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Feasibility and kinetic study of electrocoagulation with enhanced flocculation-flotation in removal of color from synthetic dye

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ABSTRACT

In electrocoagulation, the gas bubbles develop as bubble nuclei, grow in size, break off from the electrode surface and rise in the liquid. Unfortunately, over time, the gas bubbles tend to adhere and accumulate on the electrode surface, deactivate the parts of the electrode surface that reduce the mass transfer of ions. This paper aimed to investigate the feasibility of electrocoagulation with enhanced flocculation-flotation to remove color from synthetic dye. Vibration of plates was utilized in electrocoagulation treatment, with the variables studied including pH (pH 5 and pH 9), vibration intensity (1.5 – 3.5 Volts), current intensity (0.5 – 2.5 A) and operating time (15 – 60 min). From the results, the optimum color removal percentage was 74.76%, achieved at a pH of 9 with optimum vibration and current intensities of 2.5 V and 2.5 A, respectively. For the kinetic study, the rate obtained for the highest R^2 value was the first-order reaction with a rate constant of 0.0037 min^{-1} at acidic pH and 0.0022 min^{-1} at alkaline pH. The vibration of plates enhanced the rate of coagulant ions as well as bubbles dispersion during the treatment due to the oscillation of plates, which generated a stirring mechanism around the electrode plates.

Keywords: color removal; electrocoagulation; methylene blue dye; textile wastewater; vibrating electrode plates; flocculation-flotation

1. INTRODUCTION

As the demand for dyes and pigments rises annually, the textile industry has become an essential economic driver in many ASEAN countries, providing jobs and financial opportunities. According to the Grand View Research Report on dyes & pigments, Asia Pacific, China, and India dominated regional dye markets and had more than 63% of the global revenue share in 2021 (Grand View Research, 2021). Furthermore, in China, the dye industry is one of the essential parts of the textile, lighting, chemical, and other industries. According to the figures given by the National Bureau of Statistics, China currently leads the globe in fuel production and trade volume, with azo dyes accounting for 70–80% of overall output (Liu, 2020). As a result of these two countries' substantial yearly production, the wastewater generated from the textile and dye industries have now become the second largest contributor to water pollution worldwide (Fane and Wastl, 2022).

In textile processing, multiple chemicals depending on the nature of raw materials are utilized, and the chemical reagents employed also differ in chemical composition, ranging from inorganic to organic (Subki, 2017). The textile printing and dyeing processes generate other wastes from different process steps such as size, de-sizing, sourcing, bleaching, mercerizing, dyeing, printing, and finishing (Yaseen and Scholz, 2019). According to Yaseen and Scholz (2016), the effluent discharged from textile factories is a

mixture of dyes, metals, and other pollutants. It is also excessive in color, pH, suspended solids (SS), chemical oxygen demand (COD), and biochemical oxygen demand (BOD) (Yaseen and Scholz, 2016). Nevertheless, the average textile effluent is challenging to categorize, considering textile application processes vary from industry to industry, even within the same process (Yaseen and Scholz, 2016).

Since the dye composition is complicated and contains a variety of organics with biological toxicity, it is challenging to obtain the desired outcome by using a single treatment unit (Liu, 2020). For instance, the traditional biological treatment has the problem of low processing efficiency and is sometimes unable to run. In contrast, the physical and chemical treatment disadvantages include high processing costs, small processing capacity, and harsh operating conditions (Liu, 2020). Electrocoagulation (EC) process is gaining popularity in industrial wastewater treatment due to its versatility and environmental compatibility (Jagwani et al., 2019). Even though EC is an energy-intensive operation given the high use of energy, it does have benefits over other methods. For instance, it can operate at ambient temperature and pressure and has the best performance and ability to adjust to variations in influent compositions and rate of flow (Ghimire et al., 2019; Niza et al., 2019). Furthermore, another advantage of this method is that the EC equipment is simple in design and operation.

