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Application of pseudo-emulsion hollow fiber strip dispersion system for the removal of propylparaben from the aqueous solutions

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ABSTRACT

Propylparaben (PP) is one of the most common alkyl esters of *p*-hydroxybenzoic acid, used as preservatives and microbicides in the foods, beverages, pharmaceuticals and cosmetic industries. In the present work, propylparaben was removed from the aqueous solutions by using continuous pseudo emulsion hollow fiber strip dispersion system (PEHFSD) in which *n*-heptane with different carriers (trioctyl amine (TOA), tributyl phosphate (TBP) and trioctylmethylammonium chloride (Aliquat 336)) was used as a membrane phase within the pores of hollow fiber module and NaOH, Na_2CO_3 or NaCl were used as stripping solution. Effect of various parameters, like pH of feed phase, type and concentration of carriers in membrane phase, type and concentration of stripping solution, initial feed concentration on PP extraction were studied. Using the conditions giving maximum extraction of PP within the investigated levels and range, almost complete (100%) removal of PP was achieved by using PEHFSD system. The mass transfer model reported in the previous literatures confirmed its validity with the experimental results. Hence, PEHFSD system represents a highly efficient advanced separation technique for the removal of PP from aqueous solutions.

Keywords: Propylparaben; Aliquat 336; PEHFSD system

1. Introduction

Propylparaben (PP), alkyl esters of *p*-hydroxybenzoic acid, is commonly used preservatives and microbicides in the foods, beverages, pharmaceuticals and cosmetic industries. All parabens are categorized as endocrine disrupting compounds as they show endocrine activity. They also have been found in human tissues, including breast tumours. The lowest observed effect concentration of parabens on river water for the growth of aquatic life was found to be within the range of 0.12 to 9.0 mg/L [1]. Recently, it is found that parabens are frequently released in urban wastewater comparatively at high concentrations and, even with considerable removal of them using conventional treatment methods; they have been still identified in river water samples [2]. Hence, the U.S. Environmental Protection Agency (EPA) has considered these compounds as emerging environmental pollutants [3].

To reduce unwanted buildup of parabens in aquatic environments, development of efficient separation techniques is required for the extraction of these compounds from water. Separation with liquid membranes is one of the advanced extraction processes for the removal of contaminants present in waste water [4]. Chin et al. studied the removal of four parabens, methyl-, ethyl-, propyl-, and benzyl-paraben, by β-cyclodextrin (β-CD) polymer from aqueous solution using two different cross-linkers such as β-CD-HMDI (hexamethylene diisocyanate) and β-CD-TDI (toluene-2,6-diisocyanate). Role of both cross-linker in adsorption, hydrophobicity of polymers, and adsorption capacity of different parabens were compared and discussed [5]. Gonzalez et al. performed an assess-

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ment of the sewage occurrence and biodegradability of seven parabens and three halogenated derivatives of methylparaben (MeP). Stability test performed with raw wastewater showed that parabens degrade rapidly in real sewage, with half-lives lower than 10 h for *n*-butylparaben, while dihalogenated species again turned out to be more stable, with half-lives longer than a week [6]. Ara et al. developed SBA-15/polyaniline *para*-toluenesulfonic acid nanocomposite supported micro-solid-phase extraction procedure for the extraction of parabens (methylparaben, ethylparaben, and propylparaben) from wastewater and cosmetic products. pH of sample, sample and eluent volumes, sorbent amount, salting-out effect, extraction and desorption time, and stirring rate was found to be important parameters for the fast and efficient extraction of parabens [7]. Liquid membranes (LMs) technique provides high interfacial area for mass transfer and has the ability of simultaneous removal and extraction of the target solute, and the requirement of only small quantities of organic solvent [8]. Recently, Sarah Chaouchi and Oualid Hamdaoui investigated the extraction of PP through emulsion liquid membrane (ELM) from aqueous solutions [9]. Although, they indicated ELM treatment process as an effective advanced separation technique for the removal of PP even from complex matrices such as natural water, seawater and sewage water effluent, but, in general ELM experienced certain problems in scaling up the process due to swelling and breakage of emulsion globule. In view of this, recently, a more stable, continuous and efficient technique, that is pseudo-emulsion hollow fiber strip dispersion (PEHFSD) technique, has been developed which utilizes the merits of LM techniques and suitable for industrial use. Several metals and organic compounds were successfully extracted by PEHFSD using different carriers and stripping solutions [10–14]. Till date, no investigation is reported for the removal of PP through the PEHFSD, so an effort has been made to study the behaviour of the PEHFSD system for the separation of PP from aqueous solution. In the present work different process parameters such as pH of the feed phase, initial feed concentration, types of stripping phase and concentration, types of carrier and its concentration in membrane phase were optimized to achieve maximum extraction efficiency of PP. A mass transfer model was also developed to evaluate mass transfer coefficients and resistances in different phases and also to validate our experimental results.

