

Performance of PES Membrane Contactor on the Separation of Dilute Acetic Acid from Aqueous Forms

Sofiya Karunanithi* and Prabhakar Sivaraman

Department of Chemical Engineering, SRM Institute of Science and Technology, Kattankulathur- 603203, Tamil Nadu, India

Abstract: The azeotropic characteristics of acetic acid between water make its separation from its aqueous phase essential. Many methods are studied for separating such a mixture. Solvent extraction using membrane contactors can increase the contact surface area. The dilute aqueous acetic acid extraction with the solvents petroleum ether and diisopropyl ether in a Poly Ether Sulfone (PES) hollow fiber membrane contactor was studied. The solvents and membrane extraction abilities are evaluated by exchanging the fluids on the shell and tube sides based on the distribution coefficients and total mass transfer coefficient at various flow rates. The findings show that when the solvent flow is increased, whether on the shell side or the tube side, the concentration of acetic acid increases in its solvent phase. As the shell side Reynolds number increased, it was noticed that the distribution coefficient value also increased. Both solvents' extraction ability was minimal, but diisopropyl ether gave better results when compared with the petroleum ether in terms of distribution coefficient. Three film resistance parameters in the series model have determined the overall mass transfer coefficient. The Reynolds number values showed that the mass transfer occurs in a laminar zone. Changes in the petroleum ether's Reynolds number on the tube side resulted in a maximum mass transfer coefficient of 11.41×10^{-6} m/s. Data on such hydrodynamics studies on membrane solvent extraction can help recover value-added material studies.

Keywords: Membrane solvent extraction, PES membrane, Acetic acid, DIPE, Petroleum ether.

INTRODUCTION

Acetic acid is among the top twenty chemical commodities extensively used in the process industries in two different roles, as a chemical reagent and solvent in many processes. It includes the production of polyethylene terephthalate, cellulose acetate, polyvinyl acetate, etc. [1]. It is obtained as a byproduct of food, petroleum, pharmaceutical industries, etc. [2]. Additionally, the chemical reactions using the acetic anhydride either as a reactant or as an extractant can discharge a significant amount of acetic acid in its aqueous streams. The two methods of manufacture of acetic acid are synthetic, and fermentation processes also have the dilute form of acetic acid in its outlet streams [1-3]. This waste stream should be treated to meet the environmental standard and recovered to add value to that industry [4]. Several research methods were proposed to recover acetic acid in its dilute form [3]. The separation of acetic acid from its aqueous phase is difficult by distillation requires several plates. In such a case, liquid-liquid extraction is the cost-effective method to recover acetic acid in its aqueous phases [5], especially the acetic acid concentration below 50% [6]. The dilute form of acetic acid separation requires more attention due to its more aqueous phase

content; such separation is more difficult in any of the methods. The research on membrane solvent extraction is also focused on today due to its high contact area [7]. The membrane contactor with shell and tubes is also used for membrane solvent extraction [8-16]. This hollow fiber membrane technology has significant advantages over traditional extraction methods because it offers the most incredible contact area between the solvent and the feed phase [17]. The flow rates independent of one another have advantages such as less flooding and loading, dispersion-free operation, modular design that makes scaling up more feasible, and equipment retrofitting. There will not be a required density difference between the phases. The unique features of hollow fiber membranes have made several investigators examine their application in liquid-liquid extraction [18]. Many literatures reported the mass transfer study on membrane solvent extraction [19-22]. The membrane contactors are generally made with polypropylene, polytetrafluoroethylene, polyvinylidene fluoride, polyethersulfone, etc. [22-24].

