe^{3+} + OH^- \quad (4)$$

3 ***Acidic Environment (HCl):*** According to Pickett & Coyle's evaluation, acids attack the oxygen of the carbonyl group and convert it to the hydroxyl group by reducing it, then positively charge the carbon to which the hydroxyl group is attached, forming a noncovalent interaction with water or any oxygen-containing product (see Fig. 3) (Pickett and Coyle, 2013).
4 ***Alkaline Environment (NaOH and KOH):*** The mechanism of degradation of polymers by alkaline components such as NaOH and KOH has been previously reported in the literature. NaOH or KOH attack the ester groups of polymers to form sodium or potassium salt, and ethylene glycol and water are formed in addition to the polymer salt (see Fig. 4) (Al-Sabagh et al., 2016).

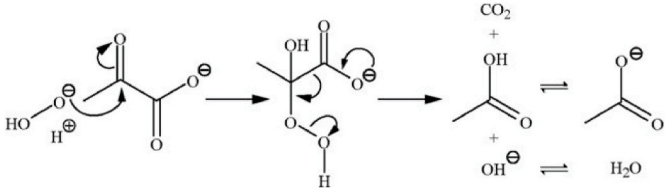


Fig. 2. Scheme of mechanism of hydrogen peroxide (Guarino et al., 2019).

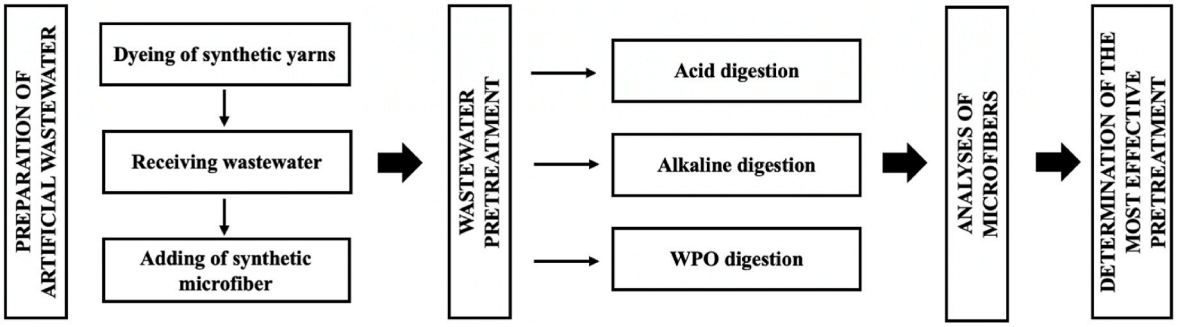


Fig. 1. Flowchart for the experimental processes.

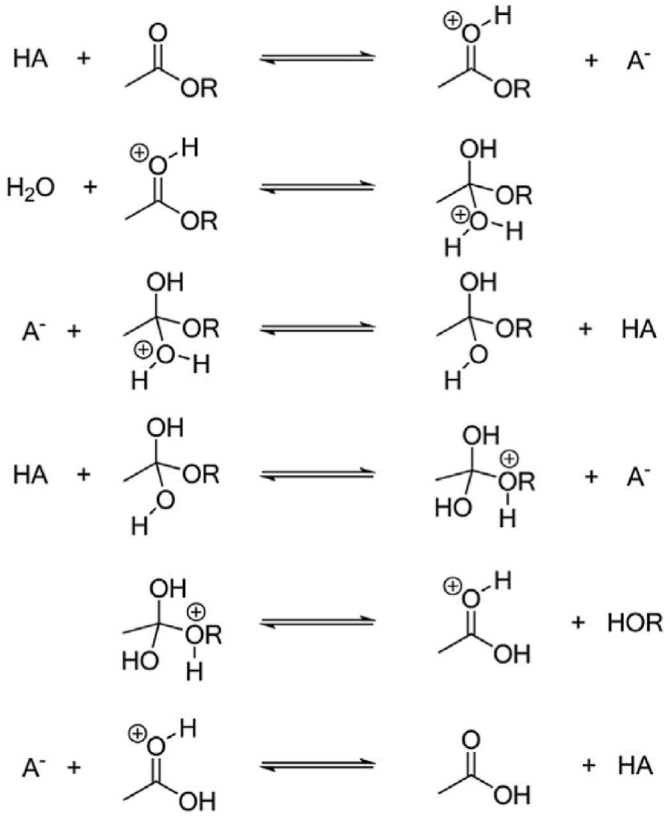


Fig. 3. Scheme of acidic hydrolysis of polymer with HCl (Pickett and Coyle, 2013).

