Effect of Laminar Pulsatile Fluid Flow on Separation of Volatile Organic Compounds from Aqueous Solution by a Hollow Fiber Membrane-Based Process

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ABSTRACT: *In this study, a laminar pulsatile fluid flow was used for the separation of benzene, toluene, ethylbenzene, and xylene isomers (BTEX) from aqueous solutions. Polyether sulfone hollow fiber membrane has been applied to this process. The effects of BTEX concentration, and feed and extraction flow rates were examined. It was found that the application of the pulsatile fluid flow with the frequency of 0.5 Hz improved the separation process significantly, and the removal efficiency increased more than twice. Moreover, the results showed that BTEX separation under pulsatile fluid flow was affected by the feed flow rate, extraction flow rate, and the BTEX concentration, as well.*

KEYWORDS: *Pulsatile fluid flow; Polyether sulfone; Ultrafiltration; BTEX; Membrane separation.*

INTRODUCTION

BTEX, which stands for benzene, toluene, ethylbenzene, and xylenes, naturally exists in crude oil [1, 2]. These components have many diverse applications in food and other industries [3]. Benzene is detected in many soft drinks, synthetic rubber, nylons, and detergents [4]. Toluene is commonly used in petroleum household products such as paints, nail polish, and gums. Ethylbenzene is found in paintings, solvents, and pesticides, and is used as an additive in fuels. Xylene is used as a solvent in the printing and leather industries. Therefore, these compounds are important in various industries[5].

On the other hand, BTEX is known as an environmental pollutant, and it can be found in many places such as chemical and petrochemical wastewaters and ground

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waters due to car exhaust, petrochemical processes, and leakage in transportation instruments [6]. Contacting with BTEX may occur by inhalation (breathing polluted air), ingestion (drinking polluted water), or absorption through the skin [7]. In this regard, these compounds have many harmful effects on human health [8]. It has been shown that many epidemic and dangerous diseases may be caused by exposure to benzene [4]. However, the other aforementioned components are not known as hazardous as benzene, but they are highly toxic materials [9]. According to US EPA guidelines, the permissible amount of benzene, toluene, ethylbenzene, and xylenes in drinking water are 5, 1000,700, and 10000 µg/L, respectively [10].

The removal of these compounds can be done with widely accepted techniques [11]. Conventional techniques such as adsorption, advanced oxidation, and chemical treatment are ineffective, so an alternative method is needed [12]. Membrane extraction techniques have attracted much attention, and they are used for the separation of volatile organic compounds from water [13]. Membrane technology has become a very useful and significant technology in many industries [14]. The major advantages of the membrane process are: (1) this process is needless of additives; (2) it has a low energy consumption; (3) it is easy to be used [15]; (4) it has fully customizable operating conditions; (5) it can be easily used as a fully automated process [16].

Various membrane materials were examined for the separation of toxic compounds from water [17]. Using polymeric membranes to separate components, suspended solids [18], and scattered oils [19] has many advantages such as high separation efficiency, small separation unit size, and low energy consumption [20]. Furthermore, polymeric membranes are cheaper than other types of membranes for example ceramic membranes [21].

The main challenge in front of membranes is fouling which adversely affects membranes' efficiency [22–24]. Fouling includes adsorption and desorption of particles to membrane pores and surfaces. On the other hand, by passing the time particles tend to stick to the membrane surface and pores which will reduce the permeate flux through the membrane [25]. Two important factors for assessing membranes' effectivity are the quality of permeate and the productivity of the membrane. Fouling will exacerbate the productivity of permeate. In addition, it reduces the quality of permeate water [17]. Therefore, finding a method to postpone fouling is really important. In addition, increasing the fouling layer will cause more energy consumption in order to push water through the membrane [26]. Regarding this problem, finding the proper method will reduce energy consumption significantly. The combination of methods for the treatment of various waters and wastewaters is a promising technique, especially for wastewaters that contain petroleum products. Although the different combinations of physical, chemical, biomedical methods or photocatalytic membrane reactors are used for the treatment before a membrane filtration unit, these methods were not effective enough to postpone fouling and increase the productivity of the membrane [22, 23].

The innovation of a low-cost process for the effective isolation of toxic materials is important. As mentioned before, the most important obstacle in the membrane process is particle deposition and concentration polarization [29] which is significant for laminar flow processes [30] and it is really important to find a solution. In this regard, using pulsatile fluid flow instead of continuous flow can increase mass transfer, uniformity of mass distribution [31], and decrease the concentration polarization in the membrane processes.

In this study, the effect of feed and extract phases' flow rate, pulsatile fluid flow, and feed concentration on the separation efficiency of BTEX was investigated. In this process, the membrane process was used to overcome the aforementioned obstacles. In addition, GC-FID was used for tracking the number of analytes that were extracted. The effect of pulsatile flow rate in terms of amplitude, frequency, and BTEX concentration in aqueous samples was studied, as well.