The data on the PES membrane (Poly Ether Sulfone) on the separation of dilute acetic acid was not presented previously in the literature. In the current investigation study, an attempt was made with a PES hollow fiber membrane contactor to extract acetic acid from its dilute aqueous phase. The shell and tube side effects are studied. The reason for selecting a PES membrane contactor is the characteristic such as

*Address correspondence to this author at the Department of Chemical Engineering, SRM Institute of Science and Technology, Kattankulathur- 603203, Tamil Nadu, India; Tel: 044-27417818
E-mail: ksofiya26@yahoo.co.in; sofiyak@srmist.edu.in;
Coauthor E-mail: sivaprabha50@gmail.com

inherently hydrophilic, which wets quickly and thoroughly the membrane pores. The pore surface area accounting for about 50-70% of the porosity, is responsible for providing the maximum interfacial area [10-12, 25]. In the case of membrane solvent extraction, the efficacy depends on the material of the membrane used, type of solvent, mode of contact, the flow rate of shell and tube side, etc.

The experiments were conducted using a PES (PolyEtherSulfone) membrane contactor. The simulated 5 % (vol %) acetic acid from its dilute aqueous phase was extracted with petroleum ether and diisopropyl ether. The solvents were selected based on the compatibility of the membrane and its environmentally friendly nature. The diisopropyl ether provides excellent low water solubility and less density and is comparatively unreacted with other compounds. The solvent petroleum ether provided a little volatile liquid hydrocarbon mixture and a cost-effective nonpolar solvent. The petroleum ether boiling point between 60 to 80°C was used in the present case. The solvents petroleum ether and diisopropyl ether are more volatile than acetic acid to reduce the energy used in the recovery section. This study included the changes in flow rates on organic and aqueous phases to exchange shell and tube side phases. The influence of the mass transfer coefficient and distribution coefficient was examined for the varied flow rates on the tube and shell sides.

MATERIAL AND METHODS

Materials

The used PES membrane module has a dimension of 300 mm in length, 32 mm shell diameter, 0.08 mm outer diameter (filament), and 0.04 mm inner diameter (filament) with 180 filament tubes. The membrane contactor was purchased from Tech Inc., Chennai, India. Solvents Petroleum ether and diisopropyl ether (all extra pure AR) were purchased from Sri chem in Chennai, India. Rankem provided glacial acetic acid in Chennai, India. The schematic view of the experimental arrangement with both streams flowing in the same direction is shown in Figure 1, and the picture of the PES membrane module is given in Figure 2. The acrylic body makes the outer tube of the membrane. The membrane and pumping material compatibility selected the organic solvents and feed concentration. The fibers were immersed in the feed and solvent for a week to confirm their compatibility with the solvents. The advantages of the PES membrane provide fast

filtration, flow rate increases, high throughputs, and uniform porosity are attractive features of this membrane. The controlled pore size allows this PES filtration membrane contactor to remove solute from solutions efficiently. The strength and durability offered by PES membranes were suited for aggressive handling and used with automated equipment [10-12].

Methods

A 5 % (vol%) acetic acid dissolved in water was taken as the aqueous phase (feed), and pure petroleum ether (or) diisopropyl ether was considered as a solvent phase. Case I) The aqueous feed is distributed over the tube portion of the membrane module. The solvent portion was distributed over the shell volume in a parallel flow. Case II) the solvent phase was spread over the tube area, and the feed phase was distributed over the shell side in a co-current manner.

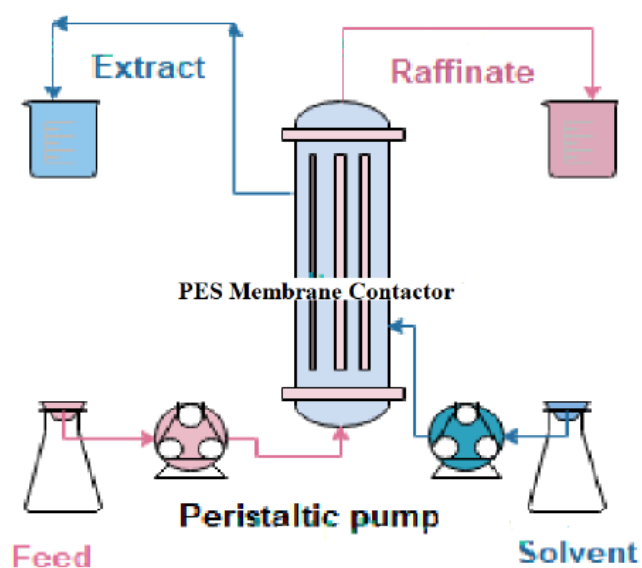


Figure 1: Membrane Solvent Extraction Experimental Schematic Diagram (MSE).



