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ORIGINAL ARTICLE

Aging effect on the adsorption behavior of microfibers obtained from cigarette butts in aqueous solutions

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Abstract

The issue of cigarette butts is an environmental crisis that has affected the world. Despite their small size, CBs are one of the most common types of solid waste found in public places, particularly in coastal areas. The aim of this study was to investigate the adsorption behavior of microfibers obtained from cigarette butts on tetracycline before and after aging. 1 g of CBs was added to 50 mL of distilled water and stirred at 220 rpm for 2 h, then filtered through Whatman 0.45 µm filter paper, and the resulting MFs were dried at 60 °C for 24 h. To simulate aging, the MFs were subjected to an ultrasonic treatment at a frequency of 80 Hz and a power of 70 W for 4 h. The adsorption behavior of aged and fresh MFs was investigated using solutions containing TTC in the range of 5–20 mg/L. This study showed that ultrasonically aged MFs had a greater tendency to adsorb TTC than fresh MFs due to an increased surface area and changes in surface chemistry. It can be concluded that as the age of MFs increases, they adsorb more concentration of pollutants. This can lead to increased contamination of MFs in the presence of contaminants.

Keywords Aging process · Microfiber · Sonication · Tetracycline

Abbreviations

CBs	Cigarette butts
MFs	Microfibers
TTC	Tetracycline
MPs	Microplastics
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectrometer
EDX	Energy-dispersive X-ray

Introduction

Aquatic environments may be polluted by urban and industrial wastewater, which often contain pollutants such as lead, cadmium (Jery et al. 2023), and chromium (CrVI) (Liu et al. 2008), as well as organic compounds (Chen et al. 2020), nitrite, sulfide (Zhao et al. 2023), cyanide (Bonyadi et al. 2012), pesticide (Pirsaheb et al. 2013; Sadeghi et al. 2019), nickel ions (Foroutan et al. 2022), fluoride (Tolabi et al. 2021), arsenic (Mohebbrad et al. 2019), antibiotics (Lotfi Golsefidi et al. 2023), azo dyes (Mazloomi et al. 2021; Nasoudari et al. 2023), phosphate (Saberzadeh Sarvestani et al. 2016), MPs (Esmaeili et al. 2023), and CBs. To eliminate these pollutants, various methods are used, including electrochemical oxidation (Dong et al. 2022; Tang et al. 2022), photocatalytic processes (Chen et al. 2020), nitrite-oxidizing bacteria (Ahmad et al. 2023), biocomposites (Kan et al. 2023a, b), *Alcaligenes* sp. (Wang et al. 2020), and biochars (Zhang et al. 2021). However, despite their effectiveness, these methods can unintentionally lead to the release of MFs from CBs during the purification process. These MFs, due to their low density, are not easily removed and can undergo aging. As a result, they can adsorb various pollutants, including antibiotics, which exacerbates water pollution and environmental concerns (Kan et al. 2023a, b).

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Cigarettes contain over 4800 chemical compounds, with more than 70 being carcinogenic and over 200 being toxic (Belzagui et al. 2021). The issue of CBs is an environmental crisis that has affected the world. Despite their small size, CBs are one of the most common types of solid waste found in public places, particularly in coastal areas (Pauly et al. 2002; Moerman et al. 2011; Mahto et al. 2022). It is estimated that about 5.6 trillion tons of CBs are discarded annually. Discarded CBs are usually transported through surface runoff, wind, rain, and storms, eventually ending up in sewers or surface water and ultimately contaminating water sources (Nitschke et al. 2023). The release of CBs into the environment not only pollutes the surroundings but also has detrimental effects on the ecosystem due to the presence of nicotine, tar, and synthetic additives (Koroleva et al. 2021). In certain studies, researchers have found various toxic elements such as arsenic, cadmium, cobalt, chromium, nickel, mercury, barium, copper, and aromatic hydrocarbons in CBs (Sen et al. 2021a; b; Soleimani et al. 2023). Leaving cigarette butts in the environment poses another risk, including the release of plastic fibers from them, which contributes to the pollution of microplastics in the environment (Koroleva et al. 2021).