EXPERIMENTAL SECTION

Chemicals and instruments

Six standard compounds (benzene, toluene, ethylbenzene, m-xylene, p-xylene, and o-xylene) and methanol were purchased from Fluka (Sleeze, Switzerland). *n*-hexane was from Sigma-Aldrich (St. Louis, MI, USA). Tubular Polyether sulfone ultrafiltration membrane was purchased from Spectrum Laboratories, Inc. (Rancho Dominguez, CA) and used for the separation of the analytes. The fibers had an Inner Diameter (ID) of 500 µm and an Outer Diameter (OD) of 1000 µm. A Varian Star 3400CX gas chromatograph (Varian Inc., CA, USA) equipped with a flame ionization was used for chromatography.

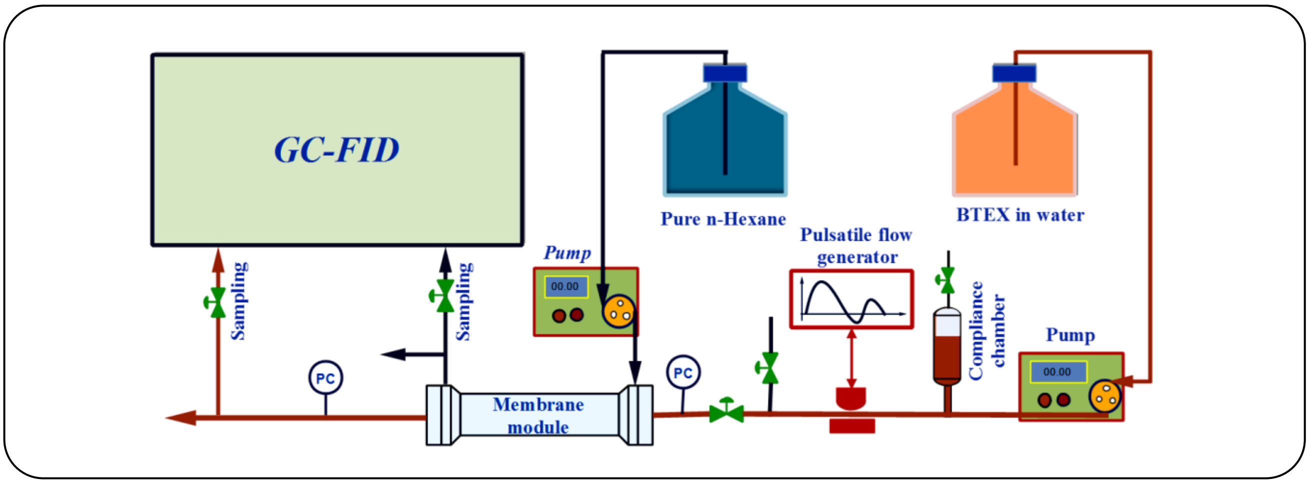


Fig. 1: Schematic of the experimental setup.

Membrane extraction

The stock solution of BTEX (100 mg/L) was prepared by weighing proper amounts of each analyte in methanol and stored in a fridge. The sample solutions for the experiments were prepared from the stock solution daily. The schematic of the experimental setup is shown in Fig. 1. Two phases were separated by Polyether sulfone (PES) membrane and *n*-hexane was used as extraction solvent. The flow rates of the two phases were controlled by two peristaltic pumps (SNJ, Isfahan, Iran). The feed and extraction phases' flow rate was calculated using Eq.$Q=\bar{V}_z.Az$ ()

$$Q = \bar{V}_z . Az \quad (1)$$

In this equation, Q shows fluid flow rate, $\bar{V}_z$ is the average velocity through the area (A) in flow direction and A_z is the cross-sectional area in which the fluid is passing [12]. A pulse generator was used to induce pulses to the feed flow with different frequencies (0-1 Hz). BTEX samples were taken every three minutes and the samples were kept in 10 mL vials. After completing the separation process, samples of outlet extraction and feed phase (raffinate), which contain BTEX, were obtained. Two milliliters of each sample were analyzed by GC-FID. Separate four-point calibration curves were drowned for benzene, toluene, ethylbenzene, and xylene isomers. The concentration of each component was calculated by these linear curves and by the area of each peak. Each peak was observed due to the amount of each component in the outlet solutions.

Analyses

All experiments were repeated at least three times and the average values of them have been reported. The standard deviation values of the measurements were small enough, so they are not included in the figures.

Theoretical analysis

Sample analysis was performed in our lab, using GC-FID. The analysis method was according to US EPA TO-17 (RL 0.5 ppbv).

RESULTS AND DISCUSSION

In this study, the effect of the concentration of BTEX in feed water samples, pulsatile fluid flow, and feed and extract flow rates were examined. The membrane process was coupled with GC-FID to determine the removal efficiency and effect of pulsatile fluid flow on the separation process. In all figures, the left-hand figures show the effect of parameters on the separation of BTEX from water, and the right-hand ones are for concentration variation of BTEX in the extraction phase.

