UNIVERSITI PUTRA MALAYSIA

REMOVAL OF NITRATE AND SULFATE FROM CONTAMINATED WATER USING NANOFILTRATION TECHNOLOGY

SEYED MOHSEN HASHEMI ARDESTANI

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By

SEYED MOHSEN HASHEMI ARDESTANI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

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DEDICATION

I lovingly dedicate this thesis to my father who is an honest doctor and my lovely mother that have supported me all the way since the beginning of my life. My lovely wife and anyone who supported me on this path.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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Chairman : Professor Thamer Ahmed Mohamed, PhD
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Nanofiltration systems are mostly applicable in removing small soluble particles in water. This study focuses on the effect conditions such as feed pressure, flow and contamination concentration on the percentage removal and permeate flux. Nitrate and sulfate are hazardous contaminants in water. To simulate them, potassium salts KNO_3 and K_2SO_4 were used to represent contaminants in water sources. In this study, a pilot plant was constructed to test the performance of nanofiltration. The results revealed that the removal of multivalent ions such as sulfate (SO_4^{2-}) ions by NF90 is not totally dependent on the water condition with a removal rate not less than 93%. On the other hand, for monovalent ions such as nitrate (NO_3^-), water condition significantly affects the system performance. The membrane performance time was stabilized around 60 minutes after the operation and membrane fouling occurred after 480 minutes. In addition, NF90 can remove more than 500 mg/L of nitrate from the polluted water which make it acceptable for drinking (Cp<50 mg/L). The impact of sulfate with concentrations of 250 mg/L and 1250 mg/L on nitrate removal (250 mg/L) was examined. Findings indicated that the rate of nitrate removals decreased by 6.2% and 30.6% at sulfate concentrations of 250 mg/L and 1250 mg/L respectively. The Response Surface Methodology (RSM) was applied to investigate the interaction effect of parameters on percentage removal and permeate flux as responses. The parameters were manipulated in five ranges in the presence of sulfate (concentration was 1250 mg/L), such that feed nitrate concentrations were 50, 100, 150, 200 and 250 mg/L, feed flow rates were 400, 600, 800, 1000 and 1200 L/h; and feed pressures were 4, 6, 8, 10 and 12 bar. Among all parameters, pressure played a positive role in the procedure in such a way that percentage removal and permeate flux increased by increasing the pressure. Percentage removal increased rapidly in the initial stages but after 10 bar of pressure, it gradually decreased. Flow variations did not significantly affect the removal rate, but it caused an increase in the permeate flux. The increase of nitrate concentration had a negative effect on nitrate removal where nitrate level increased in the permeate stream. Moreover, the flux did not change considerably by increasing nitrate concentration. Interaction effects were studied to find optimal conditions that lead to providing high quality water, saving energy and improving nanofiltration performance in water treatment industry. The optimum condition by RSM for nitrate removal is predicted to be at 10.14 bar, flow 1200 L/h and nitrate concentration 117.73 mg/L. The respective response for nitrate removal was 93.77% and 92.97 (L/m^2h) for permeate flux. Predicted results by RSM modeling were verified using experimental results where the two sets of outcomes were closed with an error less than 5%. The findings also indicated that the performance of NF90 is acceptable for removing nitrate and sulfate in polluted water with percentage removals range of 81.74-97.89% and 93.48-100% respectively. Results from this approach are applicable in water treatment procedures to improve nanofiltration performance.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENYINGKIRAN NITRAT DAN SULFAT DARI AIR TERCEMAR MENGGUNAKAN TEKNOLOGI NANOFILTRASI