Figure 2: The picture of the PES membrane contactor.

The classic membranes are used for water treatment and filtration process, and Membrane contactors are provided with shells and tubes used for solvent extraction. There is no requirement for an

external wetting agent because the PES membrane module employed in this investigation is hydrophilic. The experiments were conducted by differencing the velocity of one stream with a change in the velocity of another. The feed and solvent were circulated using a peristaltic pump from bottom to top. The resulting extract and raffinate phases were collected and analyzed using the acid-base titration method.

Theory of Membrane Process

The three distinct mass transfer coefficients influence the overall mass transfer coefficient of the membrane contactor. For the situation when the solvent is on the shell side of the aqueous feed-in tube, i.e., i) the solute movement via the filament tubes from the aqueous phase ii) transfer of solute on the membrane surface iii) solute transfer into the solvent phase.

For the aqueous feed-in tube side and solvent on the shell side with no chemical reaction, the three-resistance in the series model, which many researchers employ, was presented as follows (Based on the organic phase) [7,13-14].

$$\frac{1}{K_o d_o} = \frac{1}{k_{os} d_o} + \frac{m_i}{k_m d_{lm}} + \frac{m_i}{k_{wi} d_i} \quad (1)$$

For Case II) When the solvent in tube sides and aqueous feed on shell side with no chemical reaction of the hydrophilic membrane [7, 10] (the driving force based on organic phase condition) was given as follows

$$\frac{1}{K_w d_i} = \frac{1}{k_{oi} d_i} + \frac{m_i}{k_m d_{lm}} + \frac{m_i}{k_{ws} d_o} \quad (2)$$

The coefficient of mass transfer through a membrane's pores (k_m), which is based on the parameters of the membrane itself, such as its porosity and density as well as the tortuosity of its holes for the PES membrane, is calculated using Fick's first law. The k_m is constant in this study [7, 9-10].

$$k_m = \frac{D\varepsilon}{t\tau} \quad (3)$$

The diffusivity value of $D=1.3 \times 10^{-9} \text{m}^2 \text{s}^{-1}$ (for petroleum ether) & $D=1.8 \times 10^{-9} \text{m}^2 \text{s}^{-1}$ (for diisopropyl ether) were obtained. The Wilke, Lee, and Chang equation were used to calculate the solute transport into the solvent at 296 K. The membrane has a 2.5 tortuosity and a 0.35 porosity.

Mass Transfer Coefficient Prediction for a Shell and Tube

The parameters of the fluid and the flow rate affect each mass transfer coefficient individually, k_{wt} , k_{os} , k_{ws} , and k_{ot} . The fluid flow characteristic affects the mass transfer coefficients on the tube and shell surfaces, which determine how the membrane moves under laminar circumstances. When the G_z is more significant than 4, Leveque proposed the following equation to estimate the tube side transfer of mass [9-10].

$$k_{wt} \text{ or } k_{wt} = 1.62 \left(\frac{D^2 v}{\ell d_i} \right)^{0.33} \quad (4)$$

The relationship between the dimensionless groups can be used to anticipate the mass transfer coefficients on the membrane's shell side. Here's one way to phrase it: [15-16]

$$Sh_{shell} = \frac{k_{os} d_h}{D} = \beta(1-\phi) Re_{shell}^{0.6} Sc^{0.33} \left(\frac{D_s}{\ell} \right) \quad (5)$$

$$d_h = (4 \times \text{cross-sectional area}) / \text{wetted perimeter} \quad (6)$$

Where β is 6.1 for hydrophilic membranes, if the Reynolds no value obtained between 0 to 500 and ϕ in the range of 0.04 to 4. Reynolds numbers range from 1 to 400, $\phi=0.1$, which are both requirements that are met in the current investigation.