MPs pollution has become one of the focal points of attention around the world. Because of the increase in pollution caused by plastics and MPs, as well as their detrimental impact on environmental sustainability, the issue has reached a critical point in the oceans. This has raised awareness within the international community about the seriousness of the problem (Sen et al. 2021a, b; Nasrabadi et al. 2023a, b). MPs are pollutants that are less than 5 mm in size. They are made of chemically unmodified and non-biodegradable polymers, and they pollute every ecosystem (Nasrabadi et al. 2023a, b). MFs, released from cigarette filters, are classified as MPs due to their size, which is less than 1 mm. These pollutants enter the oceans, soil, and even the air, harming marine life, carrying toxins, and potentially entering the food chain (Ma et al. 2022; Barari et al. 2023). In one study, it was found that 80% of MPs in mangrove sediment cores are MFs (Valine et al. 2020). CBs are among the possible and important sources of MFs that are often overlooked. The CBs was designed and produced in the mid-twentieth century to capture chemicals and toxic vapors in cigarette smoke (Torkashvand et al. 2019). Most people mistakenly think that CBs are biodegradable, but this is not true. 95% of CBs consist of cellulose acetate with plastic additives. Because of the significant changes in the chemical structure of cellulose acetate, it is classified as a plastic material (Register 2000; Belzagui et al. 2021; Nitschke et al. 2023). Each cigarette butt consists of 15,000 MFs (Novotny et al. 2009). Cellulose acetate is not biodegradable because of the presence of the acetate group (Koroleva et al. 2021). Furthermore, the low nitrogen content of MFs is another contributing factor to their limited degradation. Nitrogen deficiency is known to limit microbial activity, resulting in a slower decomposition rate for CBs (Bonanomi et al. 2020).

Plastic materials that are disposed of in the environment undergo various physical, chemical, and biological processes, resulting in a change in their structure known as aging (Luo et al. 2021). Abrasion, heat, sunlight, and chemical oxidation are among the factors that contribute to the aging of plastic materials and the formation of secondary MPs (Von der Esch et al. 2020; Luo et al. 2022). During the aging process, the physicochemical characteristics of MPs change (Liu et al. 2020). The changes that occur in the structure of MPs during the aging process include a reduction in molecular weight, the formation of oxygen functional groups, a change in color of the MPs, and the cracking and brittleness of the polymer (Hüffer et al. 2018; Mao et al. 2020). One of the important features of MPs is their ability to adsorb various pollutants (Jiang et al. 2022). Shen et al. (2021a; b) demonstrated that cellulose acetate in CBs has a strong adsorption capacity and can continuously adsorb various types of pollutants from the environment. In other words, MFs that are separated from CBs can serve as carriers of harmful substances. These fibers can be ingested by aquatic organisms, leading to negative impacts (Sen et al. 2021a, b). The aging of MPs increases their ability to adsorb pollutants due to the reduction in particle size and the increase in their specific surface area (Luo et al. 2022; Zhao et al. 2022). In a study, it was reported that the adsorption of organic pollutants onto aged MPs is higher than onto virgin MPs (Bhagat et al. 2022). In another study, it was proven that aged polystyrene has a high adsorption capacity for heavy metals (Mao et al. 2020).

Antibiotics are one of the emerging pollutants that have caused serious environmental problems due to their widespread use in the treatment of bacterial diseases in humans and animals (Esmaili et al. 2023). These pollutants can cause bacterial resistance. Furthermore, they react with metal ions, leading to the production of more toxic compounds (Zhao et al. 2022). TTC is a type of antibiotic that, due to its extensive use in human and veterinary medicine, can lead to bacterial resistance and consequently have adverse effects on the environment. It is estimated that approximately 75% of this pollutant is released into the environment after it is consumed (Guo et al. 2023). Yuan Wang et al. (2021) showed that the adsorption of copper (II) and TTC onto polyethylene MPs exposed to air, water, and soil was higher than that of fresh polyethylene (Wang et al. 2021). This study specifically examines the effect of aging on microfibers extracted from cigarette butts and their ability to adsorb pollutants, such as antibiotics, in aqueous solutions. This study aimed to determine the ultrasonic performance in the aging of microfibers extracted from cigarette butts. Furthermore, the adsorption performance of aged microfibers was evaluated in comparison to fresh microfibers for tetracycline at various concentrations and over different time periods.

