Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2014.913993

55 (2015) 114–124
June

Taylor & Francis
Taylor & Francis Group

Effect of operating parameters on 2-chlorophenol removal from wastewaters by a low-pressure reverse osmosis system

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Received 5 August 2013; Accepted 30 March 2014

ABSTRACT

In this study, the performance of a commercial low-pressure reverse osmosis membrane system (TW30-1812-100) was evaluated for 2-chlorophenol removal from wastewaters. The influence of various operational conditions and system specifications such as feed pressure, feed flow rate, pH, and concentration on 2-chlorophenol rejection was investigated. The results showed that at optimum conditions (200 ppm feed concentration, 408.1 kPa feed pressure, pH 10, and 1.953×10^{-5} m^3/s feed flow rate) 79% rejection was attainable. In addition, it was found that at the feed pH was the most effective parameter on 2-chlorophenol rejection. The rejection was increased with feed pH. An increase of feed flow rate had a positive effect on 2-chlorophenol rejection. The study of the influence of feed pressure showed a maximum critical pressure for rejection. However, the study of feed concentration on 2-chlorophenol rejection did not show a clear trend.

Keywords: 2-Chlorophenol; Wastewater; Permeate flux; Reverse osmosis; Rejection

1. Introduction

Chlorophenols are commonly used as wood preservatives, as disinfectants for fungicides, herbicides and in the manufacturing of pharmaceuticals, resins and dyes [1–4]. Chlorophenols are produced by many industrial processes, such as coal gasification sites, petrochemical and oil refining plants, and in manufacture of plastics, resins, textile, iron, steel, and paper [1,5,6]. They are also produced during chlorination of drinking water [7]. These compounds are discharged into the environment and thus found in wastewaters and water resources [8]. Some chlorophenols such as 2-chlorophenol are known as carcinogens and some are known as environmental endocrine disruptors [9]. Chlorophenols' toxicity relatively increases with the degree of chlorination [7]. Chlorophenols can also be toxic for organisms and some of them may cause bioaccumulation in aquacultures [10]. It has been reported that the OH group is very effective in the toxicity of chlorophenol to fish [7]. 2-chlorophenol can cause vomiting, inflammation in the digestive system, increased blood pressure, and can cause problems in the liver and kidney. When it is absorbed through the skin, it can reach the stomach through blood circulation. In addition, when it is in contact with the skin, it can cause burns [11].

Since chlorophenols have been introduced as hazardous pollutants, development in operative processes is necessary for their removal from water [12]. Several

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techniques have been used for removal of chlorophenols such as microbial digestion, adsorption on activated carbon, biosorption, chemical oxidation, photochemical and microbial oxidation, deep-well injection, incineration, solvent extraction, liquid–liquid extraction, irradiation, membrane technology, and hybrid methods [1,2,13–16]. Membrane processes have been considered as useful methods for removal of organic pollutants from wastewater, as a single process or combined with other physical and chemical water treatment processes [4,5,17,18]. The ability of pressure driven membrane processes for removal of organic compounds from wastewaters has been reported [14]. NF/RO processes have been widely used for contaminant removal in advanced water and wastewater treatment and desalination [19–21]. Recently, reverse osmosis (RO) membrane technology has attracted much attention due to its preponderance than other separation techniques [22].

In RO process, like other membrane processes, the most important parameters are rejection (selectivity) and water flux (permeability). According to the solution-diffusion model, water flux ($m^3/m^2 s$) is directly proportional to the pressure driving force:

$$J_W = A_W(\Delta P - \Delta\pi) \tag{1}$$

where A_W is water permeability ($m^3/m^2 s$ Pa), ΔP is hydraulic pressure difference applied to the membrane (Pa), and $\Delta\pi$ is the solute osmotic pressure difference between the feed and permeate side of the membrane (Pa).

According to this theory, the solute flux is expressed as follows:

$$J_s = L_s(C_m - C_p) \tag{2}$$

where J_s is the solute flux ($kg/m^2 s$), L_s is the solute transport parameter (m/s), and C_m and C_p are the solute concentrations at the membrane surface and in the permeate stream (kg/m^3), respectively [22].

In liquid separation processes, such as RO, rejection is defined as:

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100 \tag{3}$$

where C_P and C_F are permeate and feed concentrations (kg/m^3), respectively [14].

RO process has some advantages such as low energy consumption, high recovery percent, low occupied space, simple design, and easy work-up [23]. One of the major drawbacks of the membrane processes is concentration polarization [24]. This phenomenon characterizes the accumulation of separated solutes or particles from the feed solution on the membrane surface [25,26]. The concentration of the solutes that was rejected by the membrane and accumulated on the membrane surface is high, proportional to the concentration in the bulk solution far from the membrane surface [25]. The concentration polarization forms a concentration boundary layer on the membrane surface [26]. The resulted concentration profile is expressed as:

$$\frac{C_m - C_p}{C_b - C_p} = e^{J_v/k} \tag{4}$$

where C_b is the solute concentration in the bulk solution (kg/m^3), J_v is the solvent flux ($m^3/m^2 s$), and k is mass transfer coefficient (m/s) [22].

