INVESTIGATION OF ENHANCED ELECTROCOAGULATION-MEMBRANE PROCESS FOR WATER RECLAMATION FROM PALM OIL MILL EFFLUENTS

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ABSTRACT: The process of electrocoagulation (EC) enhanced with adsorbent addition, as a pre-treatment for ultrafiltration membrane, is widely unexplored in oil palm-based wastewater treatment. Utilizing predetermined EC operational parameters and a defined activated carbon (AC) dosage for biotreated palm oil mill effluents (BPOME), membrane fouling was studied during crossflow membrane filtration at 0.5 bar transmembrane pressure and 1 kDa membrane pore size. The dominant fouling mechanism in membrane filtration without EC-AC pretreatment of BPOME, was cake formation, which was determined through Hermia's pore blocking models. However, after EC-AC pre-treatment, the membrane fouling was mitigated. Moreover, the pre-treatment process, AC assisted EC, sustainably enhanced the final treated effluent quality in addition to enhancing fouling mitigation in the subsequent membrane filtration. The removal of Total Suspended Solids (TSS), turbidity and color were nearly 100% and Chemical Oxygen Demand (COD) was 99.7% removed with final value of 5 ± 1 mg/L, which is within the range of reusable water standards.

ABSTRAK: Proses elektrokoagulasi (EC) yang ditingkatkan dengan bahan penyerap, adalah pra-rawatan bagi membran penuras ultra. Walau bagaimanapun ianya masih belum luas diterokai dalam sistem rawatan air buangan berasaskan kelapa sawit. Mengguna pakai parameter operasi EC pra-tentu dan dos karbon aktif tentu (AC) bagi bio-rawatan efluen kilang kelapa sawit yang terawat (BPOME), mendakan membran telah dikaji menggunakan teknik penurasan membran aliran silang pada tekanan transmembran 0.5 bar dan saiz liang membran 1 kDa. Mekanisme mendakan membran kotoran dominan dalam penurasan membran tanpa pra-rawatan EC-AC BPOME, adalah pembentukan kek, iaitu terhasil melalui model penyumbatan liang Hermia. Walau bagaimanapun, selepas pra-rawatan EC-AC, secara mampan dapat menambah kualiti akhir efluen terawat selain dapat meningkatkan mitigasi kotoran mendakan dalam penurasan membran seterusnya. Penyingkiran Total Pepejal Terampai (TSS) adalah 99.7%, kekeruhan dan warna adalah hampir 100%. Keperluan Oksigen Kimia (COD) tersingkir sebanyak 99.7% dengan nilai akhir sebanyak 5±1 mg/L, iaitu dalam julat piawaian air boleh guna semula.

KEYWORDS: electrocoagulation; membrane filtration; activated carbon; membrane fouling

1. INTRODUCTION

Sustainable wastewater treatment technology is a highly sought objective of the current global trend of the industrially flourishing oil palm industry. With advances in technology and global demands, the palm oil production limits tend to increase with time. However, the environmental impact of the rising industrial works and production is often partially or completely overlooked. Over time, the detrimental effects of high industrial wastewater discharge, loss of marine ecosystems, overuse of fresh water supply and environmental pollution can collectively cause a massive global downfall of the public health, the ecosystem, as well as the economy [1].

There are ongoing efforts to sustainably reclaim water from industrial effluents to cut down the overuse of fresh water supply and its global annual decline. Although most processes depicted good outcome on pollutant removal efficiency with extensive use of chemicals, oxidants, processes requiring large footprints, and complex water treatment processes, there is a crucial need for considering economic sustainability and environmental friendliness. Therefore, electrocoagulation (EC) is captivating researchers towards developing a low footprint-based technology with simplicity and versatility in pollutant removal across various wastewater types, and with low sludge production while mitigating harmful chemical-based treatments [2]. Moreover, hybrid technologies have emerged for water reclamation for industrial reuse [3]. Membrane technology is such a booming field in water treatment with its low footprint and selective pollutant rejection ability [4].

