

**STUDIES ON FORWARD OSMOSIS-NANOFILTRATION HYBRID SYSTEM
FOR THE DESALINATION OF SALINE WATERS**

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FOR THE DESALINATION OF SALINE WATERS**

by

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***I dedicate** this work to both **my father, mother and my elder sister**, whose unwavering support, guidance, and sacrifices have shaped me into who I am today. Their love and encouragement have been the foundation of all my achievements.*

CERTIFICATE

This is to certify that the thesis entitled “**Studies on Forward Osmosis-Nanofiltration Hybrid System for The Desalination of Saline Waters**” being submitted by **Mr. Ketan Mahawer** to the **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy**, is a Bonafide record of original-research work carried out by him under the guidance and supervision in conformity with rules and regulations of the institute.

The results contained in it have not been submitted in part or full to any other university or institute for the award of any degree/diploma.

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(Ketan Mahawer)

ABBREVIATIONS

ANN	Artificial neural networks
DS	Dataset
DSR	Dataset split-ratio
DT	Decision tree
FS	Feed side
HFFO	Hollow-fiber forward osmosis
MD	Membrane distillation
PAFO	Pressure assisted forward osmosis
RO	Reverse osmosis
SEC	Specific energy consumption
SES	Specific energy saving
SVR	Support vector regression
MLR	Multiple linear regression
SWFO	Spiral-wound forward osmosis
SWNF	Spiral-wound nanofiltration

THE BEGINNING OF MEMBRANE SCIENCE

*“In 1966 Israel invited a California scientist, Sidney **Loeb**, to spend a year at Ben-Gurion University in Beer-Sheva (the new Hebrew name for Beersheba). Loeb had worked for industry after taking an undergraduate degree in engineering in 1941. Feeling restless, he quit his job at the age of forty and went to graduate school at the University of California at Los Angeles. Like the researchers in Israel, scientists at UCLA had been seeking practical desalination methods. Loeb joined the quest with another student, a Canadian named **Srinivasa Sourirajan**. They developed the first successful **reverse-osmosis process in 1960**— “successful” in the sense that it worked in a laboratory, not that it could be deployed in the real world. Sourirajan soon ran into visa problems and Loeb continued alone, constantly tweaking the all-important membrane. By 1965 the technology had advanced enough that Loeb was able to build a commercial reverse-osmosis plant—the first in the Americas—in Coalinga, a town of about six thousand in the San Joaquin Valley. So thick with salts was its groundwater that residents had always brought in potable water by tanker cars.”*

*The above is the quote of **Charles C. Mann** from the book “**The Wizard and the Prophet: Two Remarkable Scientists and Their Dueling Visions to Shape Tomorrow's World**”.*

ABSTRACT

In the past few decades, rapid industrial and urban development with exponential growth in human population has significantly impacted freshwater reserves, leading to an acute scarcity of usable freshwater. Membrane-based desalination of seawater and industrial saline wastewater has emerged as a promising solution to fulfil freshwater needs. However, conventional membrane technologies, such as reverse osmosis (RO)/nanofiltration (NF) suffer critical performance limitations, including high energy consumption (3 to 6 kWh/m³), water recovery (30-40%) and fouling, which undermine their suitability for viable and large-scale desalination. Therefore, this study proposes osmotically enhanced hybrid systems like Forward Osmosis-Nanofiltration (FO-NF) and Pressure Assisted Forward Osmosis-Nanofiltration (PAFO-NF) as a potential alternative for treating salinity in wastewater effluents. Subsequently, several critical aspects of the module-scale hybrid process are evaluated using experimental and transport modeling studies and investigate the impact of continuous draw solute regeneration in these hybrid systems over standalone NF performance. Furthermore, a unique data-driven modelling for the FO-NF hybrid system is developed to predict the permeate fluxes and concentrations for different draw solutes.

Initially, in transport modelling, Speigler-Kedem model, along with the concentration polarization model, is used to describe the local mass transport in the FO and NF membranes. Following this, the combined mathematical models for FO and NF are integrated into the hybrid FO-NF system. Furthermore, FO-NF process optimization reveals that NF hydraulic pressure and inlet draw concentration control the NF retentate concentration and then simulate the desired operating conditions for the hybrid system when the NF retentate stream is directly used as a draw solution for the FO unit. Further experimental observations shows that standalone NF water recovery degrades with time from 40 to 15.6% due to fouling, while increasing the SEC

from 0.89 to 2.38kWh/m³. At the same time, the FO-NF hybrid system shows consistent water recovery 32.6% and 28.6%, with process SECs 3.26 kWh/m³ for Na₂SO₄ and 4.70 kWh/m³ for MgSO₄ as draw solutes, respectively.