Reynolds numbers across the tube portion for the current investigation ranged from 43 to 315, whereas those on the shell side were between 1 and 15, indicating the laminar nature of the situation.

RESULTS AND DISCUSSION

The hydrodynamics study on the PES membrane was carried out by exchanging the solvent and feed in the tube and shell side with varying the flow rate. The resistance in the series model provided in section 2 was taken to determine the mass transfer coefficient. Two cases of the experiment were studied solvent circulated i) on the shell side. ii) On the tube side.

Influence of Solute Concentration on Extract Phase when Feed is on the Tube Side and Solvent is on the Shell Side

The solvent was collected from the shell side, and the feed, dilute acetic acid, was obtained from the tube side. The flow rate on the shell side was altered while the flow rate on the tube side was initially kept constant. In Figure 3, the effect of variable tube side

flow rate (3-7 mL/min) is studied with the shell side steady flow of 5 mL/min. In the second case, the effect of shell side variable flow rate (3-7 mL/min) is investigated with the tube side constant flow of 5 mL/min, illustrated in red and blue, respectively.

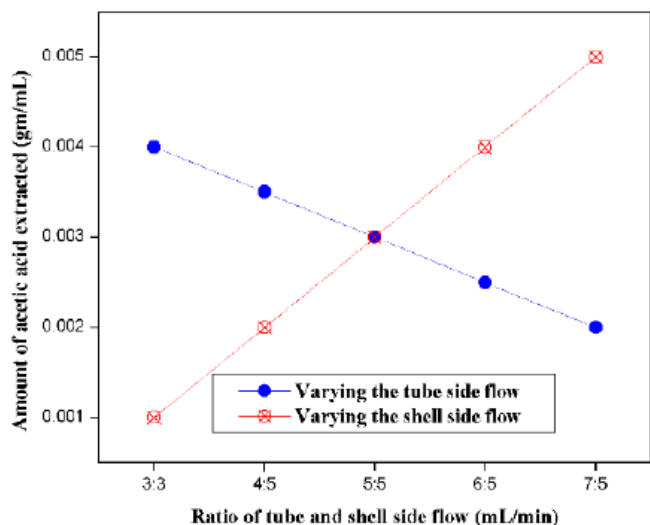


Figure 3: Solute concentration in extract phase by (Feed- in tube side solvent in shell side) by varying the shell and tube side flowrate when the petroleum ether is used as a solvent.

It was determined that the solute content in the extract phase increases with the constant tube side flow and decreases the tube side flow with every increment of the shell-side flow rate. Acetic acid was found in the extract phase at a concentration of 0.005 gm/mL when the flow rates on the shell and tube sides were 7 mL/min and 5 mL/min, respectively. The increase in concentration may be some concentration deposited on the membrane pores, the increase in solvent flow rate carries some additional concentration of acetic acid. When the shell-side flow was 3 mL/min, a lower solubility range of below 0.001 g/mL was found.

The same outcome was accomplished with success using diisopropyl ether as a solvent. When the tube side flow rate was 3 mL/min, and the shell side flow rate was 5 mL/min, 0.09 gm/mL of acetic acid was most significantly extracted. According to Figure 3, using diisopropyl ether as a solvent led to a linear rise in acetic acid solubility in the extract phase and increased shell side flow rate.

Tube flow had less of an impact on the concentration of acetic acid than changes in the shell side flow rate. It was determined that the changes in the shell flow scarcely powered the performance of the extraction process; it changed desperately with the rising velocity of the shell flow, as given in Figures 3

and 4. From the results, it can be concluded that the best extraction was obtained by keeping the shell-side flow rate as optimum level as possible; the increase in solvent flow rate maximized the extraction ability. The solvent diisopropyl ether showed better results in comparison with the petroleum ether.