2. Theoretical Background

2.1 Extraction Process

The extraction of Propylparaben ($C_{10}H_{12}O_3$) through a PEHFSD containing aliquat-336 (solvating reagent) is given by the following reaction [15]:

$$\left(C_{10}H_{12}O_3\right)_f + \left(\text{aliquat}-336\right)_{org} \leftrightarrow \left(C_{10}H_{12}O_3\cdot\text{aliquat}-336\right)_{org} \tag{1}$$

where *f* = feed phase, *org* = organic phase. In the stripping phase reaction occurs:

$$\left(C_{10}H_{12}O_3\cdot\text{aliquat}-336\right)_{org} + Na_2CO_3 \leftrightarrow \left(\text{aliquat}-336\right)_{org} + \left(C_{10}H_{11}O_3^- Na^+\right)_s + Na^+HCO_3^- \tag{2}$$

where *s* = stripping solution in pseudo emulsion phase.

The distribution ratio D_{PP} is ratio of two phases that is total analytical propylparaben concentration in the organic phase to the total analytical propyl paraben concentration in the aqueous (feed) phase at equilibrium.

$$D_{PP} = \frac{[PP]_{org}}{[PP]_f} \tag{3}$$

where $[PP]_{org}$ and $[PP]_f$ are the concentration of propylparaben in organic phase and in feed phase, respectively.

2.2 Permeability measurement

The model for the transport of solute that is PP in PEHFSD system which is used in recycling mode consists of equations describing:

- The difference in PP concentration in the feed and stripping solutions when flowing through the hollow fiber membrane module.
- The difference in PP concentration in the feed and pseudo emulsion reservoirs, where they are continuously recycled, based on the complete mixing assumption.

Similar scheme was also proposed for the recovery of various species using PEHFSD systems [10–12]. The equations describing the above material balance for PEHFSD system would result in the following equations, which show first-order kinetics:

$$V_f\left(\ln\frac{[PP]_{f,t=0}}{[PP]_f}\right) = St \tag{4}$$

where *S* is the coefficient dependent on the linear velocity of the fluids, the geometry of the fibers and module the overall permeability of the system. This overall permeability can be obtained for the system running in the recycling mode, from the experimental values of the slope S from the graph plotted between V_f (In$[PP]_{f,t=0}/[PP]_f$) versus t [4,16]:

$$P_{PP} = \frac{-Q_f}{2\pi r_i LN\varepsilon}\left[\ln\left(1-\frac{S}{Q_f}\right)\right] \tag{5}$$

The design of hollow fiber modules for the separation and concentration of PP using the overall permeability coefficient centers on three mass transfer resistances which is related to:

- the aqueous boundary layer created on the internal side of the fiber wall of the fiber,
- the diffusion of solute-carrier complex across the membrane phase immobilized in the microporous space of the fiber wall, and
- the aqueous boundary layer created on the outer side of the fiber wall of the fiber.