It is also observed that compared to the standalone NF system, the FO-NF exhibits relatively lower water recovery but higher SEC. Consequently, this observation motivates to investigate the potential of PAFO-NF systems through experimental and modeling approaches for enhancing the water recovery by simultaneously reducing SEC. The same FO-NF transport model with minor modification in FO modeling for the PAFO process is utilized for the PAFO-NF modeling. Membrane performance is then simulated for fouling prone feed stream containing 3.5 g/L of CaCl₂ using Na₂SO₄ and MgSO₄ as the draw solutes at a concentration of 40 g/L each. Simulated results under optimized operating conditions show that, FO feed side assisted pressure (ΔP_{PAFO}), FO membrane area (A_{mFO}) and the FO water permeability (A_{FO}) are the key factors for reducing the PAFO-NF process SEC and improving the water recovery, while using Na₂SO₄ as draw solute. Simulations reveal that at 1.0 m² A_{mFO} , increasing ΔP_{PAFO} (from 0 to 7.5 bar) may reduce the SEC from 2.95 to 1.47 kWh/m³ while simultaneously increasing water recovery from 60.5 to 95.54%. In fact, merely increasing the A_{mFO} from 0.25 to 1.0 m² may reduce the process SEC compared to the FO-NF process by up to 63% and increase water recovery to as much as 90 %. Similarly, increasing A_{FO} (1.22×10^{-12} to 3.0×10^{-12} m/Pa.s) may also reduce the process SEC by nearly 42 % while also increasing the water recovery by 45.73%. Alternatively, inlet feed and draw concentrations and inlet feed flowrates have negligible impact on the process SEC. In conclusion, PAFO-NF hybrid process demonstrates significant potential for treating both fouling-prone and non-fouling saline waters and surpasses the FO-NF and standalone NF (under non-fouled conditions) processes by achieving lower SEC under the same operating conditions.

The above transport modeling for the FO-NF hybrid process is complex and each draw solute needs validation to predict permeate fluxes and concentrations, which is time consuming and rigorous process. However, nowadays, enormous experimental datasets are available for FO and NF membrane processes. Therefore, artificial neural network (ANN) based data-driven model is developed and compared to the FO-NF transport modeling. to predict the permeate flux and concentration. The optimized ANN-based model showed high R^2 values: 95.03% and 96.08% for FO and NF datasets, respectively. However, model accuracy is compromised because of limited variation in training datasets, resulting in higher errors (5-12%) when compared to the transport-based model errors (2%). Despite larger errors, ANN model is competent in predicting the suitable draw solute and responses for the feed side solute.

अमूर्त

पिछले कुछ दशकों में, मानव आबादी में तेजी से वृद्धि के साथ तेजी से औद्योगिक और शहरी विकास ने मीठे पानी के भंडार को काफी प्रभावित किया है, जिससे उपयोग योग्य मीठे पानी की भारी कमी हो गई है। समुद्री जल और औद्योगिक खारे अपशिष्ट जल का झिल्ली-आधारित अलवणीकरण मीठे पानी की जरूरतों को पूरा करने के लिए एक आशाजनक समाधान के रूप में उभरा है। हालाँकि, पारंपरिक झिल्ली प्रौद्योगिकियाँ, जैसे कि रिवर्स ऑस्मोसिस (आरओ)/नैनोफिल्ट्रेशन (एनएफ) उच्च ऊर्जा खपत (3 से 6 kWh/m³), जल पुनर्प्राप्ति (30-40%) और दूषण सहित महत्वपूर्ण प्रदर्शन सीमाओं से ग्रस्त हैं, जो व्यवहार्य और बड़े पैमाने पर अलवणीकरण के लिए उनकी उपयुक्तता को कम करती हैं। इसलिए, यह अध्ययन अपशिष्ट जल अपशिष्टों में लवणता के उपचार के लिए एक संभावित विकल्प के रूप में फॉरवर्ड ऑस्मोसिस-नैनोफिल्ट्रेशन (एफओ-एनएफ) और प्रेशर असिस्टेड फॉरवर्ड ऑस्मोसिस-नैनोफिल्ट्रेशन (पीएफओ-एनएफ) जैसे ऑस्मोटिक रूप से उन्नत हाइब्रिड सिस्टम का प्रस्ताव करता है। इसके बाद, प्रयोगात्मक और परिवहन मॉडलिंग अध्ययनों का उपयोग करके मॉड्यूल-स्केल हाइब्रिड प्रक्रिया के कई महत्वपूर्ण पहलुओं का मूल्यांकन किया जाता है और स्टैंडअलोन एनएफ प्रदर्शन पर इन हाइब्रिड प्रणालियों में निरंतर ड्रा विलेय पुनर्जनन के प्रभाव की जांच की जाती है। इसके अलावा, एफओ-एनएफ हाइब्रिड सिस्टम के लिए एक अद्वितीय डेटा-संचालित मॉडलिंग विकसित किया गया है ताकि विभिन्न ड्रा विलेय के लिए पर्मेट फ्लक्स और सांद्रता की भविष्यवाणी की जा सके।