EC is one of the best approaches for eliminating color and organic pollutants from wastewater, which has also proven to reduce sludge production (Daneshvar et al., 2006). It may be employed as a potentially feasible and low-cost technique for textile dye treatment (Islam et al., 2011). According to research for color removal from synthetic dye, the removal effectiveness ranged from 65% to 92% (Islam et al., 2011). EC operates on a similar principle to the electrolysis process, where two metal electrodes that are opposed to one another will be used and then submerged into a reactor containing dye wastewater. The dye wastewater is then added with electrolyte, which gives the ions the possibility to transfer between two electrodes, for example, sodium chloride (NaCl). When current is supplied, the positively charged sodium ions (Na^+) migrate to the cathode plate and the negatively charged chloride ions (Cl^-) migrate to the anode plate. At the electrode plates, the cation of Na+ will be reduced, and the anion of Cl- will be oxidized. EC involves several chemical and physicochemical phenomena due to the electrolysis process. There will be three steps that will take place (Lekhlif et al., 2014):

- Formation of coagulants during anode dissolution (sacrificial electrode),
- Coagulation of contaminants, suspended particulate, and breakdown of emulsions,
- Aggregation of the destabilized by flocculation or adsorption on metal hydroxide flocs or its polymers.

The creation of solid oxides, hydroxides, and oxyhydroxides generates active surfaces for pollutant adsorption. The following equations show the mechanisms of electrocoagulation using aluminum plates at both basic and acidic conditions, respectively (Niza et al., 2021):

Mechanism 1 (Basic condition)

$$\text{Anode: } Al\ (s) \rightarrow Al^{3+}\ (aq) + 3e^{-1} \quad (1)$$

$$\text{Cathode: } 3H_2O\ (l) + 3e^{-1} \rightarrow 1 \cdot 5H_2(g) + 3OH^-\ (aq) \quad (2)$$

$$\text{Precipitation: } Al^{3+}\ (aq) + 3HO^-\ (aq) \rightarrow Al(OH)_3\ (s) \quad (3)$$

Mechanism 2 (Acidic condition)

$$\text{Anode: } Al\ (s) \rightarrow Al^{3+}\ (aq) + 3e^{-1} \quad (4)$$

$$\text{Cathode: } 3H^+\ (aq) + 3e^{-1}\ (aq) \rightarrow 1 \cdot 5H_2\ (g) \quad (5)$$

$$\text{Precipitation: } Al^{3+}\ (aq) + 3H_2O\ (l) \rightarrow Al(OH)_3\ (s) + 3H^+\ (aq) \quad (6)$$

When dye wastewater is electrolyzed simultaneously, oxygen and hydrogen bubbles at the anode and cathode surfaces will be produced, as illustrated in Equation. 7 and 8 (Niza et al., 2021). The bubbles generated by the EC at the electrodes contribute to eliminating some parts of the suspended particles via electro flotation (EF), and because of their tiny size, gas bubbles may easily attach to the suspended solid material (Lekhlif et al., 2014). Therefore, the role of bubbles in EC treatment is crucial, as they will affect the flocculation and flotation processes, subsequently enhancing the dye removal efficiency.

$$\text{At the anode plate: } 2H_2O\ (l) \rightarrow 4H^+\ (aq) + O_2(g) + 4e^- \quad (7)$$

$$\text{At the cathode plate: } 2H_2O + 2e^- \rightarrow H_2\ (g) + 2OH^- \quad (8)$$

During the treatment, gas bubbles form as nuclei, grow, break free from the electrode surface and float in the liquid. However, without an agitation mechanism, the nucleated bubbles tend to stick and aggregate on the electrode surface, resulting in an uneven concentration of bubbles in the floating zone (Niza et al., 2021). It has been reported in a few studies that with the application of vibration in the reactor during the treatment, there will be a reduction in air bubble size, increase in air flow rate through the aerator, and more uniform dispersion of air bubbles in water, all of it directly enhance the treatment efficiency (Muhammad Niza et al., 2020). Besides that, vibration also promotes the stirring of bubbles and pollutants in water that enhance the likelihood of collisions between contaminants and bubbles, resulting in enhanced aggregate formation (Niza et al., 2021). Nonetheless, the research on the application of vibration plates in the EC to treat dye wastewater has not been explored.

The current investigation was carried out to explore the feasibility of vibration-induced electrode plates in enhancing flocculation-flotation in batch electrocoagulation in the removal of dye color using synthetic dye methylene blue (MB) to become the pollutant model of the dye wastewater. The study focused on the effect of vibration and current intensity, treatment time, and initial pH, followed by the determination of EC kinetics with vibration-induced plates.