2.2.3. Analyses of microfibers

The wastewaters, each of which was pretreated separately, were then centrifuged at 6000 rpm for 30 min. Sediments that contain microfibers were taken and placed in individual petri dishes. They were dried overnight in petri dishes at 50 °C. The MFs isolated from organics and dried were observed using an Olympus SZ51 optical microscope and a Perkin Elmer Spectrum 65 Fourier Transform Infrared (FTIR) spectroscope to examine their physical and chemical structures, respectively.

3. Results & discussion

3.1. Microscopic analyses

The images of the individual acrylic, polyamide, and polyester MFs separated from the wastewater and subjected to various pretreatments after dyeing are given in Fig. 5.

When the results of acrylic MFs were analyzed, deterioration was seen in NaOH-treated samples, whereas acrylic fibers were visible in other samples. Acrylic fiber is well known for its low alkali resistance. In a study conducted in the literature, acrylic fibers were contacted with 50% sulfuric acid at 100 °C and it was observed that degradation began after 24 h. However, when the same fibers were exposed to the same conditions with 0.5% sodium hydroxide, the degradation process began much faster (Capone and Masson, 2003). Furthermore, when the reactions of acrylic fibers to alkalis were analyzed, it was revealed that no degradation occurs due to the low concentration of the KOH solution, whereas degradation came about due to the 20% concentration of the NaOH solution. Besides that, when the effect of NaOH treatment at 25 °C for 5 days and at 60 °C for 6 h was investigated, it was observed that long-term treatment at low temperature caused the fibers to deteriorate.

When polyester MFs dyed in red color were observed under a microscope, it was noticed that alkaline pretreatment (KOH, NaOH) made MFs' detection more challenging. This was backed by the information that high concentrations of alkalis severely degrade polyester fibers (Grishanov, 2011). Moreover, temperature changes had no impact on the unfavorable alkaline effect. On the other hand, imaging data from polyamide MFs with yellow color revealed that this sample group was quite distinct from other samples. Only H_2O_2 pretreatment, out of the 5 pretreatments investigated, permitted a healthy detection of polyamide MFs, whereas other pretreatments led to structural degradation in the MFs. Both pretreatment temperatures resulted in identical results.

Following the examination of wastewater having single fiber types (polyamide, polyester, and acrylic), microscopic images from the separation procedure used on water containing pairs of fiber types (acrylic/polyester, acrylic/polyamide, and polyester/polyamide) are shown in Fig. 6.

When the wastewaters, including pairs of fiber types, were examined, the results were found to be consistent with the outcomes of individual fibers' wastewater analysis. Aside from those that were treated with NaOH, acrylic-polyester fibers could clearly be noticed in the same sample. A similar physical deterioration was brought on by NaOH treatment at 25 °C, just as it had been in the individual analyses of both fibers. It can be noted for acrylic-polyamide sample group that acrylic MFs were clearly visible in all pretreatments except NaOH pretreatment; however, polyamide MFs were only evident at H_2O_2 and KOH treated samples at 25 °C. In a similar way, polyester-polyamide MFs were clearly visible in the H_2O_2, and HCl pretreatments.

When microscopic analysis of three different fiber types (acrylic, polyester, and polyamide) was performed, the outcomes followed a similar pattern to those of the individual results for each of the three fiber types and also to those of their pairs (Fig. 7). In order to remove organics from all types of fibers employed in this investigation, pretreatments using H_2O_2 and HCl were shown to be more effective.

