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High-efficient removal of tetrabromobisphenol A from waste mobile phone printed circuit boards leached solution by micellar enhanced ultrafiltration

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Abstract

Background: Tetrabrombisphenol A (TBBPA) is one of the major brominated flame retardants (BFRs) used in waste mobile phone printed circuit boards (WMPPCB) that accounts for approximately 60% of the total BFR market.

Methods: The potential of TBBPA removal from WMPPCB leached solution was investigated using micelle-enhanced ultrafiltration (MEUF) in the presence of cationic, anionic, and nonionic surfactants. The efficiency of several parameters including surfactant concentration, transmembrane pressure (TMP), pH, and TBBPA concentration, was evaluated to improve the MEUF. The optimal conditions were used to assess the MEUF for removing TBBPA in a real sample.

Results: The cationic surfactant cetylpyridinium chloride (CPC) showed better performance than other surfactants in removing TBBPA due to its electrostatic interactions with anionic forms of TBBPA. The removal efficiency of TBBPA increased from 48.99% to 99.10% by adding a surfactant (less than the critical micelle concentration). Increasing the pH in the range of 5 to 11 increased the efficiency of TBBPA removal due to the increase in the TBBPA solubility in the micelles. TMP had the most significant effect on permeate flux compared to other parameters but did not significantly affect the TBBPA removal efficiency. The MEUF process effectively removed (above 99%) TBBPA in the concentration range of 20 to 80 mg L^{-1} under optimal conditions. The HPLC-UV analysis of the real sample indicated the removal efficiency of 100% of TBBPA.

Conclusion: MEUF using CPC is a critical performance technology for removing TBBPA from the leached solution of electronic waste.

Keywords: Flame retardants, Surface-active agents, Micelles, Ultrafiltration, Electronic waste

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Introduction

Chemical pollution of the environment including food, water, soil and air pollution has become a global concern in the last two decades (1-3). The generation of waste electrical and electronic equipment is increasing at an exponential rate due to the rapid advancement of technology (4-6). Printed circuit boards (PCB_s), as fundamental components of electrical and electronic equipment (EEE) (7,8), consist about 20% to 30% of the total weight of a mobile phone. All PCBs generally include polymer substrates (5). Polymeric substrates threaten the environment and human health by releasing hazardous substances (9,10), such as volatile organic (4), and semi-volatile organic compounds (e.g., Tetrabromobisphenol A, TBBPA) (4,11). TBBPA are used as one of the major brominated flame retardants (BFRs) in waste PCBs with a concentration more than 100 mg kg^{-1} (12,13). TBBPA is known as a stable emerging micro-pollutant in the environment due to high lipophilicity, ability to bioaccumulation, and chemical stability (12,14), which can cause neurotoxicity, genotoxicity, biotoxicity, and endocrine dysfunction (15). So far, various techniques including physical techniques (16,17), aerobic and anaerobic biological techniques (17,18), and various chemical processes (12,14,19-23) have been proposed and administered to remove TBBPA contamination.

Most methods widely used in the past decades, including photocatalysts, oxidation, adsorption, and microbial decomposition, have relatively high economic costs (16). Micellar-enhanced ultrafiltration (MEUF) is a surfactant-based membrane separation technology that has a significant effect on the separation of micromolecular contaminants from mixed aqueous solutions (24,25). This process is cost-effective with a high percentage of pollutant recovery, low energy, and low pressure requirements, high flux through filtration, and low space requirements (25,26). In MEUF process, when a surfactant is added to the contaminated water at a concentration higher than the critical micelle concentration (CMC), the surfactant molecules form large and clear micelles in the aqueous solution. Consequently, some of the organic pollutant molecules dissolve in the micelles. The micelles containing the solutes are rejected by a ultrafiltration (UF) membrane with a smaller pore size than the micelles. Thus, additional treatment of retentate streams containing high concentrations of pollutants is much more economical than direct treatment of feed streams (6,27-29). Several researchers have studied the separation of various compounds of phenol by MEUF in recent years (24,29), but a paucity of information is available on MEUF of brominated phenols, including TBBPA. The present study aimed to investigate the effectiveness of the MEUF process for the removal of TBBPA present in waste mobile phone printed circuit board (WMPPCB) leached solution for the first time. In order to remove this compound, ionic and non-ionic surfactants were tested. After selecting the surfactant, to improve MEUF performance, the effect of some operating factors such as surfactant (0.5-5 CMC) and pollutant (20-80 mg L^{-1}) concentration, operating pressure (0.6, 0.8, 1, 1.2 bar), and pH (5-11) on removal efficiency and permeate flux were investigated.