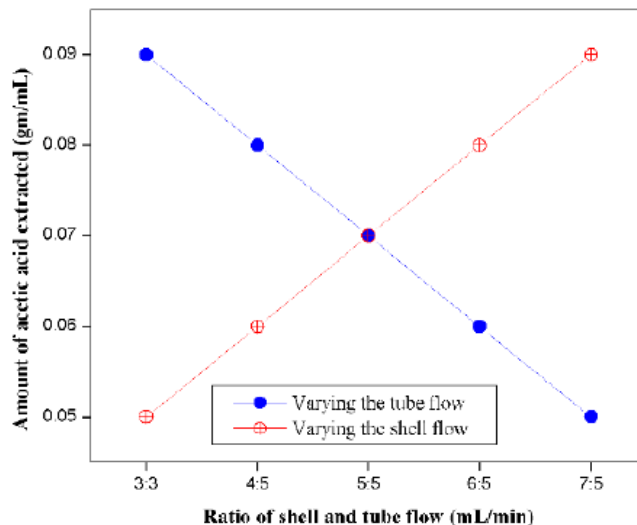


Figure 4: Solute concentration in extract phase by (Feed- in tube side solvent in shell side) varying the shell and tube side flowrate when the diisopropyl ether was used as a solvent.

Influence of Solute Concentration on Extract Phase when Feed is on the Shell Side and the Solvent is on the Tube Side

Figure 5 depicts the acetic acid concentration in the extract phase that was obtained using an aqueous feed-in shell side and solvent on the tube side. The

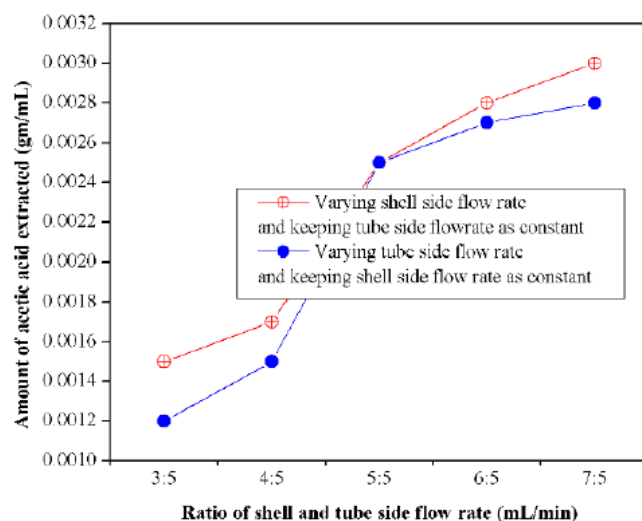


Figure 5: Solute concentration in extract phase by (Feed- in shell side solvent in tube side) by varying the shell and tube side flowrate when the petroleum ether is used as a solvent.

concentration was attained by maintaining the shell side flow rate at 5 mL/min while adjusting the tube side flow rate between 3- 7 mL/min, as shown in the Figure 5. According to the graph, using petroleum ether as a solvent, the maximum amount of acetic acid that may be present in the extract phase was 0.003 gm/mL. Figures 5 and 6 showed almost the same trend; it was observed that the solvent on the tube side, whether varying the shell flow or tube flow, had no impact on the concentration in the extract phase. The solvent diisopropyl ether obtained high concentrations in its extract phase than petroleum ether.

