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Remediation of wastewater from pulp and paper mill industry by the electrochemical technique

Sittichok Khansorthong [a], Mali Hunsom [a,b,*]

[a] Department of Chemical Technology, Faculty of Science, Chulalongkorn University, PhayaThai Road, Bangkok 10330, Thailand
[b] Center for Petroleum, Petrochemicals, and Advanced Materials, Chulalongkorn University, Bangkok 10330, Thailand

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ABSTRACT

This research attempted to find the optimum condition for color and total COD reduction in wastewater from the pulp and paper mill industry by using electrocoagulation techniques in batch and continuous modes. Six pieces of iron plates constructed in parallel configurations were used as electrodes. The effect of key parameters including the type of polyelectrolyte, current density, initial pH of the wastewater, and the circulating flow rate of wastewater in the reactor were investigated. The results indicated that the polyelectrolyte had no effect on pollutant removal. At optimum conditions, greater than 97% of color and 77% of total COD were effectively removed with a total operating cost of approximately 0.29 USD/m^3 wastewater. First order rate kinetics best explained the reduction of color and total COD concentration, the model fitting the actual data very well. For the continuous mode, the treatment process reached the steady state condition within 2.15 h and the efficiency of color and total COD reduction was greater than 91% and 77%, respectively. The properties of wastewater including color, total COD, BOD_5, TSS, TDS, pH and iron ions content were in the range of the acceptable values of current Thai Government standards.
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1. Introduction

The pulp and paper industry is an almost global, being present in most developed and developing countries and is one of the most important industries in Thailand because it has the potential to compete with other countries due to the plentiful supply of sustainable renewable fast growing forest resources and water supplies. However, this plant utilizes a high quantity of water, between 76–227 m^3 per ton of product, resulting in large amounts of wastewater generation and concomitant economic and ecological/environmental costs/problems. Indeed, such wastewater contains a large amount of pollutants characterized by biochemical oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SS), toxicity, and colorants which cause bacterial and algal slime growths, thermal impacts, scum formation, color problems, and a loss of both biodiversity and aesthetic beauty in the environment [1]. The main treatment processes used at pulp and paper mill plants are primary clarification (sedimentation or flotation), secondary treatment (activated sludge process or anaerobic digestion) and/or tertiary processes (membrane processes as ultrafiltration)[2]. Activated sludge plants have been the most common wastewater treatment process for the removal of organics in our country; however, there are several problems with the process. It produces sludges with very variable settlement properties, it is sensitive to shock loading and toxicity, and its capacity to remove poorly biodegradable toxic substances is limited. Therefore, many researchers attempted to develop the new technologies for complementing or even replace some of these treatments. The white rot fungus, *Ceriporiopsis subvermispora* CZ-3, was shown to be effective for reducing the concentration of pollutants in the effluent from a pulp bleaching process, with approximately 90% color, 45% total COD, 62% lignin and 32% adsorbable organically bound halogens (AOX) removal by the fungus within 48 h at an operating temperature and pH of 30–35°C and 4.0–4.5, respectively, in the presence of 1 g/L glucose [3]. However, in the economically and environmentally more desirable absence of glucose, the fungus could only remove color up to 62%. Subsequently, Amat et al. [4] used solar energy together with different types of catalyst, such as Fenton agent and TiO_2, to degrade pollutants from board paper industries and revealed that the Fenton reagent gave a higher degree of total COD and BOD_5 depletion compared to TiO_2. In the same year, Wong et al. [5] reported on the flocculation performances of nine cationic and anionic polyacrylamides, each with different molecular weights and charge densities, for treating pulp and paper mill wastewater. They revealed that cationic polyacrylamides, like Organopol 5415, with a very high molecular weight and low charge density achieved a reduction of 95, 98 and 93 % in turbidity, total suspended solid (TSS) and total COD, respectively, with a sludge

* Corresponding author at: Center for Petroleum, Petrochemicals, and Advanced Materials, Chulalongkorn University, Bangkok 10330, Thailand. Tel.: +66 2 2187523 5; fax: +66 2 2555831.
E-mail address: mali.h@chula.ac.th (M. Hunsom).

