

DEVELOPMENT OF INTEGRATED
ELECTROCOAGULATION AND MEMBRANE
PROCESS FOR WATER RECLAMATION FROM
BIOTREATED PALM OIL MILL EFFLUENT

BY

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ABSTRACT

Electrocoagulation (EC) is a simple, environmentally friendly and cost effective process, when integrated with membrane filtration, becomes very attractive for developing a sustainable water reclamation system. The critical parameters namely initial pH, time and current density largely impact the EC process efficiency. Few works have been done on observing the interaction of these critical parameters and the possible combined effect on the overall pollutant removal efficiency. Also, with membrane pore blocking study, the efficiency of the overall integrated process can be enhanced by determining the dominant fouling mechanism. Therefore, the knowledge of the combined effect of critical parameter interaction followed by membrane fouling study would enhance the overall efficiency of the integrated process to sustainably reclaim water. Using aluminum electrodes with interelectrode distance of 10 mm, with a set range of initial pH, current density, and time of 3-8, 40-160 mA/cm² and 15-60 minutes, respectively, the effect of the three critical variables were investigated on synthetic wastewater, representing biotreated palm oil mill effluent (BPOME). Next, a pore blocking study was undertaken with the EC treated BPOME after crossflow membrane filtration process with transmembrane pressure of 0.5 bar and pore size of 1 kDa. The optimum Chemical Oxygen Demand (COD) removal of 71.5% was determined at pH 6, current density of 160 mA/cm² (with current 1.75 A) at EC time of 15 minutes. The experiment was validated with real BPOME, resulting in the removal efficiency of 60.7% COD, 99.91% turbidity, 100% total suspended solids (TSS) and 95.7% color. The interaction of parameters observed in this study indicated a synergistic contribution of initial pH and current density in removing maximum wastewater COD in 15 minutes of EC. After following with membrane ultrafiltration process, the COD removal increased to 71.7%, and the dominant fouling mechanism prevailing was cake formation as determined by fitting with Hermia's pore blocking models. EC with activated carbon (AC) addition, run with the optimized parameters, significantly improved the final treated quality with a 100% TSS, 99% of both color and turbidity and 84.6% COD removal. The best permeate quality was achieved with 1 wt. % addition of AC in EC reactor, and the removal of TSS, turbidity and color was nearly 100% and COD was removed 99.7% with final value of 5±1 mg/L, which are within the range of reusable process water standard. Also, addition of AC in EC, sustainably enhanced the final treated effluent quality with fouling mitigation in the subsequent membrane ultrafiltration.