Effect of frequency of the pulsatile fluid flow

The effect of steady and pulsatile fluid flow with different frequencies on the separation of volatile organic compounds from water is shown in Fig. 2. The initial concentration of BTEX in water was set at 250 µg/L. The flow rates of the feed and extraction phases were 1 mL/min, respectively. As can be seen, the pulsatile fluid flow had a significant effect on the removal efficiency when compared to steady fluid flow (0 Hz in Fig. 2). Generally, it can be said that the efficiency of experiments with the pulsatile flow was higher than the steady flow. By passing the time the removal ability of membrane and pulsatile flow

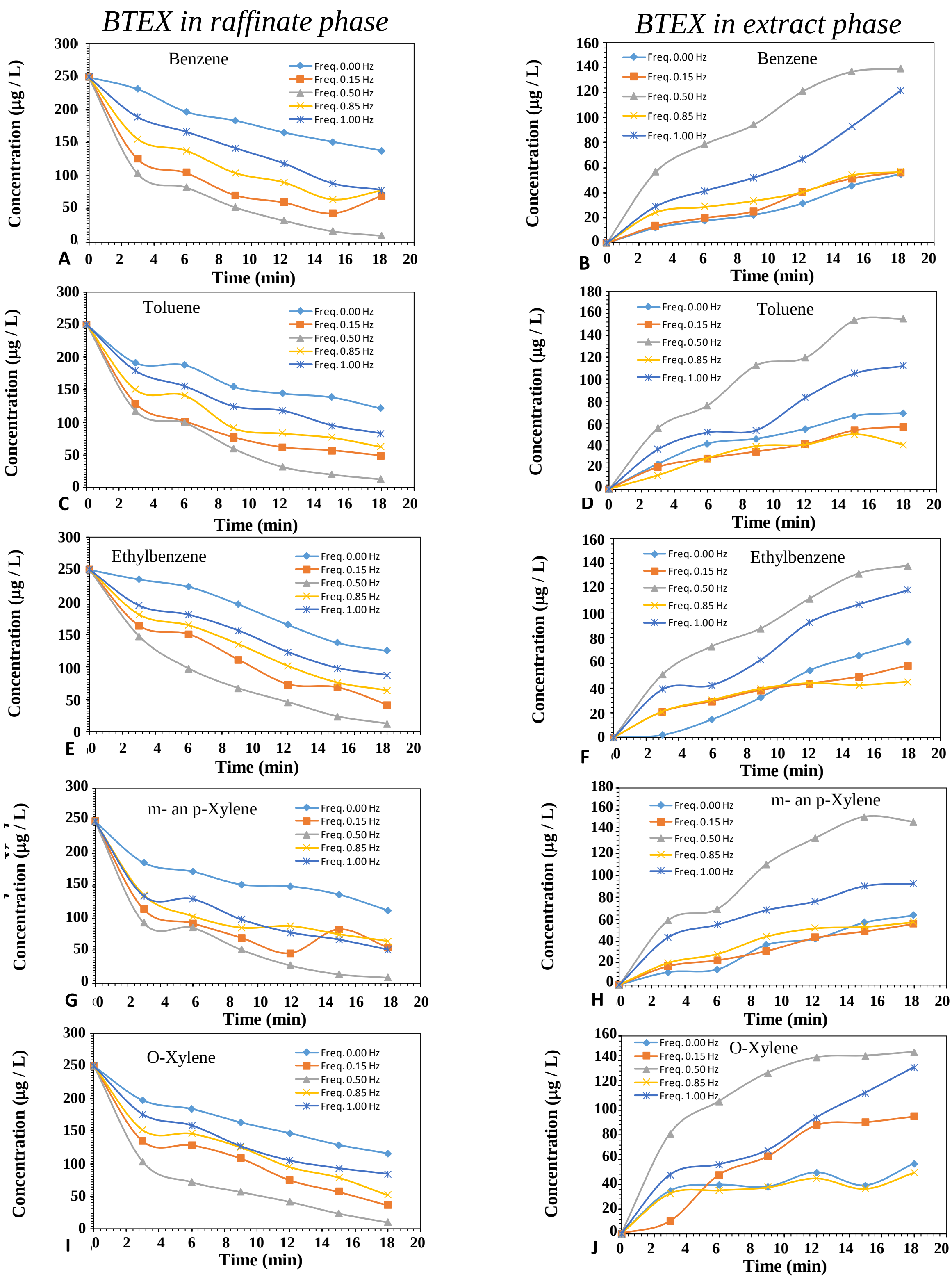


Fig. 2: Effect of pulse frequency on separation of Benzene (A, B), Toluene (C, D), Ethylbenzene (E, F), m- and p-Xylene (G, H), and o-Xylene (I, J) from water to the extract phase, BTEX concentration in feed=250 µg/L, feed flow rate=1 mL/min, the extract flow rate was I mL/min.

pulsatile flow in the improvement of water quality, as well. By looking at the charts for the extract part it can be said that the concentration of BTEX in the extract phase was higher for pulsatile flow than a steady flow. The comparison of concentration values of extract and raffinate phases showed that the system followed the mass transfer law. This means that the summation of outlet concentrations was almost equal to inlet concentration. The small difference between inlet and outlet concentrations was caused by particles that stuck to the tubes and membrane. Another point that is observed in Fig. 2 is that the highest removal efficiency was observed at the beginning of the experiment and the removal efficiency was decreasing during the experiment.