It has been reported that increasing of the feed flow rate increases the turbulency at the membrane surface, which can decrease the concentration polarization effect [26].

The polarization module (M) is a good criterion for concentration polarization of the module under different operating condition and is defined as follows [27]:

$$M = \frac{C_m}{C_b} \tag{5}$$

Obtaining the polarization module without knowing the mass transfer coefficient (k) for the membrane module is very difficult. Although several correlations have been reported for prediction of mass transfer coefficient in RO membranes, all of them are for large-scale industrial RO modules and there is no reported k value for small-scale modules (such as that we used in this study).

Lee and Lueptow reported a simple relation for calculation of the concentration factor, which is defined as the volume ratio of feed to reject flow rates as follows:

$$f_c = \frac{V_f}{V_c} = 1 + \frac{V_p}{V_c} \tag{6}$$

where V_c, V_f, and V_p are reject, feed, and permeate volumes (m^3) or flow rates, respectively [22].

The concentration polarization phenomenon decreases the efficiency of RO membranes in three ways: first, it acts as a hydraulic resistance for water flow through the membrane. Second, it increases the osmotic pressure at the surface of membrane, where the solute is accumulated and consequently lowers the driving force and water flux. Third, due to build up of the solute on the membrane surface, the solute passage through the membrane increases and thus the rejection is decreased [24].

Many authors have described that the important factors affecting the organic retention by nanofiltration and RO membranes are membrane characteristics, physicochemical properties of the solute, solution chemistry, and operating conditions [28–30]. It is reported that both membrane performance and lifetime are extremely affected by influential properties, such as concentration polarization and fouling phenomena, which are inter-related, and cause flux decline and permeate quality loss [31].

A number of studies on the removal of chlorophenols by RO and nanofiltration processes have been reported in the literature [13,32]. Unfortunately, a few studies have been reported for 2-chlorophenol removal from aqueous solution using the RO process. Arsuaga et al. studied the rejection of mono-chlorophenols by RO and described the effect of the position of the Cl atom in the aromatic ring on the rejection and reported 72% removal for 2-chlorophenol [13]. Yogesh et al. used four different TFC membranes for 2-chlorophenol removal but they did not consider the effect of operating conditions on the rejection. The maximum reported rejection for 2-chlorophenol was 33% [33].

In the present work, the effects of operating conditions such as feed pressure, feed pH, feed concentration, and flow rate on rejection of 2-chlorophenol using a low pressure RO membrane are investigated. The focus is on the effect of operating conditions and the impact of each parameter on 2-chlorophenol rejection.

2. Materials and methods

2.1. Chemicals

2-Chlorophenol with 99% purity was purchased from Merck Co. The physicochemical properties of 2-chlorophenol are presented in Table 1. The p*K*a shows the acid dissociation constant at which the organic molecule loses a hydrogen atom and becomes negatively charged and log (K_{ow}) shows the hydrophobicity of the organic molecule.

Generally, the compounds with log (K_{ow}) > 2.5 are considered as hydrophobic. The dipole moment is a criterion of the polarity of the molecules. Molecules with higher dipole moment have larger difference between positive and negative electrical charges [36].

The concentration of 2-chlorophenol in aqueous solutions was determined by sensitive 4-aminoantipyrine method. For this purpose, sodium hydroxide (97% purity), potassium ferricyanide (99.5% purity), and ammonium hydroxide (25% purity) were purchased from Merck Co., 4-aminoantipyrine (97% purity) was supplied from Alfa Aesar and used without further purification, chloroform (99% purity) and hydrochloric acid solution (25% in water) were purchased from Dr Mojallali Laboratory Chemical Co., Iran.

Table 1
The properties of 2-chlorophenol

Property	Value	Unit	Reference
Chemical formula	C_6H_5ClO	–	[13]
Molecular weight	128.56	g/mol	[34]
Density @ 20°C	1.262	g/cm^3	[35]
Melting point	7	°C	[34]
Boiling point @ 101.3 kPa	175–6	°C	[34]
Water solubility @ 20°C	22	mg/L	[35]
pKa	8.5	–	[13]
log (K_{ow})	2.15	–	[13]
Dipole moment	0.938	Debye	[33]

2.2. Experimental setup

Fig. 1 shows a schematic diagram of the designed experimental setup equipped with a spiral-wounded