प्रारंभ में, परिवहन मॉडलिंग में, स्पीगलर-केडेम मॉडल, एकाग्रता ध्रुवीकरण मॉडल के साथ, एफओ और एनएफ झिल्ली में स्थानीय द्रव्यमान परिवहन का वर्णन करने के लिए उपयोग किया जाता है। इसके बाद, एफओ और एनएफ के लिए संयुक्त गणितीय मॉडल को हाइब्रिड एफओ-एनएफ प्रणाली में एकीकृत किया गया है। इसके अलावा, एफओ-एनएफ प्रक्रिया अनुकूलन से पता चलता है कि एनएफ हाइड्रोलिक दबाव और इनलेट ड्रॉ एकाग्रता एनएफ रेटेंटेड एकाग्रता को नियंत्रित करते हैं और फिर हाइब्रिड सिस्टम के लिए वांछित ऑपरेटिंग स्थितियों का अनुकरण करते हैं जब एनएफ रेटेंटेड स्ट्रीम को सीधे एफओ इकाई के लिए ड्रॉ समाधान के रूप में उपयोग किया जाता है। आगे की प्रायोगिक टिप्पणियों से पता चलता है कि गंदगी के कारण स्टैंडअलोन एनएफ जल रिकवरी समय के साथ 40 से 15.6% तक कम हो जाती है, जबकि एसईसी 0.89 से बढ़कर 2.38 kWh/m³ हो जाती है। साथ ही, एफओ-एनएफ हाइब्रिड सिस्टम क्रमशः 32.6% और 28.6% पानी की रिकवरी दिखाता है, जिसमें प्रक्रिया एसईसी 3.26 kWh/m³ Na₂SO₄ के लिए और 4.70 kWh/m³ MgSO₄ के लिए ड्रॉ विलेय के रूप में होता है।

यह भी देखा गया है कि अकेले NF प्रणाली की तुलना में FO-NF प्रणाली अपेक्षाकृत कम जल पुनर्प्राप्ति (water recovery) लेकिन अधिक विशिष्ट ऊर्जा खपत (SEC) प्रदर्शित करती है। इस अवलोकन के परिणामस्वरूप, PAFO-NF प्रणालियों की संभावनाओं की जांच करने के लिए प्रयोगात्मक और मॉडलिंग दृष्टिकोणों के माध्यम से अनुसंधान प्रेरित होता है, ताकि SEC को कम करते हुए जल पुनर्प्राप्ति को बढ़ाया जा सके। PAFO-NF मॉडलिंग के लिए FO मॉडलिंग में थोड़े संशोधन के साथ समान FO-NF परिवहन मॉडल का उपयोग किया गया है। झिल्ली