خلاصة البحث

التخثير الكهربائي هو عملية بسيطة وصديقة للبيئة وفعالة من حيث التكلفة، عندما تتكامل مع الترشيح الغشائي، تصبح جذابة للغاية لتطوير نظام استصلاح المياه المستدام. تؤثر العوامل الحرجة وهي الرقم الهيدروجيني البدائي، والوقت وكثافة التيار إلى حد كبير على كفاءة عملية التخثير الكهربائي. تم إجراء بعض الدراسات على مراقبة تفاعل هذه العوامل الحرجة واحتمالية التأثير المشترك على الكفاءة الكلية لإزالة الملوثات. لذلك، فإن معرفة التأثير المشترك لتفاعل العوامل الحرجة من شأنه أن يعزز تحسين عوامل التخثير الكهربائي لتحقيق أقصى قدر من الكفاءة بموارد محدودة. باستخدام أقطاب كهربائية مصنوعة من الألومنيوم بمسافة بين الأقطاب الكهربائية 10 مل متر على مياه الصرف الصحي الصناعي، والتي تمثل تصريف مطحنة زيت النخيل المعالج حيويًا، مع قيم محددة من الرقم الهيدروجيني البدائي، وكثافة التيار، والوقت من 3-8، 40-160 مللي أمبير/سم مربع و15 إلى 60 دقيقة، على التوالي، وتم التحقق في التأثير على المتغيرات الثلاث الحرجة. كما تم تحديد عامل الإزالة المثلى لطلب الأكسجين الكيميائي بنسبة 71.5٪ عند الرقم الهيدروجيني 6، وكثافة التيار 160 مللي أمبير/سم مربع (مع التيار 1.75 أمبير) في وقت تخثير كهربائي لمدة 15 دقيقة. كما تم التحقق من صحة التجربة باستخدام عينة حقيقية من تصريف مطحنة زيت النخيل المعالج حيويًا، نتج عنه كفاءة إزالة بنسبة 60.7٪ من طلب الأكسجين الكيميائي، وعكارة 99.91٪، و100٪ إجمالي المواد الصلبة العالقة و95.7٪ من الألوان. إن إزالة كمية كبيرة من الملوثات في فترة زمنية مدتها 15 دقيقة مع عوامل محسنة أثناء عملية التخثير الكهربائي أمر ملحوظ بالنسبة إلى حل بديل لمعالجة مياه الصرف الصحي الذي لا يتطلب استخدامًا مكثفًا للمواد الكيميائية. أشار تفاعل العوامل الذي لوحظ في هذه الدراسة إلى مساهمة تآزيره للرقم الهيدروجيني البدائي وكثافة التيار في إزالة الحد الأقصى من طلب الأكسجين الكيميائي لمياه الصرف الصحي في 15 دقيقة من التخثير الكهربائي. بعد ذلك، أجريت دراسة لسد المسام باستخدام عينة معالجة من تصريف مطحنة زيت النخيل المعالج حيويًا بعد عملية ترشيح غشاء الجريان التقاطع مع ضغط الغشاء 0.5 بار و1 كيلو دالتون. بعد هذه العملية، زادت كفاءة إزالة طلب الأكسجين الكيميائي إلى 71.7٪، وكانت آلية تلوث الغشاء السائد هي ترسب طبقة من الجسيمات الصلبة كما تم تحديده من خلال التطبيق مع نماذج Hermia لسد المسام. التخثير الكهربائي المعزز من خلال إضافة الكربون المنشط، الذي يتم تشغيله باستخدام العوامل المحسنة، يحسن بشكل كبير جودة المعالجة النهائية مع إزالة 100٪ من المواد الصلبة العالقة، كما أنه لوحظ إزالة 99٪ لكل من اللون والعكارة. وقد تم في هذه المرحلة تحقيق الحد الأقصى لإزالة طلب الأكسجين الكيميائي (82٪) بقيمة نهائية قدرها 306 ملجم/لتر. باستخدام العوامل المحسنة، عزز التخثير الكهربائي مع إضافة الكربون المنشط من جودة تصريف النفايات السائلة المعالجة إلى حد التخفيف من التلوث في الخطوة التالية وهي ترشيح غشاء الجريان التقاطع. تم تحقيق أفضل نتيجة بإضافة الكربون النشط بنسبة وزن 1 بالمئة في مفاعل التخثير الكيميائي، وإزالة المواد الصلبة العالقة والعكارة واللون حوالي 100٪ وتمت إزالة طلب الأكسجين الكيميائي بنسبة 99.7٪ بقيمة نهائية قدرها 1±5 ملجم/لتر، والتي تقع في نطاق معيار المياه القابلة لإعادة الاستخدام. كما أن إضافة الكربون المنشط في التخثير الكهربائي قد عزز بشكل مستدام جودة النفايات السائلة المعالجة النهائية مع تخفيف الفاذورات في الترشيح باستخدام الغشاء الفائق التالي.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science in Engineering

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DECLARATION

I hereby declare that this thesis is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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TABLE OF CONTENTS