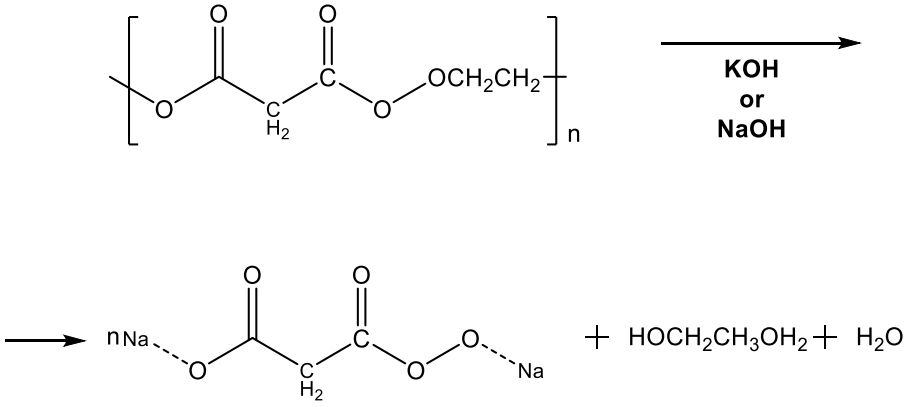


Fig. 4. Scheme of alkaline hydrolysis of polymer with NaOH or KOH (Adapted from Al-Sabagh et al., 2016).

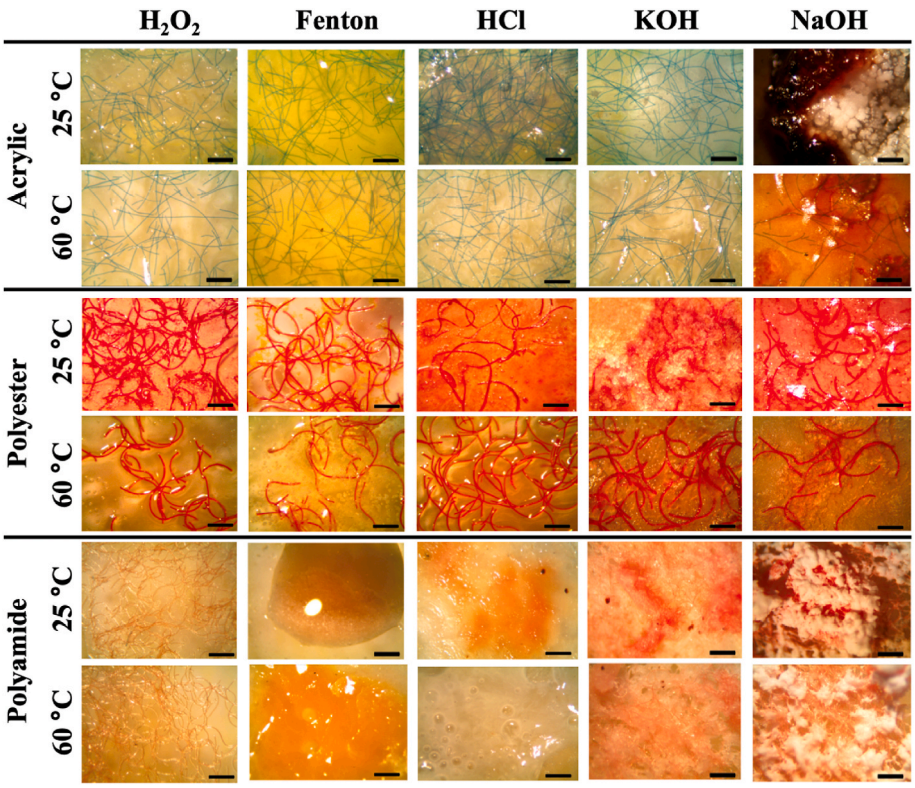


Fig. 5. Microscopic images of individual MFs' detection (scale bars indicate 0.5 mm).