Figs. 3 and 4 show the effect of pulsatile fluid flow with different frequencies on the removal efficiency of benzene, toluene, ethylbenzene, m- and p-xylene, and o-xylene from the water at t=18 min. BTEX concentration in the feed, feed flow rate and extract flow rate were set at 250 µg/L, 1 mL/min, and 1 mL/min, respectively. By increasing the frequency of pulses up to 0.5 Hz, the removal efficiency increased significantly. It should be pointed out that removal efficiency was calculated using Eq. (2)

$$R(\%) = \frac{C0 - Ci}{C0} \quad (2)$$

In this equation, *C0* and *Ci* are the initial concentration and pollutant concentration which were measured every 3 min [32].

As is shown in Fig. 3 and 4, using pulsatile flow removal efficiency increased in comparison to continuous flow. The maximum removal efficiency of BTEX (96%) occurred at 0.5 Hz and then a further increase in frequency led to lower removal efficiency of BTEX in comparison to a frequency of 0.5 Hz. The reduction in removal efficiency after a frequency of 0.5 Hz might be because of transmembrane pressure variation during the experiments or the effect of the concentration polarization layer but evaluation of these parameters was not considered in this set of experiments.

The concentration polarization on the membrane surface tended to decrease the permeation rate of the most permeable components (BTEX). It also increased the permeation rate of the less permeable component (i.e., water in this study), resulting in lesser removal efficiency of the separation process. However, an increase in feed flow pulsation frequency up to 0.5 Hz reduced the effect

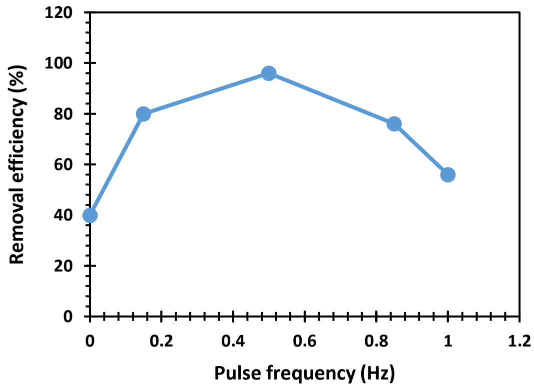


Fig. 3: Removal efficiency comparison between experiments with various pulse frequencies between 0 to 1 Hz

of concentration polarization due to variable shear force on the membrane surface so that the BTEX flux should increase as observed in Fig. 4. Shear force on the membrane surface is explained using Eq. (3)

$$T = \mu \frac{\delta u}{\delta x} \quad (3)$$

In this Equation, *u* shows the velocity of the fluid in the x-direction, and *µ* shows kinematic viscosity [33]. However, no shear force quantification was done in this study. After increasing the BTEX flux, the frequency reduced the removal efficiency of BTEX from water. It can be said that water flux, which is controlled by the rate of diffusion through the membrane, should be independent of the pulsatile flow frequency. Fig. 4 is a clearer schema that shows the ability of pulsatile flow with various pulse frequencies to separate each volatile compound from water. The error bar for each value is presented, as well. It should be pointed out that in general, the pulsatile flow had higher performance in comparison to a steady flow. Removal efficiency for pulse frequency of 0.85 and 1 Hz are almost the same and moderately 65% which increased to 96% for pulse frequency of 0.5 Hz. The lowest removal efficiency was in steady flow which was around 50%. According to *Zhang et al.* [34] by increasing pulse frequency the energy consumption will increase. However, one interesting result from this plot can be that by increasing pulse frequency from 0.5 to 1 Hz the removal efficiency was decreased and energy consumption

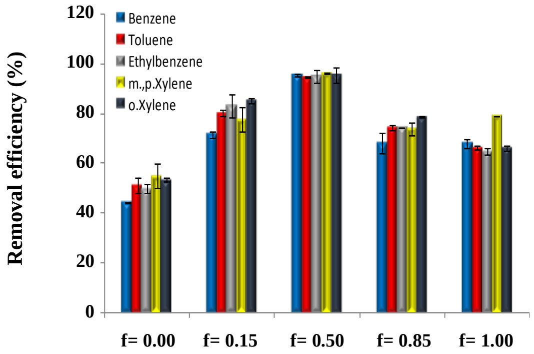


Fig. 4: Effect of pulsatile fluid flow with different frequencies on the removal efficiency of Benzene, Toluene, Ethylbenzene, m- and p-Xylene, and o-Xylene from water at the time of 18 min, BTEX concentration in feed=250 µg/L, feed flow rate=1 mL/min, extract flow rate=1 mL/min.