However, water reclamation based on membrane technology is costly and faces obstacles with inevitable fouling (pore blocking) at the membrane surfaces causing an overtime loss of flux which can be overcome by physical or chemical washing to regain original membrane functionality [5]. However, EC integration as pre-treatment for membrane process can potentially enhance wastewater treatment for water reclamation as it removes colloidal pollutants through charge destabilization, allowing a major cut down of pollutants entering the membrane. Consequently, fouling could be minimized to an extent of reversibility, helping to extend membrane functionality for an extensively long period of time and avoiding constant membrane replacements. Although chemical coagulation pre-treatment generally enhanced membrane filtration, there is a potential for EC based integrated processes to replace chemical coagulation as EC has proven to be more environmentally friendly and economical, with lower sludge production and easier operation avoiding harsh chemicals [6].

However, there are limited studies that explored EC-membrane processes enhanced with activated carbon for water reclamation from industrial wastewater. Palm oil industries treat their organic-matter-rich effluents and discharge the final effluent known as biotreated palm oil mill effluents (BPOME). BPOME is a dark brown and turbid looking wastewater, which is rich in organic matter, released in settling ponds as the final effluent. This study aims to investigate the potential of hybrid EC-membrane process with activated carbon (AC) addition to reclaim water from BPOME.

2. METHOD

BPOME was collected from the final discharge pond in a palm oil mill based in Negeri Sembilan, Malaysia. To prevent degradation due to naturally occurring biological activity, the collected sample was stored at 4 °C. The EC process was carried out with pre-optimized operating conditions of current, initial pH, and time of 1.75 A, 6, and 15 minutes just as the study carried out in [7] following the same setup, anode surface area (10.93 cm²), interelectrode distance (10 mm), and the same reactor (200 ml capacity beaker) and direct current supply unit

from Twintex (TP-2303K, Taiwan). This study incorporated further modification by activated carbon (AC) addition of 1 wt% as the study by [8].

The wastewater characterizations included COD, color, and total suspended solids (TSS) before and after EC using a spectrophotometer (HACH DR 5000, USA) according to American Public Health Association (APHA) standards [9]. A DRB 200 Reactor (HACH DRB 200, USA) was used to heat the COD vials (2 hours for 150°C) before COD measurement. The initial pH of the samples was controlled with 5% HCl and 0.1 M NaOH solutions with a pH meter (Mettler Toledo, MP220 model, USA). A multi-meter (HACH sensION5, USA) was used to measure the TDS, conductivity, and salinity of the wastewater samples. Turbidity of the samples was measured using a standard nephelometric method with a turbidity meter (EXTECH Instruments TB400, Taiwan). For separation of flocs after EC treatment, 0.45 μ m pore sized filter papers were used.

After EC, the treated BPOME was further treated with an ultrafiltration membrane. Crossflow filtration mode is one of the ways of physically reducing the concentration polarization and fouling rate by delaying the cake build up, varying the feed flow hydrodynamics [10]. Furthermore, membrane backwash via chemical cleaning contributes to removal of reversible fouling, whereas irreversible fouling diminishes the membrane performance over time, leading to membrane replacement [11]. However, having the influent feed pre-treated (i.e., EC in this study) to remove maximum possible colloids and pollutants, is one of the best ways to achieve fouling mitigation [5]. The approach of [12] was used to run the ultrafiltration membrane experiment in a crossflow mode of operation. The schematic diagram of the membrane filtration system set up used in this work is shown in Fig. 1. Before beginning the experiment, the membrane was soaked overnight in distilled water to remove impurities left over from the manufacturing processes or additives used in preserving and stabilizing the membrane. The next day, the membranes were wetted out again by circulating distilled water at 2.0 bars for 30-60 minutes. This procedure helped in preventing membrane compaction during permeation. The distilled water flux was measured before each experiment with a clean membrane at a pressure within 1 to 2 bars.



Fig.1. Schematic diagram of the crossflow filtration setup, adapted from [13].

A polyethersulfone membrane with a Molecular Weight Cut-Off (MWCO) of 1 kiloDaltons (kDa) and transmembrane pressure (TMP) of 0.5 bar was investigated on its ability to remove COD from the EC treated BPOME. These parameters resulted in the best permeate quality (highest removal of color, COD, and turbidity) in BPOME ultrafiltration in the study

reported by [12], that employed the same crossflow membrane filtration system. A lower TMP, contributed to the highest % COD removal, which was also indicated by the work of [14], where a higher applied pressure led to an enhanced gel layer formation that blocked the incoming organic matter from passing through, consequently reducing the removal of COD, color, and turbidity. The final permeate after crossflow ultrafiltration was characterized to determine the final removal efficiency in terms of COD, turbidity, TSS, and color, and were compared with reusable water standards.