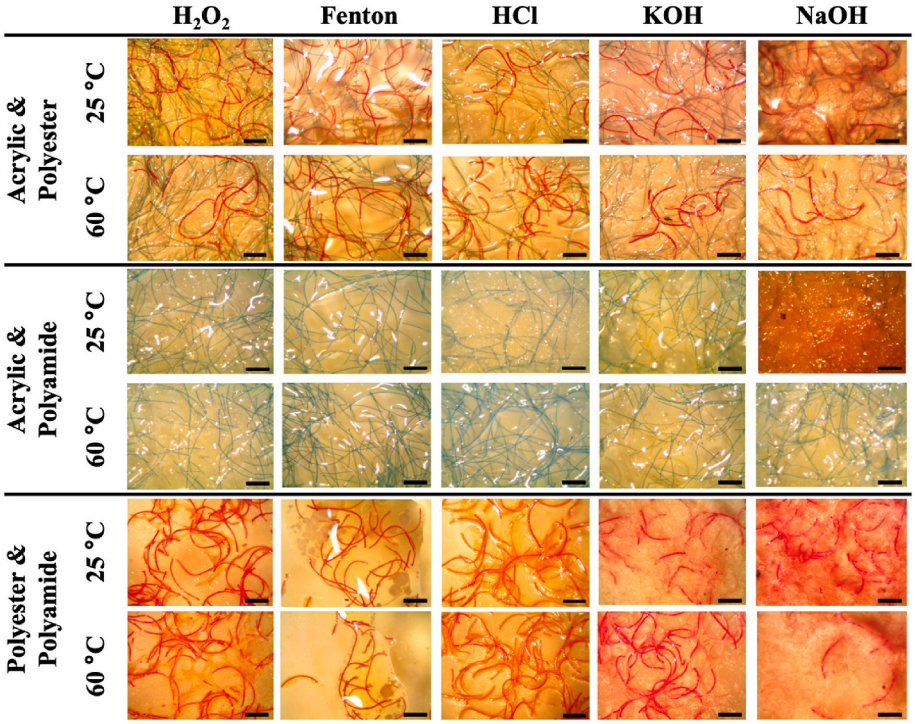


Fig. 6. Microscopic images of MFs' detection for fiber pairs (scale bars indicate 0.5 mm).

3.2. FTIR analysis

After microscopic examination, the fibers obtained from the pretreated wastewater were subjected to FTIR analysis, and it was determined whether the obtained MFs displayed the expected fibers' characteristic peaks. The FTIR analysis of the acrylic MFs extracted from synthetic wastewater after various pretreatments (Fig. 8) revealed that the transmittance intensities of the specific peaks of the acrylic structure decreased after adding H_2O_2, Fenton's Reagent, HCl, KOH, and NaOH. The characteristic acrylic peaks are C–H stretching at 2924–2853 cm^{-1},

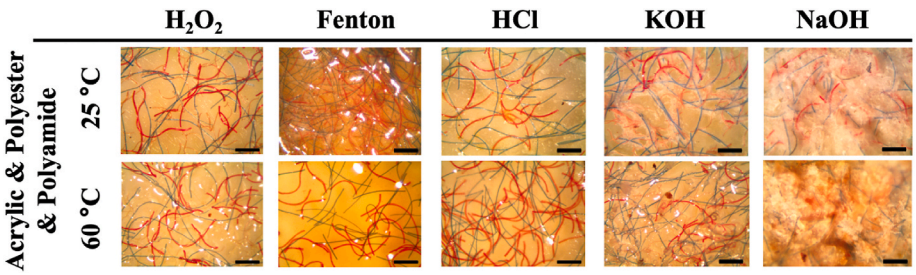


Fig. 7. Microscopic images of MFs' detection for triple combination of fibers (scale bars indicate 0.5 mm).

C≡N stretching at 2242 cm^{-1}, C═O stretching at 1734 cm^{-1}, and C–C stretch in-ring at 1452 cm^{-1} (Abdouss et al., 2012). Aliphatic C–H peaks remained intact, while –CN and -C-O peaks were relatively visible in H_2O_2 and Fenton's reagent but disappeared in acidic treatment with heat and alkaline treatments. The strong electron-withdrawing nitrile group weakens the bond on the attached carbon and accepts nucleophiles. Thus, this bond is attacked and broken by nucleophilic acids or bases. NaOH transforms acrylonitrile into acrylamide or acrylic acid. Then, a significant portion of the natural structure of acrylic acid is lost as it hydrolyzes into sodium acrylate (Dong et al., 2017; Khanlari and Dubé, 2015). As a result, it has been seen that pretreatments of H_2O_2, Fenton's Reagent or HCl without the use of heat can be preferred for acrylic MFs detection. On the other hand, the use of KOH and NaOH in the pretreatments disrupts the chemical structure of acrylic fiber and makes it difficult to detect it in the matrix.

In addition, when the analysis results of the pretreatments performed at 25 °C for 5 days and 60 °C for 6 h were compared, it was concluded that the temperature/time change did not make a significant difference (Fig. 8).