प्रदर्शन (membrane performance) को 3.5 g/L CaCl_2 वाले फाउलिंग प्रवण फीड स्ट्रीम के लिए 40 g/L सांद्रता वाले Na_2SO_4 और MgSO_4 को ड्रॉ सॉल्यूट के रूप में उपयोग कर सिम्युलेट किया गया है। अनुकूलित परिचालन स्थितियों (optimized operating conditions) के अंतर्गत सिम्युलेट किए गए परिणाम यह दिखाते हैं कि FO फीड साइड असिस्टेड प्रेशर (ΔPPAFO), FO झिल्ली क्षेत्रफल (AmFO), और FO जल पारगम्यता (AFO) प्रमुख कारक हैं जो Na_2SO_4 को ड्रॉ सॉल्यूट के रूप में उपयोग करते हुए PAFO-NF प्रक्रिया के SEC को कम करने और जल पुनर्प्राप्ति में सुधार लाने में सहायक हैं। सिम्युलेशन से यह पता चलता है कि 1.0 m^2 AmFO पर, ΔPPAFO को 0 से 7.5 बार तक बढ़ाने पर SEC को 2.95 से घटाकर 1.47 kWh/m^3 तक किया जा सकता है और जल पुनर्प्राप्ति को 60.5% से बढ़ाकर 95.54% तक किया जा सकता है। वास्तव में, केवल AmFO को 0.25 से 1.0 m^2 तक बढ़ाने से प्रक्रिया SEC को FO-NF प्रक्रिया की तुलना में 63% तक कम किया जा सकता है और जल पुनर्प्राप्ति को 90% तक बढ़ाया जा सकता है। इसी प्रकार, AFO को 1.22×10^{-12} से 3.0×10^{-12} m/Pa.s तक बढ़ाने से भी प्रक्रिया SEC को लगभग 42% तक घटाया जा सकता है और जल पुनर्प्राप्ति को 45.73% तक बढ़ाया जा सकता है। दूसरी ओर, इनलेट फीड और ड्रॉ सांद्रता और इनलेट फीड प्रवाह दरों का प्रक्रिया SEC पर नगण्य प्रभाव होता है। निष्कर्षतः, PAFO-NF संकर प्रक्रिया फाउलिंग प्रवण और गैर-फाउलिंग दोनों प्रकार के खारे जल के उपचार के लिए महत्वपूर्ण संभावनाएं प्रदर्शित करती है और समान परिचालन स्थितियों के अंतर्गत FO-NF और अकेले NF (गैर-फाउलिंग स्थितियों में) प्रक्रियाओं की तुलना में कम SEC प्राप्त कर उन्हें पीछे छोड़ देती है।

FO-NF हाइब्रिड प्रक्रिया के लिए उपरोक्त ट्रांसपोर्ट मॉडलिंग जटिल है और प्रत्येक ड्रॉ सॉल्यूट के लिए परमीएट फ्लक्स और सांद्रता की भविष्यवाणी करने हेतु सत्यापन की आवश्यकता होती है, जो समय लेने वाली और कठोर प्रक्रिया है। हालांकि, आजकल FO और NF मेम्ब्रेन प्रक्रियाओं के लिए विशाल प्रयोगात्मक डेटासेट उपलब्ध हैं। अतः एक कृत्रिम न्यूरल नेटवर्क (ANN) आधारित डेटा-संचालित मॉडल विकसित किया गया है और इसे FO-NF ट्रांसपोर्ट मॉडलिंग से तुलना की गई है ताकि परमीएट फ्लक्स और सांद्रता की भविष्यवाणी की जा सके। अनुकूलित ANN-आधारित मॉडल ने उच्च R^2 मान दिखाए: FO और NF डेटासेट के लिए क्रमशः 95.03% और 96.08%। हालांकि, मॉडल की सटीकता में समझौता होता है क्योंकि प्रशिक्षण डेटासेट में सीमित विविधता होती है, जिसके परिणामस्वरूप त्रुटियाँ अधिक होती हैं (5-12%) जब कि ट्रांसपोर्ट-आधारित मॉडल की त्रुटियाँ केवल 2% होती हैं। अधिक त्रुटियों के बावजूद, ANN मॉडल फीड साइड सॉल्यूट के लिए उपयुक्त ड्रॉ सॉल्यूट और प्रतिक्रियाओं की भविष्यवाणी करने में सक्षम है।