Materials and methods

Chemical and reagents

In this study, six common and widely used cigarette brands were collected from supermarkets. The cigarette brands were A, B, C, D, E, and F. TTC with a purity of 99% was provided by Sinadaro company, Iran. Distilled water was used in all stages of this experiment.

Characteristics and measurements

FESEM imaging was used to observe the changes in surface morphology of MFs extracted from CBs after undergoing an aging process using an ultrasonic device. The FESEM analysis was carried out using a Supra 55 electron microscope manufactured by Carl Zeiss in Germany. To determine the chemical composition, bonds, and functional groups of the MFs before and after sonication, FTIR analysis was performed using a PerkinElmer spectrometer, specifically the FT-IR/NIR FRONTIER model. Furthermore, EDX analysis was employed to determine the elemental composition of the samples. This analysis was conducted using an Oxford device connected to a JEOL-JSM-5600 SEM.

MFs extraction from CBs

First, CBs were separated from the tobacco portions and weighed. The CBs sample was poured into a beaker containing 50 mL of distilled water and stirred for 2 h at a speed of 220 rpm. MFs suspended in water after 2 h of sedimentation were separated using 0.45 µm paper and dried at 60 °C for 24 h.

Amount of turbidity produced

For this purpose, one CB (150 mg) was dissolved in 50 mL of distilled water and stirred on a magnetic shaker for 20, 30, 40, and 45 min. Then, the amount of turbidity was measured using a turbidity meter (HACH model).

Aging MFs

The MFs studied were aged using an ultrasonic process developed by Elisabeth von der Esch et al. (2020). In this study, a CBs of brand E was placed in a beaker containing 50 mL of distilled water and stirred with a shaker for 2 h. After this period, the beaker containing the MFs solution was placed in the ultrasonic device for 4 h at a frequency of 80 Hz and a power of 70 W. Finally, the supernatant was separated, and the aged MFs were dried for 24 h at 60 °C.

Adsorption experiments

Adsorption experiments were conducted using a working solution with a constant concentration of 250 mg/L of MFs obtained from the E brand (both virgin and aged MFs), along with a TTC concentration ranging from 5 to 20 mg/L. All experiments were repeated 3 times. Samples were stirred on a shaker at a speed of 110 rpm for variable times ranging from 10 to 140 min. After reaching equilibrium adsorption, approximately 1 mL of the solution was centrifuged. The remaining concentration of TTC in the supernatant was then determined using a spectrophotometer at a wavelength of 356 nm. Finally, the amount of TTC was determined based on the equations (Felista et al. 2020; Mpelane et al. 2022):

$$\text{TTC adsorption } \% = \frac{(C_0 - C_e) \times 100}{C_0} \quad (1)$$

where C_0 and C_e represent the initial and equilibrium concentrations of TTC (mg/L).

Statistical analysis

The data were analyzed using Microsoft Excel 2016 and SPSS version 26 software. The Shapiro–Wilk test was utilized to evaluate the normality of the data. If the data followed a normal distribution, the one-way ANOVA test was used. If the data did not follow a normal distribution, the Kruskal–Wallis test was utilized.

Results and discussion

Characterization

According to the findings of Fig. 1a, MFs have soft ridges and few pores and cracks. But as the aging process progresses, the number of pores and cracks gradually increases, and the fang-like threads become separated from the MFs. This study demonstrates that MFs undergo fragmentation and flaking as they age, resulting in the formation of cracks and irregular holes on their surfaces. (Guo et al. 2023). As shown in Fig. 1, the surface of aged MFs is rougher than that of new MFs and exhibits an irregular and fluff-like structure. This porous and irregular structure in aged MFs can increase the adsorption of pollutants. In one study, researchers discovered that the aging process leads to an increase in the specific surface area of MPs (Fu et al. 2021).