Abstract	ii
Abstract Arabic.....	iii
Approval Page.....	iv
Declaration	v
Acknowledgement	vii
Table of Contents	viii
List of Tables	x
List of Figures	xii
List of Abbreviations	xv
List of Symbols	xvi
CHAPTER ONE: INTRODUCTION	1
1.1 Background	1
1.2 Problem Statement and Significance	5
1.3 Research Objectives.....	7
1.4 Scope of Research.....	8
1.5 Thesis Organization	9
CHAPTER TWO: LITERATURE REVIEW.....	10
2.1 Introduction.....	10
2.2 Conventional POME Treatments.....	10
2.3 Coagulation	12
2.4 Electrocoagulation (EC).....	16
2.5 Parameters Affecting EC	18
2.5.1 Current Density	18
2.5.2 Electrode Material and Arrangement	19
2.5.3 Interelectrode Distance.....	21
2.5.4 Initial pH and Conductivity	23
2.5.5 Reaction Time	26
2.5.6 Temperature.....	26
2.5.7 Other Factors	27
2.5.7.1 Stirring Speed.....	27
2.5.7.2 Supporting Electrolytes.....	27
2.6 EC Parameter Interaction.....	28
2.7 EC Based Wastewater Treatment	29
2.8 Advancement in EC Based Hybrid Technologies	35
2.8.1 EC-Peroxidation	35
2.8.1.1 EC-CC.....	36
2.8.1.2 Photovoltaic EC	37
2.8.1.3 EC-Biological Treatment.....	38
2.8.1.4 EC-Membrane.....	38
2.9 Membrane Filtration	45
2.9.1 Pore Blocking Models for Crossflow Membrane Filtration..	47
2.9.2 Complete Blocking Model for Crossflow Filtration	48

2.9.3	Intermediate Blocking Model for Crossflow Filtration.....	49
2.9.4	Standard Blocking Model for Crossflow Filtration.....	50
2.9.5	Gel Layer Formation Model for Crossflow Filtration.....	50
2.10	Quality of Process Water	51
2.11	Research Gap	51
2.12	Summary	52
CHAPTER THREE: RESEARCH METHODOLOGY		54
3.1	Introduction.....	54
3.2	Materials and Equipment	55
3.3	Analytical Techniques	56
3.4	EC Experimental Design and Statistics	57
3.5	EC Experimental runs	59
3.6	Membrane Ultrafiltration	60
3.7	Evaluation of Integrated EC-Membrane Process.....	63
3.8	EC Coupled with Powdered Activated Carbon (AC)	64
3.9	Summary	65
CHAPTER FOUR: RESULTS AND DISCUSSION		66
4.1	Introduction.....	66
4.2	Design of Experiment (DOE) Analysis	66
4.2.1	Effect of Current Density and Time	74
4.2.2	Effect of pH	76
4.2.2.1	Observation of Aluminum Anode After EC	78
4.3	EC on Real BPOME	81
4.4	Membrane Ultrafiltration	84
4.4.1	Activated Carbon (AC) Assisted EC	91
4.5	Crossflow Filtration on EC-AC Treated BPOME	100
4.6	Evaluation of EC-Membrane Efficiency	105
4.6.1	Pollutant Removal Efficiency.....	105
4.6.2	Energy Consumption	109
4.7	Summary	111
CHAPTER FIVE: CONCLUSIONS AND RECOMMENDATIONS		113
5.1	Conclusions.....	113
5.2	Recommendations.....	114
REFERENCES.....		115
APPENDIX.....		133
	Characterization procedures.....	133
	Observation of changes in current density on COD, TSS, color and turbidity removal on BPOME, from 15 to 60 minutes	135
	Synthetic water composition representing BPOME	141
	COD, turbidity and TSS removal % when EC is assisted with AC.....	141
	Power consumption in EC process (calculations).....	143
	Power consumption in crossflow membrane filtration	143
LIST OF PUBLICATIONS		145