Since, the reaction is instantaneous on the stripping side, the contribution of the outer aqueous interface can be neglected and hence the reciprocal of the overall permeability coefficient can be written as [10,11]:

$$\frac{1}{P_{PP}} = \frac{1}{k_i} + \frac{r_i}{r_{lm}} \cdot \frac{1}{D_{PP} \cdot k_m} \tag{6}$$

2.3. The mass transfer coefficient

Mass transfer coefficients for the feed side (k_i) aqueous boundary layer and membrane phase (k_m) of the PEHFSD are estimated using empirical correlations [10]. The mass transfer coefficient for the feed side aqueous boundary layer with an average velocity u_f of the feed solution is evaluated as [10]:

$$k_i = 1.5\frac{D_f}{2r_i}\left(\frac{4r_i^2 u_f}{D_f L}\right)^{\frac{1}{3}} \tag{7}$$

where D_f is the diffusion coefficient of PP in the feed phase and evaluated by Stokes-Einstein equation described as [10]:

$$D_{(\text{form})} = \frac{k_B T}{3\pi\mu d_m} \tag{8}$$

where k_B is the Boltzmann constant, T is the absolute temperature, μ is the viscosity of the solvent (in case of aqueous phase it was taken as 7.98×10^{-4} Pa s at 30°C), d_m is the diameter of the PP molecule which is calculated to be 3.66×10^{-10} m [17]. The mass transfer coefficient in the membrane phase can be estimated as follows [4]:

$$k_m = \frac{\varepsilon D_m}{\tau \cdot d_{org}} \tag{9}$$

where ε is the porosity of the hollow fiber, d_{org} is the thickness of the membrane, τ is tortuosity of the hollow fiber and D_m is the diffusion coefficient of PP in the membrane phase which is evaluated to be 3.07×10^{-9} m^2 s^{-1} by using Eq. (8) after putting the viscosity of the membrane phase as 3.94×10^{-4} Pa s at optimum carrier concentration which is evaluated from the Bingham equation [18].

3. Materials and methods

3.1. Chemicals used

PP (Loba Chemie make, India) is used for the extraction through PEHFSD. For the preparation of pseudo-emulsion, *n*-heptane (Finar chemicals, India) of density 0.6795 g mL^{-1} and viscosity of 386 μP$_a$·s, containing TBP (Otto Kemi, India)/TOA (Merck, Mumbai, India)/Aliquat 336 (Merck, India) as carrier in membrane phase and NaOH, NaCl or Na_2CO_3 (Finar chemicals, Ahmedabad, India) in stripping phase are used. 1-Decanol (Otto Kemi, India) is used to dissolve Aliquat-336 in the *n*-heptane and hydrochloric acid (Finar chemicals, India) to adjust the pH of the feed phase before starting of each extraction experiment. The experimental conditions are given in Table 1. The details of the hollow fiber device (Membrana: Celgard®X50-215) used for the investigation is presented [10].

Table 1
Experimental conditions for PEHFSD system

Pseudo-emulsion phase	Organic phase + stripping phase
Membrane phase	*n*-heptane [19]
Carrier	TBP/TOA/Aliquat 336
Stripping phase	NaOH/NaCl/Na_2CO_3 (varied from 0.005 N to 0.1 N)
Flow rate of strip phase	200 mL min^{-1} [19,20]
Stirring speed in pseudo emulsion tank	600 rpm [19,20]
Volume of pseudo-emulsion	600 mL
Volume ratio of membrane to stripping phase	1:1 [10]
Feed phase aqueous solution	Propylparaben (5 ppm to 35 ppm)
Flow rate of feed through PEHFSD	300 mL min^{-1} [19,20]
Stirring speed in feed tank	300 rpm
pH (Feed phase)	2.0 to 6.0
Volume of feed solution	1500mL
Feed:strip ratio	5:1 [10]

3.2 PEHFSD Experiment

Authors found that few parameters such as pH, feed concentration, carrier concentration, and stripping phase concentration were process dependent. Other parameters such as volume of feed to stripping phase, stirrer speed, ratio of membrane to stripping phase and hydrodynamic conditions were system dependent which had already been optimized in their previous studies [10,19]. Hence, efforts were made to optimize process dependent parameters to achieve maximum PP extraction.