Effect of feed flow rate

As is shown in Fig. 4, feed flow rate had a significant effect on BTEX removal efficiency. The examination of the feed flow rate was done at a constant frequency of 0.5 Hz, which had the best separation efficiency. The extraction phase flow rate was set at 1 mL/min. Feed concentration was set 250 µg/L, and the feed flow rate was examined at three levels. It is clear that a flow rate of 1.35 mL/min is the most effective feed flow rate. In addition, an increase in the feed flow rate could also reduce the negative effect of concentration polarization. This means that BTEX flux should increase and water flux, controlled by mass diffusion rate through the hollow membrane. Mass diffusion through the membrane is explained using Fick's first law (Equation 4)

$$Ji = -DA\frac{dC}{dx} \qquad (4)$$

In this equation D indicates diffusion coefficient, A is the active area of the membrane, C shows concentration and dx is the indicator of membrane thickness [31]. Furthermore, flux through the membrane can be calculated using the following equation:

$$Flux = \frac{V}{At} \qquad (5)$$

was increased. Therefore, a pulse frequency of 0.5 Hz is the optimal operational condition according to the efficiency and energy consumption.

In equation 5, V represents permeate water volume, t indicates time and A is the active area of the membrane [31]. It has been shown that mass diffusion through the membrane should be also independent of the feed flow rate. Fig. 5 shows the influence of feed flow rate on the separation of BTEX/water at an effective frequency for a constant feed concentration. As a result, the BTEX/water separation increases as the feed flow rate increases as shown in Figs. 5A to J. The same results were included in the study carried out by Yahaya. He concluded that by increasing the feed flow rate from 0.1 to 1.25 m/s total flux will increase significantly [35].

Effect of extraction phase flow rate

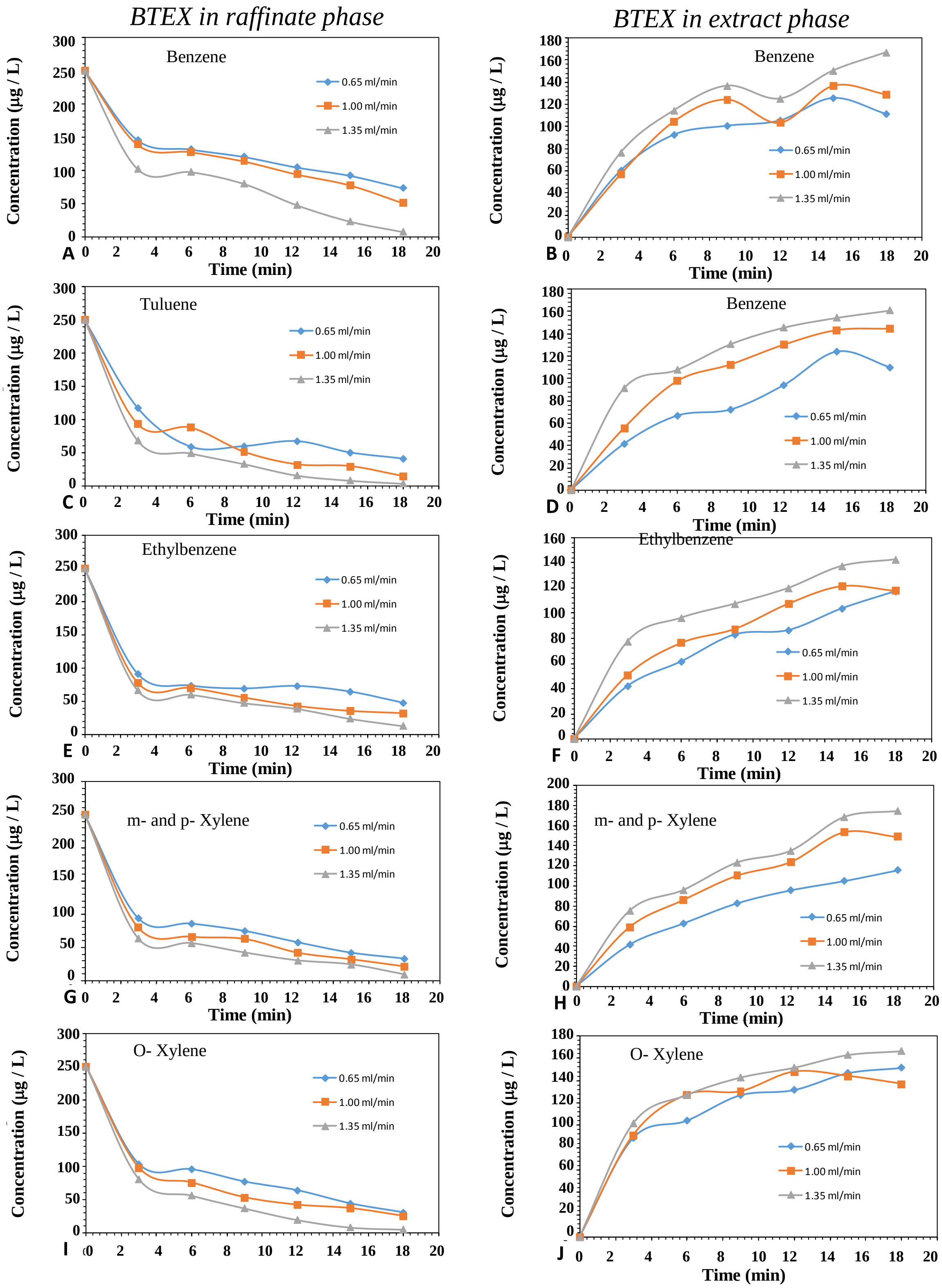
The flow rate of the extraction phase was examined at three levels. The frequency of pulses, feed concentration, and feed flow rate were set 0.5 Hz, 250 µg/L, and 1 $mL.min^{-1}$, respectively. As displayed in Fig. 6, the flow rate of the extraction phase didn't show a significant effect on the separation process, especially for the benzene. As observed, the data are very close even if the flow rate of the extraction phase was in the range of (0.65-1.35) $mL.min^{-1}$.