Methods

Chemicals

The required substances (HCl, NaOH, and CH_3OH) were purchased from Sigma Aldrich (TBBPA) and Merck, Germany. To make a working solution, a stoichiometric amount of (Sigma-Aldrich, CAS # 79-94-7, purity > 98.0%) TBBPA were dissolved in alkaline water using the ultrasonic method. This solution was freshly prepared every working day. The feed solution was prepared by adding a stoichiometric amounts of surfactant to the working solution. All surfactants (SDS, CPC, Brij 35, and TX-100) were obtained from Sigma Aldrich and used without further purification. Furthermore, HPLC grade solvents were purchased from Merck Company, Germany. Double distilled water was used for all experiments.

Membrane

A 200 kDa cross-flow hollow fiber membrane was prepared for ultrafiltration from Abtin Parsian Pishro Company in Isfahan, Iran. Membrane was used for experiments without further purification. The manufacturer's instruction was used to store and wash the membranes. The properties of membrane used in this system are listed in Table 1.

Sample of WMPPCB

To examine the removal efficiency of TBBPA, the polymer employed in WMPPCB was examined as a real sample. The WMPPCB was purchased from Pars Charkhesh Asia Company in Tehran, Iran. Initially, the accessories mounted on the PCBs were separated and crushed using a type of hammer mill. In this stage, the isolation of iron particles was performed via a magnetic separator. The decrease in the size of particles facilitates the plastic separation via the shaking table method. Thus, an industrial hammer mill was used and the particles size was reduced to an average of 1 mm. After this step, the plastics in the PCB were separated according to their density differences using the shaking table method. The method used by Kim et al to leach polybrominated diphenyl ethers in TV plastics was used to leach TBBPA in separated polymer samples of PCBs. Based on this method, 20% methanol was used as a leachant. In this study, 20 g of the polymer separated from WMPPCB was added to two liters of 20% methanol solution and kept for five days at room temperature (30°C) without ionic strength control (30). A fiberglass filter was used to separate the liquid phase. The filtered suspension was tested under the optimal conditions obtained from synthetic samples. TBBPA was not detected in the filtered suspension. Thus, TBBPA (20 mg L^{-1}) was spiked into the filtered suspension.

Micellar-enhanced ultrafiltration experiments

Experiments of UF and MEUF processes were performed with an effective membrane area of 0.08 m^2 at room temperature. The type of filtration was cross-flow, in which most of the feed flow travels tangentially across the surface of the filter. In this study, the ultrafiltration process was performed using a self-made MEUF setup (Figure 1) on a laboratory scale, which was supplied by a separate feed tank with a maximum capacity of 3 L, a separate tank for collecting permeate, a pressure gauge, a control valve to adjust the pressure along the needed equipment, and

Table 1. Characteristics of ultrafiltration membrane

Characteristics	
Molecular weight cut-off (Da)	200000
Membrane outside diameter (mm)	0.40
Effective membrane area (m^2)	0.08
Maximum number fibers	250
Length of fiber (mm)	250
Membrane material	PES
Housing material	Polyvinylchloride
Membrane type	Hollow fiber
Flow direction	Outside to inside
Flow type	Cross flow
pH range	1 - 13

PES, Polyether sulfone.

a pump to feed the solution into the membrane module. Surfactant solutions were prepared by dissolving a certain amount of surfactant in distilled water to achieve the desired concentrations. In each run of MEUF experiments, to prepare the feed solution, a specific volume of prepared TBBPA and surfactant solutions was mixed. Before filtration, approximately 2 L of feed solution were mixed for 3-4 hours at a suitable mixing rate of 100 rpm (without vortexing) at room temperature in the dark. All filtrations were carried out in the same test conditions (temperature, filtration time, and initial retentate pressure). At the beginning of each run, the permeability of the membrane was determined by measuring the distilled water flux under the same operating conditions used in the filtration. The permeate flux was measured during filtration at 5-minute intervals for one hour. Sample collection was also conducted simultaneously. Feed and retentate pressures were controlled during filtration and adjusted to the specified values if necessary. The pH of the solution was adjusted by adding the values of NaOH and HCl. After each run of MEUF, the membrane was washed with distilled water, sodium hydroxide, and distilled water, respectively.