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Nomenclature

$(\text{total COD})_t$	total chemical oxygen demand at time t ($g O_2/L$)
$(\text{total COD})_{t+\Delta t}$	total chemical oxygen demand at time $t+\Delta t$ ($g O_2/L$)
F	Faraday's constant (26.8 Ah)
I	current intensity (A)
L	volume of electrolyte (L)
V	cell voltage (V)
t	electrolysis time (h)

volume index (SVI) of 14 mL/g at the optimum dosage of 5 mg/L. In a different approach, Buzzini et al. [6] reported a system based upon precipitation with a microbial community which provided approximately 80–86 % and 75–78 % total COD removal with and without recirculation of the effluent, respectively. By using the combined processes of coagulation-flocculation and heterogeneous photocatalysis to treat the post-bleaching effluent from a cellulose and paper industry, Rodrigues et al. [7] reported that the optimal conditions for coagulation were pH 6.0, 80 mg/L of $FeCl_3{\cdot}6H_2O$, and 50 mg/L of chitosan while the optimized photocatalysis conditions were found at pH 3.0 in 0.50 g/L of TiO_2 and 10 mmol/l of H_2O_2. Effluent turbidity decreased sharply after coagulations (from 10 FTU of in natura samples to 2.5 FTU without chitosan and 1.1 FTU with chitosan) and a decrease in the concentrations of N-ammoniac, N-organic, nitrate, nitrite, phosphate, and sulfate ions after coagulation. This combined method (coagulation followed by photocatalysis) resulted in a biodegradability index of 0.71, transparency, and absence of color and odor in the treated water.

However, without disregarding the already summarized treatment techniques, one of the most interesting and effective processes for treating some pollutants in wastewater are the electrochemical process because of their convenience and typically greater effectiveness than the above traditional methods [8]. Electrochemical processes can be applied in various configurations such as electrooxidation, electroprecipitation, electrocoagulation, electrodeposition, electro-Fenton, etc. Using three-dimensional electrodes with a $Ti/Co/SnO_2–Sb_2O_5$ anode to treat paper mill wastewater, Wang et al. [9] reported that pH and current density had a significant effect on the total COD and color removal efficiency. Removal of chromium and organic pollutants simultaneously by electroprecipitation lead to good (approximately 98%) chromium removal within 60 min but whilst the organic pollutants contained in wastewater such as oil and grease, color and the level of BOD_5, total COD and total kjeldahl nitrogen (TKN) were also markedly reduced [10], they were still higher than the acceptable values of the government standard. However, using electrocoagulation instead of electroprecipitation was found to be effective at both reducing the amount of chromium (within 10 min) together with up to 96 % of the organic pollutants present in the wastewater [11].

In the studies reported here, importantly, real wastewater from a pulp and paper mill plant, instead of using synthetic wastewater as in many previous works, was treated by the electrocoagulation technique both in batch and continuous processes. The effects of various parameters on color and total COD removal were explored to optimize the efficiency and cost effectiveness of the system.

2. Experimental

The experiment was carried out in a bench-scale laboratory at ambient temperature both in batch (Fig. 1(a)) and continuous modes (Fig. 1(b)) by employing actual wastewater from