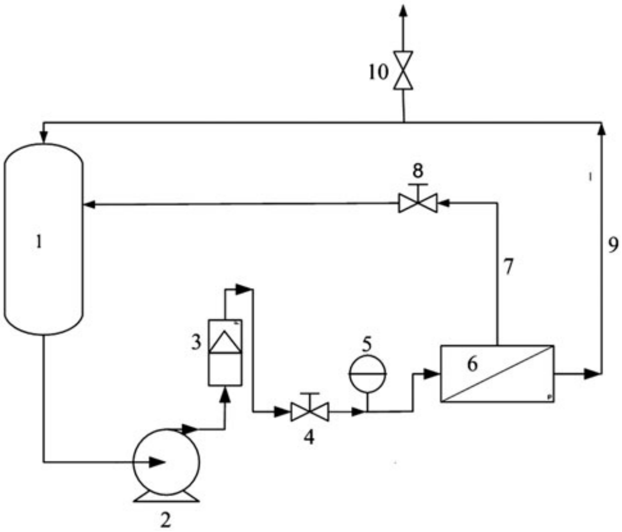


Fig. 1. The schematic diagram of the RO setup. 1. Feed tank, 2. Diaphragm pump, 3. Flow meter, 4. Needle valve, 5. Pressure indicator, 6. Membrane module, 7. Reject line, 8. Pressure regulating valve, 9. Permeate line, and 10. Sampling valve.

RO membrane. The feed tank was a 2 L glass container. A diaphragm pump (model HF-8367) with maximum working pressure and flow rate of 125 psi and 1.2 L/min was used for the experiments. A stainless steel pressure gauge (Marsh), two high-precision regulating needle valves (AISI 316L, from Fujikin Co.), a diaphragm valve (Nupro, SS-4DAL), and a flow meter (model 10A1227, Bailey Fischer & Porter) were used for controlling of the system. A low-pressure TFC polyamide composite membrane (TW30-1812-100) from Dow Filmtec Co. was used in this work. Table 2 shows the technical specifications of the applied membrane.

2.3. Procedure

The stock solution was prepared by diluting a predetermined amount of liquid 2-chlorophenol with distilled water. This stock solution was used for the preparation of different feed solutions having a concentration within the range of 100–500 ppm. After adjusting the pH of the feed solution at the desired value, it was pumped into the membrane module. A flow meter, one manual needle valve, and a pressure gauge were installed on the feed flow line. The flow meter was used to measure and control the feed flow rate and the needle valve was used to adjust the flow rate of the feed stream. The pressurized feed stream after passing through the membrane module was divided into two streams; one stream containing low 2-chlorophenol concentration (permeated stream) and another stream with higher 2-chlorophenol concentration (rejected stream). Both rejected and permeated streams were recirculated into the feed tank for keeping the feed concentration practically constant and establishing a continuous process in a quasi-steady state condition. In the reject line, a manual needle valve was installed for adjusting the feed line pressure. Sampling from the permeate stream was performed using the diaphragm valve at different times. In each run, the system was allowed to operate for about one hour (equilibration time) before samplings were carried out. It was determined that after one hour, permeate and reject streams flow rates and the streams concentrations reached their steady-state conditions. Fig. 2 shows the permeate flux and reject stream flow rates vs. "time on the stream" at 476 and 544 kPa feed pressure, 100 ppm feed concentration, pH 5, and $1.953 \times 10^{-5}\ m^3/s$ feed flow rate. This figure shows a steady flux decline within the initial 50 min; after that the changes become negligible and the system shows a steady response. This flux decline is due to the formation and development of the concentration boundary layer on the membrane surface. Fig. 3 shows the concentration of the permeate stream during the initial period of the experiments until equilibrium is established. The concentration of the permeate stream increased continuously during this period and reached a steady value within 60 min. It seems that the time needed for attaining the steady concentration is greater than that for permeate flux. This is due to the solution-diffusion mechanism of transport in RO membranes, which implies that the time for achieving steady concentration is more than that for permeation flux. In addition, as reported by other researchers, due to the increase in concentration on the surface of the membrane, because of concentration polarization, the concentration of the permeate stream increased [28,38].

Table 2
The technical specifications of the applied membrane [37]

Characteristic	Value	Unit
Manufacturer	Dow/Filmtec	–
Structure type	Polyamide TFC	–
Active membrane area	4.8	ft^2
Diameter	1.75	in
Membrane length	10	in
Max. working pressure	300	psi
Max. flow rate	7.6	L/min
Max. feed water temperature	45	℃
Maximum pH range	2–11	–

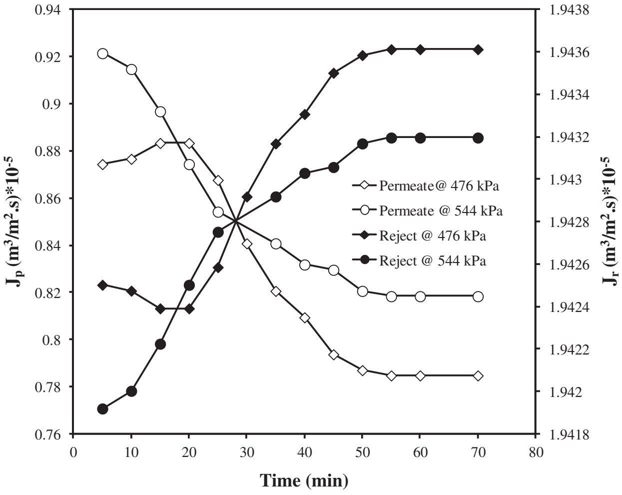


Fig. 2. Permeate and reject flux and concentration vs. time on stream for 100 ppm 2-chlorophenol feed concentration, pH 5, $1.953 \times 10^{-5}\ m^3/s$ feed flow rate and at room temperature.