After each experimental run, chemical cleaning was performed by passing 1 N NaOH at 1 bar (TMP) through the filtration membrane to preserve the integrity of the membrane. Additionally, the permeate volume collected was plotted against time to observe the flux. Next, the linearized pore blocking model equations (Table 1) were employed to fit the data, to determine the dominant pore blocking model, where J, J_0 and t represent the flux, initial flux, and time respectively, and K_{cb} , K_s , K_i and K_c represent the pore blocking constants for complete, standard, intermediate, and cake filtration models, in Eq. (1) to Eq. (4), respectively.

Pore blocking model	Linearized equation	
Complete	$\ln J^{-1} = \ln J_0^{-1} + K_{cb}t$	(1)
Standard	$J^{-0.5} = J_0^{-0.5} + K_s t$	(2)
Intermediate	$J^{\text{-}l} = J_0^{\text{-}l} + K_i t$	(3)
Cake filtration	$J^{-2} = J_0^{-2} + K_c t$	(4)

Table 1: Linearized pore blocking equations for membrane filtration [15]

The energy consumption of the treated BPOME volume, at optimized conditions, was calculated with Eq. (5) and Eq. (6) in Table 5 for EC and membrane filtration, respectively. The symbols I(A), U(V) and t(hours) represent the current applied, resulting voltage, and the time of EC operation, respectively. The flux J (L/(m².min)), effective membrane surface area of 0.1 m² and transmembrane pressure, TMP (bar), are the parameters for the membrane filtration energy consumption in Eq. (6). V represents the working volume (m³) for EC and permeate volume (m³) for crossflow filtration.

Although COD removal was monitored after the EC process, concerns remain for the possibility of the presence of aluminum ions from the aluminum cathode in the final treated water. Aluminum contamination can be detrimental to human health as it has been known to increase the risk of dementia and Alzheimer's disease [16]. Therefore, the final treated effluent was tested for the presence of aluminum and possible trace metals to observe the impact of the EC and EC membrane process on BPOME. The ICP-MS technique is greatly beneficial for water analysis to determine trace metals, due to its sensitivity and the ability of multi-element detection, hence, the USEPA have employed this technique for water and waste analyses [17].

3. RESULTS AND DISCUSSION

With the EC optimized parameters and addition of powdered AC, the treated effluent quality was enhanced to the extent of fouling mitigation in the subsequent crossflow membrane filtration step of the hybrid process. The best outcome was achieved with 1 wt. % addition of AC in the EC reactor, and the removal of TSS, turbidity and color were nearly 100 % and COD was removed 99.7% with final average value of 5 mg/L, which lie within the range of process water standards (in terms of the stated key parameters, namely COD, TSS, turbidity, and color, as in the scope of this study).

Implementing the method carried out previously in [8], BPOME was treated with EC assisted with 1 wt% of AC addition. The resulting treated wastewater was used for studying pore blocking behavior in crossflow membrane filtration. Not all COD was removed, and a considerable amount of COD was left in the EC treated solution alone. However, with AC addition, the fastest removal % noted was of TSS from BPOME for all concentrations of powdered AC added, followed by turbidity, color, and COD. The combined action of the adsorption of colloids by both AC along with the metal coagulants generated from the Aluminum metal anode in the EC reactor, greatly enhanced the efficiency of the EC process, that contributed to a much higher pollutant removal rate. Membrane pore blocking studies were performed to further study the mechanism of the fouling in membrane ultrafiltration of BPOME for both with and without EC-AC pre-treatment.