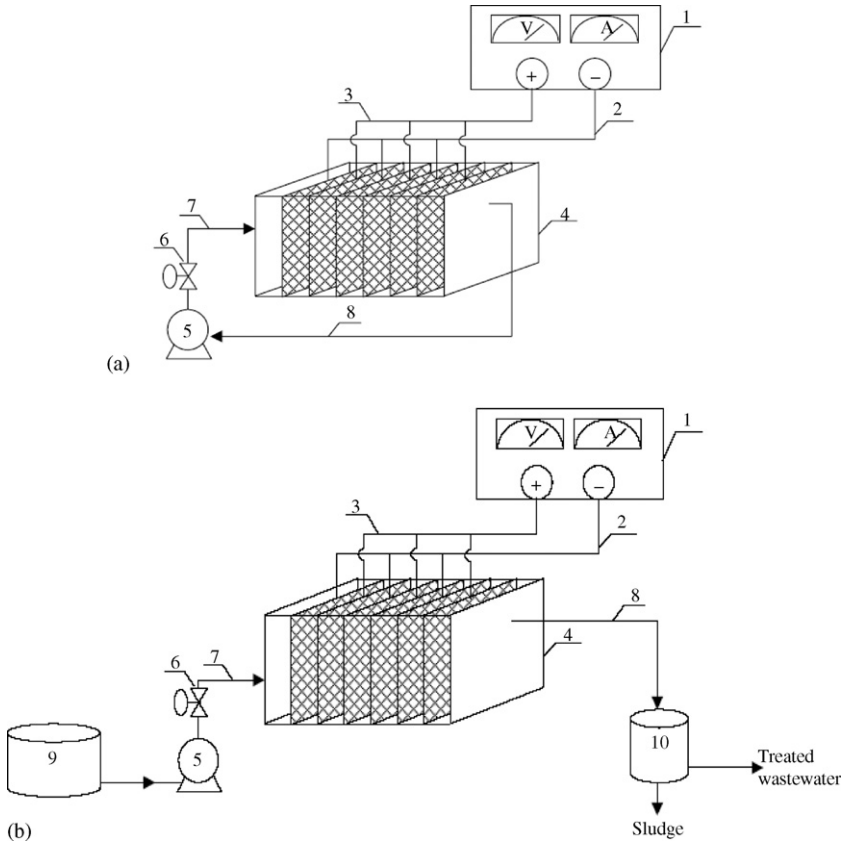

Fig. 1. Experimental set up for batch (a) and continuous modes (b): (1) power supply; (2) cathode; (3) anode; (4) reactor; (5) pump; (6) valve; (7) inlet stream; (8) outlet stream; (9) wastewater reservoir tank; (10) filtration tank.

Table 1
Chemical and physical properties of pulp and paper mill industry waste water in Thailand, and after both batch and continuous electrocoagulation treatment.

Properties	Thai standard	Values (before treatment)[a]	Values (after treatment) batch	Values (after treatment) continuous[b]
pH	5.5–9	6.6–10	8.84	8.05
Temperature (°C)	≤40	27–29	NA[c]	NA
Conductivity (μS/cm)	NA	1650–1790	NA	NA
Total dissolved solid (mg/L)	≤3000	1310–1560	865	825
Total suspended solid (mg/L)	≤150	40–260	5	25
Total COD (mg/L)	≤400	490–1114	127	255
BOD_5 (mg/L)	<60	810–1095	74	240
Color (Pt–Co unit)	Not objectionable	1000–1984	51	167

[a] Wastewater obtained from pulp and paper mills plant using wood pulp as raw material and the final product is paper pulp for making writing paper and packaging paper.
[b] Data obtained at optimum condition of batch treatment with a feed rate of wastewater of 66.6 l/min after 4.5 h start-up time.
[c] NA: not applicable.

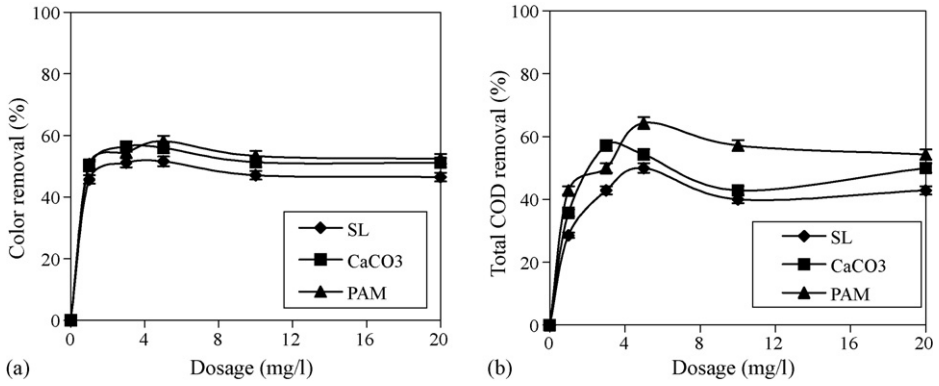

Fig. 2. Removal percentages of color (a) and total COD (b) by using different types of polyelectrolyte.

the pulp and paper mill industry (Table 1). The electrocoagulation cell was constructed from Plexiglas having a dimension of 0.12 m × 0.21 m × 0.15 m. The total volume of wastewater in each experiment was approximately 0.003 m^3. A low-cost iron plate with a total surface area of 0.0161 m^2 was used for the sacrificial electrodes, arranged in monopolar configurations. In each batch, six iron plates were constructed in the electrochemical reactor and the distance between plates was fixed at approximately 0.05 m. To achieve a good mass transfer in the system, a magnetic pump (Model NH-5PX type) was used to circulate the electrolyte in the reactor. A regulated DC power supply (ZS 3205-2X type) was employed to supply the external electricity. During the treatment, the properties of wastewater including color, total COD, BOD_5, TSS and TDS were analyzed at various stated time intervals following standard methods [12]. The data reported in this experiment were the average values obtained from the duplicate of experiments and the acceptable error in this work was 3%.