LIST OF TABLES

Table 2.1: Characteristics of biotreated POME	11
Table 2.2: Summary of EC process on various types of wastewater in recent studies	31
Table 2.3: Recent studies on EC based hybrid systems for wastewater treatment	43
Table 2.4: Pre-treatment processes before membrane filtration	46
Table 2.5: Boiler-feed/process water standards for low-moderate pressure boilers	51
Table 3.1: Characteristics of the membrane used in this research	56
Table 3.2: Membrane filtration operating conditions	62
Table 3.3: Linearized pore blocking equations for membrane filtration	63
Table 3.4: General coal based powdered AC characteristics	65
Table 4.1: Completed DOE table with output of COD removal % on synthetic wastewater for optimization	67
Table 4.2: ANOVA and fit- statistics for the reduced quadratic model	69
Table 4.3: Summary of DOE validation experiment	71
Table 4.4: Characterization of EC treated synthetic wastewater	71
Table 4.5: Characterization of BPOME	79
Table 4.6: Characterization of EC treated BPOME	80
Table 4.7: Effect of current density (160 mA/cm^2) on BPOME EC at pH 6	82
Table 4.8: Comparison of the removal output with optimized parameters on synthetic and real BPOME	83
Table 4.9: Crossflow filtration with 0.5 TMP on deionized water	84
Table 4.10: Crossflow filtration with 0.5 TMP on EC treated BPOME	85
Table 4.11: Summary of pore blocking constants, R^2 and J_0	89
Table 4.12: Summary of characterization after EC-membrane process	90

Table 4.13: Wt % of AC and its equivalent mass per working volume	92
Table 4.14: Characterization of EC treated BPOME with 0.5 wt % AC	93
Table 4.15: Characterization of EC treated BPOME with 1.0 wt % AC	93
Table 4.16: Characterization of EC treated BPOME with 1.5 wt % AC	93
Table 4.17: Crossflow filtration with 0.5 TMP on deionized water	100
Table 4.18: Crossflow filtration with 0.5 TMP on EC-AC treated BPOME	101
Table 4.19: Characterization of permeate after crossflow filtration and comparison with Environmental Protection Agency (EPA) reusable limits	106
Table 4.20: ICP-MS analysis on BPOME before and after EC-AC and membrane ultrafiltration, comparison with EPA water reuse guidelines	108
Table 4.21: Energy consumption of EC-membrane process	109

LIST OF FIGURES

Figure 2.1: Diagram of electrical double layer (EDL)	13
Figure 2.2: Schematic diagram of the forces on two colloid particles	14
Figure 2.3: Schematic visualization of particle removal through EC-membrane hybrid technique	40
Figure 2.4: Schematic diagram of overall hybrid EC-membrane process plan for process water reclamation and recycle	42
Figure 2.5: Schematic diagram of membrane operation (a) crossflow and (b) dead-end filtration	47
Figure 2.6: Schematic Diagram of the Four Blocking Filtration Laws: (a) Complete Blocking, (b) Standard Blocking, (c) Intermediate Blocking, and (d) Cake Filtration	48
Figure 3.1: Flow chart of the overall research methodology	54
Figure 3.2: Schematic diagram of simple EC setup	60
Figure 3.3: Crossflow membrane filtration setup	61
Figure 3.4: Schematic diagram of the crossflow filtration setup	61
Figure 4.1: Optimized variables depicted in ramps, generated using Design Expert 13.0	70
Figure 4.2: EC treatment on synthetic wastewater, before treatment (left) and after treatment (right)	71
Figure 4.3: Combined effect of time and initial pH on COD removal a) 3D surface, b) Contour plot	72
Figure 4.4: Combined effect of time and current density on COD removal a) 3D surface, b) Contour plot	73
Figure 4.5: Combined effect of current density and initial pH on COD removal a) 3D surface, b) Contour plot	73
Figure 4.6: Perturbation plot % COD removal	77
Figure 4.7: SEM image of aluminum anode before EC (left) and after EC (right)	78
Figure 4.8: Visual observation of color removal on BPOME after every 15 minutes of EC (left to right) 160 mA/cm ² pH 6	82

