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Performance studies of phosphorus removal using cross-flow nanofiltration

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ABSTRACT

Over-enrichment of phosphorus in water bodies can have a serious impact on aquatic life. In this study, the removal of phosphorus from aqueous solution was investigated with a cross-flow filtration process system using nanofiltration membranes (NF and NF90). The effect of different operating parameters, such as pressure, temperature and pH of the model solution, was studied. The result obtained from this study showed a near complete removal of phosphorus, from the feed solution, i.e. 99.9% rejection for NF90 membrane and 99.2% for NF membrane. It was found that the rejection of phosphorus decreased as the pressure and temperature increased resulted from the concentration polarisation and diffusivity of phosphorus, respectively. An increase in pH of the feed solution gave a higher phosphorus rejection. The permeate flux increased with pressure, temperature and pH for both membranes tested. Amongst the parameters investigated, pH had the most significant effect on the rejection of phosphorus.

Keywords: Cross-flow filtration; Flux decline; Isoelectric point; Membrane; Phosphate ion

1. Introduction

Phosphorus is an important element for all living organism. While it plays an important role in human's daily lives, phosphorus in excess can cause a major impact to the environment. It is found that as little excess as 100 μg/L of total phosphorus will cause enrichment of water bodies [1]. This will lead to eutrophication and result in an increased growth of algae and aquatic weeds leading to water quality decay and imbalance in aquatic populations. Human activities, such as deforestation, phosphorus mining and agricultural practices are, the source of these enrichments which caused as much as 75% of the phosphorus being stored in soil and freshwater bodies as compared to pre-industrial era [2].

There are several methods that are ready available for removing phosphorus from liquid sources, such as chemical precipitation and biological treatment [3]. However, these methods are sensitive to temperature and pH and produce sludge that requires further treatments. A more recent method to remove phosphorus pollutant from waste stream is through

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membrane filtration, in specific, nanofiltration. With the molecular weight cut-off (MWCO) of 150–1,000 Da [4], nanofiltration is a technology which ranges between ultrafiltration and reverse osmosis. Nanofiltration adopts the combination of sieving and diffusion methods, which are found in ultrafiltration and reverse osmosis, respectively, to produce permeate flux and retain organics or multivalent salts at an operating pressure lower than that of reverse osmosis [5]. Moreover, the shear stress experienced by ions in a cross-flow filtration is lower than that of a dead-end filtration system because in cross-flow filtration system, the liquid flow is parallel to the membrane surface [6]. Hence, cross-flow filtration has a higher rejection with respect to dead-end filtration system. Becht et al. [7] found that cross-flow filtration unit gave a higher permeate flux and lower flux variance as compared to dead-end stir cell.

Till date, there is only a handful of researcher reported the removal of phosphorus from liquid feed. Visvanathan and Roy [3] achieved more than 95% removal of phosphorus from a feed solution containing 2–10 mg/L of phosphorus in a pressure range of 4–10 bar using Desal-5 membrane. Niewersch et al. [8] used three different membranes (Desal-5, NP030 and MPF34) to remove phosphorus in a concentration of 3–5 g/L, and it was reported that MPF34 gave the highest rejection of about 80% at a pressure and temperature of 25 bar and 25°C, respectively. Upon further studies, Niewersch et al. [9] found that DK5, DL and NF270 achieved the highest phosphorus rejection of less than 90% at a pH of 4 and phosphorus concentration of 10.5 g/L. Leo et al. [10] demonstrated that phosphorus rejection of above 70% can be obtained using NF90 at a pH less than 2 from the phosphorus solution of concentration 2.5 g/L. The latest study by Blöcher et al. [11] combined a wet oxidation and nanofiltration to recover 54% of phosphorus using DL membrane. It is noted that these researchers used dead-end stir cell in their process studies, and there are only few data available in the literature about the removal of phosphorus using nanofiltration membrane in a cross-flow system. Therefore, the aim of this work is to investigate the feasibility of nanofiltration membrane in the removal of phosphorus from liquid feed via a cross-flow system. Three main operating parameters, representing pressure, temperature and pH, were studied to optimise the performance of NF and NF90 membranes in the phosphorus rejection. The surface properties of both membranes were characterised by contact angle goniometer, atomic force microscope and zeta potential.