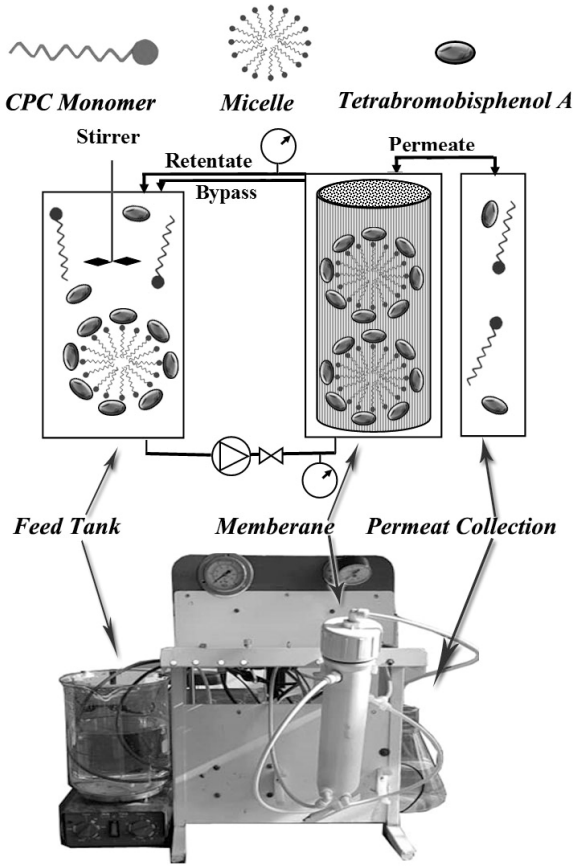

Figure 1. Schematic diagram of lab-scale cross-flow MEUF setup

Measurements and analyses

Feed solutions were always prepared at initial pH (9), except in pH-effect experiments that used specific values. The samples were taken at specified intervals and prepared for HPLC analysis. To ensure the accuracy of the experiments results, each sample was analyzed twice. TBBPA was detected by RP-HPLC apparatus, Smart line manager 5050 (Knauer), pump 1050, and UV detector 2520 equipped with Eurospher 100-5 C18 column (250×4.6 mm with precolumn). The elution was done with 5% water and 95% acetonitrile at a flow rate of 1 mL min^{-1}. The wavelength of the detector was set at 310 nm, and TBBPA removal efficiency was calculated using the following equation:

$$R = (1 - \frac{C_p}{C_f}) * 100 \quad (1)$$

Where R is the removal efficiency of TBBPA (%), Cp is the concentration of TBBPA in permeate (mg L^{-1}), and C_f is the concentration of TBBPA in feed (mg L^{-1}).

Results

Surfactant nature

Figure 2 shows the removal efficiency of TBBPA in the presence of nonionic surfactants (TX-100, Brij 35), cationic (CPC), and anionic (SDS), as well as the removal

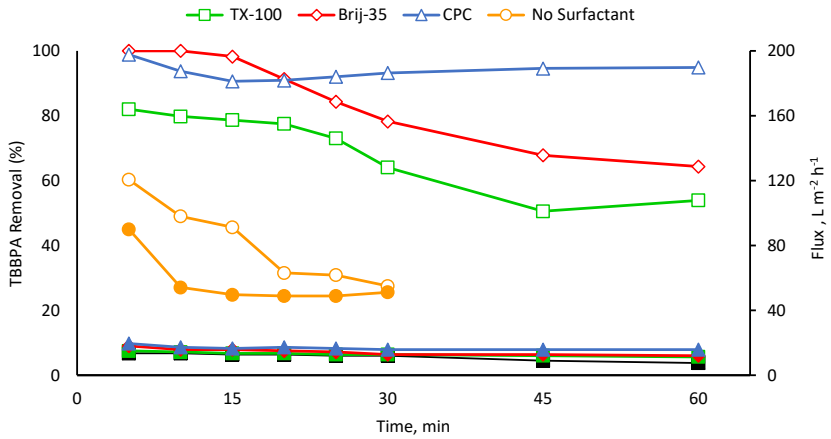

Figure 2. The removal efficiencies of TBBPA (no solid) and the permeate flux (fill solid) in different surfactants (TMP = 0.6 bar, surfactants concentration = 5*CMC, TBBPA concentration = 20 mg l^{-1}, pH = 9)

efficiency in the absence of a surfactant. The results indicated that CPC surfactant has higher efficient removal of TBBPA than others. Over time, the removal percentage of TBBPA only decreased 5.68% in the presence of CPC surfactant (from 98.86% to 93.18%) after 30 minutes of filtration, while in the absence of surfactant, the removal efficiency decreased from 60.30% to 29.55% at the same times. The removal of TBBPA with non-ionic surfactant TX-100 and Brij 35 was 82.02 and 100%, respectively, at the beginning of filtration. No removal of TBBPA was observed with SDS anionic surfactant. The flux was almost the same in all experiments with different surfactants, which decreased during filtration. However, the highest flux was observed for CPC surfactant.