The PEHFSD process involves a single membrane contactor for extraction and stripping (Fig. 1). One stirred tank for preparing and as a reservoir of the pseudo-emulsion containing carrier + *n*-heptane and strip solution in which stirrer was immersed in the stripping phase and another stirred tank as a feed phase reservoir are used. The experimental set-up contains two gear pumps of variable flows for both phases with flow meters. Initially, a series of experiments was performed to select the best hydrodynamic conditions in terms of the flow rates of feed solution and pseudo-emulsion and the results were found to be similar as in our previous work (i.e. 300 mL/min for feed solution and 200 mL/min for pseudo-emulsion) [10,19]. The organic solution wets the porous wall of the fiber because of its

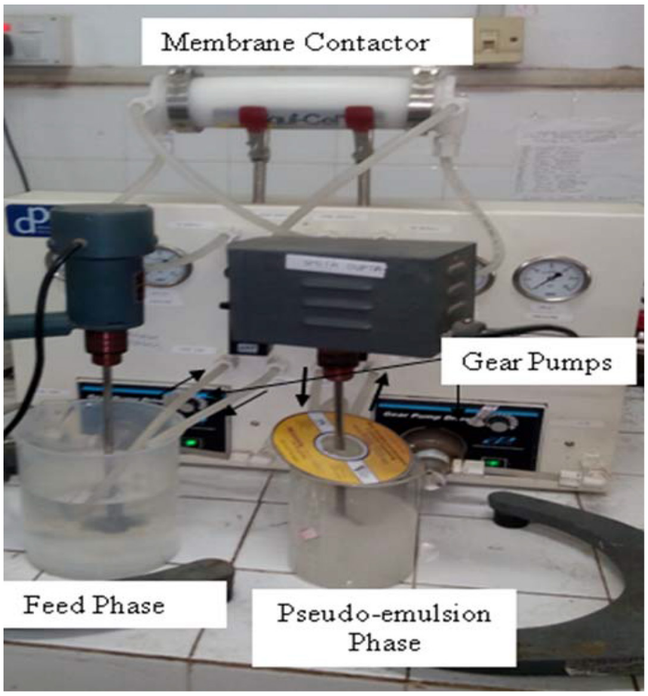

Fig. 1. Experimental setup of pseudo-emulsion hollow fiber strip dispersion system for the continuous removal of propylparaben from aqueous solution in recycling mode.

hydrophobic nature. The boundary layer is maintained at the opening of the pores by applying 1.38 × 10^4 N m^{-2} higher pressure to the feed side (tube side) than to the pseudo-emulsion side (shell side). The operation is performed by circulating an aqueous feed containing PP through the tube side and pseudo-emulsion through the shell side in counter-current recirculation mode. Continuous stirring is provided to maintain the homogeneity in both the tanks. The recovery of PP from pseudo-emulsion can be done by breaking down of the pseudo-emulsion and separating organic and stripping solutions.

Small volume of the aqueous streams are taken at different time interval and analyzed for PP concentration by UV–vis spectrophotometer (HACH make, Germany) at 256 nm wavelength. Permeability values are evaluated from Eq. (5) and the percentage extraction of PP through PEHFSD can be calculated by:

$$\%\ \text{Extraction}\ (Y) = \frac{[PP]_{f,t=0} - [PP]_f}{[PP]_{f,t=0}} \times 100 \tag{10}$$

Experiments are performed in triplicates and it is found that the data are reproducible.

4. Results and Discussion

4.1. Effect of pH

As solubility and diffusivity of PP in membrane phase is maximum in molecular form, so we need to optimize the pH of the feed phase (pH < pKa of PP = 8.47) to maintain the molecular form of propylparaben for PEHFSD experiments. A series of experiments was performed to determine the distribution ratio of PP by using equal volumes of 20 mg L^{-1} of PP in aqueous phase at different pH (pH 2 to 6) and 0.2% (w/v) of carrier (Aliquat 336/ TBP/TOA) in *n*-heptane. The distribution ratio of PP at equilibrium was found to be higher at pH 4 (Fig. 2) with all of carriers in membrane phase. Hence, for further experiments, pH 4 was used as feed phase pH to achieve maximum extraction efficiency of PP.

