

ELECTROCOAGULATION USING NOVEL CONFIGURATIONS
ELECTRODES FOR TREATMENT OF TEXTILE WASTEWATER

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DEDICATION

To Allah (SWT), my beloved mother and father

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ABSTRACT

Textile wastewater is considered as one of the most polluted wastewater. Conventional chemical coagulation is the most famous technique used to treat the textile effluent. However, this method produces low removal rate, long retention time and a large quantity of sludge and chemicals. The electrocoagulation (EC) technology is an active wastewater treatment used before sedimentation and filtration. It is still in the "black box" stage where process design, control, and optimization have been primarily empirical. In this research, the EC process was performed in two models for the treatment of textile wastewater. The first model is the conventional reactor with a new configuration that has static electrodes, classified in two phases. The two phases are the EC process alone, using Mp Al-Bp Al or Mp Al-Bp Fe and EC combined with electrooxidation process (EO) in the same reactor using MpTi-Bp Al or MpTi-Bp Fe. The second model is a novel reactor design with a rotating anode using aluminum electrodes. The rotating anode consists of 10 impellers supported by a shaft with 10 rings used as a cathode. The operational parameters were investigated, and the process was performed under optimal conditions. For model one, the results showed that the Mp Al-Bp Al was more effective than the Mp Al-Bp Fe and it was more efficient than chemical coagulation. The optimal combination (EC-EO) treatment was established with Mp Ti-Bp Al, and it is found to be more active than EC alone. The optimal parameters of the EC process with a novel rotating anode were 4 mA/cm² current density, 150 rpm rotational speed, and 10 minutes reaction time. EC process with a rotating anode was more active than model one and chemical coagulation, where the removal efficiency was higher, and the operational cost were reduced significantly. The increase of impellers anode diameter led to enhance the mass transfer coefficient of ionic aluminum. This result was confirmed by computational fluid dynamic (CFD) simulation results. By solar cell supply, the EC process with rotating anode reactor using batch and continuous flow regime had almost similar removal rate. Zeta potential tests showed that reaction was chemo-adsorption, and this was validated by the X-ray diffraction (XRD) analysis. Moreover, the reaction product was environmental friendly. Hydrogen production was improved at a rotation speed of anode. The electrode passivation was reduced by increasing rotational speed of anode which led to an improved in the EC process performance and validated the reactor design. Finally, the contribution of this study is a novel EC reactor with a rotating anode which is more efficient and economic as compared to the conventional coagulation process in the textile wastewater treatment.