FTIR spectroscopy was used to analyze and modify the functional groups present on the surface of MFs samples

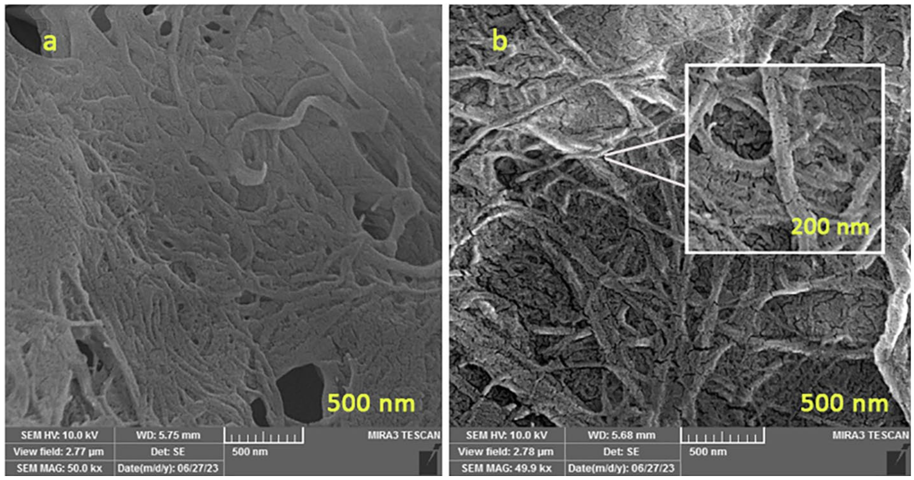

Fig. 1 FESEM of **a** virgin cigarette MFs and **b** aged cigarette filament

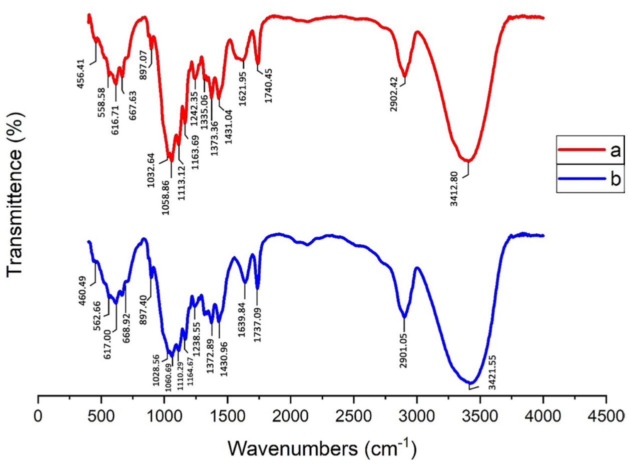

Fig. 2 FTIR Spectra of **a** before and **b** after aging cigarette

before and after undergoing ultrasonication. Figure 2a illustrates the FTIR spectrum of MFs before sonication. The peaks observed at 3412.80 cm^{-1}, 2902.42 cm^{-1}, and 1431.04 cm^{-1} correspond to the stretching vibration of O–H, bending vibration of C–H, and vibration of C–H (You et al. 2021). The peak at 1335.06 cm^{-1}, which disappeared after the ultrasonic process, is within the range associated with C–H bending vibrations. Hence, it can be inferred that this peak is connected to the C–H bending vibrations (Atiku et al. 2014). The peak at 1373.36 cm^{-1} is associated with CH_3, while the peak at 897.07 cm^{-1} is attributed to the stretching of the aromatic ring (Veerasingam et al. 2021). Figure 2b displays the FTIR spectrum after sonication. The peak corresponding to the O–H stretching vibration has shifted to 3421.55 cm^{-1}, indicating the formation of hydroxide resulting from the reaction between oxygen and the formed radicals (Wang et al. 2022). It was reported that there is a positive correlation between the presence of the O–H functional group and the adsorption capacity of metals on aged MPs (Huang et al. 2020). The peak associated with the C–H bending vibration has slightly decreased after ultrasonication, reaching a value of 2901.05 cm^{-1}. The peak at 1740.45 cm^{-1} observed before sonication was attributed to the stretching bands of C=O. After the aging process, this peak exhibited a slight reduction, measuring 1737.09 cm^{-1}. This decrease occurred due to the oxidation and separation of a portion of the carbon from the carbonyl group. Aged MFs exhibits a peak at 1639.84 cm^{-1}, which is attributed to the adsorption of specific carbonyl components. Following sonication, the peak associated with aromatic rings reached 897.40 cm^{-1} due to the environmental characteristics of the material (Asemani et al. 2020).