Table of Contents

CERTIFICATE	iii
ACKNOWLEDGEMENTS	iv
ABBREVIATIONS	v
THE BEGINNING OF MEMBRANE SCIENCE	vi
ABSTRACT	vii
LIST OF FIGURES	xiv
LIST OF TABLES	xvii
Chapter One	1
Introduction	1
1.1 Background	1
1.2 Motivation of the study	6
Chapter Two	8
Literature Review	8
2.1 Introduction	8
2.2 Saline waters and its environmental impact	8
2.2.1 Saline water sources	10
2.2.2 Environmental impact	13
2.3 Existing membrane separation processes	14
2.4 Forward osmosis (FO)-hybrid systems and their applications	17
2.4.1 History of draw solute development	17
2.4.2 FO-integrated draw solute regeneration technologies	19
2.4.2.1 Pressure-driven	19
2.4.2.2 Temperature-driven	21
2.4.2.3 Magnetically-driven	22
2.4.2.4 Electrically-driven	22
2.4.2.5 Precipitation	23
2.4.3 Studies on FO-hybrid systems	23
2.5 Research gaps	28
2.6 Research objectives	30
Chapter Three	31
The FO-NF hybrid system: Transport modeling theory, membrane parameter estimation, and experimental validation	31
3.1 Introduction	31

3.2 Theory of FO-NF transport modeling	31
3.3 Membrane local mass transport equations using the Speigler-Kedem model	33
3.4 External and internal concentration polarization (ECP & ICP) model equations	34
3.5 Mass balance and pressure drop equations for FO and NF modules	37
3.6 Parameter estimation technique	40
3.7 Materials and methods	42
3.7.1 Materials	42
3.7.2 Selection criteria for draw solute	43
3.7.3 Experiments on solute permeation through FO membrane	43
3.7.4 Experimental conditions, setup, and procedure	45
3.8 Membrane parameter estimation and model validation for spiral wound FO and NF modules	48
3.8.1 Numerical estimation of FO and NF hydraulic permeabilities (A & A_r)	48
3.8.2 Numerical estimation of solute permeability (B), reflection coefficient (σ), solute resistivity (K), and mass transfer correlation constants (k_a & k_b)	50
3.8.3 Parameter Estimation and Validation for PAFO Spiral-Wound Module	57
3.9 Membrane parameter estimation and model validation for hollow fiber FO module	60
3.9.1 Theory of hollow fiber FO module	61
3.10 FO-NF hybrid system evaluation terms	67
Chapter Summary	69
Chapter Four	70
A modeling and experimental comparative study of a closed-loop FO-NF and standalone NF systems for the treatment of fouling-prone feed solution	70
4.1 Introduction	70
4.2 Influence of operating variables	70
4.2.1 Effect on FO Dilution	71
4.2.2 Effect on FO permeate/water flux (J_{vFO})	72
4.2.3 Effect on FO-NF hybrid recovery (HR)	74
4.2.4 Effect on FO-NF hybrid permeate concentration (C_{pNF})	75
4.3 The FO-NF performance evaluation	78
4.4 NF retentate concentration (C_{rNF})	80
4.5 Process optimization and comparison	82
4.6 The FO-NF process operating variable optimization	83
4.7 The FO-NF comparison with standalone NF	86
4.8 Indicators of effective draw solute for the FO-NF system	87
Chapter Summary	88
Chapter Five	90

The PAFO-NF process modeling and performance comparison with the FO-NF process	90
5.1 Introduction	90
5.2 Permeate flowrate comparison between PAFO and FO processes	91
5.3 PAFO-NF hybrid process optimization	93
5.4 Performance comparison of PAFO-NF and FO-NF hybrid systems	94
5.5 Impact of FO process variables on PAFO-NF process SEC and water recovery	95
5.5.1 FO membrane solvent permeability (A_{FO})	95
5.5.2 Effect of FO membrane area (A_{mFO}) with change in feed side assisted hydraulic pressure (ΔP_{PAFO})	98
5.5.3 Effect of inlet feed and draw concentrations (C_{Fin} & C_{Din}) with change in FO membrane area (A_{mFO})	102
5.5.4 Effect of inlet feed flowrate (Q_{Fin}) with change in FO membrane area (A_{mFO})	105
5.6 PAFO-NF comparison with non-fouled standalone NF	107
Chapter Summary	108
Chapter Six	110
A modeling-based comparison study of data-driven and transport models for FO-NF hybrid system	110
6.1 Introduction	110
6.2 Data collection	111
6.3 Predictive data-driven models	113
6.3.1 Support vector regression (SVR)	113
6.3.2 Decision tree (DT)	114
6.3.3 Multiple linear regression (MLR)	114
6.3.4 Artificial Neural Networks	115
6.3.4.1 Dataset split ratio (DSR) and pre-processing	115
6.3.4.2 Hyper-parameter tuning	116
6.3.4.3 ANN training algorithm	117
6.4 Terminologies	120
6.4.1 Correlation	120
6.4.2 Error measurement	120
6.4.3 Software used	121
6.5 Results and Discussions	121
6.5.1 Data analysis using correlograms	121
6.5.2 The FO-NF model selection from predictive or data-driven models	123
6.5.3 Development of FO-NF hybrid data-driven model using ANN	125
6.5.3.1 Hidden layers (HL) and Neurons (N) tuning	125