Oleh

SEYED MOHSEN HASHEMI ARDESTANI

Jun 2016

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Sistem penapisan nano kebiasaannya digunakan untuk mengeluarkan zarah-zarah larutan air. Kajian ini memberi tumpuan kepada kondisi-kondisi yang membawa kepada keberkesanan proses ini, seperti tekanan suapan, aliran dan kepekatan pencemaran terhadap peratusan penyingkiran dan flux penyerapan. Nitrat dan Sulfat adalah bahan pencemaran berbahaya di dalam air. Bagi mensimulasikan pencemaran di dalam punca-punca air, garam kalium KNO_3 dan K_2SO_4telah digunakan. Dalam kajian ini, kilang perintis telah dibina bagi menguji keberkesanan sistem penapisan nano. Keputusan kajian telah menunjukkan bahawa penyingkiran ion multivalen seperti ion-ion sulfat (SO_4^{2-}) oleh NF90 tidak sepenuhnya bergantung kepada kondisi air, iaitu dengan kadar penyingkiran tidak melebihi 93%. Bagi ion manovalen pula seperti nitrat (NO_3^-), kondisi air sangat mempengaruhi kebolehupayaan sistem. Kebolehupayaan membran berada dalam keadaan stabil sekitar 60 minit selepas operasi dan pencemaran membran berlaku selepas 480 minit. NF90 juga boleh menyingkirkan lebih daripada 500 mg/L nitrat daripada air tercemar yang boleh digunakan untuk minum (Cp<50 mg/L). Kesan sulfat dengan konsentrasi 250 mg/L dan 1250 mg/L terhadap penyingkiran nitrat (250 mg/L) juga diperiksa. Hasilnya menunjukkan bahawa kadar penyingkiran nitrat berkurangan sehingga 6.2% dan 30.6% bagi kepekatan sulfat, masing-masing 250 mg/L dan 1250 mg/L. Kaedah tindakan permulaan (RSM) telah digunakan untuk mengkaji kesan interaksi parameter-parameter ke atas peratusan penyingkiran dan penyerapan flux. Parameter ini telah di manipulasikan dalam lima julat dengan keberadaan sulfat (konsentrasi 1250 mg/L), iaitu kepekatan nitrat suapan pada 50, 100, 150, 200 dan 250 mg/L, kadar aliran adalah pada 400, 600, 800, 1000 dan 1200 L/h; dan tekanan suapan pada 4, 6, 8, 10 dan 12 bar. Daripada kesemua parameter ini, tekanan memainkan peranan penting yang menyebabkan peratusan penyingkiran dan penyerapan fluk meningkat dengan peningkatan tekanan. Peratusan penyingkiran meningkat dengan cepat pada permulaanya tetapi selepas tekanan pada 10 bar, ia perlahan-lahan menurun. Variasi aliran tidak memberi kesan ketara kepada kadar penyingkiran tetapi ia menyebabkan kesan negatif terhadap penyingkiran nitrat, iaitu kadar nitrat meningkat semasa aliran meresap. Tambahan pula, fluk tidak berubah dengan ketara dengan peningkatan konsentrasi nitrat. Kesan-kesan tindakbalas dikaji untuk mencari kondisi optimum yang membawa kepada penyediaan air berkualiti tinggi, penjimatan tenaga dan penambahbaikan prestasi dalam industri rawatan air. Kondisi optimum oleh RSM bagi penyingkiran nitrat dijangka berlaku pada 10.14 bar, aliran 1200 L/h dan konsentrasi nitrat 117.73 mg/L. Tindak balas penyingkiran nitrat ialah 93.77% dan 92.97 (L/m^2h) untuk resapan fluk. Jangkaan keputusan oleh RSM model telah disemak menggunakan keputusan sebenar dan keputusan tersebut menunjukkan persamaan dengan ralat kurang dari 5%. Keputusan daripada kajian ini juga menunjukkan bahawa prestasi NF90 adalah baik dan sesuai untuk penyingkiran nitrat dan sulfat daripada air tercemar dengan peratus penyingkiran sebanyak 81.74-97.89% dan 93.48-100%. Keputusan dari pendekatan ini adalah bersesuaian untuk prosedur rawatan air untuk memperbaiki prestasi penapisan nano.

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In The Name of GOD, The Most Merciful and Most Beneficent

All praises do to GOD, Lord of the universe. Only by His grace and mercy this thesis can be completed.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science.The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

WHO	World Health Organization
MNDWQS	Malaysian National Drinking Water Quality Standard
STC	FILMTEC™ Membranes, Standard Test Conditions
DES	Design-Expert® Software version 7
NF90	Nanofiltration membrane model 90-2540 from Dow-Filmtec CO
NO_3^-	Nitrate
SO_4^{2-}	Sulfate
K_2SO_4	Potassium sulfate
KNO_3	Potassium nitrate
C_f	Concentration of feed water
C_p	Concentration of permeate water
R%	Percentage removal
Flux	Permeate flux (L/m^2h)
Flow	Feed flow (L/h)
Pressure	Feed pressure (bar)
Concentration	Feed concentration (mg/L)
R^2	Regression
NF	Nanofiltration
RO	Reverse Osmosis
RSM	Response Surface Methodology
UV	Ultraviolet