Other influencing parameters in the MEUF process

Figure 3 shows changes in cationic surfactant concentration on TBBPA removal efficiency and permeate flux. TBBPA removal efficiency increased from 48.99% (without surfactant) to 99.10% by adding CPC surfactant (below CMC) after 10 minutes of filtration. At concentrations above CMC, the removal efficiency of TBBPA did not significantly change. In addition, adding surfactant decreased the permeate flux. Figure 4 shows the changes of TMP on the TBBPA removal efficiency and permeate flux. The percentage of TBBPA removal was similar in different TMPs (0.6, 0.8, 1, 1.2 bar) so that the percentage of TBBPA removal reached from 98.80 to 99.70 by changing the pressure from 0.6 to 1.2 bar. During filtration, the decrease in the TBBPA removal efficiency is more evident at high pressures than at low pressures. Figure 5 shows the changes in TBBPA removal efficiency at pH 5-11 during filtration. An increase in TBBPA removal efficiency was observed with increasing pH. The removal efficiency of TBBPA in the MEUF at different initial concentrations of TBBPA is shown in Figure 6. The results indicated minimal changes in TBBPA removal efficiency so that after 30 minutes of filtration, the removal efficiency reached from 99.40% at a concentration of 20 to 99.78% at a concentration of 80 mg L^{-1}. The chromatogram of the leached solution of WMPPCB (a), the sample spiked into the leached solution before (b) and after (c) the MEUF process is illustrated in Figure 7.

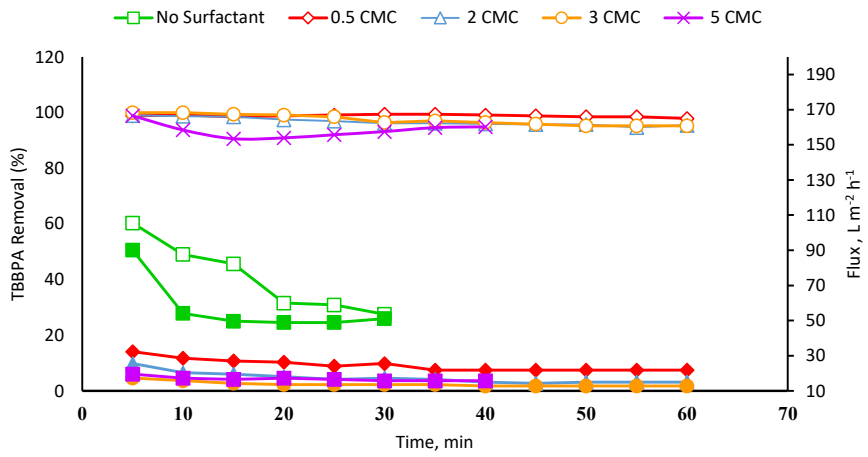

Figure 3. The removal efficiencies of TBBPA (no solid) and the permeate flux (fill solid) in CPC different concentrations (TMP = 0.6 bar, TBBPA concentration = 20 mg l^{-1}, pH = 9)