2. Materials and methods

2.1. Nanofiltration membrane and chemicals

The membranes used in these experiments were NF and NF90 supplied by Film Tec (Dow). The functional groups of NF and NF90 membranes were polypiperazine amide thin film composite and polyamide thin film composite, respectively. According to the manufacturer, retention of magnesium sulphate for both the membranes is greater than 99%. Other properties of the membranes are shown in Table 1. Phosphate salt used in this study (NaH_2PO_4) with purity of 99% was provided by R&M Chemicals. In order to adjust the pH environment of the bulk solution to acidic or basic condition, 1 M sodium hydroxide (NaOH) and 0.18 M sulphuric acid (H_2SO_4) were added. Other chemicals used were supplied by HACH in the test kit including phosphate acid, potassium persulphate, sodium hydroxide and molybodovanadate reagent. All chemicals were used without any purification.

2.2. Membrane preparation

The surface area of the membranes tested was 0.082 m^2. Prior testing, the cut membranes were soaked overnight in deionised water (0.055 μS/cm) to remove any chemicals originated from the manufacturing or additives used for stabilisation [12,13]. Membrane compaction was then carried out at a pressure of 16 bar at 25°C with deionised water. This was done for both NF and NF90 membranes in order for the membranes to reach stationary membrane behaviour.

2.3. Membrane characterisation

The membranes were soaked in deionised water and dried in a desiccator, before hydrophobicity tests. Hydrophobicity of the membranes was determined by sensile drop method using a contact angle goniometer (Rame-Hart Instrument 250). The air/water/membrane surface contact angle was measured at first second after the water droplet was dropped. The contact angles were obtained from the average of 10 measurements. The pure water flux of the membrane was tested at pressures ranged from 5 to 15 bar at 25°C using dead-end a stir cell (Sterlitech HP4750). The properties of NF and NF90 are summarised in Table 1. Atomic Force Microscopy (AFM) (XE-100 Park System) was performed on both the membranes using tapping mode to study the membrane surface topography. This method uses the tip on the AFM-cantilever to oscillate close its resonance frequency when the tip goes into

Table 1
Specification of membranes used in experiment

	NF	NF90
Manufacturer	Film tec (Dow)	Film tec (Dow)
Functional group[a]	Polypiperazine amide thin-film composite	Polyamide thin-film composite
Contact angle (°)[b]	45.29	88.75
Range of pure water flux ($g/min\,cm^2$)[c]	0.103–0.107	0.145–0.157
Operating pressure (bar)[a]	41 (max)	41 (max)
Operating temperature (°C)[a]	60 (max)	60 (max)
pH range[a]	3–10	3–10

[a]From manufacturer. [b]Contact angle was done in the laboratory. [c]Pure water flux was tested at 25°C and 15 bar using a dead end stir cell by Sterlitech HP4750.

close proximity with the membrane surface (Thormann Ultramicroscopy 2009). Field emission-scanning electron microscopy (FE-SEM) is a common imaging tool for membrane structural and surface analysis. The model of the instrument used is Hitachi SU8010. Membranes selected for FE-SEM imaging were dried in a desiccator a day prior to testing. Images were taken at 10 k times magnification with an accelerating voltage of 0.5 kV. Zeta potential tests were carried out using Delsa Nano HC by Beckman Coulter at several different pH values using electrophoretic light scattering method. Electric field was applied to the charged particles in the suspension which caused the particles to move in the direction of an electrode opposed to their surface charge. Zeta potential was calculated using Smoluchowski equation, Eq. (1),

$$v_E = 4\pi\varepsilon_0\varepsilon_r \frac{\varsigma}{6\pi\mu}(1 + kr) \quad (1)$$

2.4. Flux and rejection studies

The filtration experiments were carried out in a laboratory scale which comprises a cross-flow membrane unit, a diaphragm pump, mechanical stirrer and a 10-L feed tank with a built-in heat exchanger. The performance of NF and NF90 membranes were tested at different temperature, pressure and pH to determine their effect on phosphorus rejection and flux in a cross-flow membrane filtration unit. The model phosphorus solution of 120 ppm was prepared, and the solution mimics the phosphate ion concentration of rinse water from an automotive assembly plant [14]. Mechanical stirrer was used for mixing the chemicals to produce a well-mixed solution. Besides that the stirrer was also used to ensure that heat was not localised and well distributed across the whole solution. The percentage of phosphorus rejection was determined using Eq. (2).