The FTIR analysis of the polyester MFs extracted from wastewater after various pretreatments (Fig. 9) revealed that the transmittance intensities of the specific peaks of the polyester structure decreased after addition of Fenton's Reagent, KOH, and NaOH. The characteristic polyester peaks are C–H stretching at 2968–2908 cm^{-1}, C═O stretching at 1710 cm^{-1}, aromatic ring at 1504–1405 cm^{-1}, the carboxylic acid (C–O) at 1242 cm^{-1}, ester (O═C–O–C) at 1092 cm^{-1}, and aromatic (C–H) at 719 cm^{-1} (Bhattacharya and Chaudhari, 2014). When H_2O_2 and HCl were added, there was no significant change in the peaks, and no new peaks were formed. Fenton method is able to degrade organic materials at a high reactive rate while maintaining a consistent level of effectiveness due to its strong oxidization capabilities (Wang et al., 2020). This occurs as a result of the Fenton reagent's ability to produce highly reactive oxidants like the OH radical at ambient temperature (Kavitha and Palanivelu, 2005). This radical attacks the terminal methyl groups, alkene bonds, and axis bridges on the polyester and breaks down the polymer (Wang et al., 2020; Ibrahim et al., 2016). While polyester MFs are affected by the oxygen formed as a result of the Fenton's reaction and undergo oxidative decomposition, it has not undergone oxidative decomposition due to H_2O_2. When polyester is treated with dilute bases, the ester bonds are broken, forming the sodium or potassium salt of the carboxylic acid together with the ethane-1,2-diol and water (Ömeroğulları Başyiğit, 2018). Once again, it was concluded that the change in pretreatment ambient conditions (25 °C for 5 days and 60 °C for 6 h) was not significant for the degradation of polyester MFs (Fig. 9). While polyester fibers can withstand the effects of weak acids, alkalis, and organic solvents, high concentrations can cause serious damage (Grishanov, 2011). Thus, while pretreatments with H2O2, HCl and relatively Fenton's Reagent (at 60 °C) in polyester microfiber-containing matrices are found to be suitable chemicals since they remove organics and enable the identification of MFs, it is not

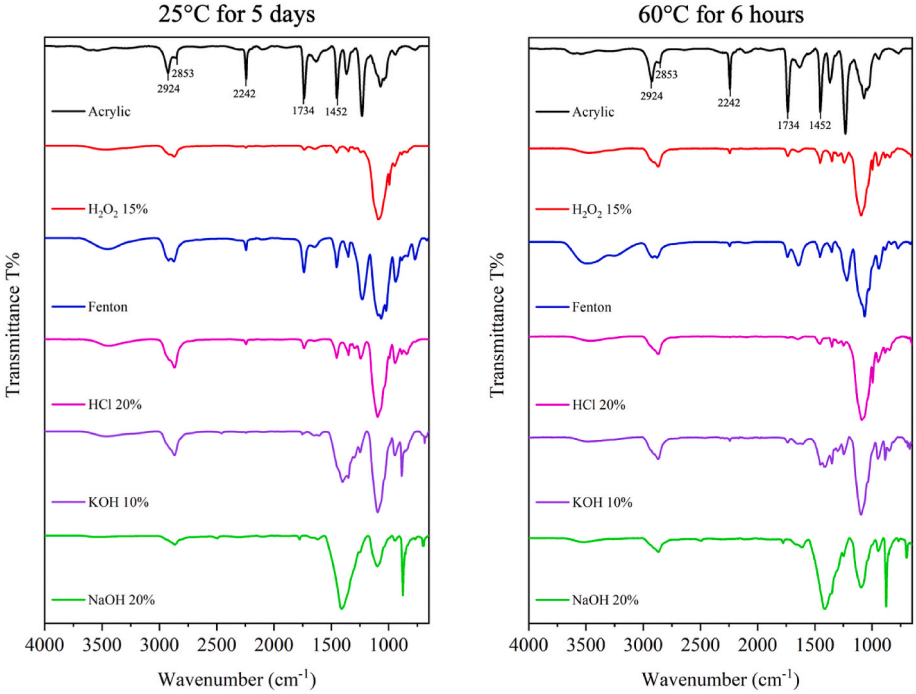


Fig. 8. FTIR spectra of the acrylic MFs separated from the samples treated at 25 °C for 5 days, and at 60 °C for 6 h.