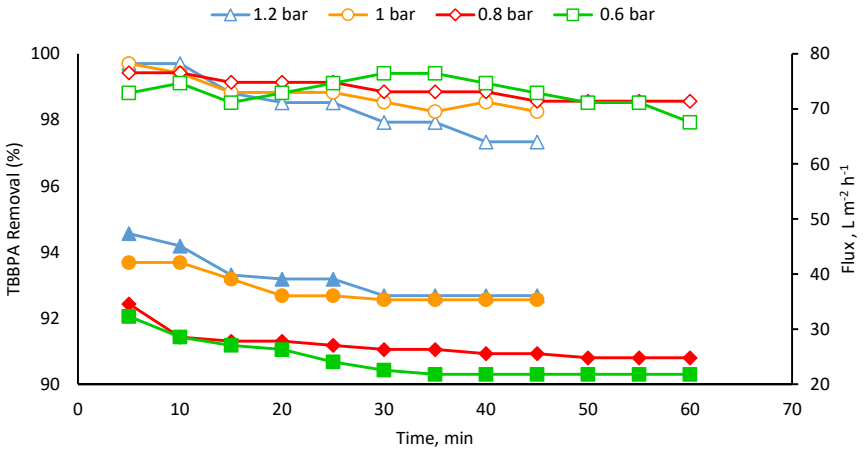

Figure 4. The removal efficiencies of TBBPA (no solid) and the permeate flux (fill solid) in different TMP_s (CPC concentration = 0.5 CMC, TBBPA concentration = 20 mg l^{-1}, pH = 9)

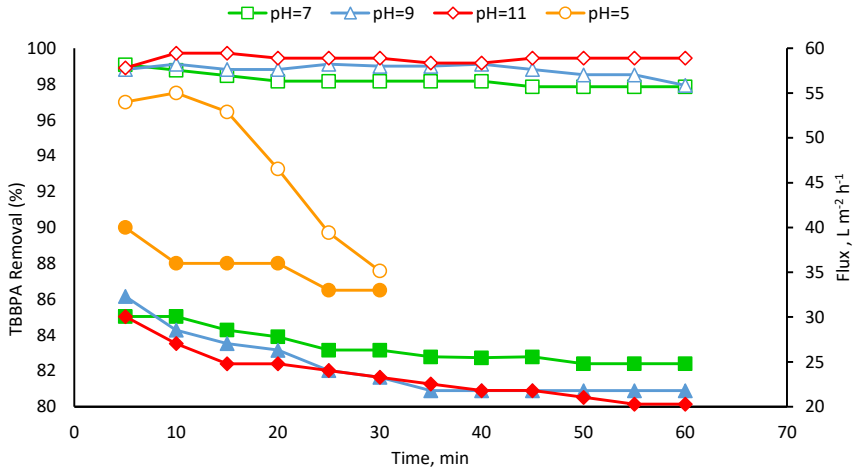

Figure 5. The removal efficiencies of TBBPA (no solid) and the permeate flux (fill solid) in different pHs (CPC concentration = 0.5 CMC, TBBPA concentration = 20 mg l^{-1}, TMP = 0.6 bar)

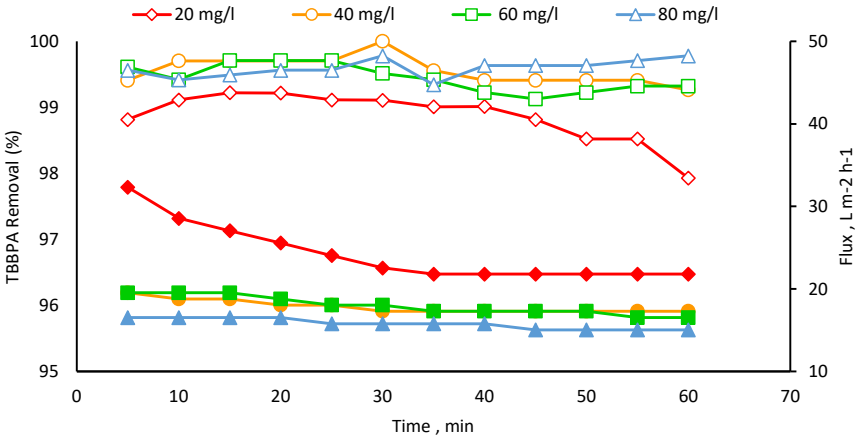

Figure 6. The removal efficiencies of TBBPA (no solid) and the permeate flux (fill solid) in TBBPA different concentrations (CPC concentration = 0.5 CMC, pH = 11, TMP = 0.6 bar)