$$R = \left(1 - \frac{C_{\text{permeate}}}{C_{\text{initial concentration}}}\right) \times 100\% \quad (2)$$

The experiments were carried out over the temperature range 25–55°C and pH 3–8, while pressure varied between 5 and 15 bar. The pressure built-up was controlled by adjusting the return valve. Heater and temperature controller were installed in the unit to control the operating temperature, while pH was adjusted using 1 M NaOH and 0.18 M H_2SO_4. A schematic representation of the filtration system is shown in Fig. 1. Permeates were collected at the end of 1 h.

3. Result and discussion

3.1. Membrane characterisation

The wetting properties and surface topography of NF and NF90 membranes were characterised with contact angle goniometer and AFM. Fig. 2 shows the contact angle of water droplet on both membranes. The average contact angles of water on NF and NF90 membranes were found to be 45.29 and 88.75°, respectively, revealing that NF membrane is more hydrophilic than that of NF90 membrane. It is known that the water flux for hydrophobic membrane is higher as compared to hydrophilic membrane [15]. This finding supported the higher pure water flux observed in NF90 (Table 1). Akin and Temelli [16] stated that a boundary would be formed when a hydrophobic surface met with a hydrophilic compound, giving the surface a greater selectivity of polar components. Therefore, it is expected that NF90 membrane will perform better in terms of phosphorus rejection as it possesses greater hydrophobicity as compared to NF membrane.

Fig 3(a) shows the AFM image of NF membrane. While NF90 on the other hand (Fig. 3(b)) has an

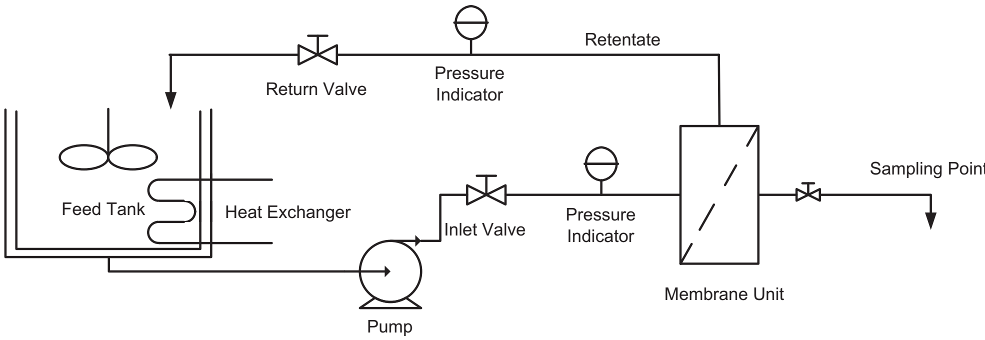

Fig. 1. Schematic diagram of nanofiltration set up.