4.2. Effect of types of carrier

Since, membrane phase (*n*-heptane in this case) act as a water resistant barrier in liquid membrane process, some carrier is required to enhance the system performance by increasing the diffusion rate through the membrane phase. In this regard, to evaluate the effect of different types of carrier, a series of experiments in PEHFSD system, was conducted with different types of carrier (TOA, TBP and Aliquat 336) of 0.2% (w/v) concentration in *n*-heptane, keeping PP concentration at 20 ppm in feed phase, Na_2CO_3 concentration at 0.01 N in stripping phase. Taking same compositions of feed phase, membrane phase (with different carriers) and stripping phase, another series of experiments was conducted to determine the distribution ratios (calculated using Eq. (3) for each carrier in *n*-heptane. From Fig. 3, it was found that Aliquat 336 extracted the maximum amount of PP (97 %) in 40 min.

From Fig. 3, it was observed that Aliquat 336 is more reactive than the other two carriers which might be due to its higher capacity of loading more acid (PP which is slightly acidic in nature) [15]. Hence Aliquat 336 was chosen as carrier for further experiments.

4.3. Effect of types of stripping phase

In order to select the stripping phase to strip PP from the membrane phase, three stripping phases namely of NaOH, NaCl and Na_2CO_3 of 0.01 N concentration were tested for the extraction of PP. For this purpose, an aqueous solution of 5.0 mg L^{-1} of PP at 4.0 pH was transported

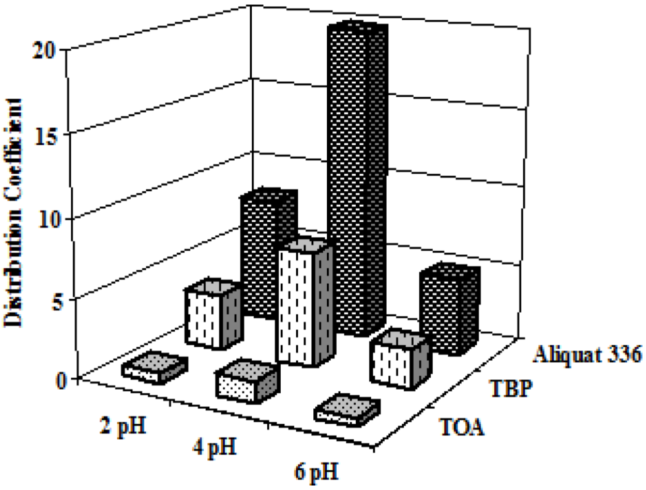

Fig. 2. Effect of pH on distribution coefficient with different carriers in membrane phase.

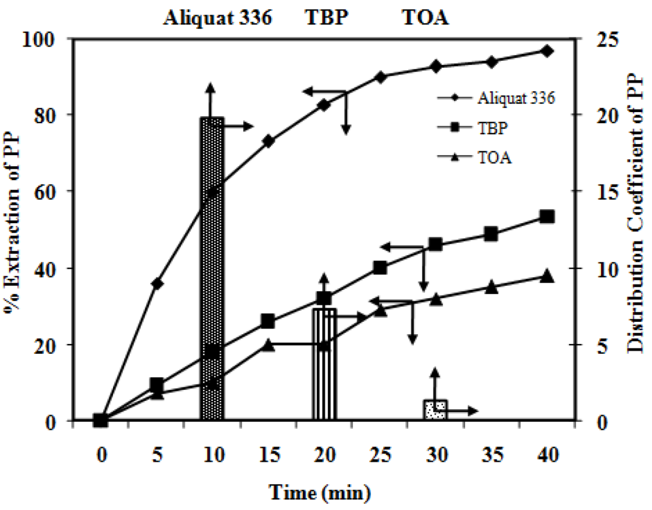

Fig. 3. Effect of different types of carriers on distribution coefficient and % extraction of PP through PEHFSD system.

through the membrane phase with 0.2% (w/v) of carrier in PEHFSD system. From Fig. 4, it was found that using Na_2CO_3 and NaOH as stripping phase the degree of stripping was higher as compared with NaCl. The reason behind this result might be due to the formation of HCl (H^+ generated from the PP combined with Cl^- of NaCl solution) in stripping phase in case of NaCl which would resist the extraction in the stripping phase due to the decrease in concentration gradient of PP molecule between membrane phase and stripping phase. Whereas in case of Na_2CO_3 and NaOH, % extraction was high (~97 %) due to higher concentration gradient of PP. In our experiment, as feed phase volume was 5 times more than strip phase volume, so 5 times (25 mg L^{-1}) more concentrated PP solution was recovered (Fig. 4). Hence, Na_2CO_3 was chosen as a stripping phase for the PP extraction through PEHFSD system for further optimization.