ABSTRAK

Air sisa tekstil dianggap sebagai salah satu air sisa yang paling tercemar. Penggumpalan bahan kimia konvensional adalah teknik yang paling terkenal untuk merawat efluen tekstil. Namun begitu, kaedah ini mempunyai kadar penyingkiran yang rendah, masa penahanan yang lama, dan kuantiti enapcemar dan bahan kimia yang banyak. Teknologi elektroenap cemar (*electrocoagulation*, EC) merupakan rawatan air sisa aktif yang digunakan sebelum proses pemendapan dan penapisan. Teknologi ini masih dalam peringkat percubaan memandangkan reka bentuk proses, kawalan dan pengoptimuman masih di tahap empirikal. Dalam kajian ini, proses EC telah dilaksanakan dengan menggunakan dua model untuk rawatan air sisa tekstil. Model pertama ialah reaktor konvensional dengan konfigurasi baru yang menggunakan elektrod-elektrod statik, yang diklasifikasikan dalam dua fasa. Dua fasa tersebut ialah proses EC sahaja yang menggunakan Mp Al-Bp Al atau Mp Al-Bp Fe dan proses EC digabungkan dengan elektropengoksidaan (*electro-oxidation*, EO) dalam reaktor yang sama menggunakan Mp Ti-Bp Al atau Mp Ti-Bp Fe. Model kedua ialah reka bentuk reaktor baharu dengan anod berputar menggunakan elektrod aluminium. Anod berputar terdiri daripada 10 pendesak yang disokong oleh aci dengan 10 cecincin sebagai katod. Parameter operasi disiasat dan proses ini dilaksanakan dalam keadaan yang optimum. Keputusan bagi model pertama menunjukkan Mp Al-Bp Al lebih berkesan berbanding Mp Al-Bp Fe dan lebih cekap berbanding penggumpalan kimia. Rawatan gabungan optimum (EC-EO) dibina dengan Mp Ti-Bp Al dan rawatan ini didapati lebih aktif berbanding EC sahaja. Parameter optimum untuk proses EC dengan anod berputar baharu adalah 4 mA/cm^2 untuk ketumpatan semasa, kelajuan putaran pada 150 rpm, dan 10 minit untuk masa tindak balas. Proses EC dengan anod berputar adalah lebih aktif berbanding model pertama, kecekapan penyingkiran proses penggumpalan kimia adalah lebih tinggi dan kos operasi berjaya dikurangkan dengan ketara. Penambahan diameter anod pendesak menjurus kepada peningkatan pekali pemindahan jisim bagi aluminium ionik. Keputusan ini disahkan dengan keputusan simulasi pengiraan dinamik bendalir (CFD). Berbekalkan sel solar, proses EC dengan reaktor anod berputar menggunakan rejim kelompok dan rejim aliran berterusan menunjukkan kadar penyingkiran yang hampir sama. Ujian potensi Zeta menunjukkan bahawa tindak balas yang berlaku ialah kemo-jerapan, sebagaimana yang disahkan oleh analisis belauan sinar-X (XRD). Selain itu, produk tindak balas ialah mesra alam. Pengeluaran hidrogen adalah bertambah baik dengan kelajuan putaran anod. Pempasifan elektrod dikurangkan dengan meningkatkan kelajuan putaran anod yang mana meningkatkan prestasi proses EC dan mengesahkan reka bentuk reaktor. Akhir sekali, sumbangan kajian ini adalah reaktor EC baharu dengan anod berputar yang lebih efisien dan lebih ekonomi berbanding dengan unit kimia konvensional untuk rawatan air sisa tekstil.

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LIST OF SYMBOLS

I	-	Current intensity (A)
Mw	-	Molecular weight of the substance (g/mol)
F	-	Faraday's constant (96485 C/mol)
z	-	Number of electrons (2 for Fe ²⁺ and 3 for Fe ³⁺ and Al ³⁺)
m	-	Quantity of metal dissolved (g)
XRD	-	X-ray diffraction
Mp Al- Bp Al	-	Monopolar aluminum –Bipolar aluminum
AC	-	Alternating current
DC	-	Direct current
η_{AP}	-	Applied overpotential (V)
η_k	-	Kinetic overpotential (V)
η_{Mt}	-	Concentration overpotential (V)
η_{IR}	-	Overpotential caused by solution resistance or IR-drop (V)
IR-drop	-	Drop of internal resistance between electrodes (V)
d_e	-	Distance between the electrodes (cm)
A	-	Surface area of the cathode (cm ²)
w	-	Specific conductivity of the solution ($\mu\text{S}/\text{cm}$)
Km	-	Mass transfer coefficient (cm/sec)
i_L	-	Limiting current (A)
n	-	Electron moles (3 for Al and 2 for Fe)
C_b	-	Bulk concentration (mol/cm ³)
D	-	Diffusion coefficient or particle diffusivity (cm ² /s)
K_B	-	Boltzmann constant ($1.388 \times 10^{-16} \text{ g}\cdot\text{cm}^2/\text{s}\cdot\text{k}$)
T	-	Temperature (298 K)
μ	-	Viscosity of solution (g/cm \cdot s)
d_o	-	Particle (ionic aluminum or iron) size (cm)
Re	-	Reynolds number