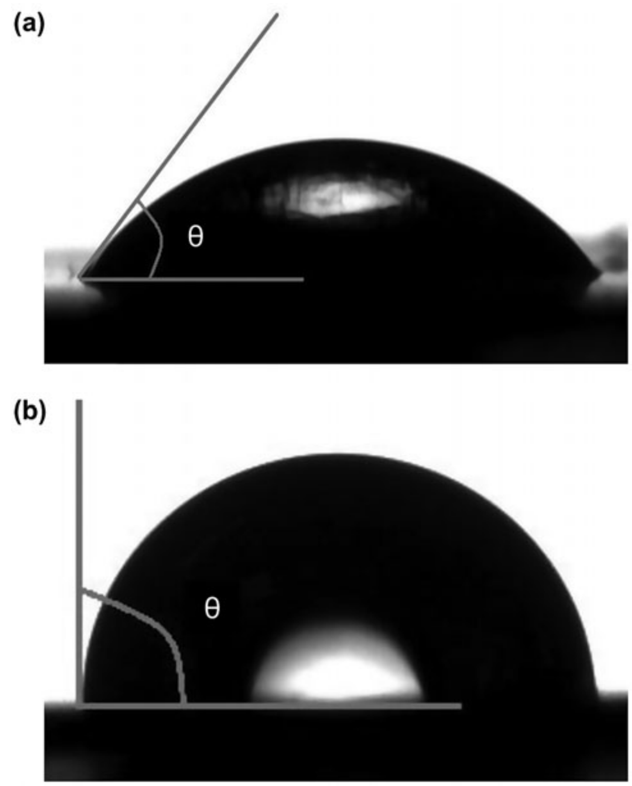

Fig. 2. Contact angel measurement for (a) NF and (b) NF90.

irregular geometry surface which would give the ions more interference while passing through the membrane. Comparing both the images, it is clear that the surface roughness of NF90 membrane is higher as compared to NF membrane and this will offer more surface area for adsorption. A linear relationship can be established between the surface roughness of the membrane and the permeate flux whereby the higher the surface roughness, the higher the permeate flux would be [17]. This explains the higher permeate flux of NF90 membrane as shown in Table 1.

FE-SEM is a common tool to study the surface morphology of a membrane. Fig. 4 shows the FE-SEM images of (a) NF membrane and (b) NF90 membrane. These images were taken at 10 k times of magnification with an accelerating voltage of 0.5 kV. Fig. 4(a) shows that NF membrane has a smooth surface, whereas the surface of NF90 membrane is rough as uneven structure could be observed. These findings are in line with those of AFM analysis which disclosed that the roughness of NF90 membrane is greater than that of a NF membrane.

Zeta potential for NF and NF90 membranes were tested with the solution containing 120 ppm of phosphorus at pH values studied in this paper and the result is shown in Fig. 5. The isoelectric point was found at pH 3.36 for NF membrane and pH 3.61 for NF90 membrane. At isoelectric point, the surface charge of the membrane is 0. At a pH value lower than the isoelectric point, the membranes are positively charged, whereas above this point, the membranes are negatively charged. When the membrane was at the isoelectric point, the selectivity of the membrane would be primarily governed by steric hindrance provoked by the solute size, in other words, sieving effect [18]. The changes in the surface charge were due to the dissociation of the functional groups in the membranes [19]. Functional group of the membranes, i.e. polyamides, contains faintly charged carboxyl group and amine group whereby both are dissociable. Freger et al. [20] claimed that the membrane active layer with strong negative charge at alkaline conditions would have higher concentration of carboxyl group than amine group. As seen in Fig. 5, NF membrane had a higher negative charge as compared to NF90 membrane divulging that the higher concentration of carboxyl group on NF membrane in alkaline condition.

3.2. Effect of pressure

The effect of operating pressure on phosphorus rejection was shown in Fig. 6. The performance of

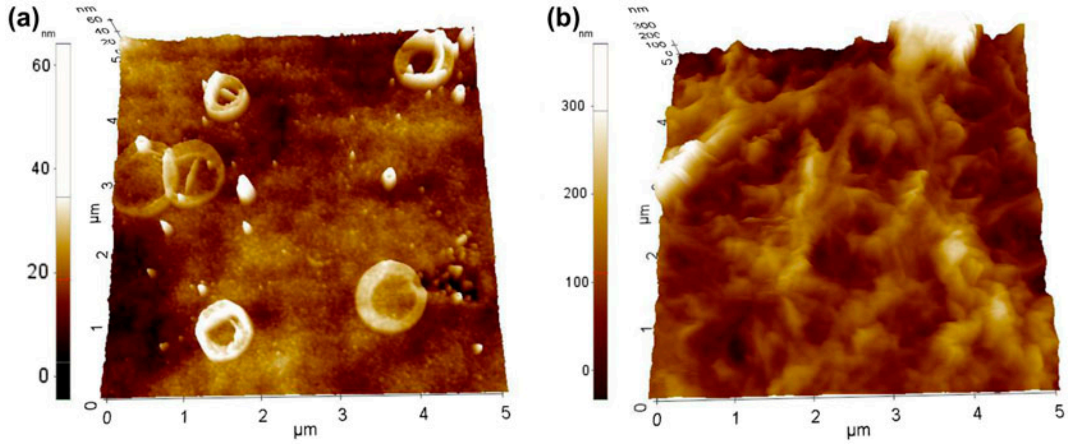

Fig. 3. AFM images for (a) NF and (b) NF90.