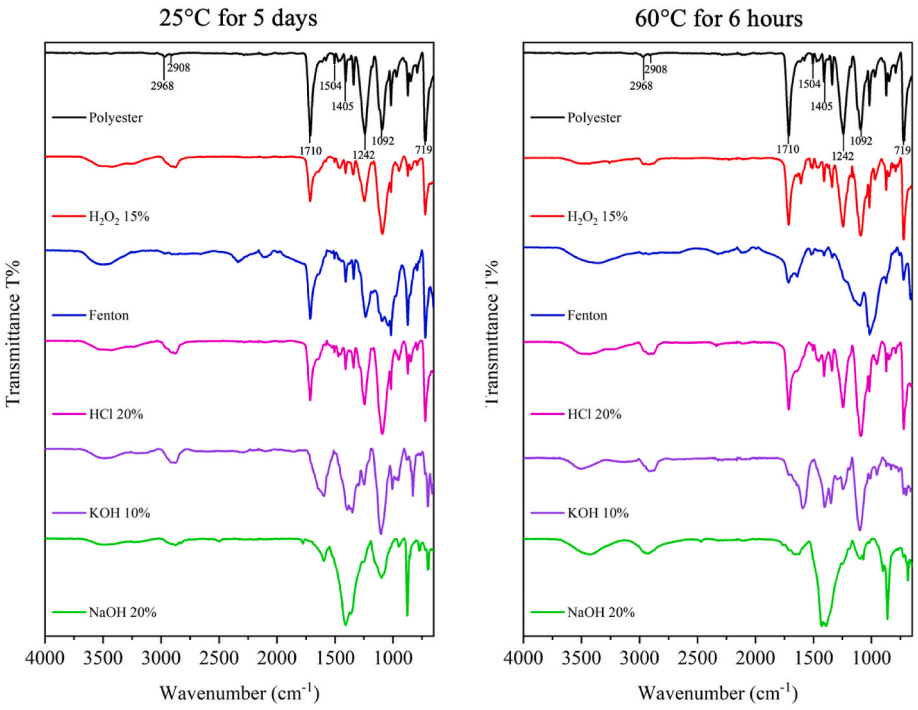


Fig. 9. FTIR spectra of the polyester MFs separated from the samples treated at 25 °C for 5 days, and at 60 °C for 6 h.

possible to make accurate inferences from the FTIR results because KOH and NaOH chemicals degrade the polyester structure.

The FTIR analysis of the polyamide MFs are given in Fig. 10. The characteristic polyamide peaks are N–H stretching at 3293 cm^{-1}, C–H stretching at 2932- 2857 cm^{-1}, C═O stretching at 1631 cm^{-1}, C–H stretching at 1536-1460 cm^{-1}, and C–N stretching at 1373 cm^{-1} (El-Newehy et al., 2011). MFs extracted from synthetic wastewater after various pretreatments revealed that when treated with H_2O_2 and HCl, no change was observed in the –N–H, –C═O and –C═C– bonds, and the transmittance intensities of the relevant peaks remained the same. Thus, H_2O_2 did not affect the polyamide structure and did not decompose it chemically. When the polyamide was decomposed with HCl, adipic acid and hexamethylenediamine moieties were formed (Achhammer et al., 1951). As a result of treatment with Fenton's Reagent, oxidative decomposition occurred in the polyamide, but the intensity of the amine peak around 3290 remained decreasing and new peaks were formed in the aromatic region. In the fingerprint region, wide uninterpretable bands were formed due to deterioration. In the alkaline treatment, it was observed that the polyamide had slight resistance to KOH and NaOH (Fig. 10). In Ullrich's patent, he stated that polyamides are very weak