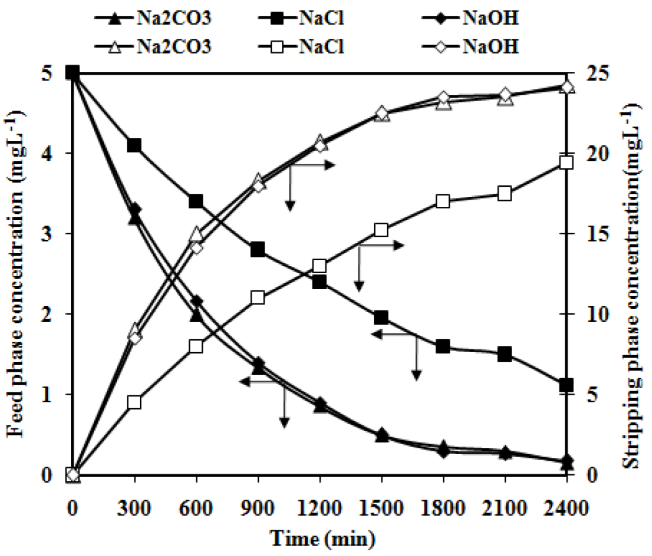

Fig. 4. Concentration profile of PP in feed phase and stripping phase using different stripping phases.

4.4. Effect of concentration of stripping phase

Keeping feed concentration at 5.0 mg L^{-1} of PP at pH 4 and Carrier concentration at 0.2% (w/v) in *n*-heptane, a series of experiments were performed in PEHFSD system varying Na_2CO_3 concentration (0.005 N to 0.1 N) in the stripping phase. From Fig. 5, it was observed that on increasing the stripping phase concentration from 0.005 N to 0.01 N % extraction of PP increased from 74 to 97% which may be due to presence of insufficient CO_3^- at lower concentration of Na_2CO_3 to strip all the H^+ ions of PP [20]. Further increase in Na_2CO_3 concentration from 0.01 N to 0.05 N showed no significant effect (superimposition of permeability data in Fig. 5) on the degree of PP extraction whereas a significant decline in the degree of extraction was observed when Na_2CO_3 concentration increased from 0.05 N to 0.1 N. This might be occurred either because of the back extraction of PP or because of unavailability of free Aliquat 336 in the membrane phase as Aliquat 336 could also participate in the anion exchange reaction [21]. Hence 0.01 N Na_2CO_3 was used in stripping phase for further experiment. Permeability of PP at different concentration of Na_2CO_3 was also calculated using Eq. (4) (Fig. 5).

4.5. Effect of initial feed concentration

Effect of initial feed concentration (5 to 35 mg L^{-1}) was studied keeping carrier concentration 0.2% (w/v) in membrane phase and stripping phase 0.01 N Na_2CO_3. From Fig. 6, it was observed that on increasing the feed concentration from 5.0 mg L^{-1} to 20 mg L^{-1}, no significant change was observed in % extraction of PP which might be due to the presence of sufficient amount of stripping phase concentration. Further increase in feed concentration resulted decrease