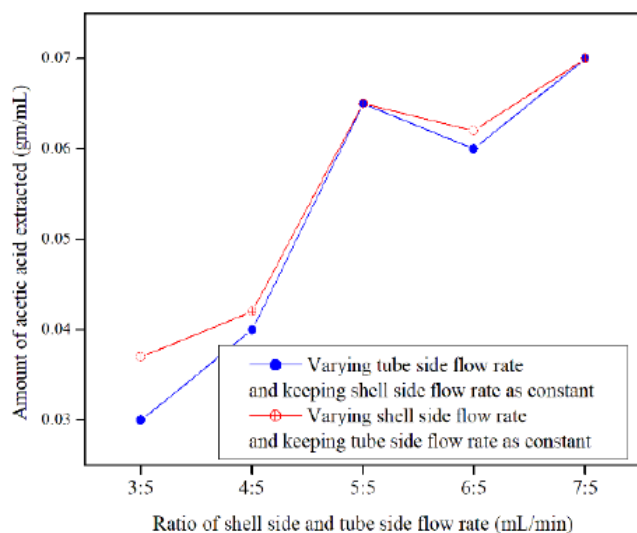


Figure 6: Solute concentration in extract phase by (Feed- in shell side solvent in tube side) by varying the shell and tube side flowrate when the Diisopropyl ether is used as a solvent.

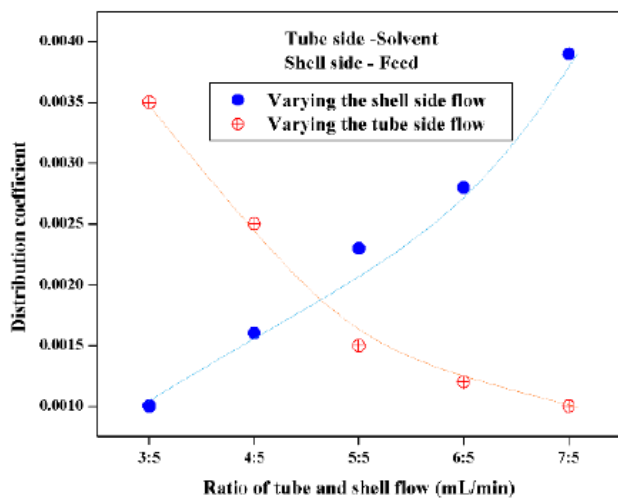
Effect of Variable Tube and Shell side Flow on the Partition Coefficient

The distribution coefficient was calculated using the extract phase to raffinate phase ratio of acetic acid. The distribution coefficient comparison between the solvent and aqueous feed when i) solvent phase dispersed at the tube section and ii) solvent phase dispersed at the shell section was analyzed and shown in Figures 7 and 8. The results show that the distribution ratio raised the shell flow, whether the solvent or the feed on the shell side. In the alternative scenario, whether solvent in shell or tube, the distribution ratio falls as tube flow increases.

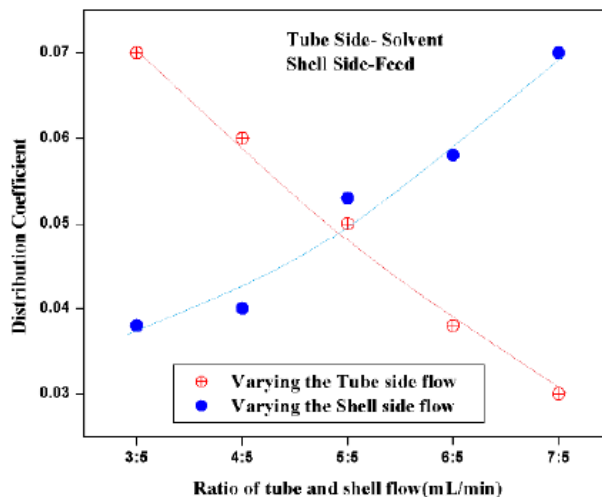
When comparing the distribution coefficient by altering the flow on both the tube side and the shell side (Reynolds No), adjusting the shell side flow enhanced the distribution coefficient. The exchange of feed and solvent in a tube or shell does not affect the distribution coefficient. The distribution coefficient was impacted by the variable flow of the shell and tube; it increased with an increase in shell flow and decreased with an increase in tube flow.