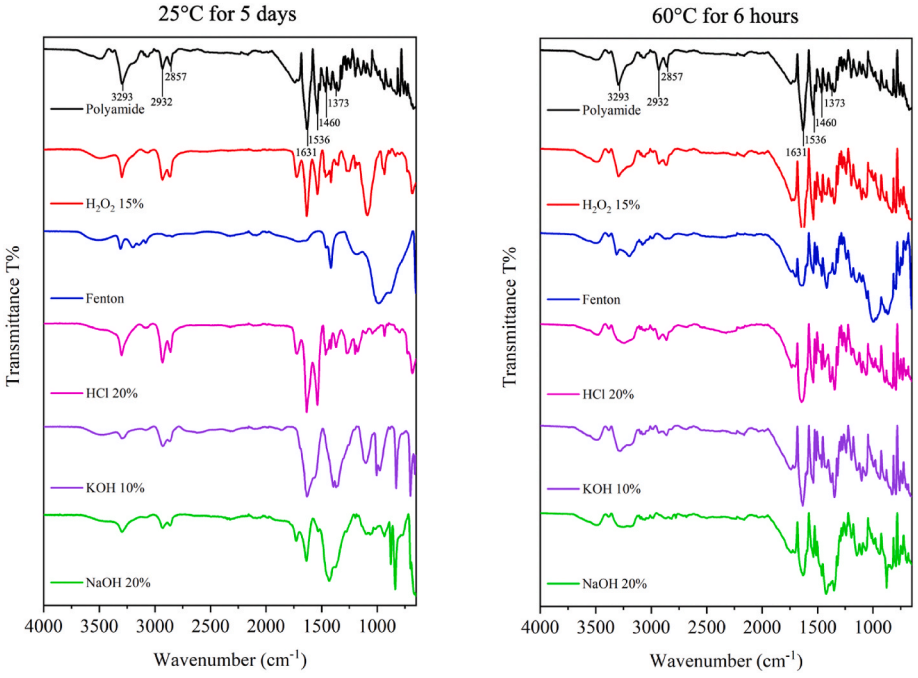


Fig. 10. FTIR spectra of the polyamide MFs separated from the samples treated at 25 °C for 5 days, and at 60 °C for 6 h.

against radicals formed as a result of the Fenton's radical reaction, and they are also resistant to alkalis (Ullrich, 1994). This correlates with the results of this study. In the treatment process at 60 °C for 6 h, it exhibited similar behavior as at 25 °C, while it was resistant to H_2O_2 and HCl, fragmentation was observed in Fenton's Reagent and alkaline environment (Fig. 10). It has been concluded that the use of H_2O_2 and HCl chemicals can be preferred due to the degradation caused by Fenton's Reagent, KOH and NaOH in the determination of polyamide fibers by removing organic components.

The changes in the FTIR spectra of the double and triple combinations of microplastics showed parallel results with the tests in their independent state, and fragmentation and resistances were correlated. Therefore, Fenton's Reagent, HCl, and H_2O_2 were generally more effective than KOH and NaOH in removing organic compounds from textile wastewater in order to perform FTIR analysis.

4. Conclusion

The main purpose of this study is to optimize the pretreatment process, which allows the detection of microplastics and microfibers in textile wastewater. In this context, controlled wastewater was prepared by dyeing polyamide, polyester, and acrylic yarns in the laboratory. These enriched wastewaters were treated with various chemicals (Fenton's Reagent, H_2O_2, HCl, KOH, and NaOH) at predetermined concentrations over two different process conditions (5 days at 25 °C and 6 h at 60 °C) to determine the most suitable method for organic removal thus MFs' detection.

Highlighted outcomes are as follow:

- In general, Fenton's Reagent, HCl, and H_2O_2 were more effective than KOH and NaOH in terms of removing organic substances from textile wastewater.
- Treatments with HCl and Fenton's Reagent caused physical damage for polyamide MFs.
- Treatment with NaOH and KOH caused physical and chemical deterioration to almost all polymers thus their usage in pretreatment of textile wastewater was not found appropriate.
- Pretreatment with H_2O_2 was the most suitable treatment since textile wastewater in real life would likely contain a wide diversity of MPs, MFs, and organic components.
- Using heat during pretreatment decreased processing time but it had no effect on MF detection. Given that using heat would cost more in this situation, it is feasible to select the best option in terms of optimizing time and cost.

As a result, chemicals such as KOH, NaOH, Fenton's Reagent, HCl and H_2O_2, which are used for similar purposes in a wide variety of matrices in the literature, were used together for the first time in this study to compare their performance at the point of removing organics from textile wastewater. However, it has been observed that especially alkali chemicals cause damage to the MFs' structure while removing organics. For this reason, the findings of this study emphasize that KOH and NaOH based pretreatments cannot be used effectively for matrices containing significant MPs, especially textile wastewater, as they damage the MPs structure. In the light of all these evaluations, it was concluded that H_2O_2-based pretreatment can be chosen as the safest method for the detection of acrylic, polyester and polyamide MFs in textile wastewater.

CRediT authorship contribution statement

Sinem Hazal Akyildiz: Writing – original draft, Methodology, Visualization, Writing – review & editing. **Hande Sezgin:** Methodology, Visualization, Writing – review & editing. **Bahattin Yalcin:** Supervision, Conceptualization, Writing – review & editing. **Ipek Yalcin-Enis:** Supervision, Conceptualization, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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