Effect of feed concentration

The study of BTEX concentration in water was done to investigate how the concentration affects mass transfer operation in the membrane module. Pulses' frequency and flow rates of feed and extraction phases were set 0.5 Hz, 1 mL/min, and 1 mL/min, respectively. As shown in Fig. 7, mass transfer increased by increasing the inlet concentration of BTEX in water. This behavior can be explained easily using equation 4. One of the driving forces for mass transfer through the membrane is concentration difference. By increasing the concentration difference between the two phases mass transfer increased. The same result was concluded in other studies as well [35].

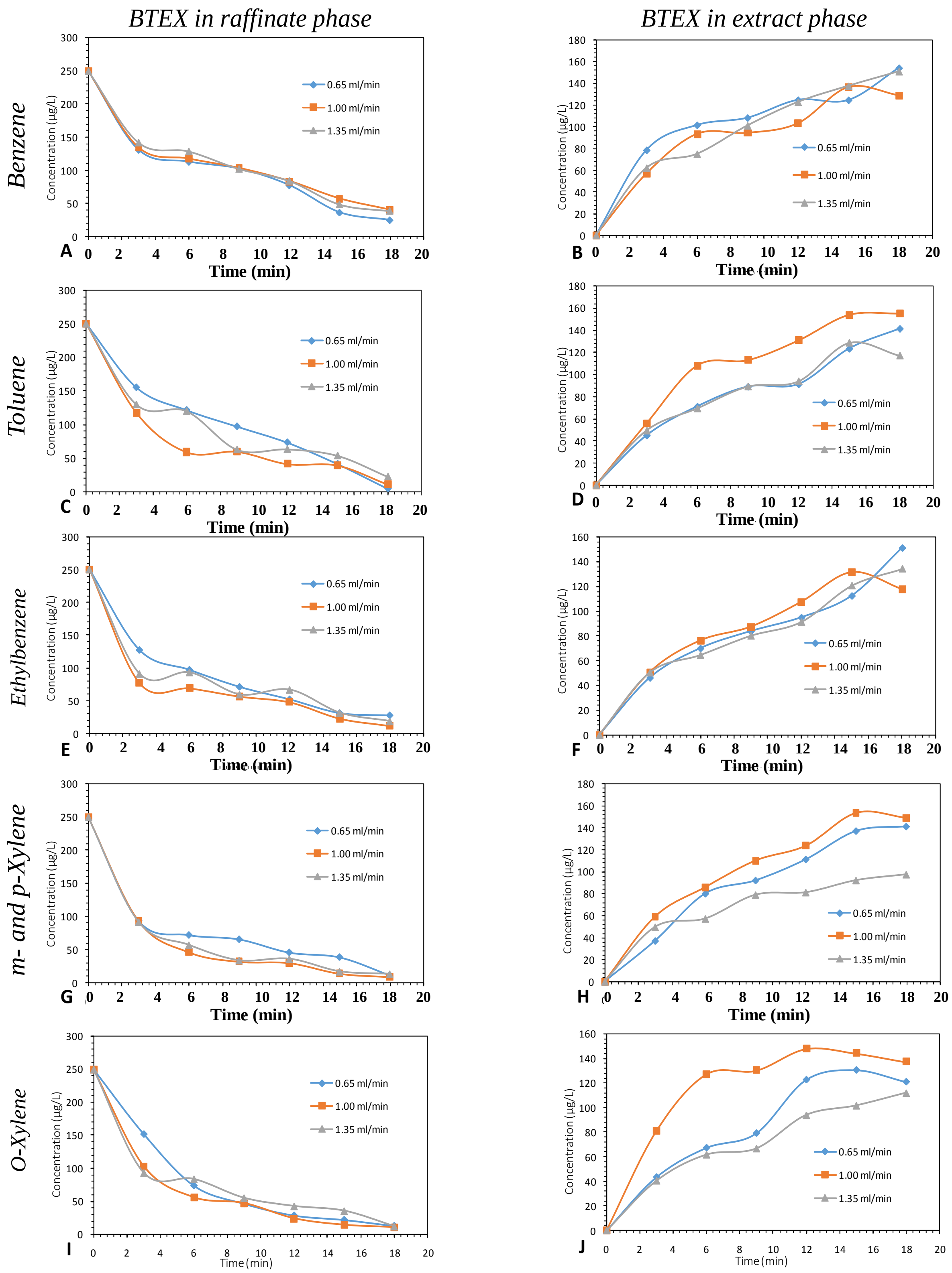
DISCUSSIONS