6.5.3.2 ANN model fitting and activation function tuning	127
6.5.4 ANN model predictive performance	130
6.6 FO-NF Modeling comparison	132
6.7 Potential implication of FO-NF data-driven process	133
Chapter Summary	134
Chapter Seven	136
Conclusions and Recommendations	136
7.1.1 The FO-NF transport modeling	136
7.1.2 The PAFO-NF and FO-NF processes comparison	138
7.1.3 The FO-NF data-driven modeling	140
7.2 Scope for Future work	141
Supplementary data (Chapter 6)	142
Biodata of the Author	181

LIST OF FIGURES

	Page no.
Fig. 1.1: RO module design configurations adapted from the literature	2
Fig. 1.2: Number of FO-hybrid Published articles a) Documents by year and b) documents by FO-Hybrid.	4
Fig. 1.3: Countries focused on FO-hybrid research based on documents published yearly from 2005 to 2023.	6
Fig. 2.1: Schematic diagram of Fracking process.	12
Fig. 2.2: Schematic diagram of conceptual relations in FO, PAO, OARO, MD and RO processes.	15
Fig. 2.3: Schematic diagram of FO-MD hybrid system for recovering water from feed side.	21
Fig. 3.1: Concentration profile diagram of forward osmosis-nanofiltration (FO-NF) hybrid system using a continuous flow of divalent draw solution.	32
Fig. 3.2: Illustration of modeling flowchart for a) determination of the actual membrane parameters for FO-NF hybrid process; b) optimization program to search the final optimized parameters.	41
Fig. 3.3: A schematic diagram of laboratory-scale FO-NF hybrid experimental setup.	45
Fig. 3.4: Plot shows the estimated mass transfer coefficients (k_D , k_F , and k_r) for both FO and NF membranes against inlet draw flowrate (Q_{Din}) and diluted draw flowrate (Q_{Dout}).	54
Fig. 3.5: The Unwounded structure of the hollow fiber FO membrane. However, if hydraulic pressure is applied towards the feed side, the FO membrane ($\Delta P_{PAFO} = (P_D - P_F)$) is converted to PAFO. Note: the pressure is shown in the schematic diagram.	61
Fig. 4.1: Influence of inlet draw concentration (C_{Din}), inlet feed and draw flowrates (Q_{Fin} and Q_{Din}) and applied pressure across FO module (ΔP_{FO}) on FO dilution (%) at $T=25\pm 2^\circ\text{C}$ for both for Na_2SO_4 and MgSO_4 based draw solutions.	72