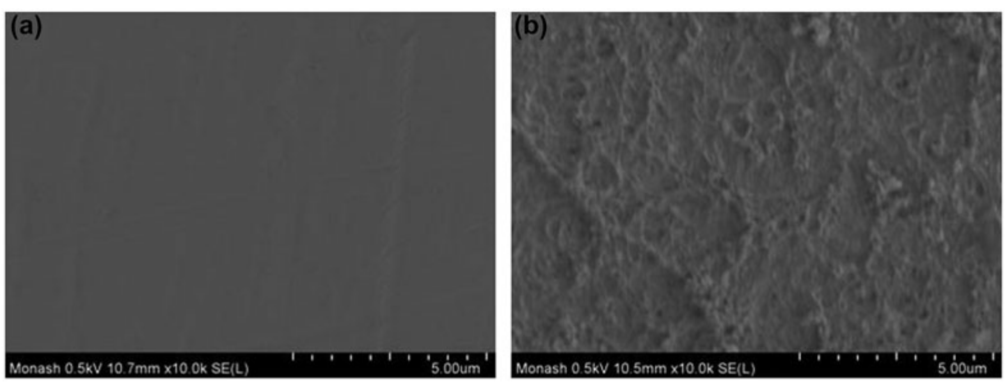

Fig. 4. Surface analysis using FE-SEM on (a) NF membrane and (b) NF90 membrane at 10 k times magnification.

NF90 membrane in removing phosphorus was better than that of NF membrane. Phosphorus rejection using NF membrane showed a peak as the pressure increased, while NF90 membrane showed a decreasing trend with increasing pressure. At the pressure of 5 bar, the transport of phosphorus through the membrane was dominated by diffusive transport while convective transport remained constant [21]. However, as the pressure increased, convective transport became dominant and hence increasing the rejection of phosphorus. As the pressure increased further, concentration polarisation increased and outweighed the convective transport [22]. Hence, the retention of phosphorus decreased. The mentioned reason explains the noticeable peak in Fig. 6 by NF membrane. Possible justification for the reduction in rate of rejection of phosphorus with pressure by NF90 membrane was that the concentration polarisation occurred on the membrane was too high for the diffusive transport of the phosphorus to overcome. Therefore, phosphorus rejection decreased.

Fig. 7 shows the permeate flux of pure water and phosphorus solution at 40°C at varying pressure. From this figure, pure water flux increased linearly with pressure. It was reported by Mehiguene et al. [23] that linearity of the membrane permeate flux signified that concentration polarisation was insignificant. However, in these experiments, the permeate flux deviated from the pure water flux as the pressure increased, indicating that polarisation of phosphorus on the membrane surface contributed to the decrease in the phosphorus rejection as found in this work. This polarisation effect can be explained by membrane zeta potential. In acidic condition, both NF and NF90 membranes were negatively charged. This causes repulsion between the negatively charged phosphate ion and membrane surface, forming a polarisation layer on the membrane surface and hence affecting the permeate flux.

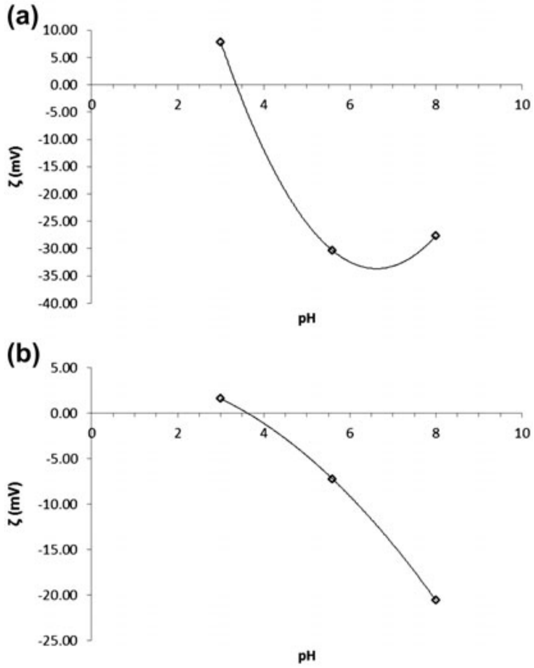

Fig. 5. Zeta potential on (a) NF and (b) NF90 membranes at the pH value studied in this experiment.