Sh	-	Sherwood number
Sc	-	Schmidt number
d	-	Effective diameter (cm)
N	-	Rotational speed (rps)
ρ	-	Fluid density (g/cm^3)
a and b	-	Constants of model mass transfer
\emptyset	-	Variable velocity along x, y, and z direction (cm/sec)
C_0	-	Concentrations of pollutants before treatment (mg/L)
C	-	Concentrations of pollutants after treatment (mg/L)
$m_{\text{theoretical}}$	-	Theoretical amount of aluminum or iron generated (mg/L)
M	-	Molecular mass of Al (26.98 g/Mol) and Fe (55.84 g/Mol)
RT	-	Reaction time of treatment (min)
V	-	Volume of wastewater (L)
DO ₀	-	Dissolved oxygen concentration immediately (mg/L)
DO ₅	-	Dissolved oxygen concentration after 5 days(mg/L)
SRF	-	Specific resistance to filtration (m/kg)
Kb	-	Slope of the volume vs time/volume plot
P	-	Pressure during sludge filtration (Pas)
a_w	-	Weight of the solid per unit volume of filtrate (g)
m_2 and m_1	-	Mass of the cup (with the membrane) before and after filtration
m_3	-	Mass of cup after drying process
SVI	-	Sludge volume index (mL/g)
VD ₃₀	-	Volume of settled sludge after 30 min (mL/L)
C_{energy}	-	Energy consumption (kWh/m^3)
$C_{\text{electrode}}$	-	Electrodes consumption (kg/m^3)
C_{sludge}	-	Amount of sludge (kg/m^3)
$C_{\text{chemicals}}$	-	Quantity of chemicals (kg/m^3)
a	-	Total electricity costs (US\$/kWh)
b	-	Cost of aluminum or iron (US\$/kg)
d	-	Cost of sludge disposal(US\$/kg)
e	-	Cost of chemicals added (US\$/kg)
U	-	Voltage (volt)
$(C_{\text{energy}})_s$	-	Energy consumption of DC power supply (kWh/m^3)

$(C_{\text{energy}})_M$	-	Energy consumption of DC motor anode rotation (kWh/m ³)
C.E	-	Current efficiency (%)
M_{rpm}	-	Mixer rotational speed (rpm)
CD	-	Current density (mA/cm ²)
K_B	-	Boltzmann constant (1.388×10^{-16} g·cm ² /s·k)
d_0	-	Aluminum size (cm).
n_{H_2}	-	Amount of hydrogen generated (mole)
H	-	Number of hydrogen molecules per electron in the redox reactions (1/2)
V_{H_2}	-	Volume of the cumulative hydrogen (L)
m_{H_2}	-	Mass of the produced cumulative hydrogen (g)
M_{H_2}	-	Hydrogen's molar mass (2 g/mol)
R	-	Gas constant (0.082 L atm. mol ⁻¹ K ⁻¹)
E_{H_2}	-	Energy contents of the hydrogen gas (kW.h/m ³)
Q	-	Discharge flow rate (L/min)
R_{ct}	-	Passivation resistance (Ω)
R_{ads}	-	Adsorption resistance (Ω)
C_{ads}	-	Adsorption capacitance (μF)
C_{dl}	-	Double layer capacitance (μF)
R_s	-	Solution resistance (Ω)
U_0	-	Flow velocity (cm/sec)
T	-	Turbulence intensity (%)
SD	-	Standard deviation
R1	-	Simulation results
R2	-	Experimental results
I_1	-	Applied current of power supply (A)
I_2	-	Applied current of motor rotational anode (A)
U_1	-	Voltage of power supply (V)
U_2	-	Voltage of motor rotational anode (V)

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The advancement in the search for new wastewater treatment technologies seems lacking as, fresh water sources are being polluted with increasing population (Ntwampe *et al.*, 2016, Cosgrove, 2010). The developing countries are soft targets of water-borne diseases due to a lack of the knowledge in water decontamination and less authoritative support. A limited resource of water is consumed by the industry acquiescing lower quality raw water, whereas a larger fraction of fresh water is required for human consumption. Wastewater pollutants produced from industry have become a central issue, as these contaminants cause environmental problems which are harmful to public health. It is estimated that nearly half a million organic compounds are synthesized and 10,000 new compounds are added each year (Sarala, 2012).