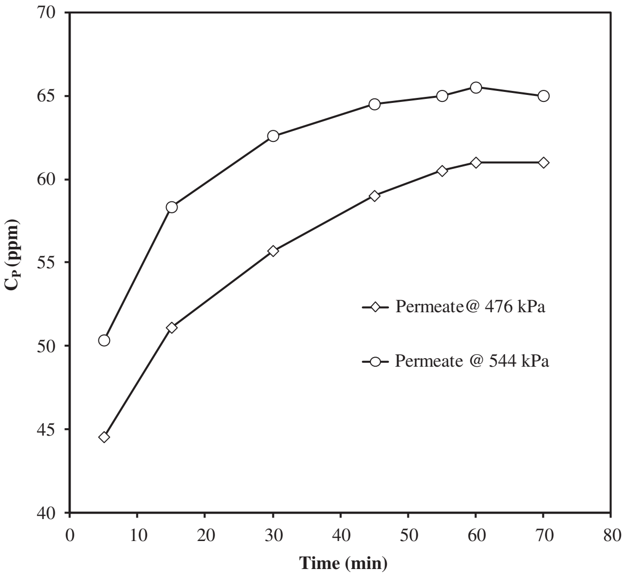


Fig. 3. The influence of time on stream on the permeate concentration for a 100 ppm feed, pH 5, $1.953 \times 10^{-5}\,m^3/s$ feed flow rate and at room temperature.

The permeate flux was calculated as follows:

$$J_p = \frac{Q_p}{A} \tag{7}$$

where J_p is the permeate flux, Q_p is the permeate flow rate, and A is the active membrane area [1].

Prior to each experiment, the membrane was washed by circulating distillated water to remove any adsorbed contaminants during the storage, handling, and mounting. The temperature of the experiments was at room temperature (25 ± 2˚C). The experiments were carried out at four different feed flow rates (0.416×10^{-5}, 0.733×10^{-5}, 0.933×10^{-5} and $1.953 \times 10^{-5}\,m^3/s$), seven feed pressures (136–544 kPa), and five pH values (pH 2–10). The concentration of 2-chlorophenol in the streams was determined using sensitive 4-aminoantipyrine method at 460 nm [39] using a visible-range spectrophotometer (Cecil, model CE1010).

3. Results and discussion

3.1. Membrane adsorption

For testing the adsorption of 2-chlorophenol onto the polymeric membrane, 50 cm^2 of the membrane was cut into small pieces and was taken into contact with 250 ml of 2-chlorophenol solution with different concentrations at a closed vessel. The mixture was stirred at 300 rpm and at room temperature. The sampling was done at different times until the equilibrium and the samples were analyzed for 2-chlorophenol content. Fig. 4 shows the results of this test. This figure shows that the amounts of adsorbed 2-chlorophenol for the concentrated feeds are more than that for the dilute feeds.

3.2. Effect of feed pressure

The influences of feed pressure on 2-chlorophenol rejection at different concentrations are shown in Fig. 5(A)–(D). These results were obtained at $1.953 \times 10^{-5}\,m^3/s$ feed flow rate, room temperature, and at different pH values. The results showed that the rejection increases steadily as the pressure increased. These figures show that 2-chlorophenol rejection increases with pressure from 136 up to 408 kPa (critical pressure) and then decreases as the pressure is increased from 408 to 554 kPa. When the pressure is increased up to the critical pressure, the rejection is increased. According to Eq. (1), by increasing feed pressure, the water flux across the membrane also increases. Thus, increasing the feed pressure causes enhancement in the permeate flux, which increases the rejection coefficient.

Fig. 6 shows the effect of pressure on the permeate flux for different feed concentrations at two different pH values. The figure shows that at low feed pressures, for all concentrations, the permeation fluxes are low and close to each other, which shows a low concentration polarization. As the feed pressure is increased, the differences between permeation fluxes become considerable and as the feed concentration is

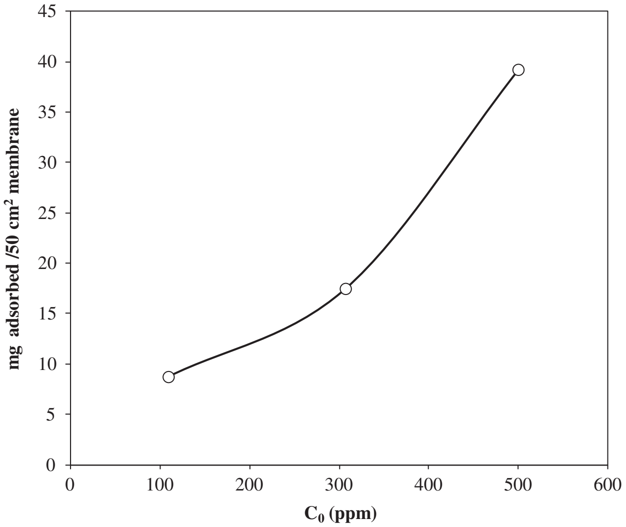


Fig. 4. Equilibrium adsorption of 2-chlorophenol onto the membrane at different initial concentrations.