3.1 Membrane Pore Blocking Study without EC-AC Pre-Treatment

The linearized pore blocking equations by [15], listed in Table 1, have been employed in this work to establish the prominent fouling mechanism prevailing in the crossflow filtration step. The linearized pore blocking models contribute not only to establish the dominant fouling mechanism taking place to explain the flux decline in the ultrafiltration membrane but also significantly contribute to the scale up design in the industrial scale, providing the pore blocking constant values [18]. Moreover, understanding the fouling mechanism enables the devising of antifouling strategies to overcome the loss of flux and preserve membrane integrity for longer time. Based on the linearized pore blocking model plots observed in Fig. 2 to Fig. 5, the trendlines after 10 minutes, beyond initial stage of crossflow operation, represent the relationship between the permeate flux with time with the respective pore blocking model. The pore blocking constants, along with the R^2 and J_0 (y-intercept) of each model, are summarized in Table 2, depicting the fitness of the experimental data with different pore blocking models.

	Table 2: Summary of pore blocking constants, R^2 and J_0				
	Pore blocking model	Pore blocking constant	J ₀ (intercept) L/m ² .min	R ²	
1	Standard	$K_{\rm s} = 0.0078$	0.3452	0.9534	
2	Intermediate	$K_{\rm i} = 0.0296$	0.3484	0.9605	
3	Complete	$K_{\rm cb} = 0.0083$	0.3425	0.9450	
4	Cake filtration	$K_{\rm c} = 0.2109$	0.3570	0.9708	



Fig. 2: Linearized plot for permeate flux vs time for standard pore blocking model.



Fig. 3: Linearized plot for permeate flux vs time for intermediate pore blocking model.



Fig. 4: Linearized plot for permeate flux vs time for complete pore blocking model.



Fig. 5: Linearized plot for permeate flux vs time for cake filtration model.

The most suitable fouling model was determined by assessing the regression of the models (Table 2). With the highest R^2 of 0.9708, cake filtration stood out as the dominating pore blocking mechanism in this study. After the cake filtration model, the intermediate pore blocking model depicted the next highest R^2 of 0.9605. Therefore, the intermediate pore blocking contributed the most, leading towards the cake formation. This outcome of prevailing cake filtration fouling corresponds to the work of [14] where the membrane filtration depicted cake filtration as the dominant pore blocking mechanism for oil palm effluent, feedwater, and dairy wastewater respectively. Hence, overall, the conclusions established in these studies suggest the cake filtration model to be the most universal model that describes the fouling mechanism of wastewater membrane filtration.



Fig. 6: Comparison of flux decline with and without EC-AC treatment.

Moreover, the flux decline in the ultrafiltration membrane was observed with and without EC-AC treated BPOME and is presented in Fig. 6. Notably, there is a decline in the flux after the initial phase of filtration, the flux was constant throughout, denoting no fouling took place as it was free from pollutants that cause membrane fouling for EC-AC treated BPOME ultrafiltration. To further explore the membrane fouling conditions, a membrane pore blocking study was carried out.

3.2 Membrane Pore Blocking Study with EC-AC Pre-Treatment

After collecting the EC (coupled with AC) treated BPOME, a crossflow ultrafiltration membrane was run with TMP 0.5 bar, effective membrane surface area of 0.1 m^2 and noting the time taken after every 100 mL volume of permeate achieved, freshwater flux was determined with deionized water, followed by BPOME.

As the observed permeate flux was nearly constant with time corresponding to the gradient of the linearized flux graphs for all the models (Fig. 7 to Fig. 10), it was concluded that the level of purity obtained in the EC with the addition of powdered AC prevented any type of fouling to take place in the crossflow filtration membrane. This result agrees with the notable work of [19], where incorporation of powdered AC pre-treatment by adsorption, significantly contributed to fouling mitigation along with pigment removal before carrying out membrane filtration. Therefore, exploring addition of powdered AC in this work, enhanced color removal in the EC process and greatly contributed to fouling mitigation of the subsequent crossflow membrane filtration in BPOME treatment.

3.3 Characterization of Permeate after Membrane Filtration

The permeate obtained after membrane filtration of BPOME pretreated with EC-AC, was characterized and compared with EPA standards for reusable water. The main parameters monitored for pollutant removal efficiency in this work were COD, turbidity, TSS, and color.

Besides, the final characterization of the permeate (Table 3) after the EC-membrane process, where EC was enhanced with addition of powdered AC in the reactor, 99.7% of COD was removed, with 5 ± 1 mg/L of final COD value. Besides COD, almost 100% of TSS, color and turbidity were removed from the BPOME, with hybrid process. This water quality (based on color, TSS, COD, and turbidity) meets not only the standards of irrigation water but also process water for industrial reuse [20].