Some of the wastewater sources, for example, textile wastewater, oily and greasy wastewater from restaurant or canteen, cannot be collated and discharged to municipal drainages directly due to the faultiness of the existing wastewater treatment system. Discharging these wastes into municipal drainages directly, may spoil the drainage system as it will make the treatment process difficult and unviable due to the presence of different types of pollutants (Xu and Zhu, 2004, Vepsäläinen, 2012). With the passage of time, wastewater treatment technology have developed by improving the reliability and competence of treatment systems to meet the certain criteria and lessen the land engaged by treatment works by speeding up the natural treatment rates under defined conditions. Despite the improvements in wastewater

treatment systems, they are still mainly focused on the removal of suspended and floatable materials, the treatment of biodegradable organic and in some cases the removal of pathogenic organisms. Filtration, sedimentation or flotation is the focused areas of particle separation in wastewater treatment processes. Yan *et al.*(2008) reported that to know destabilization of particles by coagulants and flocculants, it is necessary to know the mechanism, which stabilizes particles in aqueous solutions. Chemical coagulation and flocculation are used in both industrial and municipal raw water and wastewater treatment systems due to their efficacy in removal of several types of pollutants from the water streams (Verma *et al.*, 2012, Dorea, 2009).

In past two decades, electrochemical technologies, like electrodeposition, electrocoagulation (EC), electroflotation (EF), electrooxidation and electrokinetic remediation emerged as effective processes due their distribution and smaller amount and number of chemicals required. Furthermore, resilient and compact instrumentation is also available with minimal effort (Mollah *et al.*, 2004a, Barrera-Díaz *et al.*, 2012). These technologies benefit in terms of environmental compatibility, adaptability, energy effectiveness, safety, selectivity, and amenability to automation, and cost reduction (Rajeshwar and Ibanez, 1997). This indicates the potential of electrochemical technologies to serve as localized treatment facilities to substitute sophisticated processes (chemical coagulation and flocculation), which need large volumes and/or number of chemicals, massive containers in a typical wastewater treatment plant (Barrera-Díaz *et al.*, 2012, Chen, 2004, Comminellis and Chen, 2010).

Chemical coagulation has been studied extensively compared to electrocoagulation (EC). For water and wastewater treatment, electrocoagulation is a good technique that avoids many complicated processes involving many chemicals and physical phenomena, e.g. formation of coagulants, destabilization of contaminants, particulate suspension and breaking of emulsions, and flocculation (Mollah *et al.*, 2004a, Chaturvedi, 2013). This process has received substantial consideration so far. It has been widely studied depending on the process behaviours and the operational parameters to remove the pollutants from various type of water or wastewater such as drinking water (Holt *et al.*, 2005), domestic wastewater (Sarala,

2012), laundry wastewater (Janpoor *et al.*, 2011), electroplating wastewater (Verma *et al.*, 2013, Adhoum *et al.*, 2004), oil refinery wastewater (Un *et al.*, 2009), restaurant wastewater (Chen *et al.*, 2000), metal plating wastewater (Al Aji *et al.*, 2012, Kabdaşlı *et al.*, 2009a) and poultry slaughter house wastewater (Bayar *et al.*, 2011, Kobya *et al.*, 2006). However, no attempts have been made to present two different models with a new style for the process optimization.

1.2 Problem Statement

Nowadays, high quantities of wastewater are being produced, especially from the industrial and residential sectors that may affect the environmental life. Industrial activities always cause a problem to the environment and the society. Textile mill effluent needs proper treatment before it is discharged into the environment due to high pollutants characteristics such as chemical oxygen demand (COD), total suspended solid (TSS), biological oxygen demand (BOD), turbidity, and color (Verma *et al.*, 2012). There are many *batiktextile* mills in Malaysia. The effluents of these textile mills mostly have high COD due to intensive dyes use. Moreover, the electrical conductivity of the effluent is high as a result of the presence of sodium silicate which was used during the textile process (Rashidi *et al.*, 2016). Conventional treatment of these textile mills contains primary sedimentation unit, chemical coagulation unit, sedimentation unit, filtration unit, and a collection unit for disposal to the river. In the present study, the conventional chemical coagulation units were focused. The drawbacks of this treatment unit are; low removal efficiency, creates weak and low dense addition of coagulant, long detention time, and high costs coming from large quantity of sludge after treatment, and large quantity of chemicals needed for coagulation and flocculation, and requirements of pH and conductivity (Un *et al.*, 2009, Moreno C *et al.*, 2009, Yildiz *et al.*, 2008, Barrera-Díaz *et al.*, 2012).