Effect of Overall Mass Transfer Coefficient

Depending on the organic phase on the Shell and Tube sides, the Reynolds number was plotted against the overall coefficient of mass transfer, or "Ko," as illustrated in Figures 9 and 10. The graph showed the overall coefficient of mass transfer along with the increase in the Reynolds numbers of the shell and tube



(a)



(b)

Figure 7: Distribution coefficient of acetic acid when Solvent in Tube side (a. Petroleum ether & b. DIPE).

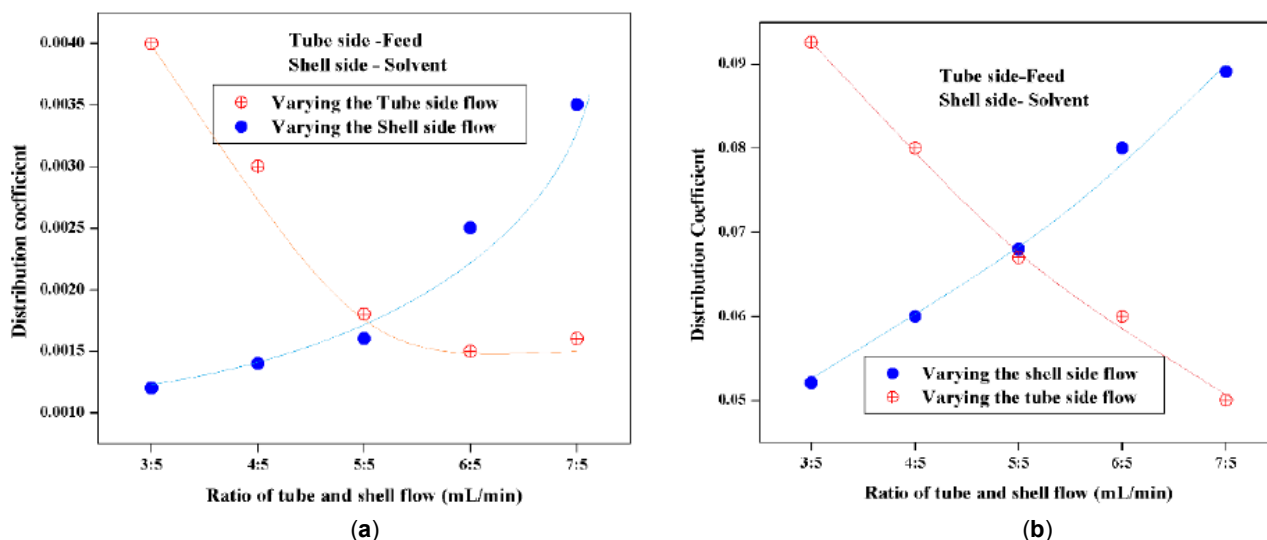


Figure 8: Distribution coefficient of acetic acid when Solvent in shell side (a. Petroleum ether & b. DIPE).

sides. The mass transfer coefficient was not significantly altered when the solvent was on the shell side, and the aqueous feed was on the tube side. Petroleum ether improved its flow rate to attain a more significant mass transfer coefficient, whether in a shell or a tube.

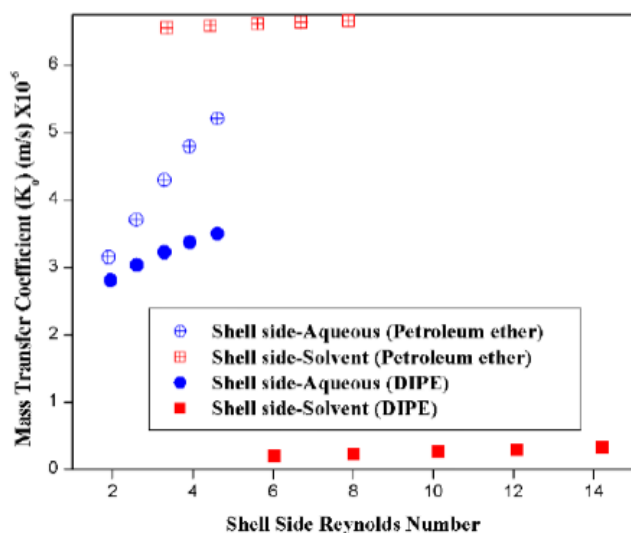


Figure 9: Total mass transfer coefficient (Based on Organic phase) with a change in Shell side Reynolds No.