EDX technique is a powerful method for analyzing the composition of MPs (Gniadek et al. 2019). This technique identifies the type and number of elements present in the MFs separated from the CBs before and after the ultrasonic process. A range of oxygen, carbon, nitrogen, phosphorus, calcium, and sulfur elements in virgin MFs is identified in Fig. 3a. The highest percentage of detected elements was related to oxygen and carbon, with 48.71% and 45.76%, respectively. Other elements also have small amounts. Figure 3b shows the changes in the amounts of the mentioned elements in MFs after the ultrasonic process. Based on the findings of Fig. 3b, there was a decreasing trend in carbon and nitrogen elements, while the other elements showed an increase. The amount of carbon after sonication reached 45.62%. The carbon content in the aged MFs decreased slightly to 45.62%. Hadiyanto et al. (2021) confirmed that the carbon content of MPs decreased slightly after the aging process (Hadiyanto et al. 2021). A slight increase in the oxygen content of the aged MFs can be attributed to the fact that the MFs have undergone oxidation due to the influence of ultrasonic waves and prolonged exposure. In a study, it was shown that the oxygen content in MPs increased when exposed to oxidation agents (Zhou et al. 2020).

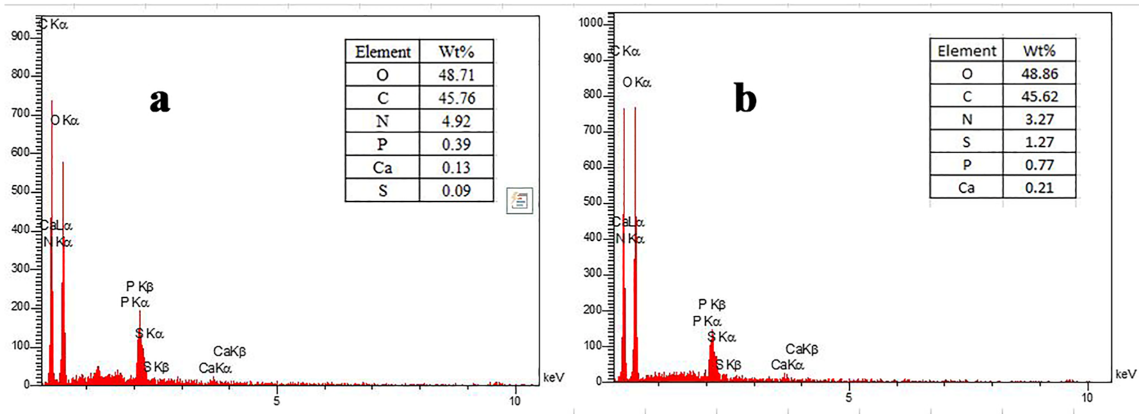

Fig. 3 EDX spectrum **a** before and **b** after aging

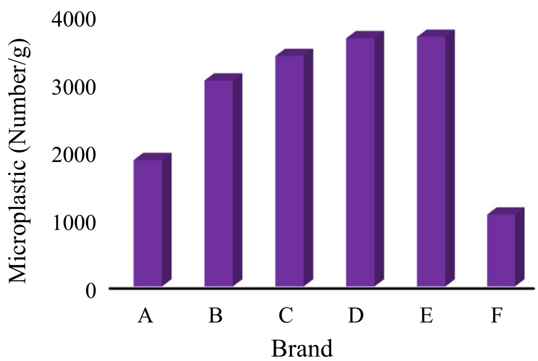

Fig. 4 Percentage of microplastics released from studied masks based brand

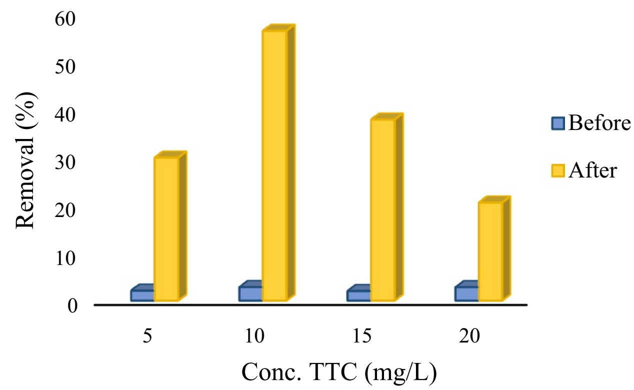

Fig. 5 Effect of TTC concentration on removal efficiency

Effect of different variables on the rate of MFs produced

Figure 4 indicates the percentage of MFs released from the studied cigarettes based on brand. The results showed that the amount of MFs produced from CBs varied among different brands (*P* value < 0.05). According to Fig. 4, brands D and E had the highest MFs emissions compared to other brands, with