For environmental protection, it is important to find the best method to overcome the above problem. Some technologies like biological oxidation, chemical oxidation, advanced oxidation, nanofiltration and adsorption, and electrochemical

technologies such as electrooxidation (EO), electrodeposition and electrocoagulation (EC) can be used to reduce this problem. However, most of the discussed methods have some major drawbacks. For example, biological oxidation is undoubtedly the inexpensive process, but the existence of toxic or biorefractory molecules may hinder this approach due to effluents polluted with organic compounds (Barrera-Díaz *et al.*, 2012, Panizza and Cerisola, 2009). Chemical oxidations have low capacity rates and need transportation and storage of dangerous reactants (Panizza and Cerisola, 2009); advanced oxidation processes require high investment costs (Martinez-Huitle and Ferro, 2006, Panizza and Cerisola, 2009); nanofiltration and adsorption processes are not always sufficient to achieve the discharge limits (Bousher *et al.*, 1997, Wachinski, 2012); and electrooxidation and electrodeposition processes need long reaction times to achieve the treatment (Chen, 2004, Martinez-Huitle and Ferro, 2006).

According to previous research of the EC process, no set configuration is applicable to all needs, and the problem of EC reactor is due to the generating of hydrogen (H₂) and oxygen (O₂) bubbles that accumulate onto the electrode surface and forms an insulating layer (oxide film) (Gunukula, 2011, Mollah *et al.*, 2004a, Khandegar and Saroha, 2013c, Nasrullah *et al.*, 2012). Due to this layer, the electrodes will be passive and it will affect the active time of the electrodes, large electrical energy is required to achieve optimum removal efficiency of pollutants, IR drops between electrodes will increase, and long reaction time gives way to high operational cost.

This study explored the method of EC for the treatment of textile wastewater in two models. The first model is the conventional reactor with new configurations of parallel electrodes. The second model is a rotated bed of EC reactor in a unique design, which was not subjugated in previous studies. Thereby, developing this new reactor will overcome the passivation problem and improve the electrolyte homogeneity, and making efficient improvements such as improve efficiency and quality of waste water treated, low power consumption, low sludge production, and reduction of the reaction time of EC process.

1.3 Research Question

Following are the research questions for this study:

1. What are the operational parameters of the EC technique used for the treatment of textile wastewater?
2. How to improve the wastewater effluent quality of textile manufacturing using the EC technique?
3. Why the EC technique is used instead of the conventional treatment to remove wastewater pollutant in textile manufacturing?
4. What are the methods, materials and equipment that are used to improve the performance of the EC treatment in economic terms?

1.4 Research Objective

The aim of this research is to evaluate the effectiveness of electrocoagulation process, compared with the chemical coagulants, as a treatment unit before the sedimentation unit in textile wastewater treatment plant. This research had two models. Each case produced data that was necessary to observe trends and support conclusions suitable for publication in peer-reviewed research journals. The specific objectives of this study are:

1. To investigate the best conditions of the operational parameters affecting removal efficiency of textile wastewater using new EC configurations with static electrodes under combined electrical connections and compare with conventional coagulation process.
2. To combine EC - EO processes static electrodes under combined electrical connections for the treatment of textile wastewater with optimum operational parameters and compare with EC alone.
3. To develop EC technology using a novel reactor design with rotating anode by investigating the operational parameters affecting removal efficiency of

pollutants. The enhancement of the mass transfer coefficients, the mechanism of treatment, and hydrogen production were also investigated.