Figure 4.9: Bar graph representation of EC treatment efficiency on BPOME in terms of removal % vs time with current density 160 mA/cm ² and pH 6	83
Figure 4.10: Change of permeate flux with time for EC treated BPOME crossflow membrane filtration	85
Figure 4.11: Linearized plot for permeate flux vs time for standard pore blocking model	87
Figure 4.12: Linearized plot for permeate flux vs time for intermediate pore blocking model	88
Figure 4.13: Linearized plot for permeate flux vs time for complete pore blocking model	88
Figure 4.14: Linearized plot for permeate flux vs time for cake filtration	89
Figure 4.15: Visual observation of color removal on BPOME from 15 to 60 minutes of EC (left to right) with AC addition	92
Figure 4.16: Turbidity and TSS removal % within 5 minutes of EC (combined with AC at 0.5 wt.% - 1.5 wt. %)	94
Figure 4.17: Bar graph representation of EC treatment efficiency on BPOME in terms of color removal % vs time with current density 160 mA/cm ² and pH 6	95
Figure 4.18: COD removal % with and without AC coupled with EC	96
Figure 4.19: Observation of the best pollutant removal efficiency with respect to time (min) with EC and 1% AC	97
Figure 4.20: XRD spectra of AC before and after EC treatment on BPOME (as sludge)	99
Figure 4.21: Observation of flux with time for the crossflow filtration with deionized water	102
Figure 4.22: Linearized plot for permeate flux vs time for standard pore blocking model for crossflow filtration of EC-AC treated BPOME	102
Figure 4.23: Linearized plot for permeate flux vs time for intermediate pore blocking model for crossflow filtration of EC-AC treated BPOME	103
Figure 4.24: Linearized plot for permeate flux vs time for complete pore blocking model for crossflow filtration of EC-AC treated BPOME	103

Figure 4.25: Linearized plot for permeate flux vs time for cake formation model for crossflow filtration of EC-AC treated BPOME	104
Figure 4.26: Permeate after crossflow filtration of EC-AC treated BPOME	105
Figure 4.27: Appearance of BPOME after EC (with AC addition) treatment and crossflow filtration	106

LIST OF ABBREVIATIONS

AC	Activated Carbon
AOP	Advanced Oxidation Process
BPOME	Biotreated Palm Oil Mill Effluent
BET	Brunauer, Emmett and Teller
BOD	Biochemical Oxygen Demand
CC	Chemical Coagulation
COD	Chemical Oxygen Demand
CPO	Crude Palm Oil
DLVO	Derjaguin Landua Verwey Overbeek
DOE	Department of Environment
EC	Electrocoagulation
ECP	Electrochemical Peroxidation
EDL	Electrical Double Layer
EFB	Empty Fruit Bunch
FFB	Fresh Fruit Bunch
K_c	Pore blocking constant for cake formation
K_{cb}	Pore blocking constant for complete pore blocking
kDa	Kilo Daltons
K_i	Pore blocking constant for intermediate fouling
K_s	Pore blocking constant for standard fouling
MF	Microfiltration
MOH	Ministry of Health
MWCO	Molecular Weight Cut-Off
NTU	Nephelometric Turbidity Units
NWQS	National Water Quality Standard
PES	Polyether Sulphone
POME	Palm Oil Mill Effluent
PtCo	Platinum-Cobalt scale
RSM	Response Surface Methodology
SDG	Sustainable Development Goals
TMP	Transmembrane Pressure
TDS	Total Dissolved Solids
TN	Total Nitrogen
TP	Total Phosphorus
TSS	Total Suspended Solids
UF	Ultrafiltration
UN	United Nations
WEF	World Economic Forum

LIST OF SYMBOLS

J	Permeate Flux
Al	Aluminum
Fe	Iron
kg	Kilogram
L	Litre
mg	Milligram
μm	Micrometer
nm	Nanometer
mS	milliSiemens
Q	Flowrate
rpm	Rotation Per Minute
T	Time
Δ	Transmembrane pressure drop
R_m	Membrane Resistance
μ	Absolute viscosity
g	Gram