3. Results and discussion

3.1. Experiment in batch mode

3.1.1. Effect of flocculants

As mentioned by Mollah et al. [13], the efficient removal of pollutants by the electrocoagulation technique requires an effective coagulants formation step in order to achieve better coagulation between the coagulants generated in the system and the pollutants. Therefore, the effects of a polyelectrolyte as a coagulant aid were first explored using sodium silicate (SL), calcium carbonate ($CaCO_3$) and polyacrylamide (PAM) as the polyelectrolyte at a dose ranging from 0–20 mg/L. This experiment was first conducted by the direct addition of the chosen polyelectrolyte and dose into the wastewater with subsequent analyses of the residual levels of color and total COD. The results indicated that all three polyelectroloytes were effective at concentrations from 2 mg/L for color removal but

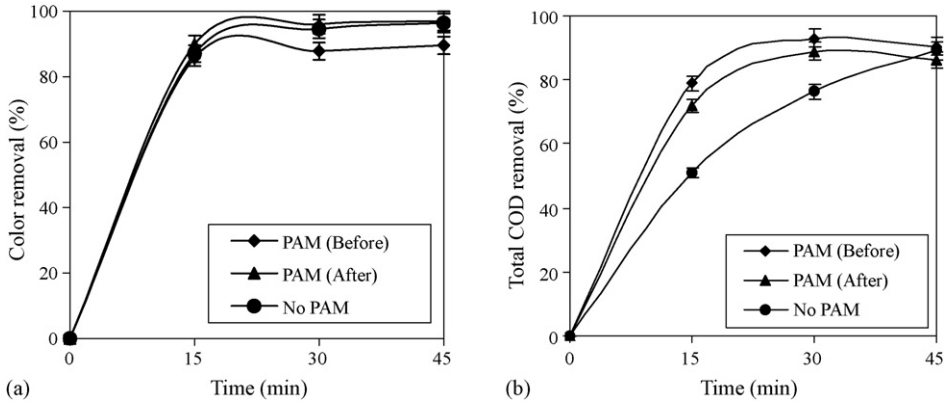

Fig. 3. Removal percentages of color (a) and total COD (b) by PAM as polyelectrolyte in electrocoagulation process.

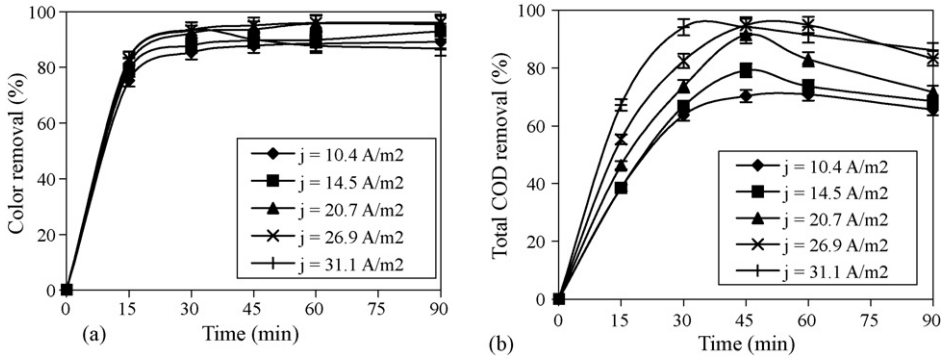

Fig. 4. Removal percentages of color (a) and total COD (b) as a function of operating time at various current densities.

at 5 mg/L for the optimal reduction of total COD levels (Fig. 2). Also, whilst all three polyelectrolytes yielded similar color removal levels, and especially PAM and $CaCO_3$, PAM provided by far the better reduction in total COD levels at 5 mg/L, with color and total COD levels reduced around 58.2% and 64.3%, respectively (Fig. 2). Therefore, for the next experiment, PAM at a dose of 5 mg/L was employed as the polyelectrolyte in the electrocoagulation step.

The effect of the addition of PAM (5 mg/L) before and after treatment by the electrocoagulation process on the removal of color and total COD was investigated using a current density of 20.7 A/m^2 and a circulating flow rate of wastewater in the electrochemical reactor of 2.6 l/min, with the initial pH of wastewater being 7.58. The results revealed that the extent of color removal both in the presence and in the absence of PAM were very similar (Fig. 3(a)), whilst PAM addition lead to some 20–30% higher total COD reduction only if added during the first 30 min, and after that had no discernible effect on the total COD removal (Fig. 3(b)). The clear suggestion from these results is that, under these conditions the addition of PAM polyelectrolyte in the electrocoagulation process may not be necessary or advantageous. Although it may lead to the ability to reduce the electrocoagulation time from 45 to 30 min to attain the degree of reduced total COD, and thus enhance the output rate of the process, against the cost of PAM is expensive (4500 USD/ton).