CHAPTER 1

INTRODUCTION

1.1 Background of the study

In most developing countries, water supply and sanitation are among the most critical concern. It is worth mentioning that water is a limited resource and demands for consumption are increasing rapidly. In spite of the fact that 70% of the earth is covered by water, less than one percent is available as freshwater for human use (Grifen, 2010). Ocean contains the main part of water on this “blue planet”. This water is too salty to drink and inappropriate for many other applications. Around two-thirds of the freshwater accessible on Earth resides in ice caps and glaciers in the frozen form; a small fraction of which is available for potable water uses. Twenty percent of the world’s population is currently living without access to safe water for drinking, personal hygiene, and domestic use. The World Health Organization (WHO) Commission on Health and Environment has reported that waterborne diseases have significant negative health impacts world-wide (Castellano, et al., 2007). The problem of waterborne-associated illnesses has been decreased by the improvements in water treatment methods; although, outbreaks due to viral contamination continue to occur (Anderson et al., 2003). Water pollution means any chemical, physical or biological change in the quality of water. Such changes impose harmful effects on living conditions and environment. Consuming polluted drinking water by people or animals can lead to serious problems in their health and life conditions. Human activities directly and indirectly usually engender water pollutions. Water pollution problem has expanded today and an appropriate policy is required to hinder it; therefore, a study in this area seems inevitable. Water pollution originates from two sources: point and nonpoint sources as demonstrated in Figure 1. Point sources discharge pollutants at specific locations through pipelines or sewers into the surface water. For examples sewage treatment plants, factories, underground mines, oil wells, oil tankers, chemical products in agriculture, especially fertilizer. Nonpoint sources are the ones that cannot follow single sources of discharge. For instance traffic, acid deposition from the air, pollutants that enter the water through groundwater; and pollutants that are spread through rivers. Actually nonpoint pollution is difficult to control and supervised due to the difficulty in tracing its perpetrators (Lenntech and Internships, 2014).

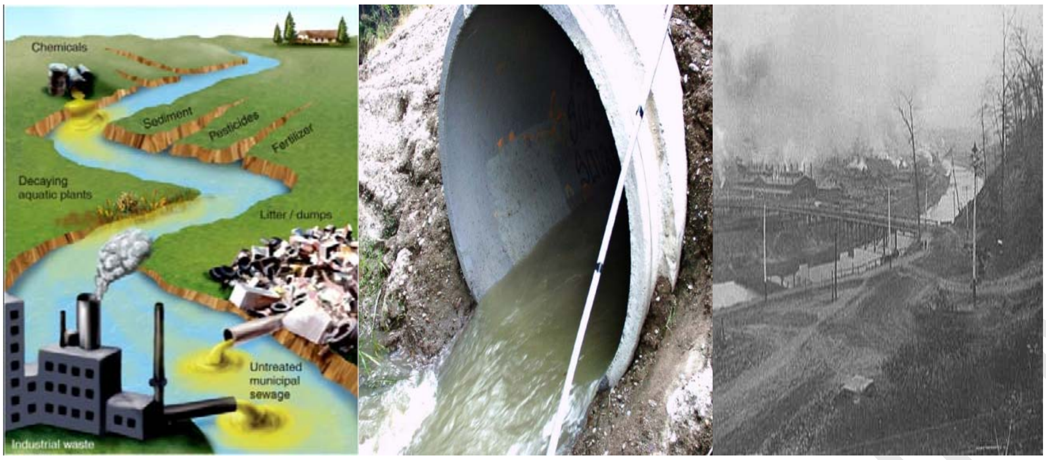

Figure 1.1 : Various sources of water pollutions

The presence of nitrate and sulfate in surface and ground waters is attributed to wastewater outfalls and agricultural runoffs. A huge portion of nitrate and sulfate in surface and ground waters occurs as a result of over-fertilizing, which can result in accelerated growth of algae and weed (eutrophication).