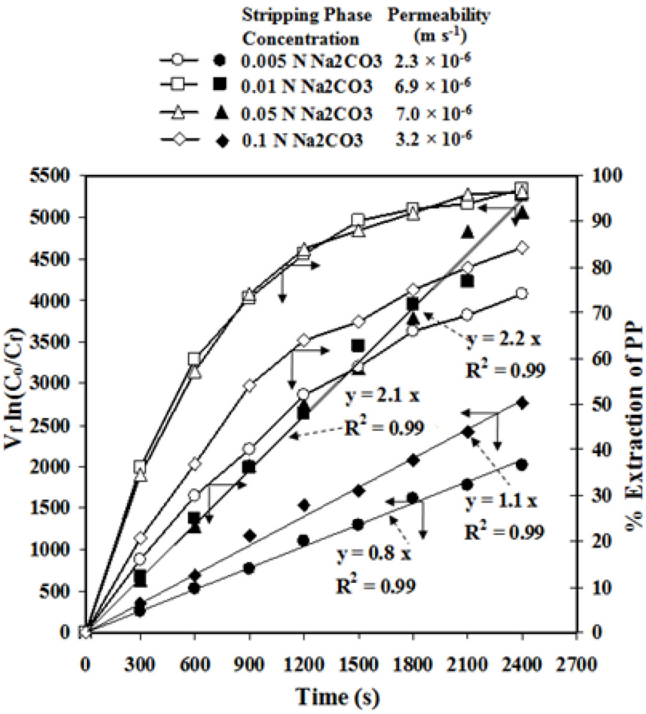

Fig. 5. Effect of concentration of Na_2CO_3 in stripping phase on permeability and % extraction of PP.

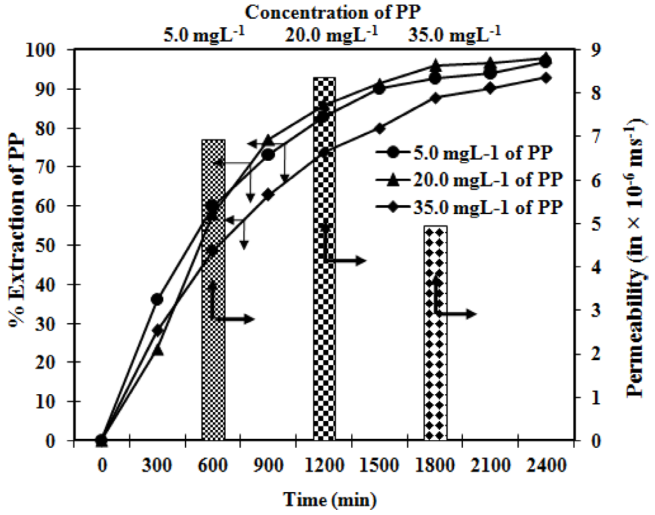

Fig. 6. Effect of initial PP concentration in feed phase on the permeability and % extraction of PP through PEHFSD.

in % extraction of PP due to unavailability of the sufficient amount of Na_2CO_3 to extract PP from feed phase. Same trend of extraction was observed in our previous work for the recovery of bisphenol A through hollow fibre supported liquid membrane (HFSLM) from its aqueous solution [20].

4.6. Effect of carrier concentration

Keeping initial feed concentration at 20 mg L^{-1} and 0.01 N Na_2CO_3 as stripping phase, a series of experiments were executed varying Aliquat 336 concentration (0.2 to 1.8% (w/v)) in membrane phase. It was found from Fig. 7 that on increasing carrier concentration from 0.2 to 1.4% (w/v) in membrane phase resulted almost complete removal of PP (100%) in 40 min and further increase in carrier concentration beyond 1.4% (w/v) would lead to decrease in permeability and % extraction. This may be due to the formation of more complex of PP with Aliquat 336 in membrane phase because of the presence of larger amount of carrier in membrane phase and also due to increase in viscosity in membrane phase which resulted decrease in diffusivity of PP in membrane phase. Maximum permeability (i.e. 3.23 × 10^{-5} m s^{-1}) was achieved with 1.4% (w/v) Aliquat 336 in membrane phase.