3.1.2. Effect of current density

Consequently, the effect of current density was then explored for this treatment process because the supply of current influences the amount of ferrous ions produced from the sacrificial electrodes. A large current allows the use of a small electrocoagulation unit but wastes electrical energy in heating up the water [14]. Different current densities in the range of 10.4–31.1 A/m^2 were investigated with a fixed circulating flow rate of wastewater of 6.9 l/min. The initial wastewater pH, color and total COD values were 7.90, 1909 Pt–Co unit and 623 mg/L, respectively. Color reduction was very fast with approximately 80% reduction within 15 min at all current densities, and attained a constant level by 30 min. Furthermore, a higher applied current density, a higher color reduction was obtained according to the Faraday's law. (Fig. 4(a)). However, at the highest tested current density of 31.1 A/m^2, the degree of color reduction decreased with operating times longer than 30 min. This might be attributed to the fact that a large amount of iron ions was produced at the high current density and long operating times, leading to the resurgence of color in the treated wastewater. With respect to reduction in the total COD levels at the same operating conditions, increasing the current density led to an increased removal of total COD, until 45 min, in an applied current density manner (Fig. 4(b)). Thereafter, the total COD levels slightly increased from 45 min onwards for all applied current densities, presumably due to the fact that large amounts of iron (II) ions (themselves a total COD component) were generated at long electrolysis times which can react with the dissolved oxygen in the wastewater according to Eq. (1) leading to lower amount of oxygen in the treated wastewater.

$$4Fe^{2+} + O_2 + 10H_2O \rightarrow 4Fe(OH)_3 + 8H^+ \quad (1)$$

The optimum current density was then determined in terms of the current efficiency calculated at any particular time by Eq. (2) [15]. The relationship between the current efficiency and time, plotted in Fig. 5, indicated that the current efficiency increased sharply from zero to approximately 20–50% during the first 15 min and then dropped rapidly with increasing time to around 1–7% by 60 min. This was presumably attributed to the adsorption of lignin on the electrode surface, or due to the passivation formation by the reaction between the metallic chloride and calcium or magnesium salts present in the wastewater [16]. Lignin is a common component of wood and it is an integral part of the cell walls of plants, especially in tracheids, xylem fibres and sclereids, and thus unavoidable in pulp and paper waste water samples. From the curve (Fig. 5), the intensities of the current efficiency at current densities of 20.7, 26.9 and 31.1 A/m^2 were similar, that is, they were around 46–50%. Therefore, the optimum current density was chosen at 20.7 A/m^2 with 93.7% and 73.6% reduction in color and total COD levels (Fig. 4), respectively, yielding remnant levels of color and total COD in the treated wastewater at around 119 Pt–Co units and 237 mg/L, respectively.

$$\mathrm{CE}(\%) = \frac{(\text{total COD})_t - (\text{total COD})_{t+\Delta t}}{8I\Delta t}\mathrm{FL}100 \quad (2)$$

3.1.3. Effect of initial pH of wastewater

Depending upon the degree of acid re-treatment and bleaching of wood pulp along with the local water pH for washings,

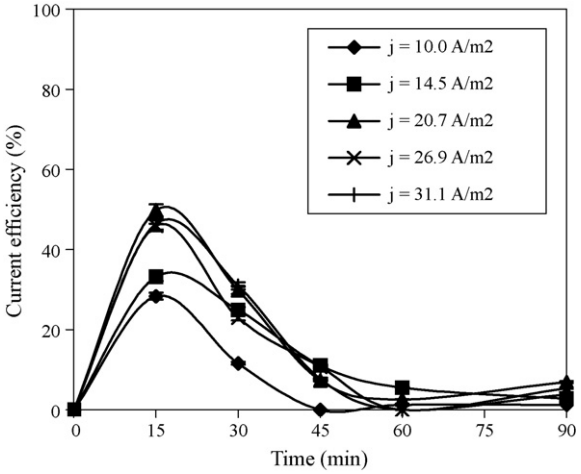

Fig. 5. Variation of current efficiency as a function of electrolysis time at various current densities.