1.2 Problem statement

According to Malaysian National Drinking Water Quality Standard (MNDWQS) and WHO, the maximum concentration of nitrate in drinking water is 50 mg/L as NO_3^- (Jamaludin et al., 2013). Statistical analyses indicated that nitrate is strongly correlated with urea in water samples; which points to the fact that surface and groundwater contamination is mainly because of nitrogenous fertilizers (Di and Cameron, 2002; Di and Cameron, 2007). Industrials wastewater from dairy and swine are reported to contain nitrate more than 200 mg/L (Almeida et al., 1995). Furthermore, some industries produced wastewater containing nitrate greater than 1000 mg/L such as those producing explosives, fertilizers, cellophanes, pectins, and finishing metal industries (Glass and Silverstein, 1999; Peyton et al., 2001; Watanabe et al., 2001). According to recorded data in Bachok Malaysia, the existing nitrate levels are higher than the National Standard at about 150 mg/L as NO_3-N (Roslan et al., 2014). Nowadays, an important area in Malaysia economy is agriculture, where there has been a substantial development in rubber cultivation, oil palm, cocoa, fruits and vegetables. This in return has resulted in the increase usage of nitrogenous fertilizers (Yusoff et al., 1990). For instance, such contaminants exist in the maters of Johor Bahru and Kelantan. The nitrate level in drinking water wells has raised due to high levels of nitrogenous fertilizers used in tobacco and rice agro systems in Kelantan, especially during the months of fertilizer application (Libra et al., 1986; Sharma and Willett, 1994, 1996; Yusoff et al., 1990). Formerly nitrate levels had been below 10 mg/L as NO_3-N coordinates set by National Guidelines for Raw Drinking Water Quality (Huang et al., 2015). The aforementioned study in Kelantan, also illustrated that nitrate contamination is an outstanding and an insistent factor in groundwater. On average, 15% of well water samples contain nitrate concentrations more than the maximum acceptable level (Libra et al., 1986; Sharma and Willett, 1994, 1996; Yusoff et al., 1990). Recently most of the rivers involve more than 10 mg/L NO_3-N and occasionally some exceed 50 mg/L NO_3-N (Horne, 1995). According to Engineering Services Division Ministry of Health Malaysia (2004) the allowable maximum level of sulfate in drinking water is 250 mg/L. All the 106 surface water samples contain sulfate with concentrations ranging from 2 to 358 mg/L (Bast, 1990). In another study on surface water, sulfate was detected in 101 of 154 samples ranging from 15 to 321 mg/L (Bast, 1990). The highest sulfate concentration was detected in the spring water and the results demonstrated that sulfate levels in groundwater were more than 1000 mg/L (Abdalla and Scheytt, 2012). One of the main constituents of fertilizers is potassium sulfate (Xinpeng Xu et al., 2014) termed as sulfate of potash (S.O.P) in the market (Nor et al., 2013). This fertilizer is one of the sources of surface water contamination (Nagarajan and Rajmohan, 2010). According to other recordings as in Gebeng river, Pahang, Malaysia, the rate of sulfate (SO_4^{2-}) was 639.1667 mg/L (Hossain and Mir, 2013). There are reports on samples that are extracted from groundwater pollution in Spain with 275 mg/L and 500 mg/L of nitrate and sulfate concentrations, respectively (Santafé-Moros et al., 2005a). In this study, the removals of contaminants namely, nitrate and sulfate in water were examined using nanofiltration method. The current study investigates the effects of main parameters (feed pressure, feed flow and feed concentration) on percentage removal and permeate flux (produced water) by nanofiltration technology. Furthermore the study aims at determining the optimum conditions for removing nitrate and sulfate.