Fig. 7: Linearized plot for permeate flux vs time for standard pore blocking model for crossflow filtration of EC-AC treated BPOME.



Fig. 8: Linearized plot for permeate flux vs time for intermediate pore blocking model for crossflow filtration of EC-AC treated BPOME.



Fig. 9: Linearized plot for permeate flux vs time for complete pore blocking model for crossflow filtration of EC-AC treated BPOME.



Fig. 10: Linearized plot for permeate flux vs time for cake formation model for crossflow filtration of EC-AC treated BPOME.

Table 3: Characterization of permeate after crossflow filtration of EC-AC treated BPOME

	Parameters	Initial BPOME	Final permeate	Reusable limits (EPA)	Overall removal %
1	COD (mg/L)	1981	5±1	<30	99.7
2	Turbidity (NTU)	332	0.3	<2	99.9
3	TSS (mg/L)	192	0	<10-30	100
4	Color (PtCo)	2882	0	<5	100

After addition of powdered AC, the enhanced EC process with the destabilization of colloids with the action of AC adsorption enabled the removal of maximum TSS and turbidity, and a large quantity of COD and color. However, without AC addition, the resulting EC treated solution was not compatible enough to be treated with crossflow filtration to achieve process water COD. The physical observation of the treated BPOME at each stage is presented in Fig. 11. Notably, the final EC-AC treated output was transparent but not colorless and had a significant quantity of COD remaining without the addition of AC. However, with added AC, the EC process was enhanced with rapid removal of color, TSS and turbidity, with 100% color removal and maximum COD removal (5 ± 1 mg/L) subsequently after crossflow ultrafiltration membrane process from BPOME was proven in this study, after EC was enhanced with AC addition in the same EC reactor, incurring no additional unit operation and harsh oxidants.



Fig. 11: Appearance of BPOME after EC (with AC addition) treatment and crossflow filtration.

Metals	Initial concentration in BPOME, mg/L	Concentration after EC-membrane treatment, mg/L	Reusable EPA standard, mg/L [20]
Al	5.93	0.20	5
Fe	1.51	0.09	5
Cu	2.92	0.24	0.2
Zn	0.26	0.02	2
Ni	8.45	0.52	0.2
Cr	29.99	1.81	0.1
Pb	1.05	0.06	5
Со	0.62	0.03	0.05
Cd	0.03	0.00	0.01

Table 4: Concentration of trace metals in BPOME after EC and EC-membrane process

According to the USEPA, 5 mg/L is the discharge limit for aluminum for water reuse in agriculture [20]. From the ICPMS analysis data presented in Table 4, EC-membrane process does not cause the possible leaking of aluminum ions in the final discharge from aluminum electrodes in EC. Moreover, the final concentration after the process is almost 97% less than the initial concentration in the BPOME sample, besides being lower than the EPA standard for reuse. Hence, it was confirmed that no possible aluminum contamination takes place in the treated effluent due to aluminum anode dissociation in EC. Rather, EC with AC assistance, removed over 90% of most of the trace metals present in the real BPOME, and additionally removed most of them to the reusable limits for irrigation water according to EPA guidelines

[20]. Investigating possible metal removal from BPOME was not in the scope of this research, but the finding supports the versatility of the EC process in removing various types of pollutants with its effective colloidal destabilization. Hence, the quality of EC being a sustainable environmentally friendly water treatment process is notably exhibited in this study, which corresponds to the work of [21].



Fig. 12: Trace metal removal through ICPMS after EC and EC-membrane process on BPOME.

Figure 12 presents the percentage removal of trace metals namely Al, Fe, Cu, Zn, Ni, Cr, Mn, Pb, Co, and Cd, after EC and EC-membrane process on BPOME. The graph shows that EC alone removes the maximum possible percentage of trace metals, and no significant removal was observed after the ultrafiltration membrane. The coagulant formed from anode dissolution in EC results in the pollutant-coagulant complex formation after destabilization [2], hence separating it from the BPOME along with COD. Although the study aimed at COD removal from BPOME, the additional ICPMS result proved a subsequent removal of metals that were initially present in the wastewater. This occurrence of a versatile pollutant removal ability of EC from wastewater opens possibilities for further exploration of the process and its integration with other processes for a desired water purity for reusability in the industry as well as in the household or as drinking water. Besides the removal efficiency of the overall EC-membrane hybrid process, the energy consumption was evaluated in the following section.