The pulsatile flow was used to increase the efficiency of the ultrafiltration membrane process and we have seen that adding pulses to the flow affected the process significantly. Membrane separation is an efficient method for improving water quality. As mentioned before, the main problem in membranes is fouling which exacerbates membrane productivity. For the aim of postponing and preventing concentration polarization in membranes, pulsatile fluid flow was used. By increasing the frequency of the applied pulse up to 0.5 Hz, the removal efficiency increased nearly twice which



Time (min)

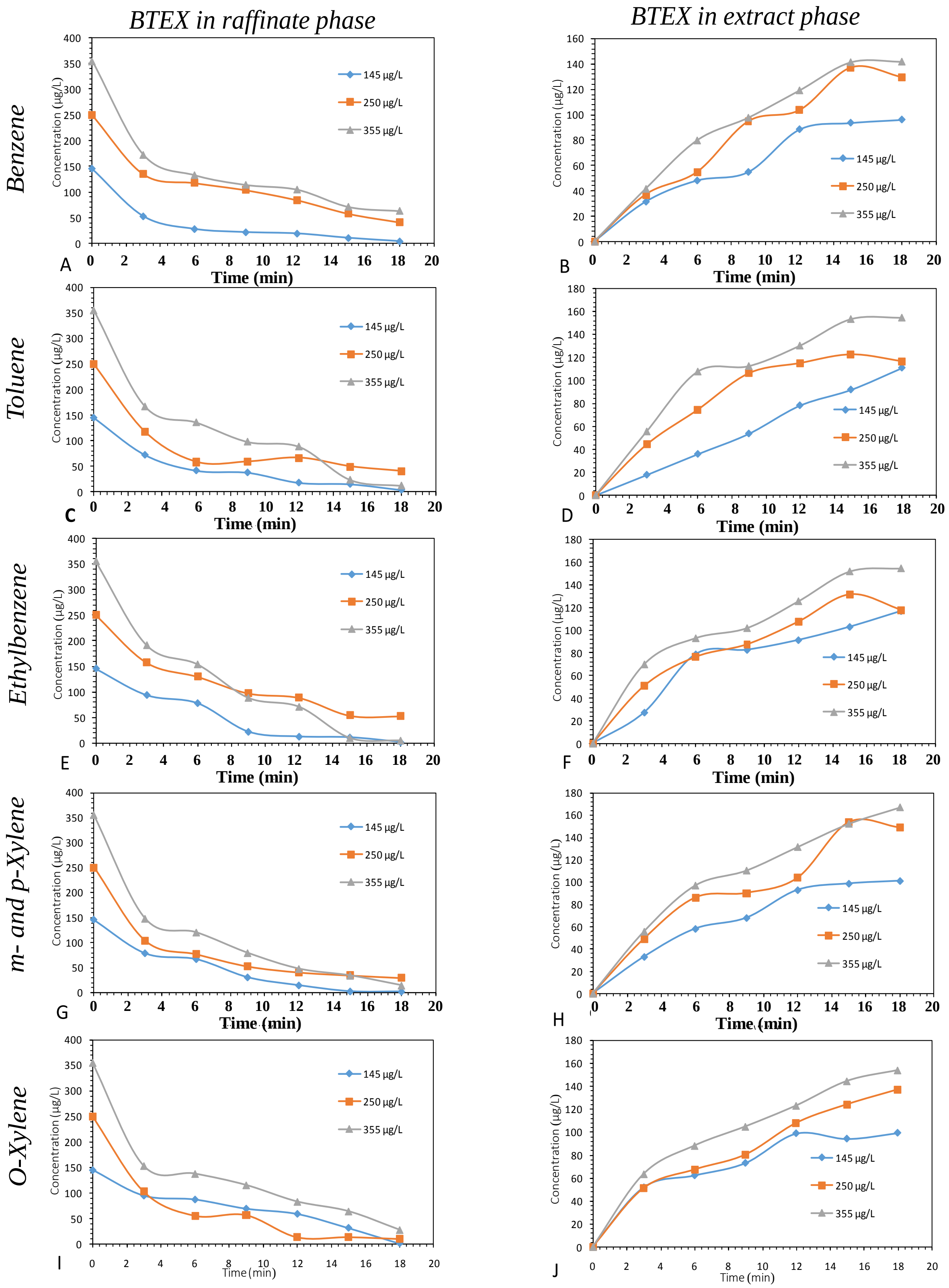
Fig. 5: Effect of feed flow rate on the separation of Benzene (A, B), Toluene (C, D), Ethylbenzene (E, F), m- and p-Xylene (G, H) and o-Xylene (I, J), f=0.5 Hz, BTEX concentration in feed=250 μg/L and extract flow rate=1 mL/min.