1.3 Importance of Study

In the recent years, nanofiltration has been vastly used for water treatment all over the world. Concerning the surface and groundwater pollution, a new cost-effective technology is required for water treatment. Homeowners are increasingly concerned on contaminants in their water supply since it may result in serious health, taste and odor problems. To consider the human health, nitrate as an important element must be controlled in drinking water due to its negative effects. This study and modeling was performed by taking into consideration the extended removal of the hazardous ions to produce high quality drinking water. High concentrations of nitrate will cause methemoglobinemia in infants and could cause cancer. Nitrate converts hemoglobin to methemoglobin in the blood and as a result of which oxygen is not carried out in the body cells; and it may lead to death from asphyxiation or oxygen deficiency (Purushotham et al., 2011). In addition, gastrointestinal disorders, indigestion and inflammation of the stomach, multiple digestive tract impairment, abdominal pain, gastroenteritis, diarrhea; blood in urine and feces can all be attributed to nitrate content in potable water (Moore, 1952; Suthar et al., 2009). Sulfate ion is one of major anions occurring in natural waters (Daniels, 1988). A higher sulfate content may also have a laxative effect on water distribution systems (Raju et al., 2011). Sulfate concentration more than 250 mg/L can lead to bitter or medicinal taste of water. High sulfate levels may also be corrosive for plumbing; particularly copper pipe. Applying corrosion resistant plumbing materials like plastic pipe is common practise in areas at high sulfate level water (Lewis, 2004). The respiratory problems and diarrhea may cause due to high concentration of sulfate in drinking water (Maiti, 1982; Majidano et al., 2010). Because of the increase in potable water demands, investigation on alternative methods for preparation of high quality water seems essential. This is not only due to accelerating demand for water resources, but also developments in the standards and increase in water pollution levels. Therefore, engineers have been looking for new methods such as membranous approach that is widely extended. Nanofiltration is one of the most accepted methods for purifying water in order to supply healthy drinking water from surface and ground waters with high nitrate and sulfate concentrations (Van der Bruggen et al., 2001). Regarding water pollution, pilot study and performance review are essential in enhancing new technology and identifying economic advantages in water treatment. The rejection of ions such as nitrate and sulfate via nanofiltration membranes is a complicated procedure. This study leads to more profound understanding of ions behaviour in the process of nitrate and sulfate removal by nanofiltration membrane. Although the surface characteristics and pore size in all membrane are specified, experimental tests on the membrane are inevitable (Torabian et al., 2009). The results of this study are practical and useful in water treatment industry, which can improve nanofiltration performance in water treatment.

1.4 Research objectives

In order to examine the removal of contamination elements to reach drinking water standard by nanofiltration membrane method, a set of variables such as water pressure, flow rate and contaminant concentration were investigated. This method leads to optimizing the operating condition.

This study aims to achieve the following objectives:

1. To design a pilot plant in order to study the effects of feed pressure, feed flow and contaminant concentration on nitrate removal and permeate flux rate by NF90.
2. To test the fouling performance and find the maximum permeated nitrate and sulfate concentration using single membrane element.
3. To determine the optimal conditions for nitrate removal and flux in the presence of sulfate.

1.5 Scope and limitation of the study

Membrane Filmtec NF90 is a commercial nanofiltration membrane with spiral modules in which all subsystems are affected by this technology where all components comply with FDA standards (Dow, 2013; Gray, 2008). The reasons for choosing the proposed membrane, compared to other membrane models, is as it follows:

1) Providing high productivity performance while removing a high percentage of salt, nitrate, sulfate, hardness, iron and organic compounds used in a wide variety of industrial, municipal and commercial water treatment applications (DOW, 2005).
2) High permeate flux output in high quantities by implementation of proper rejection technique.
3) Using low energy leads to the reduction of power consumption and costs.
4) Durability of tool with favorable cleanability features for long life of the element.

5) Agreeable quality of constituent parts.

Chemical fertilizers such as potassium sulfate [K_2SO_4] and potassium nitrate [KNO_3] are among the main causes of water pollution. Due to their importance in water pollution, these contaminants used in this study and the other particles are neglected.

As to the limitations of this study, the positively charged basic cations such as potassium are united with the negatively charged nitrate and sulfate ions to maintain their electric balance. During this procedure, they turn into soil particles which ultimately are dissolved in surface and ground waters. In addition, the manipulated samples of water pollutants used in this study, which exceed the acceptable drinking water standards, comply with the actual pollutants. In order to simulate the concentrations of sulfate and nitrate, potassium salts (K_2SO_4 and KNO_3) were used. Meanwhile, the concentration of sulfate and nitrate were measured before and after each experiment. The samples were manipulated in five ranges for nitrate, and two ranges for sulfate. The nitrate concentrations used are 50, 100, 150, 200 and 250 mg/L; sulfate 250 and 1250 mg/ and the flow rates are 400-1200 L/h. The range applied of feed pressure are 4 to 12 bar as maximum level, based on the proposed recommendations by membrane housing manufacturers and safety requirement (DOW, 2005).

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