Discussion

Effect of surfactant nature

As shown in Figure 2, CPC cationic surfactant had the highest efficiency in removing TBBPA during the experiment (98.86%). The removal efficiency decreased by only 4% after one hour of filtration. Since TBBPA is a hydrophobic and ionizable compound, it is converted into the anionic form of $TBBPA^{-}$ and $TBBPA^{2-}$ in alkaline pHs (31,32). Its interaction with the cationic group of pyridinium hydrophilic in CPC surfactant causes electrostatic interaction, which improves the removal efficiency of TBBPA with cationic surfactant. In most phenol MEUF research, CPC is commonly used to form micelles to dissolve phenol (24,33). The non-ionic surfactant cannot ionize to dissolve the TBBPA molecule, so the pollutant does not interact with the micelle through electrostatic forces at the surfactant head (24). However, the hydrophobic interaction force is responsible for trapping TBBPA in the micelle core. In addition, the polyether sulfone (PES) membrane is hydrophobic and has a high ability to absorb non-ionic surfactants/micelles, so the micelles containing TBBPA are removed by the membrane, and the removal efficiency increases. The pollutant removal efficiency decreased during filtration due to the phenomenon of concentration polarization near the membrane surface so that after one hour of filtration, the removal efficiency reached 53.93% and 63.35% for TX-100 and Brij 35, respectively. CPC surfactant was more efficient in removing TBBPA than TX-100 and Brij 35 non-ionic surfactants. This high efficiency can be caused by the difference in the interaction between the pollutant and the surfactant. The interaction of non-ionic surfactants with TBBPA is hydrophobic, but the interaction of CPC with pollutants is electrostatic attraction, which is much stronger than hydrophobic interactions. The removal efficiency of TBBPA was negative for SDS anionic surfactant, which is caused by the electrostatic repulsion between the anion of the surfactant and the anionic form of TBBPA. The flux is almost the same in all surfactants. The decrease in flux during filtration is due to the formation of a concentration polarization layer near the membrane surface. The flux reduction in the cross-flow mode reaches an almost constant value after some time (34). However, the highest

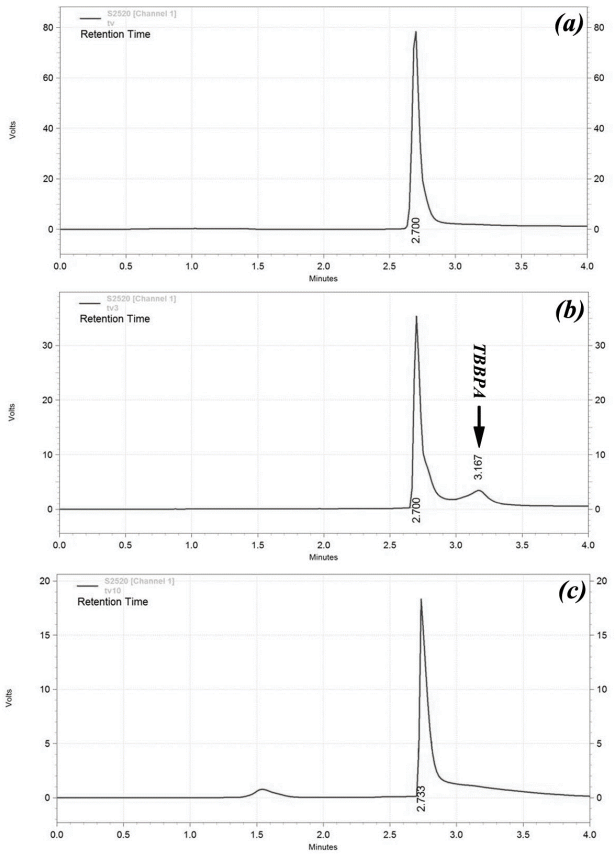

Figure 7. HPLC-UV chromatogram of TBBPA in samples: (a) real sample; (b) real sample spiked with 20 mg l^{-1} of TBBPA before the MEUF process; (c) real sample spiked with 20 mg l^{-1} of TBBPA after the MEUF process (TMP = 0.6, PH = 11, CPC concentration = 0.5 CMC)

flux was observed for CPC surfactant. The decrease in flux in non-ionic surfactants (Brij35 and TX-100) was more than the CPC surfactant, which is related to the nature of the surfactant and the interaction between micelle and membrane. The low solubility of hydrophobic surfactants in aqueous solutions leads to an increase in the viscosity of the micelle solution and, as a result, a decrease in flux (34). In addition, PES tends to absorb non-ionic surfactants, which causes a double decrease in flux compared to CPC surfactant. The decrease in flux during MEUF operation has also been observed by Víctor-Ortega et al, which was performed to recover phenolic compounds from wastewater with cationic surfactant (35). According to the results, CPC cationic surfactant was selected to remove TBBPA by the MEUF method.