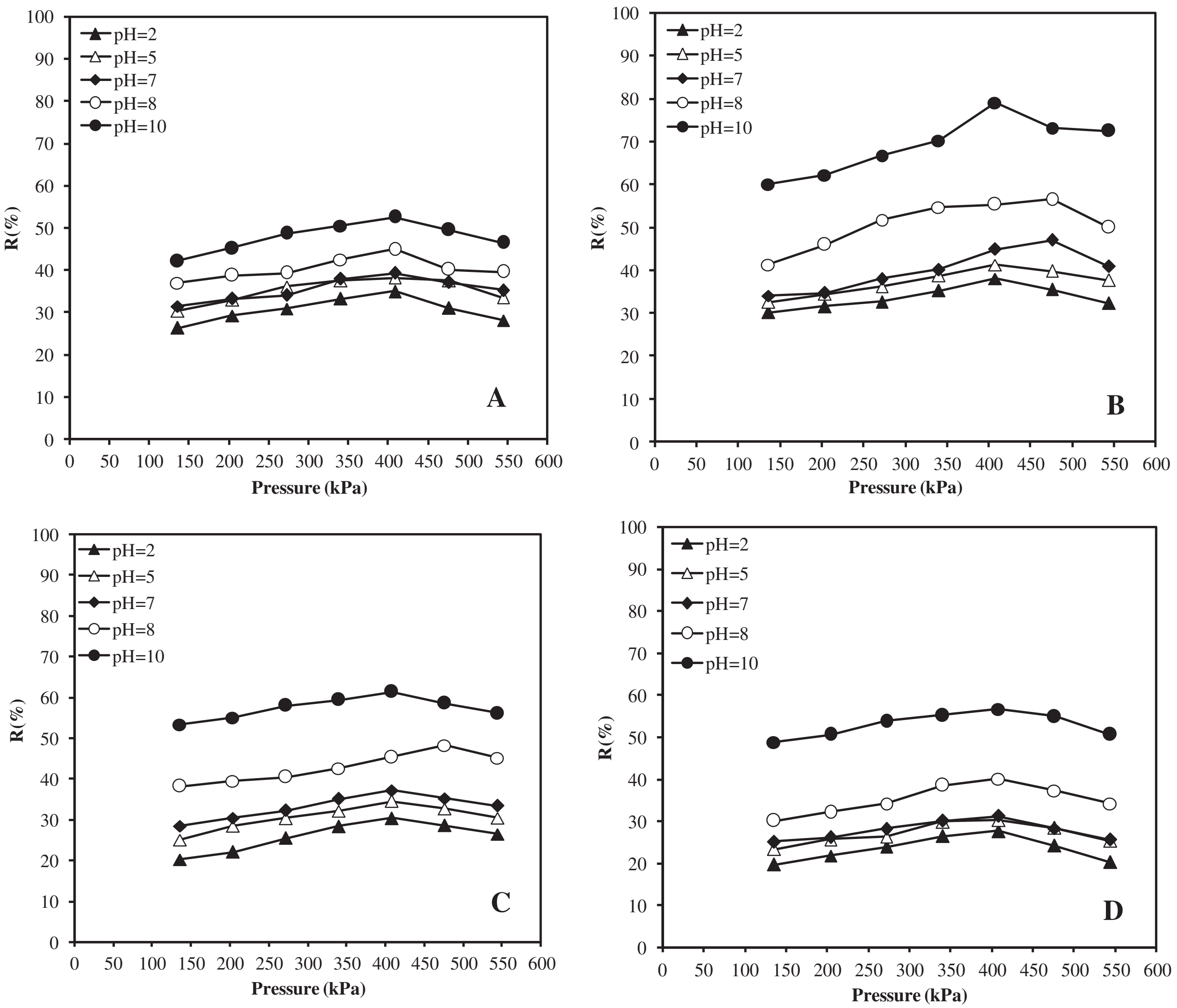


Fig. 5. Rejection percent vs. pressure at $1.953 \times 10^{-5}\,m^3/s$ feed flow rate and room temperature, (A) 100 ppm, (B) 200 ppm, (C) 300 ppm, and (D) 500 ppm concentrations.

increased, the permeation flux is decreased, respectively. The presence of the critical pressure could clearly be observed in these figures. By increasing the permeate flux, the solute which is rejected accumulates on the membrane surface and concentration of the solute on the membrane wall becomes higher than that in the bulk stream. Thus, by increasing the permeation through the membrane, the concentration polarization is also increased [26].