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND

Rising scarcity of fresh water is a global concern. According to the World Economic Forum (WEF), the global water crisis ranks as the number four risk in terms of impact on the society till date. A significant decrease in the available freshwater quality and quantity is raising concerns on consequent impact on not only human health and ecosystem but also the world economy (WEF, 2020). Besides, the United Nations (UN) realizing the importance and urgency of freshwater crisis mitigation, placed “Clean Water and Sanitation” as the number six goal to be achieved by 2030 under the movement of Sustainable Development Goals (SDG). Therefore, Target 6.3 under goal 6 (SDG) is to improve water quality, wastewater treatment and safe reuse. By UN definition, the accomplishment of Target 6.3 (one of the eight targets to achieve SDG goal 6) is visualized by breaking it down to the following four measures (SDG 6 Synthesis Report, 2018):

1. Reducing pollution
2. Abolishing dumping and minimizing the emission of hazardous materials and chemicals
3. Cutting down the quantity of untreated wastewater by half
4. Substantially increasing recycling and safe reuse globally.

It is evident that emerging industries and urban societies are major drivers for global economic boost. However, disregarding the environmental consequences strictly on appropriate waste treatment and disposal, will sooner or later, not only tax the world

economy, but also can build up to the collapse of the ecosystem sustainability, even impacting climate change in the long run.

For instance, Malaysia being one of the top global palm oil producers, generated 19.86 million tons of crude palm oil (CPO) in 2019 alone, which is a marginal increase by 1.8% (Malaysian Palm Oil Board, 2020). With every ton of CPO produced, huge amount of water is employed for extraction processes on fresh fruit bunches (FFB) and about 50% of the water is disposed as effluents (Ahmad et al., 2003). Besides, the effluents are high in organic matter and nutrients that are nontoxic but carry potential to induce algal growth and eutrophication overtime (Reilly et al., 2019), while the conventional treatment methods fail to meet the environmental discharge standards set by Department of Environment (DOE) of Malaysia (Kamyab et al., 2018). Similarly, most wastewater effluents besides treated palm oil mill effluents (POME) (Bashir et al., 2019; Daud et al., 2013; Kamyab et al., 2018; Othman et al., 2014), from several industries such as cheese whey effluent (Tirado et al., 2018), municipal wastewater (Nawarkar & Salkar, 2019), mineral processing wastewater (Wu et al., 2019) and many other types of wastewater are discharged into rivers after conventional treatments. Typical treatments before discharge into rivers involve physical, biological and/or chemical processes.

The type of treatment varies depending on the different types of wastewater and pollutants. The conventional processes involved in wastewater treatment include advanced oxidation processes (Boczka & Fernandes, 2017), biological processes (Huang et al., 2017; Iskandar et al., 2018; Liew et al., 2014), physico-chemical processes (Bhuptawat et al., 2007; Lin & Chen, 1997; Sher et al., 2013), and emerging technologies namely membrane filtration (Teng et al., 2018) and adsorption (Amosa et al., 2016). Advanced oxidation processes require strong oxidants making the

wastewater treatment taxing in terms of safety and cost. Biological processes on the other hand, demand strictly controlled conditions with long retention times, larger footprint, and unwanted by-products generation (Abu Hasan et al., 2020; Deveci et al., 2019). Chemical processes need extensive chemical dosages that not only adds to process cost, but also makes downstream processes complex, with increased risk of secondary contamination (Jiang, 2015). Membrane filtration and adsorption alone cannot efficiently treat wastewater, unless integrated with thorough pre-treatment processes (Khan & Boddu, 2021; Saleem et al., 2019). Or else, the processes become unproductive overtime due to pore blocking with pollutants and loss of flux (flowrate of clean treated effluent per unit surface area). Therefore, wastewater treatment research is greatly allured to electrochemical processes (Hakizimana et al., 2017). In 1889, electricity employed water treatment was first proposed in UK, while electrocoagulation (EC) achieved its first patent in the US in 1909 (Chen, 2004). Even though EC was successfully applied in the US in large scale drinking water treatment in 1946, it failed to gain global popularity for wider applications limited by power supply costs and huge capital investment (Chen, 2004).