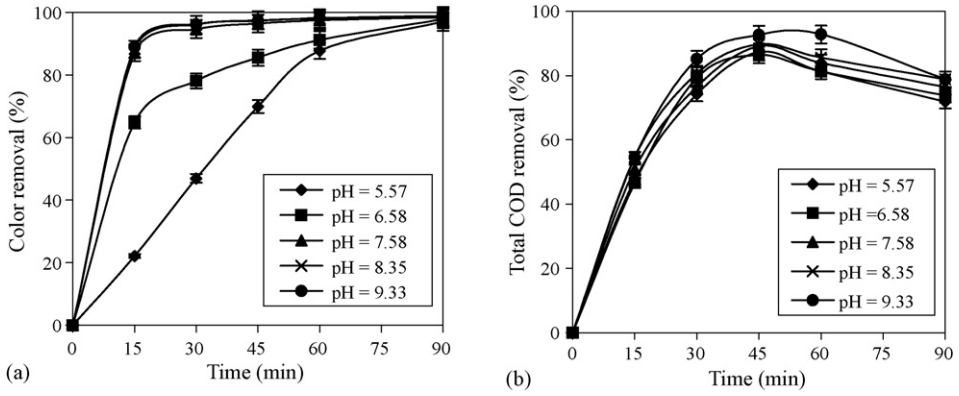

Fig. 6. Removal percentages of color (a) and total COD (b) as a function of operating time at various initial pH of wastewater.

paper and pulp mill wastewater can range in pH from acidic to mildly alkaline and these will potentially effect the electrocoagulation process, for example via shifting of the redox equilibrium between iron (II) and iron (III) ion complexes. The effect of the initial pH of wastewater on the removal of color and total COD was explored in the range of pH 5.57–9.33 using a fixed current density of 20.7 A/m^2, circulating flow rate of 6.9 l/min and time of 90 min. For color removal, the efficiency reduced with decreasing acidic pH and no detectable difference was seen in color reduction at mildly alkaline pHs in the tested range of pH 7.58–9.33 which all lead to removal of approximately 90% and 95% color within 15 and 30 min, respectively (Fig. 6(a)). This is most likely explained by the fact that in basic solutions the redox equilibrium is shifted to iron (III) ions which, in monomeric forms such as $Fe(OH)_3$, or in polyhydroxyl iron (III) complexes such as $Fe(OH)^{2+}$, $Fe(OH)_2{}^+$, $Fe(H_2O)_5OH^{2+}$ and $Fe(H_2O)_4(OH)_2{}^+$, are reactive and can couple with the pollutants in wastewater [12,13] leading to lower amount of pollutants in wastewater. In contrast, at the weak acidic conditions tested (pH 5.57 and 6.58), a large amount of iron ions are in the form of Fe^{2+}, which is stable and soluble in water resulting in a reduced formation of coagulant and pollutant removal. For reduction in total COD levels, the initial pH of wastewater in the investigated range did not significantly affect the efficiency of the reduction in total COD levels (Fig. 6(b)).

3.1.4. Effect of the circulating flow rate of wastewater in the reactor

The effect of wastewater flow rate on the efficiency of color and total COD removal by electrocoagulation was investigated using flow rates between 2.6–6.9 l/min at a fixed current density of 20.7 A/m^2 and an initial pH of 7.58. The flow rate, within the tested range, appeared to have no significant effect upon the removal of color in terms of rate or final level attained (Fig. 7(a)). However, with respect to reduction of total COD the high flow rate (6.94 l min^{-1}) was the least efficient whilst low and medium flow rates were essentially the same in terms of both the kinetics and final levels of total COD reduction (Fig. 7(b)). This flow rate dependent effect upon the efficiency of total COD reduction may be attributed to the dissociation of the coagulants at high circulating flow rate conducting to a small amount of coagulants attached to the pollutants.

3.1.5. The amount of iron ions produced, and distribution in sludge versus treated wastewater in the electrocoagulation process

Finally, the amount of iron generated as ions from the anode by oxidation, and their final distribution in terms of the coagulated sludge or as free (soluble) ions in the treated wastewater at optimum condition (current density of 20.7 A/m^2, an initial pH of wastewater of 7.58 and circulating flow rate of wastewater of 2.6 l/min) was evaluated. The quantity of iron generated from the anode as ions and found in the sludge co-increased as a function of the electrolysis time whereas that of ions in treated wastewater reached its maximum value of 0.395 mg/L at 30 min electrolysis time and consequently decreased sharply (Fig. 8). The presence iron ions in large quantity in the treated wastewater during the first 30–45 min might be attributed due to the in-situ generation of coagulating agents by electrolytic oxidation of the sacrificial electrode. Subsequently, these generated ions mostly combined with the pollutants species leading to the formation of coagulants throughout the electrolysis time. Thereafter, the amount of iron ions diminished rapidly due to the precipitation of flocculated pollutants in the sludge.