4. To conduct the comparative study of the unique EC reactor with CFD simulation for ionic mass transfer and investigate the passivation phenomenon.

1.5 Scope of Study

In the pursuit of meeting all the objectives, the scope of this project is crucial. The scope of this research focuses on EC method using two models for the treatment of textile wastewater. The first model was the EC process with a new configuration of static electrodes in two phases, first, using the EC process alone, and then combining EC process with another process technology (electrooxidation, EO) to reduce the amount of produced sludge. The second model is a unique design of EC with a rotating anode. Two types of effluent textile wastewater were examined and tested before and after using the suggested treatment process. The first sample was taken from a textile industry located at Hilla-Iraq. Here, the experimental investigations were achieved for the first and second model to find the differences of effluent quality and to optimize the operational parameters affecting removal rate of treatment. The effluent samples before and after treatment were analysed and organized according to the chemical oxygen demand (COD), total suspended solid (TSS) and colors of textile wastewater. For model two (EC with rotating anode), advanced investigations were achieved to ionic mass transfer study. For more experimental investigations and verifications about the unique design (model two), the second type of effluent (textile wastewater) was used which was taken from the textile industry located in Kajang, Selangor-Malaysia.

1.6 Engineering Significance

The EC technology investigated in this research is different (in composition, but smaller scale) to those currently used as the option for chemical coagulation

(historically, alum) prior to the sedimentation unit in the textile industry. Historically, the lacks of practical experience and predicted high energy requirements have eliminated EC as a membrane pretreatment option. Recent technological improvements and the desire to reduce liquid chemical storage and usage have made electrocoagulation a more desired pretreatment option for the textile wastewater.

However, the EC system requires a better understanding of the mechanisms of coagulation involved, the species of coagulant generated, and the impact of EC coagulated wastewater on removal efficiency. This research provides further data on the treatment of textile wastewater using electrocoagulation as a pretreatment before sedimentation and filtration units.

The option of EC systems (with doctrinal approval from previous research) would increase the quality of wastewater produced with less energy consumption, and low operational and maintenance costs. The most immediate application of this research is the employment of EC treatment process with the new configuration and the incorporation of EC process with electrooxidation in a single reactor (model one). The model two is a novel EC reactor with a rotating anode will improve wastewater quality, reduction of retention time and reduction of the passivation phenomenon in the EC treatment.

1.7 Organization of Thesis

This thesis consists of nine chapters. Chapter 1 introduces the subject of this study by demonstrating the relevance of electrocoagulation to the textile industry. Published reports are summarized in a literature review as fundamental principles and operational parameters effecting on EC process in Chapter 2. The comparison with conventional coagulation process, combination EC with another technologies, and operational cost analysis of the previous study are discussed in this chapter. Chapter 3 presents the methodology of this study where research layout, collection and sampling of textile wastewater, models design, measurement tools and methods for chemical and cost analysis are discussed.

In Chapter 4, the EC static electrodes with the new configuration (model one, phase one) were examined and the results were observed under optimal conditions. The comparison with conventional coagulation was also discussed in this chapter. Chapter 5 presents the combination model with static electrodes (EC-EO, model one, phase two) with the results obtained under optimal conditions. The comparison with EC static electrodes was also presented in this chapter. Chapter 6 presents a novel EC reactor with the rotating anode (model two) and the results under optimal conditions were discussed and validated. The comparison of EC rotating anode performance with a model of the conventional static electrode (model one, EC and EC-EO) was also discussed. Chapter 7 presents the investigations for the enhancement of ionic mass transfer using EC rotating anode reactor by the experimental work and CFD simulation. The verification of experimental results was studied using statistical method. Chapter 8 presents EC rotating anode reactor using solar energy feed. The investigations included batch and continuous flow mode, mechanism of treatment, enhancing of hydrogen production, and passivation phenomenon study by impedance. Finally, conclusions, and recommendations for further study were made in Chapter 9.

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