However, constant progress in EC research remarkably upheld the significance of EC and its promising impact in wastewater treatment. Amongst all electrochemical processes, EC stands out as the most sustainable alternative to treat wastewater due to its simple setup, small footprint, ability to treat large quantity of water with no extensive chemical treatment (Moussa et al., 2017; Sahu et al., 2014). Moreover, the versatility of the process and its setup enables EC to treat a wide range of wastewater across industries and domestic works with different types of pollutants. Many researchers worldwide had conducted several EC studies till date, to treat various types of wastewater and achieved promising outcomes. Some EC studies were taken further by integrating with other

processes, progressing into advanced wastewater systems to produce cleaner effluents. Some of the notable works on EC are carried out by Bashir et al. (2019), Changmai et al. (2019), Deveci et al. (2019), Dimoglo et al. (2019), Khemila et al. (2018), Nasrullah et al. (2020), Nawarkar & Salkar (2019), Sher et al., (2020) and more.

Of all the current processes employed in the POME and wastewater treatments namely aerobic/anaerobic digestion, adsorption, chemical coagulation, etc., EC is an attractive alternative as it does not require heavy chemical extensive processes and is relatively quick and inexpensive and a simple process that has immense potential to sustainably treat large quantities of water at once (Naje et al., 2017). EC is an attractive electrochemical process with simple set up, inexpensive, environmentally friendly procedure requiring less carbon footprint in terms of space and chemical requirement and therefore, carries a promising potential to be scaled up to treat huge amount of POME at the industrial level. However, it is important to understand the critical parameters of EC and the parameter interaction effect on wastewater treatment efficiency. Determining the synergistic or antagonistic influence of possible parameter interaction on pollutant removal, contributes to deliver an enhanced EC performance.

To treat the wastewater to the standard of water reclamation for reusability, EC needs an effective integration with an additional separation process (Afanga et al., 2020). Strictly considering the environmental and economic sustainability, along with the simplicity in set up, in situ operation and maintenance, EC is a desirable fit as a pre-treatment for the booming membrane technology, greatly mitigating membrane fouling. Typically, strong oxidants such as peroxides are added to enhance wastewater treatment efficiency with EC (Bashir et al., 2019). Even though, oxidants can breakdown colloidal pollutants in the wastewater, they are hazardous and not environmentally friendly. Hence, combining activated carbon (AC), a relatively green support (compared to

peroxides), has been found to work very well to enhance EC performance with remarkable reduction in the wastewater pollutant quantity (Barhoumi et al., 2019; Sher et al., 2021).

Membrane technology is popular for producing consistent permeate quality to meet more stringent water requirements such as for drinking, urban reuse, industrial reuse etc. (Ezugbe & Rathilal, 2020). To tackle the issue of membrane pore blocking (fouling) over time, that declines membrane performance and reduces membrane integrity, hybrid membrane systems are increasingly being studied for an overall enhancement of treatment efficiency (Khan & Boddu, 2021). The emerging studies of hybrid membrane-based processes with integrated EC depict immense potential for treating wastewater for water reclamation for specific applications such as irrigation, industrial or urban reuse. Therefore, integration of EC with membrane process, for water reclamation from BPOME is a promising direction as EC is able to sustainably remove a huge amount of pollutants as colloidal particles, that can significantly reduce membrane fouling. Hence, the EC-membrane hybrid process is not only a promising treatment process for contributing to mitigating environmental pollution from the final industrial effluents, but also would lead to freshwater scarcity mitigation with the resulting water reusability.