rates of 3636 numbers/g and 3657 numbers/g, respectively. The lowest rate of MFs is related to the F brand with a release rate of 1049 numbers/g. Belzagui et al. (2020) reported that CBs release approximately 100 MFs daily, and 0.3 million tons of MFs annually (Belzagui et al. 2020). Nitschke et al. (2023) reported that more than 100 MFs are released from each CBs during a day (Nitschke et al. 2023).

Figure 5 shows the effect of TTC concentration on the adsorption rate. The results show that the adsorption rate of TTC in both aged and virgin MFs is higher at a concentration of 10 mg/L compared to concentrations of 5, 15, and 20 mg/L. The adsorption rate of TTC in aged MFs, at a concentration of 10 mg/L, was 56.23%, whereas the adsorption rate of virgin MFs was only 2.81%. One possible reason for the higher adsorption rate of TTC at a concentration of 10 mg/L compared to other concentrations could be the saturation of MFs. At lower concentrations, MFs may not be fully saturated with TTC, leading to reduced adsorption rates. But at higher concentrations, MFs may become saturated, resulting in increased surface adsorption rates. In a study, it has been shown that as the concentration of TTC increases, its adsorption rate on PVC decreases. This is attributed to the saturation of active sites for adsorption in PVC. Furthermore, the competition among TTC molecules to bind to the MPs surface as an adsorbent has resulted in a reduction in the adsorption capacity (Zahmatkesh Anbarani et al. 2023). Khademi et al. (2015) demonstrated that an increase in the initial concentration of the pollutant results in a decrease in its adsorption rate onto the adsorbent (Khademi et al. 2015). The difference in adsorption rate between aged and virgin MFs can be attributed to the surface area of the MFs. Over time, the surface area of MFs may increase due to the aging process, resulting in more adsorption sites available for pollutants. Additionally, the surface chemistry of MFs