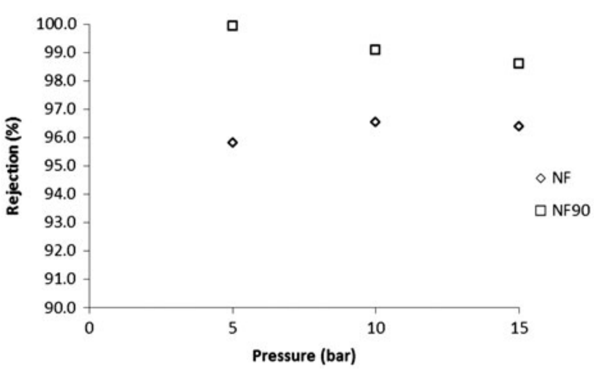

Fig. 6. Effect of pressure on phosphorus rejection of NF and NF90 membranes at 40°C and pH 8.

3.3. *Effect of temperature*

The influence of temperature on the rejection of phosphorus is shown in Fig. 8. Temperature had a greater impact in phosphorus retention by NF membrane than that of NF90 membrane. This might be due to NF membrane was sensitive to the environmental change caused by the changes of temperature. The observed reduction in rejection for NF membrane can be attributed to the diffusivity of phosphorus across the membrane. With increasing temperature, the solutes were thermally excited and more solutes

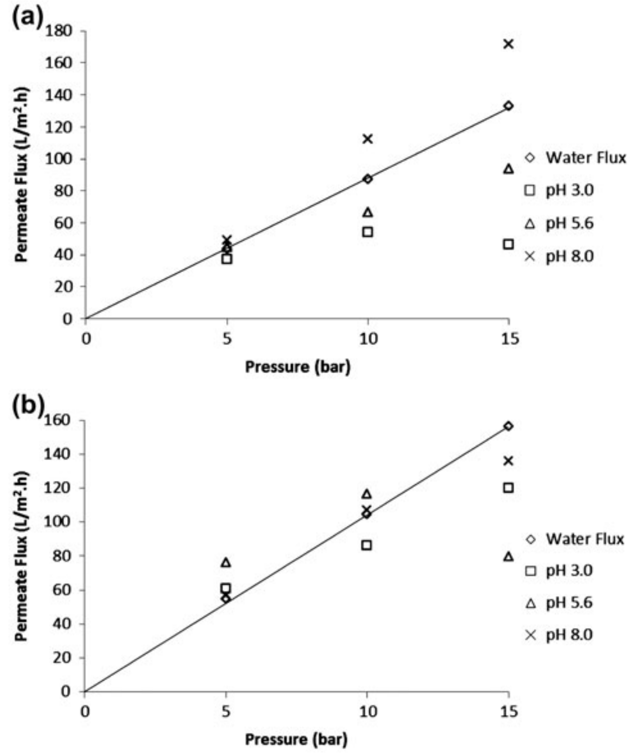

Fig. 7. Effect of pressure on permeate flux of (a) NF and (b) NF90 at 40°C.

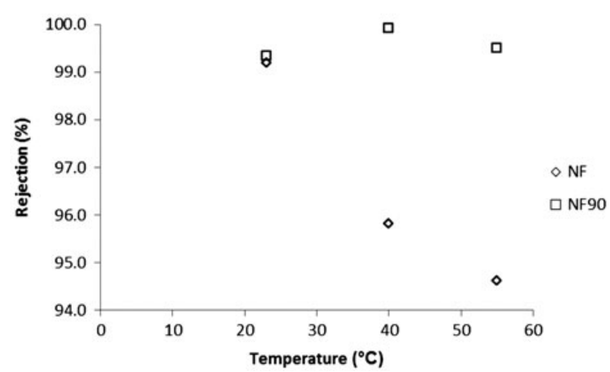

Fig. 8. Effect of temperature on the rejection of phosphorus at 5 bar and pH 8.