Fig. 7 shows the effect of pressure on concentration factor for a 100 ppm feed concentration, $1.953 \times 10^{-5}\,m^3/s$ feed flow, and at room temperature. This figure shows that concentration polarization is increased with an increase in pressure and as it was previously shown in Fig. 6, the permeate flux increased with increasing pressure. According to Eq. (2), solute transport occurs due to the concentration gradient across the membrane, so the accumulated 2-chlorophenol molecules at the polarized layer are adsorbed on the membrane surface because of the solute concentration difference between the concentration of the solute at membrane wall and permeate side and then 2-chlorophenol molecules pass through the membrane which reduces the solute rejection. This is in agreement with the results obtained in adsorption experiments (Fig. 4) and confirms that the concentration polarization is the reason for decrease in apparent rejection of the organic solute.

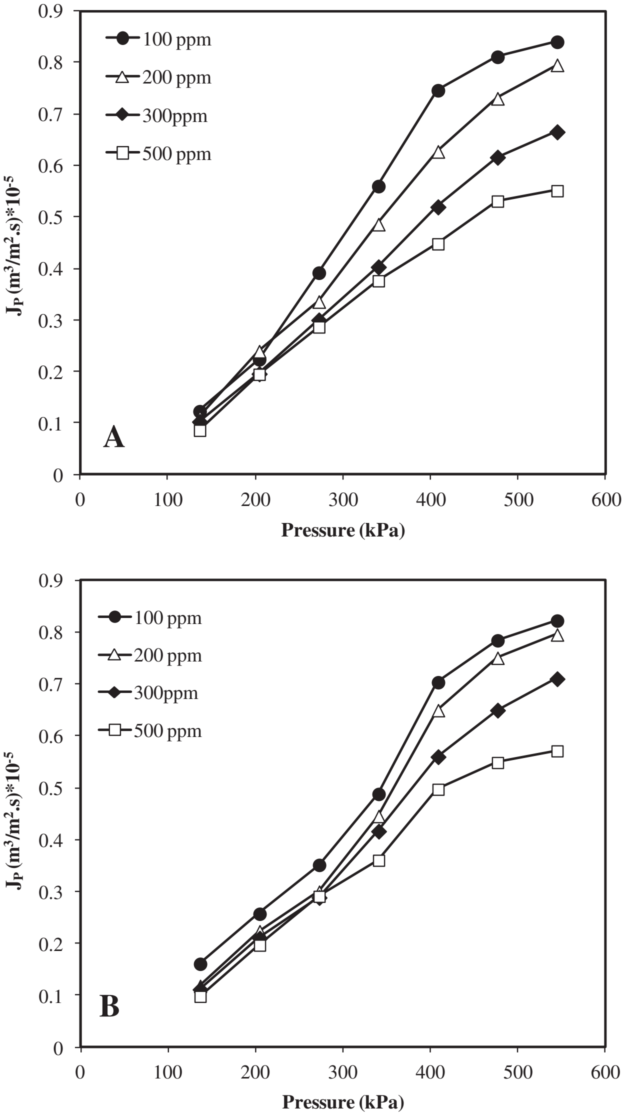


Fig. 6. Effect of pressure on permeate flux at 1.953×10^{-5} m^3/s feed flow rate and room temperature, (A) pH 5, (B) pH 8.

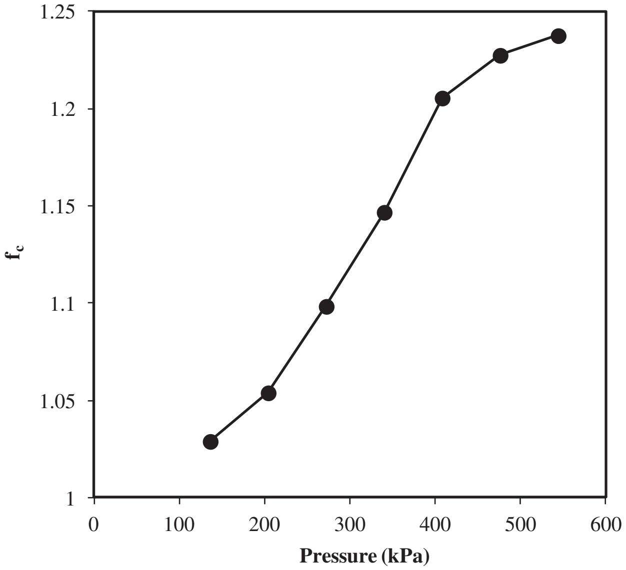


Fig. 7. Concentration factor vs. pressure at 100 ppm feed concentration, 1.953×10^{-5} m^3/s feed flow rate, and room temperature.

In the pressure range of 136 up to 408 kPa, the feed flow rate is sufficient to overcome the concentration polarization effect, and the effect of pressure is stronger than the effect of concentration polarization on the rejection and consequently the rejection is increased. As the pressure is increased, the slope of the curve is decreased. At feed pressure of 408 kPa and higher, the concentration polarization effect becomes very strong and feed flow rates were unable to overcome it and thus the rejection was decreased. If the turbulency of the feed becomes lower than a certain level, it cannot reduce the boundary layer thickness and consequently the effect of concentration polarization [26]. It has been reported that for organic solutes that have strong interaction with the polymeric membrane materials, retention may reduce with pressure [40]. An increase in water permeation through the membrane with an increase in pressure increases 2-chlorophenol permeation, which results in the co-permeation of the solute with solvent (friction theory) and thus a decrease in rejection is observed at high pressures [41].