3.4 Energy Consumption

The calculated energy consumption by the EC-membrane process is presented in Table 5.

Energy consumption	Formula	Results
EC	$\frac{I \times U \times t}{V} $ (5)	27.34 kWh/m ³
Membrane filtration	$\frac{J \times A \times TMP}{V} (6)$	0.0138 kWh/m ³
Total EC-membrane p	27.354 kWh/m ³	

Table 5: Energy consumption of EC-membrane process

It is evident from this outcome, that EC is the process in the overall hybrid system that holds the main contributing role to the energy consumed in the overall hybrid process. As EC is primarily based on applied current for colloidal destabilization, it is predictable that it consumes the most energy to treat the volume of BPOME in this study. Though the power consumed varies based on reactor geometry, wastewater type, electrolyte conductivity and pollutants present, a similar result was reported by [22], where maximum removal % of a specific pollutant (dye) was achieved from textile wastewater by EC treatment with a power consumption of 17.04 kWh/m³. However, working with a larger volume with addition of supporting electrolytes and employing multiple sacrificial anodes are reported to reduce power consumption in EC [23]. The resistance of the wastewater is a crucial determining factor of how much voltage is generated in the EC reactor, at a specified current.

When scaled up with respect to electrode surface area to volume ratio in a 500 ml beaker, at the same applied current, the voltage dropped to about 6.5 V. The energy requirement by EC can be minimized by scaling up the reactor volume, for instance with 2.5 L working volume, despite the low pollutant removal efficiency in EC on wastewater, a very low amount of energy was consumed (0.36 kWh/m³). Therefore, exploring the scale up of the EC reactor volume for a reduced power consumption is a strong recommendation to carry this study forward.

The energy consumed in the crossflow filtration process is mainly due to the pressure applied for the feed inlet, retentate and permeate flow. The pressure driving the overall process is denoted by the TMP (0.5 bar), the value of which was used in Eq. (6) to calculate the energy consumption. The energy consumed in the crossflow filtration of EC treated BPOME was 0.0138 kWh/m³, which is very small, and therefore, very economical for treating huge volume of feed [24]. Similar conclusion was made in the study by [25], where using an ultra-low TMP for crossflow filtration, significantly reduced the amount of energy consumed (<0.03 kWh/m³) for the ultrafiltration of oil/water emulsions. Further reduction of TMP leads to a much lower overall energy consumption, but the overall flux and time taken for the desired permeate volume are compromised.

The total energy consumption of the overall hybrid EC-membrane process was calculated to be 27.35 kWh/m³. As a small volume of BPOME had been treated as a batch process in the EC study, the energy consumed appears to be large in terms of per m³ of treated sample. However, this can be combatted by scaling up the EC process to treat a larger BPOME volume. As BPOME undergoes natural biodegradation, its quality was not maintained completely constant throughout the experiment. However, storing the sample at 4°C helped to minimize biodegradation. The BPOME samples were characterized in every experiment and the average value was used to compare the overall treatment efficiencies. Besides, exploring the scale up of the EC reactor volume with multiple anodes for a more reduced power consumption is a recommendation to carry this study forward.

4. CONCLUSIONS

The fouling mechanisms in crossflow membrane filtration of BPOME were analyzed with and without adding powdered AC in the EC process prior to the membrane filtration. When powdered AC was coupled with the EC process in the BPOME treatment, fouling mitigation was noted. The final permeate obtained was 100% free of TSS and color, 99.9% of turbidity and 99.7% of COD, with a final COD value of 5 ± 1 mg/L which met the agricultural and industrial water reuse standards. Further, the overall energy consumption (27.35 kWh/m³) largely resulted from EC, as it was solely based on application of direct current. Based on the small working volume used in this study, the high amount of energy consumption is reasonable, and can be overcome with effective scale up strategies, which is recommended to carry this research forward. Moreover, the subsequent removal of metals with EC, that were initially present in the wastewater, measured with ICP-MS, suggests possibilities of further exploration of the process for enhanced water reclamation from wastewater.

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