diffused across the membrane [24]. Also, the pore size and MWCO of the membranes increased with increasing feed temperature, as they are temperature-dependant [25]. Consequently, this led to the reduction in phosphorus retention as more phosphorus was able to pass through the membranes. On the contrary, the effect of temperature on the retention of phosphorus by NF90 membrane was marginal. This may be due to the difference in functional groups that constitutes the

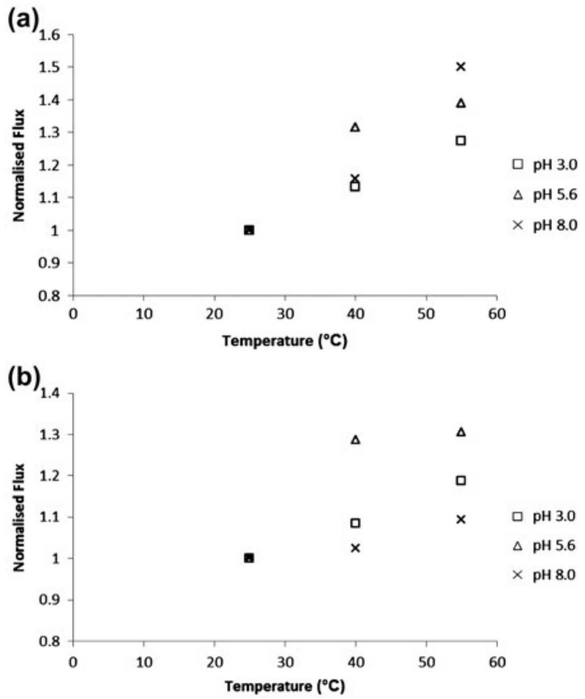

Fig. 9. Permeate flux of (a) NF and (b) NF90 membranes at different temperature (Pressure = 5 bar).

membrane active surface and further studies have to be carried out to identify the reasons.

Fig. 9 shows the normalised flux for (a) NF membrane and (b) NF90 membrane at different temperature and pH. The permeate flux was normalised at 25˚C. There is a general trend observed for both membranes, i.e. permeate flux increased with temperature. We believe that the increase in permeate flux with temperature would be caused by the reduction in solute viscosity, increase in both solvent diffusion coefficient and polymer chain mobility with increasing temperature [26,27].

3.4. Effect of pH

Fig. 10 shows the effect of pH on the retention of phosphorus at 5 bar and 40˚C. The effect of pH on NF90 was more significant than that of NF membrane. This finding demonstrated that the rejection for NF90 membrane is higher than NF membrane at pH 5.6 onwards. At pH 3, which was below the isoelectric point, both of the NF and NF90 membranes were positively charged (referring to Fig. 5). Therefore, there was an electrostatic attraction between positively charged membrane and the negatively charged phosphate ions, leading to a low phosphorus rejection. On the contrary, at pH 5.6 and 8, phosphorus retention

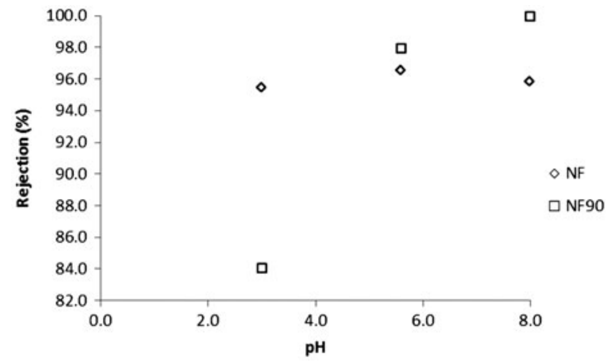

Fig. 10. Effect of pH on removal of phosphorus at 5 bar and 40˚C.

increased due to the repulsion occurred between the negatively charged membrane surface and phosphate ions. However, the rejection decreased at pH 8 for NF membrane. This was caused by an increase in zeta potential at pH 8 as shown in Fig. 5(a), leading to less repulsion between the membrane surface and the phosphate ion. Zeta potential for NF90 membrane as shown in Fig. 5(b), however, decreased at pH 8, and thus leading to the increase in rejection.