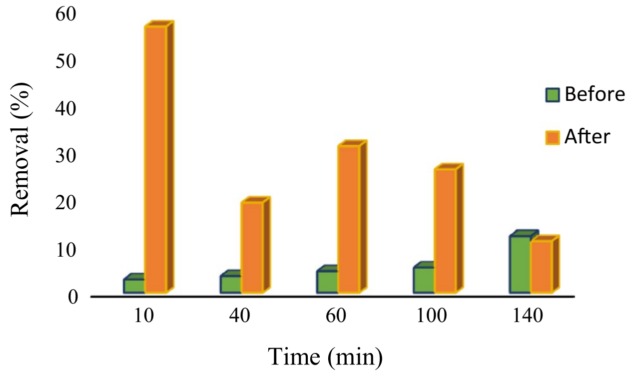

Fig. 6 Effect of contact time on removal efficiency

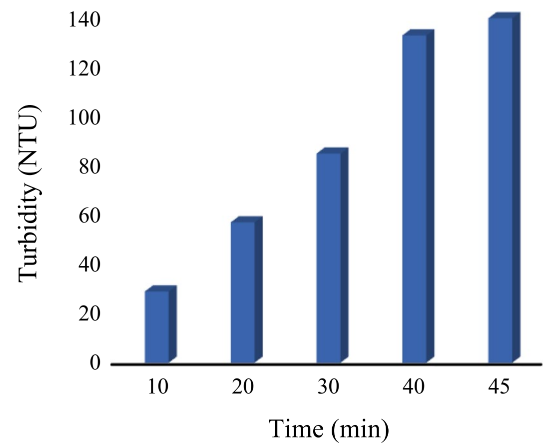

Fig. 7 Turbidity values at different times

changes during the aging process, which can impact the adsorption of pollutants (Yu et al. 2021).

Figure 6 shows the effect of contact time on the rate of adsorption. According to Fig. 6, the highest adsorption rate of TTC in aged MFs with a contact time of 10 min was 56.23%. The lowest adsorption rate was 10.91% after 140 min. On the other hand, the lowest and highest adsorption rates of TTC in virgin MFs were 10 min with a rate of 2.81% and 140 min with a rate of 11.97%, respectively. As time passes, the surface area of MFs may increase due to the aging process. This leads to an increase in the number of adsorption sites available for pollutants. Additionally, the surface chemistry of MFs changes during the aging process, which can impact the adsorption of pollutants. The saturation of the MFs and the available contact surface area may be the reasons for the variation in adsorption rate during different time period (41). In a study, ciprofloxacin absorption to MPs decreased over time (Atugoda et al. 2020). At shorter time intervals, aged MFs may not be fully saturated with TTC. Additionally, due to the aging process, there is an increase in surface area and pores, leading to higher adsorption rates. In fresh MFs, adsorption occurs over a longer period of time due to a smaller contact surface area in the fibers (42).

Determining the amount of turbidity produced. According to Fig. 7, the test results show that the turbidity caused by the diffusion of MFs in an aqueous solution increases over time (P value < 0.05). According to Fig. 4c, the turbidity level reached 29 NTU within the first 10 min after placing the CB in the water. With the passage of time, the turbidity steadily increased. After 45 min, the turbidity of the solution reached a maximum value of 140 NTU. These results show that the presence of MFs released by CBs in aquatic environments can have significant effects on water quality, particularly over an extended period of time. Alvim et al. (2023) stated that MPs particles ranging from 1 mm to 5 μm in size are associated with water turbidity (Alvim et al. 2023). Another study found a direct relationship between turbidity and the abundance of MPs (Li et al. 2021).

Conclusion

The study utilized FESEM, FTIR, and EDX techniques to characterize the MFs. FESEM analysis showed that aged MFs had a rough and porous surface, which enhanced pollutant adsorption. FTIR analysis indicated changes in functional groups after ultrasonication, including the formation of hydroxides and the oxidation of carbon. EDX analysis revealed a decrease in carbon content and an increase in oxygen content in aged MFs. This study showed that the price of cigarettes does not have a significant effect on the release of MFs. However, the amount of MFs produced from CBs varies among different brands. However, the concentration of TTC and the contact time did influence adsorption rate. Aged MFs exhibited higher adsorption rates due to an increased surface area and altered surface chemistry. The adsorption behavior of aged and fresh MFs was investigated using solutions containing TTC in the range of 5 to 20 mg/L. This study showed that ultrasonically aged MFs had a greater tendency to adsorb TTC than fresh MFs due to an increased surface area and changes in surface chemistry. It can be concluded that as the age of MFs increases, they adsorb more concentration of pollutants. This can lead to increased contamination of MFs in the presence of contaminants.

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Authors' contributions MZA performed the experiments of the paper; AEN wrote the paper, ZB wrote and edited the paper, and conceived and designed the experiments.

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Data availability All necessary data are included in the document.

Declarations

Conflict of interest The authors declare that they have no conflict of interests.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

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