1.2 PROBLEM STATEMENT AND SIGNIFICANCE

This study focuses on EC-membrane process as post treatment for BPOME and investigation of its ability to sustainably reclaim process water for industrial reuse. BPOME is the final discharge effluent of the palm oil industries, that hold the potential to be reused in the industry with a sustainable water reclamation system, mitigating fresh water scarcity and environmental pollution. To study the hybrid process, the

optimization of critical operational parameters of EC are mandatory to establish for BPOME treatment. Many researchers have studied the effect of operational parameters on EC efficiency. However, few studies are found that investigate the parameter interaction and the combined parametric effect on pollutant removal % in EC for BPOME. Therefore, observing the effect of critical operating parameters namely current density, initial pH and time, and their combined effect on pollutant removal efficiency and EC optimization in this study, propels the advancement of this sustainable technology in the palm oil industries. Besides, the fouling studies of the following membrane filtration is necessary to explain the pore blocking mechanism in play while purifying the EC treated effluent that started with BPOME, and providing information for industrial scale up and specific fouling mitigation strategies. As another stepping stone for sustainably producing cleaner effluents for discharge and potential water reclamation, this study paves a way to the direction of accomplishing the SDG 6, that strongly aims to reduce and reuse industrial effluents with its Target 6.3 (SDG 6 Synthesis Report, 2018).

The effluent from the proposed EC-membrane process is expected to not only meet the discharge standards, but also to be reusable in the palm oil industry as process water. EC process involves application of current on the wastewater through metal electrodes, destabilising the charge of the pollutants and separating them in the form of flocs. In the process of EC, the electrodes (anodes) reduce in size due to metal dissolution. Therefore, the electrodes need to be replaced from time to time. Besides EC, membrane technology is a booming field for separation processes. However, the COD, turbidity, color and TSS in BPOME are too high to be treated with membrane leading to instant fouling and membrane dysfunctionality. To combat this issue, EC-

membrane for BPOME stands out as the most suitable, sustainable and environmentally friendly alternative.

Studies have shown promising outcomes employing EC-membrane process for treating various types of wastewater namely textile wastewater, oily wastewater and bilge water. However, a gap remains in exploring the integrated process for treating BPOME. The EC critical process parameters vary with different wastewater types. There is a need to understand the critical parameter (current density, pH and time) interaction effects on pollutant removal efficiency. Also, in the studies relating EC-membrane integrated processes, there is a lack of membrane fouling study, which is important to understand the dominant fouling mechanism for EC treated BPOME. The resulting fouling constants determined contribute as raw materials for industrial scale up of the membrane filtration system and also enable strategic development of appropriate antifouling strategies. Also, it is interesting to investigate the potential enhancement of the EC-membrane process by coupling EC with powdered activated carbon (AC). A consequent improved flux and potential fouling mitigation along with improved permeate quality can support a long term membrane integrity, maintaining low footprint, easy maintenance and environmental sustainability, besides enhanced treatment efficiency.

1.3 RESEARCH OBJECTIVES

The overall aim of this research is to propose a sustainable water reclamation process for BPOME reusability in the palm oil industry. Therefore, by exploring the potential of the EC-membrane integrated process, to achieve the aim, this research can be broken down to the following objectives:

1. To optimize the operational conditions namely current density, initial pH and time of EC process on synthetic wastewater using RSM and verify with real BPOME
2. To establish the best pore blocking model on the crossflow ultrafiltration process for the EC treated BPOME
3. To enhance EC-membrane process by coupling EC with the addition of commercially available adsorbent and investigating its effect on final effluent quality and membrane fouling

1.4 SCOPE OF RESEARCH

This research focuses on EC process followed by membrane filtration as hybrid process for water reclamation.

1. First, optimization of operational variables was carried out on synthetic wastewater which was prepared in the laboratory to model BPOME with COD, nitrogen and phosphorus ratio adapted from Nopens et al. (2001). This approach of employing synthetic POME preparation for this research led to a more solid characterization, reliability and repeatability of results for optimization of operational variables compared to BPOME as their values vary from time to time due to natural degradation and irregular effluent discharge.
2. Then, the optimized parameters were verified with EC-membrane hybrid process on actual BPOME. The BPOME was stored at 4°C to prevent biodegradation.
3. This research concentrated on modification of initial pH (3-8), EC time (15-60 min) and current density (40-160 mA/cm² i.e. 0.44-1.75 in terms of applied current) for optimization using Al electrodes, following the experimental runs designed via Design Expert software version 13.0.