Fig. 4.2: Influence of (a-b) inlet draw concentration (C_{Din}), (c-d) applied pressure across FO (ΔP_{FO}), (e-f) inlet draw flowrate (Q_{Din}) and (g-h) inlet feed flowrate (Q_{Fin}) on FO water flux (J_{vFO}) and specific reverse salt flux (SRSF).	73
Fig. 4.3: Describing the influence of operating variables: a) inlet draw flowrate (Q_{Din}), b) inlet feed flowrate (Q_{Fin}), c) applied pressure across FO (ΔP_{FO}), d) inlet draw concentration (C_{Din}) and e) hydraulic pressure across NF (ΔP_{NF}) on FO-NF hybrid permeate concentration (C_{pNF}).	75
Fig. 4.4: Describing the influence of operating variables: a) inlet draw flowrate (Q_{Din}), b) inlet feed flowrate (Q_{Fin}), c) applied pressure across FO (ΔP_{FO}), d) inlet draw concentration (C_{Din}), and e) hydraulic pressure across NF (ΔP_{NF}) on FO-NF hybrid permeate concentration (C_{pNF}).	77
Fig. 4.5: The influence on FO-NF specific energy consumption (SEC) behaviour with change in operating variables: a) inlet draw concentration (C_{Din}), b) applied pressure across FO (ΔP_{FO}), c) inlet draw flowrate (Q_{Din}), d) inlet feed flowrate (Q_{Fin}) and hydraulic pressure across NF (ΔP_{NF}).	78
Fig. 4.6: Study the Solute rejection (R_r %) from diluted draw solution using NF membrane with respect to change in operating variables: a) inlet draw concentration (C_{Din}), b) applied pressure across FO (ΔP_{FO}), c) inlet draw flowrate (Q_{Din}), d) inlet feed flowrate (Q_{Fin}) and hydraulic pressure across NF (ΔP_{NF}).	79
Fig. 4.7: Influence of FO-NF hybrid operating variables on NF retentate concentration (C_{rNF}) after the regeneration of diluted draw solution by NF module.	81
Fig. 4.8: The optimization procedure of the FO-NF process operating variables (Q_{Din} and ΔP_{NF}).	86
Fig. 4.9: Permeate flux (Q_{pNF}) and concentration (C_{pNF}) as a function of time in (a) standalone NF and (b) FO-NF hybrid processes at constant feed and draw flowrates (Q_{Fin} or Q_{Fb} and Q_{Din}), concentration (C_{Fin}) and hydraulic pressure (ΔP_{NF}).	84
Fig. 5.1: Comparison between the PAFO and FO processes in terms of permeate flowrate (Q_p) with respect to osmotic pressure difference ($\Delta\pi$), inlet draw flowrate (Q_{Din}) and inlet feed flowrate (Q_{Fin}) using Na_2SO_4 and $MgSO_4$ based draw solutions.	94

Fig. 5.2: Effect of FO solvent/water permeability on SEC and water recovery with varying feed-side hydraulic pressure (ΔP_{PAFO}) at $A_{mFO} = 0.25 \text{ m}^2$, using a calcium-rich feed solution and Na_2SO_4 -based draw solution in the PAFO-NF process.	98
Fig. 5.3: Effect of FO feed side assisted hydraulic pressure (ΔP_{PAFO}) and its membrane area (A_{mFO}) on a) SEC, b) water recovery (R) c) CaCl_2 concentration diffusion towards the draw side and d) draw solute SRSF in the PAFO-NF hybrid process using a Na_2SO_4 -based draw solution.	100
Fig. 5.4: Influence of (a and c) 20g/L inlet feed concentration (C_{Din}) (red color) and (b and d) 10 g/L inlet draw concentration (C_{Fin}) (red color) on the PAFO-NF process SEC and water recovery using Na_2SO_4 -based draw solution with change in FO membrane area (A_{mFO}) and FO feed side assisted hydraulic pressure (ΔP_{PAFO}).	104
Fig. 5.5: Effect of inlet feed flowrate ($Q_{Fin} = 30\text{L/h}$) (red color) on PAFO-NF hybrid SEC and recovery using Na_2SO_4 -based draw solution with change in FO membrane area (A_{mFO}) and FO feed side assisted hydraulic pressure (ΔP_{PAFO}).	106
Fig. 6.1: Flowchart showing artificial neural network training.	117
Fig. 6.2: Neural network architecture of one neuron or node in an artificial neural network.	119
Fig. 6.3. Correlograms of the two datasets (a) DS-FO and (b) DS-NF.	122
Fig. 6.4: Predicted vs. true response curves of outputs Y1 and Y2 for DS-FO and Y3 and Y4 for DS-NF of the training and validation of MLR, DT and SVR predictive models.	125
Fig. 6.5: Network architecture a) for the ANN model trained on DS-FO, b) for the ANN model trained on DS-NF.	127
Fig. 6.6: Trained ANN_{FO} model results trained on DS-FO a) Performance analysis, b) Error analysis, and c) Regression analysis.	128
Fig. 6.7: Trained ANN_{NF} model results trained on DS-NF a) Performance analysis, b) Error analysis, and c) Regression analysis.	129
Fig. 6.8: ANN model prediction curves FO-NF permeate flux ($J_{v, FO-NF}$) and permeate concentration ($C_{p, FO-NF}$) of four draw solutes Na_2SO_4 , MgSO_4 , $(\text{NH}_4)\text{SO}_4$ and K_2SO_4 .	131