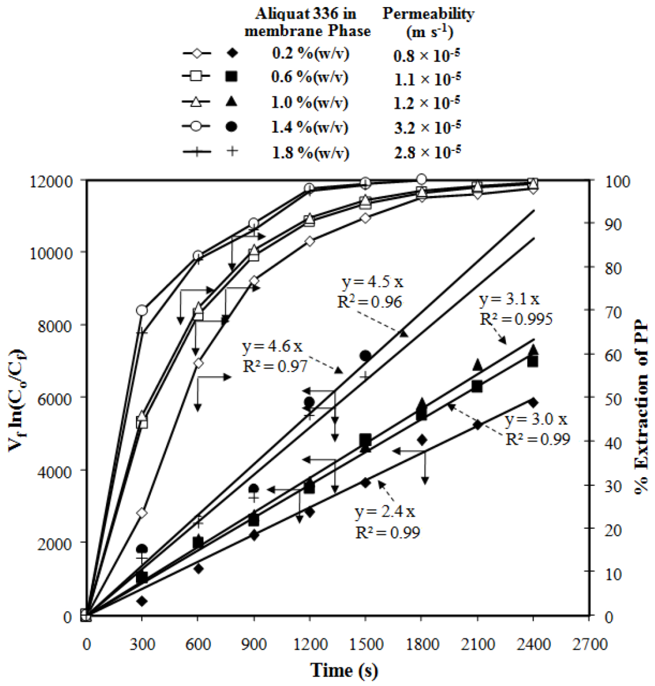

Fig. 7. Effect of carrier concentration in membrane phase on the permeability and % extraction of PP through PEHFSD.

4.7. Evaluation of mass transfer coefficients and diffusional parameters

The mass transfer coefficients (aqueous mass transfer coefficient (k_i), membrane mass transfer coefficient (k_m)) and diffusivity were calculated using Eqs (7)–(9). The obtained values of k_i, k_m and D_m from these equations were 3.4 × 10^{-5} m s^{-1}, 1.0 × 10^{-5} m s^{-1} and 3.1 × 10^{-9} m^2 s^{-1}, respectively. The distribution ratio of PP at optimum carrier concentration (i.e. 1.4% (w/v) of Aliquat 336 in *n*-heptane) was found to be 42.14. Eq. (6) indicated that the theoretical value of the total resistance was a sum of the theoretical values of the individual resistances. The deviation between the experimental (from Fig. 7) and predicted (from Eq. (9)) values of the overall resistance (1/P), was found to be less than 1.0%. This shows the validity of the mass transfer model. It was found that the resistance due to aqueous phase was dominating under studied experimental conditions.

5. Conclusions

The present work was aimed for the separation of PP from aqueous solution through PEHFSD system in recycling mode. For this purpose, three different carriers (TOA/TBP/Aliquat 336) and stripping agent (NaOH/Na_2CO_3/NaCl) were tested and among which Aliquat 336 and Na_2CO_3 was identified to provide maximum PP extraction. Optimum experimental conditions within the investigated range for higher degree of PP extraction through PEHFSD system were: Carrier: 1.4% (w/v) in *n*-heptane; Feed: 20 mg L^{-1}; Stripping solution: 0.01 N Na_2CO_3; Extraction time: 25 min. The results of the permeation experiments also confirmed the validity of the mass transfer model. On the basis of the contribution of fractional resistances, it was concluded that the rate-controlling step was the diffusion through the boundary layer of the feed side when operated under the optimum process conditions. By using the optimal process parameters, it was possible to achieve 100% extraction of PP from the aqueous solution using PEHFSD technique.

Symbols

C_o — initial PP concentration in feed phase (mg L^{-1})
C_t — PP concentration in feed phase at time *t* (mg L^{-1})
d_m — molecular size of PP (m)
d_{org} — thickness of the fiber (m)

D_{PP} — distribution ratio
D_f — diffusivity of PP in aqueous feed phase ($m^2 s^{-1}$)
D_m — diffusivity of PP in organic membrane phase ($m^2 s^{-1}$)
k_B — Boltzmann constant (1.38×10^{-23} m^2 kg s^{-2} K^{-1})
k_i — mass transfer coefficient corresponding to the inner aqueous boundary layer (m s^{-1})
k_m — mass transfer coefficient in membrane phase (m s^{-1})
L — length of fiber (m)
N — number of fibers in module
P — permeability coefficient (m s^{-1})
Q_f — total flow rate of feed solution flowing through tube side ($m^3 s^{-1}$)
r_i — inner radius of hollow fiber (m)
r_o — outer radii of the hollow fiber (m)
r_{lm} — the log-mean radius of the hollow fiber (m)
S — Slope of plot $V_f \ln(C_0/C_f)$ versus t
t — time (s)
T — absolute temperature (K)
u_f — average velocity of the feed solution (m s^{-1})
V_f — volume of the feed solution in the reservoir (m^3)
e — porosity of hollow fiber (%)
m — viscosity of solvent (Pa s)
t — tortuosity

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