In the studies that were carried out on chlorophenols removal using nanofiltration and RO membranes, the effect of pressure on rejection have had different trends. In the work of Hidalgo et al. the rejection increased with an increase in pressure [1], but Nghiem et al. reported that an increase in pressure decreased chlorophenol rejection [40].

3.3. *Effect of feed pH and feed concentration*

The influence of feed pH and feed concentration on 2-chlorophenol rejection is shown in Fig. 8. All of these experiments were carried out at 1.953×10^{-5} m^3/s feed flow rate and room temperature. The maximum rejections were observed for 200 ppm feed concentration. According to the results, by an increase in pH of the feed, the rejection was increased but this increase is not uniform. At pH values lower than 7, the rejection is less sensitive to the feed pH but as the feed pH is increased over 7, a sharp increase in rejection was observed for all feed concentrations. The maximum rejection was measured at pH 10 for a 200 ppm feed

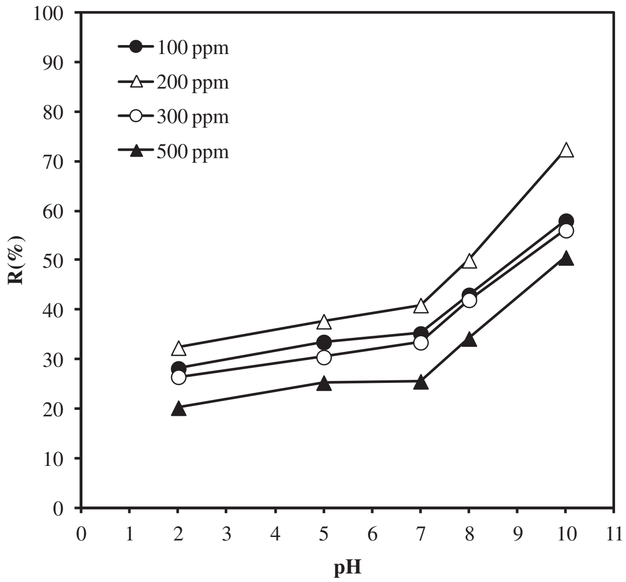
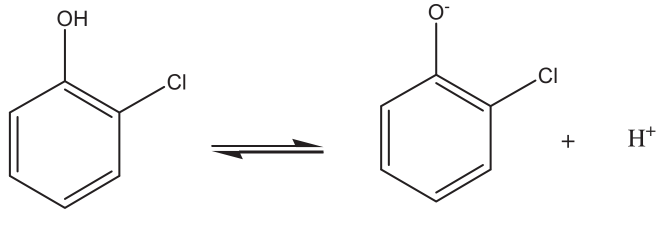


Fig. 8. Effect of feed pH on rejection of 2-chlorophenol at $1.953 \times 10^{-5}\,m^3/s$ feed flow rate, room temperature, and various feed concentrations.

concentration. The reason is completely clear. At alkaline solutions, 2-chlorophenol molecules become ionized to their ionic form and this ionic form is insoluble in the membrane. Then the rejection was increased.

At low pH values, 2-chlorophenol is mainly at its molecular state. In this form, the molecules can be adsorbed on the membrane surface through hydrogen bounding or hydrophobic interactions and is dissolved in the membrane, and thus, the solute rejection is decreased. Polyamide has polar nature and can act as both hydrogen acceptor and donor [36]. Since the membrane used in this study is polyamide, then the hydrogen bounding is the most probable interaction between the membrane and the solute. As the pH of the feed is increased, 2-chlorophenol is dissociated and forms the negatively charged 2-chlorophenolate according to the following equilibrium reaction [13]:



$$pk_a = 8.5 \qquad (8)$$

The concentration of the 2-chlorophenolate ions depends on the pH of the solution. The more pH values give more 2-chlorophenolate ions.

These phenolate ions cannot be dissolved in the polymeric membrane. Then the rejection is decreased considerably at higher alkaline solutions.

Hidalgo et al. reported similar results for the rejection of 4-chlorophenol using three nanofiltration membranes that confirms our measurements [1].

As the feed concentration was increased from 100 to 200 ppm, the rejection was increased. For the concentrations higher than 200 ppm, the rejection was decreased so that the least rejection was observed for 500 ppm feed concentration. The most important reason for such a behavior is the effect of concentration polarization. According to Eq. (2), the solute transport through the membrane is proportional to the concentration gradient between the permeate side and membrane wall. As the concentration is increased from 100 to 200 ppm, no considerable concentration polarization was formed and the feed flow rate is sufficient to overcome the concentration polarization effect and can decrease the solute accumulation on the membrane surface. However, for the feed concentrations higher than 200 ppm, the feed flow was not sufficient to decrease the concentration polarization effect. As the feed flow rate is increased, the turbulency at the membrane surface is increased which caused the decrease in the effect of concentration polarization at the membrane surface. Consequently, the permeate flux is increased which resulted a permeate stream with lower solute concentration (Eq. (2)).