Effect of surfactant concentration

The unexpected removal of TBBPA in the absence of surfactant can be attributed to the uptake of the pollutant by membrane at the beginning of the process. After one hour of filtration, the membrane becomes saturated due to surface absorption of the pollutant, causing an increase in the concentration in the permeate and a decrease in the removal efficiency. This result was also reported by Víctor-Ortega et al, who performed MEUF to recover phenolic compounds from wastewater (35). The removal efficiency of TBBPA increased significantly with the addition of CPC surfactant so that in the first minutes of filtration, the removal efficiency increased from 60.30% to 98.80%. This behavior can be attributed to the electrostatic interaction of the surfactant positive head groups with the TBBPA anionic forms, which causes the TBBPA to bind to the micelle and be rejected by the membrane. Thus, adding a surfactant to the solution reduces TBBPA concentration in the permeate and increases its removal efficiency compared to the absence of a surfactant. The reduction in removal efficiency in the presence of surfactant was negligible compared to the absence of surfactant during filtration because in the presence of surfactant, the free solutes on the membrane surface are much lower than that in the absence of surfactant (24). Although micelle formation is not possible at concentrations lower than CMC, in this study, the TBBPA removal efficiency was reported to be 98.80% by adding a surfactant at a concentration lower than CMC. The accumulation of surfactants in the membrane adjacent layer causes its concentration in this area to increase to the CMC level and forms the micelles. During filtration, TBBPA molecules are also absorbed on the surface and pores of the membrane. In addition, surfactants in aqueous solutions at concentrations close to or lower than CMC form pre-micelles to dissolve pollutant molecules (24,35). All these factors increase the removal efficiency of TBBPA due to the addition of surfactant to the solution. No significant change was observed in TBBPA removal efficiency in the above CMC value. This behavior can be explained in this way that the shape and number of micelle formation (the number of molecules present in the micelle in CMC) change with the increase of CPC concentration (36), but the number of binding sites of the pollutant to the micelle does not increase in the same proportion. In addition, increasing the surfactant concentration is not cost-effective from an environmental and economic point of view (37). Therefore, the concentration of 0.5 times the CMC of CPC was considered the optimal concentration. After one hour of filtration, the TBBPA removal efficiency decreased slightly in all tests related to the effect of surfactant concentration. This behavior is caused by micelle accumulation near the membrane surface. Convection transport of accumulated solutes increases the TBBPA concentration in the permeate and reduces its removal efficiency. Figure 3 shows the changes in permeate flux at different concentrations of CPC. The flux is decreased by adding a surfactant to the solution compared to the absence of a surfactant. Flux reduction increased with increasing surfactant concentration. It can be justified by the fact that increasing the concentration of surfactant forms more micelles and raises the thickness of the deposited layer on the membrane surface. Consequently, the membrane resists the solvent flux, which leads to a sharp decrease in the permeate flux compared to the permeate flux in the

test without surfactant. Similar behavior of flux reduction is observed during MEUF of phenolic compounds and MEUF of nitrite and nitrate using cationic micelles (35,38). Considering the removal efficiency, the concentration of surfactant, 0.5 CMC, was used to continue the experiments.

Effect of transmembrane pressure

Figure 4 shows that pressure changes do not affect the TBBPA removal efficiency. The only influential factor in filtration efficiency is micelle, pollutant, and membrane interactions. Since the pressure does not affect the interactions in the filtration process, it is not an influential factor in the removal efficiency. The decrease in pollutant removal efficiency at high pressures can be explained by mentioning that micelles cannot dissolve well at higher pressure levels due to the compression. Furthermore, increasing TMP improves the concentration of TBBPA transferred to the permeate stream leading to a slight decrease in TBBPA removal efficiency (39). A similar result was observed in a study that used the MEUF process to remove emerging pollutants from secondary wastewater (40). Figure 4 shows the increased practical driving force due to increased TMP caused more volumes transfer of stream through the membrane (flux from 32 to 47 L m^{-2} h^{-1} to change the pressure from 0.6 to 1.2 bar). Furthermore, the observed reduction in permeate flux can mainly be attributed to the concentration polarization near the membrane surface during the filtration. The reduction of the permeate flux is reported in olive oil wastewater treatment and in the removal of the emerging compounds from secondary effluent using MEUF (35,41). TMP 0.6 bar was preferred to continue the MEUF experiments due to the lack of influence of TMP changes on the efficiency of TBBPA removal and the reduction of energy consumption at low pressures.