0 2 4 6 8 10 12 14 16 18 20
Time (min)

0 2 4 6 8 10 12 14 16 18 20
Time (min)

Fig. 6: Effect of extract flow rate on the separation of the benzene (A, B), toluene (C, D), ethylbenzene (E, F), m- and p-Xylene (G, H) and o-Xylene (I, J), f= 0.5 Hz, BTEX concentration in feed=250 µg/L and feed flow rate=1 mL/min.



0 2 4 6 8 10 12 14 16 18 20
Time (min)

0 2 4 6 8 10 12 14 16 18 20
Time (min)

Fig. 7: Effect of the concentration of BTEX on the separation of benzene (A, B), toluene (C, D), ethylbenzene (E, F), m- and p-Xylene (G, H) and o-Xylene (I, J), f= 0.5 Hz, feed flow rate=1 mL/min and extract flow rate=1 mL/min.

showed the significant effect of pulsatile flow on improving the efficiency of the membrane process and postponing fouling. These results are much better than Yahaya's observation in regard to process efficiency in which a combination of membrane process and pervaporation was used for BTEX removal [35]. Unsteady flow which was created by flow pulsation caused thinning of the boundary layer on the membrane surface.

Therefore, it can be said that pulsatile flow improved the mixing process by disturbing flow patterns and particles were swept to the bulk flow, and largely prevented phenomena like concentration polarization. Using pulsatile flow, the boundary layer on the membrane surface was thinner which resulted in reducing the boundary layer effect and in general, improving mass transfer and concentration distribution uniformity in the membrane module [36]. The best removal efficiency occurred at 0.5 Hz. Then a further increase in the frequency led to a lower removal efficiency which might have had different reasons like the transmembrane pressure (TMP) effect. On the other hand, it should be pointed out that further increase in pulse frequency unexpectedly did not always increase the efficiency and sometimes acted against the flow [37].

According to the applied Reynolds number (Re=22.05), the flow was laminar and the concentration polarization phenomenon is common in the laminar flow near the surface of the membrane. Reynolds number was calculated using Eq. (6):

$$Re = \frac{\rho V d}{\mu} \tag{6}$$

In this Equation, ρ and μ are the fluid density and viscosity and V indicates the fluid velocity and d is the diameter of the tube [38]. The concentration polarization may have decreased the permeation of the high permeative components which caused a lower removal efficiency. By increasing the feed flow rate, higher shear stress on the membrane surface was expected which increased the mass transfer coefficient by disrupting the mass boundary layer and may have decreased the concentration polarization. It has been shown that BTEX flux through the membrane is controlled by the mass transfer coefficient of the boundary layer in the feed phase. Therefore, the flow rate of the extraction phase was not the main parameter in the separation of BTEX from the water. Mass transfer increased by increasing the concentration of BTEX in water. Based on the above explanation, the mass transfer operation in the membrane can be attributed to the concentration driving force between two sides of the membrane. In addition, higher feed flow velocities resulted in higher removal efficiency. However, the membrane process is mainly a compromise between efficiency and energy consumption which was not the main focus of this study but it is obvious that a higher feed flow rate needs higher energy consumption.

CONCLUSIONS

An alternative method was used for the separation of BTEX from polluted water. The conventional membrane processes suffer from fouling which happened so fast, especially when the feed samples had a high concentration of pollutants the efficiency and productivity of the membrane process declined significantly. To overcome the problems which arise from particle deposition and concentration polarization on the membrane, the pulsatile flow, and PES ultrafiltration membrane system were combined. The effect of pulsatile fluid flow with different frequencies was studied and observed in a membrane system. In addition, the effect of feed concentration, and feed and extract phases flow rate was investigated. In comparison to the conventional UF process, a combination of pulsatile fluid flow and ultrafiltration membrane had a significant impact on the removal efficiency of BTEX from the water. And this performance enhancement was more and more by increasing pulse frequency to 0.5 Hz and by increasing feed flow rate from 0.65 to 1.35 mL/min. It should be mentioned that increasing pulse frequency from 0 to 0.5 Hz increased removal efficiency from 40 to 96% and increasing flow rate from 0.65 to 1.35 mL/min increased the efficiency from 56 to 95% on average. Moreover, BTEX flux increased with increasing the flow rate of the feed. It's due to the enhancement of shear force on the surface of the membrane. An increase in the flow rate of extract had no effect on the removal efficiency. Enrichment of BTEX in the extraction phase increased by increasing feed concentration due to a concentration driving force between two sides of the membrane. As observed, pulsatile fluid flow can be simply used in waste water treatment industries. In addition, this process is simple, it can save time, and it needs only a small quantity of solvents. Since the application of the pulsatile flow is novel in this study, further study is needed for a mechanistic understanding of the phenomenon.

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