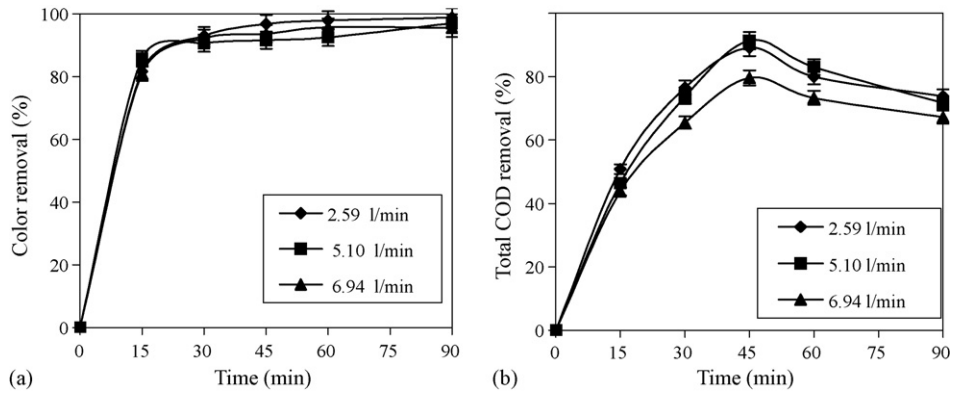

Fig. 7. Removal percentages of color (a) and total COD (b) as a function of operating time at various circulating flow rates of wastewater.

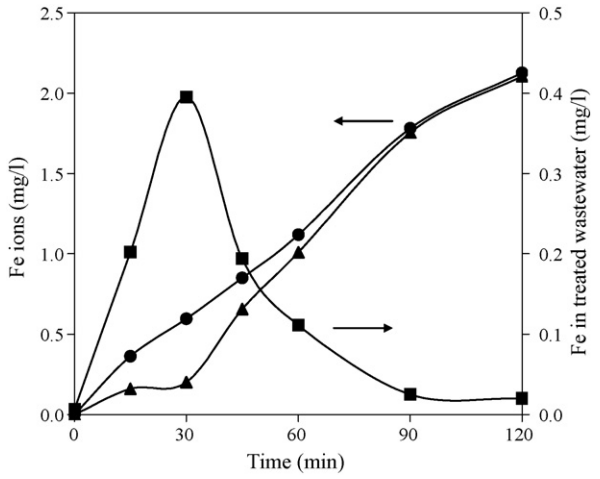

Fig. 8. Amount of iron ions generated from anode (•); iron ions in sludge (▲) and iron ions in the treated wastewater (■).

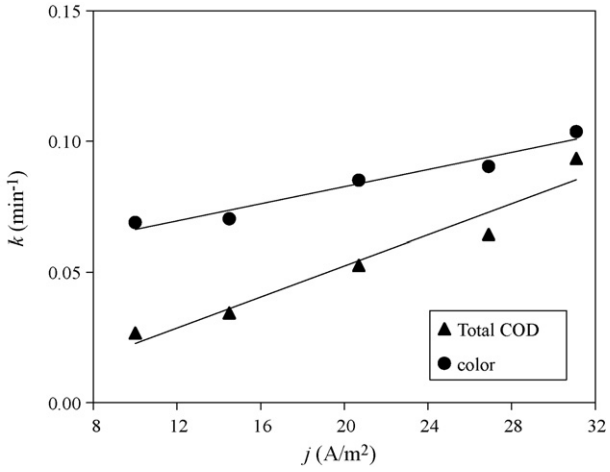

Fig. 9. Plot of first-order kinetics rate constant of color total COD as a function of current density at optimum condition.

The operating cost of this system, related to the costs of electricity, electrodes and sludge treatment, were calculated for operation at the optimum condition. The energy consumption of this batch electrocoagulation process was lower than 1.2 kW h/m^3 wastewater and had a total operating cost of approximately 0.29 USD/m^3 (including electricity cost of 0.08 USD/m^3, electrode cost of 0.21 USD/m^3 and sludge disposal cost of 0.01 USD/m^3). The kinetics of color and total COD removal were calculated using a macro-kinetics model and both the reduction of color and total COD concentration were best and very well, explained by a first-order rate kinetics model. In support of this, the coefficient of determination, R^2, for a plot based upon first-order rate kinetics was greater than 0.96. A series plot of first-order kinetics rate constant as a function of current density for color and total COD is shown in Fig. 9, where the rate constant is directly proportional to the applied current density. This result is similar to that of some previous works [11,17]. In addition, the kinetics rate constant for color reduction was higher (faster rate) than that of total COD by about 1.1–16 times, which also agrees with the previous results.