Effect of pH

The solution pH is a critically influential factor in the dissolution of TBBPA in CPC surfactant micelles in the MEUF system because many phenolic compounds have weak acidic properties (12). Therefore, the optimum pH must be determined to achieve the highest surfactant efficiency in dissolving TBBPA. As TBBPA is a weak bifunctional acid, increasing the pH is effective on its acid-base balance and causes its more significant dissolution, which causes an increase in its removal efficiency at a higher pH, so that after 30 minutes of filtration, by changing the pH from 5 to 11, the removal rate increased from 87.58 to 99.45. In this research, the increase in pH caused a decrease in the permeate flux. pH 11 was chosen as the optimal pH.

Effect of initial concentration of TBBPA

The removal efficiency of TBBPA at different initial concentrations of pollutant is shown in Figure 6. TBBPA removal efficiency is not significant even after one hour of filtration. This finding may be justified by the fact that TBBPA is dissolved in the range of studied concentrations (20-80 mg l^{-1}) at the micelle-water interface by ionic interaction between the anionic forms of TBBPA and $C_5H_5N^+$. Also, it is dissolved in the hydrophilic head region CPC micelles by polar interaction. TBBPA may penetrate deeper layers and reach the micelles nucleus if these areas are saturated (33,34). Given that the removal efficiency remained high even after increasing the initial concentration of TBBPA, it can be concluded that these areas were not saturated with pollutant yet. Therefore, it can be concluded that in the concentration range of 20 to 80 mg L^{-1} of TBBPA, the removal efficiency is not affected by the initial concentration and the conditions for measuring adsorption isotherms are impossible.

Real sample analysis

The HPLC chromatogram of the real sample did not show the TBBPA peak. Therefore, the amount of 20 mg L^{-1} of TBBPA spiked into the real sample. The HPLC chromatogram (Figure 7) before and after the process showed that 20 mg L^{-1} TBBPA is removed by the MEUF method with 100% efficiency.

Conclusion

The present study investigated the removal efficiency of TBBPA in leached solution of WMPPCB by MEUF in the presence of various surfactants. The TBBPA removal efficiency can be arranged in the following surfactant order: CPC > TX-100 > Brij35 > SDS. In addition, the lowest removal efficiency of TBBPA was observed in surfactant-free conditions indicating the ability of TBBPA to be dissolved on surfactant micelles and retained by the ultrafiltration membrane. Since CPC surfactant has a higher solubility for TBBPA than other surfactants, it showed better removal efficiency during MEUF. The efficiency of several effective parameters in the MEUF process, including surfactant concentration, TMP, pH, and TBBPA concentration, was evaluated. Using different CPC concentrations (0.5, 2, 3, 5 CMC) showed an increase in the removal efficiency of TBBPA. The number of binding sites of the pollutant to the micelle does not change above CMC values; therefore, TBBPA removal efficiency did not change significantly with the increase in surfactant concentration. Although increasing TMP improved permeate flux, it did not affect TBBPA removal efficiency significantly. Increasing the pH in the range of 5 to 11 increased the efficiency of TBBPA removal due to the ionization of TBBPA and increase in the TBBPA solubility in the micelles. Although the initial concentration of TBBPA increased, the removal efficiency remained high due to the unsaturation of the empty sites in the formed micelles. The HPLC-UV analysis of the real sample indicated a very high removal efficiency of TBBPA. Thus,

ultrafiltration using CPC can be identified as an efficient technology to remove TBBPA from the leached solution of WMPPCB.

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Ethical issues

The authors declare that there are no ethical issues.

Competing interests

The authors declare that there is no conflict of interests.

Authors' contribution

Conceptualization: Fatemeh Sahlabadi, Akbar Eslami.
Data curation: Nadali Alavi.
Formal Analysis: Mohsen Sadani.
Funding acquisition: Akbar Eslami.
Investigation: Nadali Alavi, Mohsen Sadani, Marzieh Torabbeigi, Mahdokht Arshadi.
Methodology: Marzieh Torabbeigi.
Project administration: Akbar Eslami.
Resources: Mahdokht Arshadi, Marzieh Torabbeigi, Nadali Alavi, Mohsen Sadani.
Supervision: Fatemeh Sahlabadi, Akbar Eslami.
Validation: Mohsen Sadani, Marzieh Torabbeigi.
Visualization: Nadali Alavi.
Writing–original draft: Akbar Eslami.
Writing–review & editing: Mahdokht Arshadi, Nadali Alavi, Mohsen Sadani, Marzieh Torabbeigi.

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