Fig. 11 shows the effects of pH on permeate flux for both NF and NF90 membranes. Permeate flux of

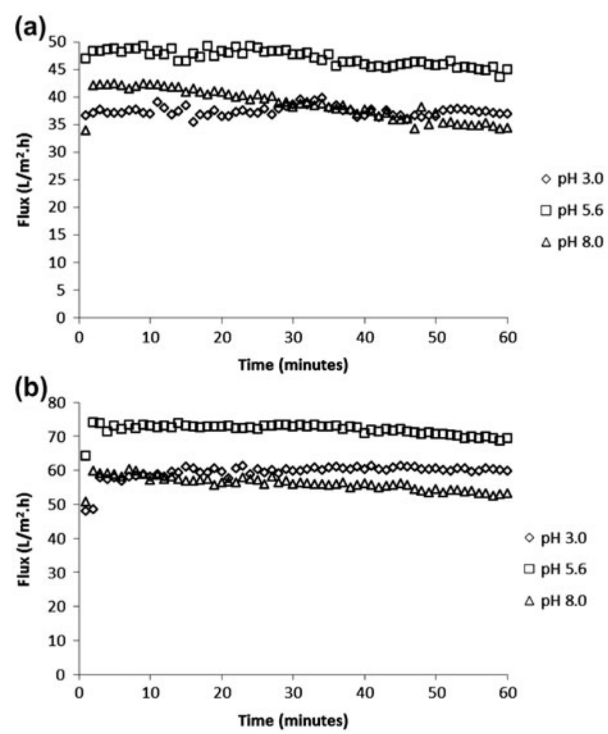

Fig. 11. Flux decline of (a) NF and (b) NF90 at 5 bar and 40˚C and different pH values.

NF90 membrane was higher than NF membrane at all the pH tested. This was caused by the hydrophobicity of NF90 membrane as explained in Section 3.1. According to Kaya et al. [28], pore size can be reduced due to the electrostatic repulsion between the charged functional groups in the membrane. While at isoelectric point, the membrane surface charge is neutral. Therefore, the membrane pore size cannot be reduced, causing the permeate flux to have a lower flux decline. This phenomenon can be observed in Fig 11 and was explained by Childress and Elimelech [29] that when the solution was in alkaline condition, the carboxyl group present at both NF and NF90 membrane surface could deprotonate to $\equiv COO^-$ and bring about a reduction in membrane pore size. Therefore, the flux decline in this condition was found to be higher than the flux at pH closer to the isoelectric point.

4. Conclusions

Phosphorus removal using cross-flow nanofiltration was studied by varying the pressure, temperature and pH of the feed solution using two different commercial membranes, i.e. NF and NF90. The experiments findings show that pressure, temperature and pH had a noteworthy influence on the retention of phosphorus. The phosphorus rejection by NF90 membrane was higher as compared to NF membrane for most of the conditions studied. The retention of phosphorus was found to decrease with an increase in pressure and temperature for both the membranes investigated. It was noted that both membranes performed better in alkaline condition (pH 8) rather than acidic conditions (pH 3.0 and pH 5.6). For both NF membrane and NF90 membrane, the flux increased with increasing pressure and temperature. pH value closed to isoelectric point (pH 3) gave a lower flux decline, due to the limited pore size reduction when the surface charge of the membrane was nearly neutral. From the results obtained in this investigation, cross-flow filtration can be deemed as a feasible approach to remove phosphorus from a liquid feed as the highest rejection of 99.9% was achieved over NF90 membrane. Nevertheless, in order to produce a permeate stream with reasonable phosphorus concentration and flux, the pH of the feed solution cannot be ignored.

Acknowledgement

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Symbols

C_{permeate}	—	permeate concentration
$C_{\text{initial concentration}}$	—	initial concentration
ε_0	—	relative dielectric constant
ε_r	—	electrical permittivity of vacuum
K	—	Debye–Hückel parameter
r	—	particle radius
v_E	—	particles mobility
ζ	—	zeta potential

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