As the feed concentration is increased, the permeation flux across the membrane decreases as shown in Fig. 6(A) and (B) for different concentrations at $1.953 \times 10^{-5}\,m^3/s$ feed flow rate, room temperature, and two pH values. Fig. 6 illustrates that with an increase in feed concentration, the permeate flux decreases for both pH values, as the other researchers were reported [1].

At feed concentrations more than 200 ppm, the concentration polarization increases considerably

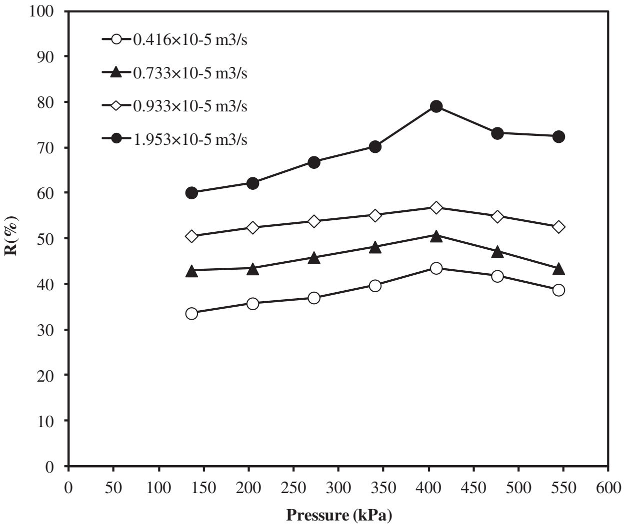


Fig. 9. Effect of feed flow rate and feed pressure on rejection of 2-chlorophenol at pH 10, room temperature, and 200 ppm feed concentration.

because at higher concentrations more solutes accumulate on the membrane surface that causes an increase in the solute transport and consequently a decrease in solute rejection according to Eq. (2).

Bolong et al. worked on bisphenol A removal by a hollow fiber nanofiltration membrane and reported that the rejection was decreased with an increase in feed concentration [42].

3.4. Effect of feed flow rate

The influence of feed flow rate on rejection of 2-chlorophenol is illustrated in Fig. 9. These experiments were carried out at 200 ppm, pH 10, and room temperature. The figure shows the importance of the effect of concentration polarization on 2-chlorophenol rejection. The highest rejections were obtained at 1.953×10^{-5} m^3/s while the lowest were at 0.366×10^{-5} m^3/s. The results showed a direct dependency between the rejection and feed flow rate in the range of current study. The most important effect of the feed flow rate on rejection is related to a decrease in concentration polarization via increase in flow turbulency at the membrane surface, which was discussed earlier. Moreover, it was found that the effect of feed flow on the rejection value in the range of 0.366×10^{-5}–0.933×10^{-5} m^3/s was nearly 3.5 times more than that for the range of 0.933×10^{-5}–1.953×10^{-5} m^3/s. This shows that the increase in the feed flow rate over 1.953×10^{-5} m^3/s is not very effective.

4. Conclusions

The effects of feed flow rate, pH, pressure, and concentration on 2-chlorophenol rejection by a low pressure RO membrane were studied. Results of feed pressure showed that with pressure increasing from 136 to 408 kPa, the rejection increases and for higher pressures the rejection decreased. In addition, when pH was increased from 2 to 10, the rejections were continuously increased. The influence of feed concentration on the rejection was different and from 100 to 200 ppm, the rejections were increased but for higher concentrations, rejections were decreased. In the case of feed flow rate, with an increase in feed flow, the rejections were increased. This study showed that the RO process is an effective method for 2-chlorophenol removal from aqueous wastewaters.

Symbols

A — membrane area (m^2)
A_w — water permeability (m/Pa s)
C_b — solute concentration in the bulk solution (kg/m^3)
C_F — solute concentration in feed stream (ppm)
C_m — solute concentration at the membrane wall at feed side (kg/m^3)
C_P — solute concentration in permeate stream (ppm)
f_c — concentration factor (dimension less)
J_p — permeate flux (m^3/m^2 s)
J_r — reject flux (m^3/m^2 s)
J_s — solute flux (kg/m^2 s)
J_v — Solvent flux (m^3/m^2 s)
J_w — water flux (m^3/m^2 s)
K — mass transfer coefficient (m/s)
L_s — solute transport parameter (m/s)
M — polarization module (dimensionless)
Q_p — permeate flow rate (m^3/s)
R — rejection (dimensionless)
V_c — reject volume (m^3)
V_f — feed volume (m^3)
V_p — permeate volume (m^3)
ΔP — hydraulic pressure difference (Pa)
$\Delta\pi$ — osmotic pressure difference between feed and permeate side of the membrane (Pa)

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