The solvent petroleum ether showed a greater mass transfer coefficient value than diisopropyl ether. The maximum mass transfer coefficient of 11.41×10^{-6} m/s was reached by switching the tube side flow. The overall mass transfer coefficient obtained was comparable to the literature value [22, 23]. Reynolds No, we got a result that agrees with the value given by

utilizing both shell and tube side flow as constants [14].

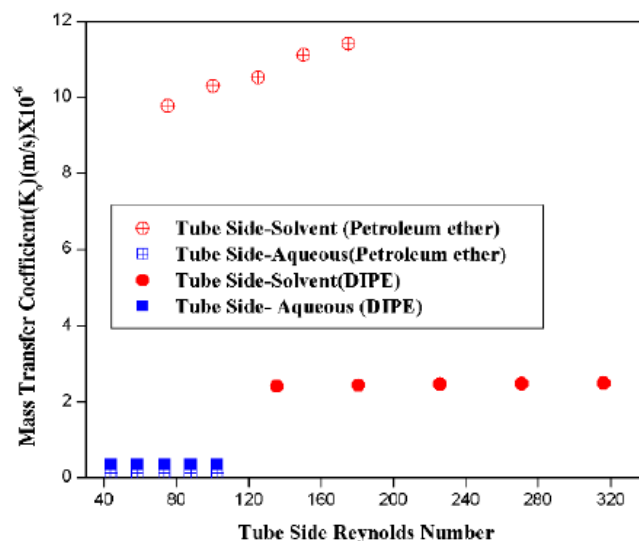


Figure 10: Total mass transfer coefficient (Based on Organic phase) with a change in Tube side Reynolds No.

CONCLUSIONS

The removal of dilute acetic acid from aqueous feed was studied through the PES membrane with petroleum ether and isopropyl ether as solvents. The process was carried out in a hydrophilic hollow fiber membrane contactor without contacting the solvent or aqueous phase. The feed and solvent flow rate influenced the performance of the PES membrane. The maximum acetic acid concentration in the extract was 0.005 g/mL. This was accomplished by using petroleum ether as a solvent with shell side and tube side flows of 7 mL/min and 5 mL/min, respectively. A

concentration of 0.09 g/mL was observed with diisopropyl ether as a solvent with the shell side and tube side flow rates of 3 and 5 mL/min, respectively.

The overall diffusion mass transfer coefficient increased as the Reynolds number rise. The Reynolds number value indicates that the mass transfer occurs in a laminar region. By adjusting the feed and the solvent flow rates, the highest mass transfer coefficient of 11.41×10^{-6} m/s was discovered to boost the solvent flow on tube sides. The mass transfer hypothesis revealed that the regulating step was the solute transfer from the bulk aqueous solutions to the feed-solvent interface.

NOMENCLATURE

K_o - Overall mass transfer coefficient based on organic phase (m/s)

K_{ot} , K_{os} - Organic phase local mass transfer coefficient for organic flow in tube & shell (m/s)

K_m - Membrane transfer coefficient (m/s)

K_{wt} , K_{ws} - Aqueous phase local mass transfer coefficient for aqueous phase in tube or shell

d_{im} - Hollow fiber Log mean diameter (m)

d_i - Hollow fiber inner diameter (m)

d_o - Hollow fiber outer diameter (m)

m_i -Partition coefficient

D -Diffusion coefficient of solute in the organic phase (m^2/s)

ϵ - Membrane Porosity

τ -Tortuosity of the membrane

t - Membrane Thickness (m)

v -Tube side velocity of the fluid (m/s)

d_h - Hydraulic diameter (m)

D_s -Shell diameter (m)

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