LIST OF TABLES

	Page no.
Table 2.1: Describes the concentrations of solute ions in various industrial wastewaters and compares them with the WHO drinking water standard.	9
Table 2.2: Water classification by the Food and Agriculture Organisation of the United Nations (<i>FAO</i>).	10
Table 2.3: Produced water specifications with crude oil as by product.	13
Table 2.4: Draw solutes development in FO hybrid process.	18
Table 2.5: FO-hybrid processes used for wastewater treatment.	24
Table 3.1: List of inorganic draw solutes selected from literature for FO-NF hybrid system.	43
Table 3.2: Experimental observation of the feed and draw solutes permeation in deionized water placed at the opposite side of the FO membrane.	44
Table 3.3: Describing the lab-scale FO-NF hybrid experimental operating conditions and FO and NF spiral module specifications.	46
Table 3.4: The experimental data used for estimating the FO and NF membranes hydraulic permeabilities (A & A_r).	49
Table 3.5: Data used for parameter estimation and validation of (a) FO and (b) NF membranes for Na_2SO_4 based draw solution.	50
Table 3.6: Data used for parameter estimation and validation of (a) FO and (b) NF membranes for MgSO_4 based draw solution.	52
Table 3.7: Represents the experimental data of non-fouled standalone NF membrane for model validation in which CaCl_2 used as a feed solute.	56
Table 3.8: Represents the estimated parameters of FO and NF membranes for both draw solutions (Na_2SO_4 and MgSO_4) using CaCl_2 as feed solute.	57

Table 3.9: Shows the estimated membrane parameters of PAFO and NF processes for CaCl_2 based feed solution.	58
Table 3.10: Experimental data for membrane parameter estimation and validation of PAFO process for feed and draw side solute species: CaCl_2 and Na_2SO_4 . Where C_{Fin} and C_{Din} are the inlet feed and draw concentrations.	59
Table 3.11: Experimental data for membrane parameter estimation and validation of PAFO process for feed and draw side solute species: CaCl_2 and MgSO_4 . Where C_{Fin} and C_{Din} are the inlet feed and draw concentrations.	60
Table 3.12: The experimental data used for estimating hydraulic permeabilities (A_{FO} & A_{NF}) for the FO, PAFO and NF processes.	65
Table 3.13: Experimental data for membrane parameter estimation and validation of PAFO process for feed and draw side solute species: CaCl_2 and Na_2SO_4 . Where C_{Fin} and C_{Din} are the inlet feed and draw concentrations.	66
Table 3.14: Estimated membrane parameters for the PAFO process.	67
Table 4.1: The FO-NF process simulated data of specific energy consumption (SEC) and water recovery (R) at optimized operating conditions.	85
Table 4.2: Calculate the total operating cost of water production per m^3 of permeate volume between FO-NF hybrid and standalone NF.	89
Table 5.1: The FO-NF, and PAFO-NF processes SEC and water recovery (R) comparison under the optimized operating conditions.	94
Table 5.2: Describing the PAFO-NF process operating conditions utilized for the simulation of SEC and water recovery with variation in FO solvent/water permeability, as shown in Fig. 4.	97
Table 5.3: Initial optimized operating conditions of the PAFO-NF process simulation performed in Fig. 5. 3.	99
Table 5.4: Initial optimized operating conditions of the PAFO-NF process simulation performed in Fig.5.4.	103
Table 5.5: Initial optimized operating conditions of PAFO-NF process simulation performed in Fig. 5.5	107

Table 5.6: Comparison between the non-fouled standalone NF and the PAFO-NF process.	108
Table 6.1: Input variables and ranges of DS-FO and DS-NF datasets.	112
Table 6.2: Finding the best dataset split ratio distribution for ANN model training, validation, and testing.	118
Table 6.3: Inputs and outputs of collected datasets: DS-FO and DS-NF.	123
Table 6.4: Comparison among the predictive models for DS-FO and DS-NF datasets.	123
Table 6.5: Testing DS-FO and DS-NF datasets to find the optimal combination of hidden layer and neurons for their respective ANN model.	126
Table 6.6: FO-NF hybrid accuracy-based comparison between the data-driven modeling using ANN and transport modeling.	133
Table 7.1: Process SEC and water recovery (R) comparison between the FO-NF, PAFO-NF and Standalone NF processes at optimized conditions.	139