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/cm^2 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/cm^2 (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 حيوياً، 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 حيوياً، 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 محسّنة 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 تآزريه 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 حيوياً 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 Hermiaلسد المسام. التخثير الكهربائي المعزز من خلال إضافة الكربون المنشط، الذي يتم تشغيله باستخدام العوامل المحسّنة، 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 المحسّنة، 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 التقاطيع. 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 5±1 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 القاذورات 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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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 (Boczkaj & 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/cm^2 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.