3.2. Experiment in continuous mode

The above data was performed as batch electrocoagulations, but it would typically be more ideal for applied use in the paper and pulp industry to have a continuous operation mode. These experiments were consequently carried out in a continuous mode using the optimum condition discovered in the batch mode (current density of 20.7 A/m^2, initial pH of 7.58 and circulating flow rate of wastewater of 2.6 l/min). Two feed rates of wastewater to the electrochemical reactor were investigated, namely either 33.3 mL/min or 66.67 mL/min. The degree of both color and total COD reduction was reduced in the higher flow rate than in the lower rate (Fig. 10), and both continuous modes were less efficient at color and total COD removal compared to the batch mode. This is most likely because the retention time of wastewater in the electrochemical reactor was highest in the batch mode followed by the low and then lastly by the high flow rates in the continuous mode. With respect to the operation in continuous mode, reduction of both color and total COD reached their steady state conditions after a start up time of 2.15 h. The feed rate of wastewater at 33.33 mL/min provided a slightly higher reduction rate than that at 66.67 mL/min, as already mentioned, and is likely attributed to the retention times of 90 and 45 min, respectively, within the electrocoagulation unit. The characteristics of wastewater after continuous treatment at 66.67 mL/min and batch treatment are summarized in Table 1. It can be seen that in both the batch and the continuous process all pollutants were markedly reduced to below the current maximum allowed levels for discharge permission by the Thai Government, except for BOD_5 which was still higher than the standard by around 3–4 times. However, at this relatively low level of BOD_5, this wastewater can be further reduced in BOD_5 by settling in aeration ponds.

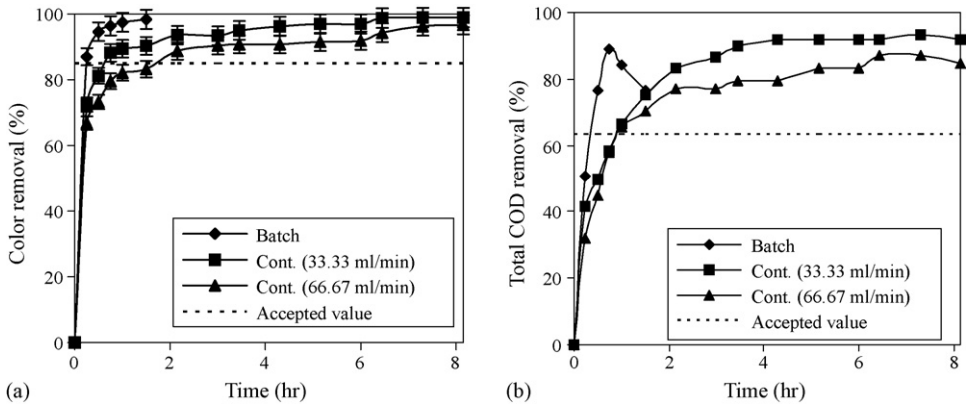

Fig. 10. Comparison of the removal percentages of color (a) and total COD (b) by electrocoagulation process in batch and continuous modes.

4. Conclusions

According to this work, for the electrocoagulation process, the presence of polyelectrolytes such as sodium silicate, calcium carbonate and polyacrylamide have no significant effect on the treatment process of wastewater from the pulp and paper mill industry and thus can be omitted saving on operational and environmental costs. The optimum condition of treating such wastewater in a batch electrochemical reactor was found, assuming independence of each of the variables, to be at a current density of 20.7 A/m^2, an initial pH of wastewater of 7.58, a circulating flow rate of wastewater of 2.6 l/min and a 45 min operating time. Under this condition, color, total COD, BOD_5, TSS and TDS were reduced by 97.0, 87.8, 91.2, 89.8 and 37.5 %, respectively, with all bar BOD_5 being below current limits for discharge into the environment. The pH of the treated wastewater was around 8.84. Low energy consumption and operating costs were obtained at 1.2 kW h/m^3 and 0.29 USD/m^3, respectively. The kinetics of the rate of color and total COD removal were directly proportional to the applied current density, and the rate constant of color removal was a little higher (faster) than that of total COD under the same conditions. For the continuous process, using the same optimum batch process conditions, the system reached its steady state condition within 2.15 h and, at the higher and less efficient feed rate of wastewater into the electrochemical reactor of 66.67 mL/min, all properties of the treated wastewater, including color, total COD, TDS and TSS, except BOD_5 were acceptable to discharge into the environm. In addition, the color of the treated wastewater was not objectionable, its pH was around 8.05 and the residual iron concentration was lower than 0.4 mg/L.

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