2. MATERIALS AND METHODS

2.1 Methylene blue (MB) dye preparation and EC reactor set up

The methylene blue was used as the material sample of dye wastewater. Table 1 shows the physical properties of MB. The MB dye was prepared at five different concentrations between 5 to 25 ppm. The weight of MB dye powder for each concentration was determined by using the following formula:

$$\text{Concentration of MB dye (ppm)} = \frac{\text{Dye power weight} \times 10^6\ (g)}{1000\ mL} \quad (9)$$

Table 1. Physical properties of methylene blue dye

Parameters	Value
Color index (CI)	52030
Trade name	Methylene blue crystal or powder
Color	Dark green to blue
Maximum wavelength (nm) (λ_{max})	665
Solubility in water	Soluble in water
Chemical formula	$C_{16}H_{18}N_3OS{\bullet}3H_2O$
Molecular weight	375.5 (g/g mol)
Molecular volume	390.2 (cm^3/g mol)
Cationic/anionic dye	Cationic dye (strongly acidic at pH of 2 and 3.5)

The MB dye powder was weighted and diluted into 1 L of beaker containing distilled water. After that, the pH of the sample was adjusted by using 1 M solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH) solution. The pH of each dye sample was measured before and after EC treatment using the pH meter (HACH model Sens ion 1, Loveland, USA) and the analysis of the color was based on the true color using a spectrophotometer (HACH model DR3900, Loveland, USA) at 455 nm.

The schematic design of the electrocoagulation reactor is depicted in Figure 1. The main equipment used in the experiment were the mini vibration motors with a DC voltage of 1.5–6 V (25 × 13 mm) and a DC power supply with 0–10 A, 0–30 V (QJE PS3010). To induce vibration, the vibration motors were attached to each cathode and anode of the aluminum plate, with a total active surface area of the plate immersed in the solution of 240 cm^2. The electrode plate had a dimension of 200 mm x 50 mm (L x W), and a thickness of 1 mm. While the vibration motors had a dimension of 25 mm in height and 13 mm in diameter.

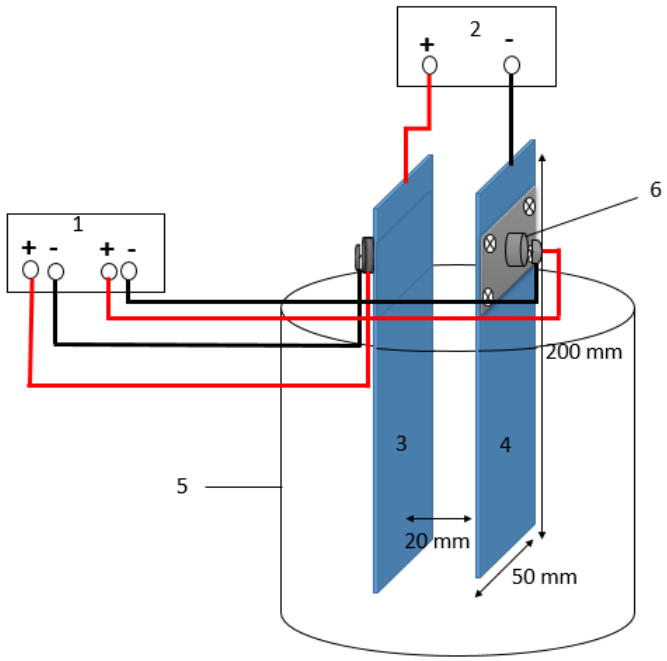

Figure 1. Schematic diagram of batch EC with vibration-induced electrode plates
Note: (1) Multi-input DC power supply (2) single-input DC power supply (3) anode plate, (4) cathode plate (5) EC reactor (1 L beaker) (6) vibration motor

2.2 EC study

The experimental design was divided into three stages. The first stage was to determine the optimum MB dye concentration, followed by the determination of the removal of color at varying vibrations (1.5 – 3.5 Volts) and current intensities (0.5 – 2.5 A), pH (5 and 9), and treatment time (15 – 60 min). The third stage involved the kinetic study of electrocoagulation. The optimum concentration of each independent variable was determined based on the highest color removal efficiency. The optimum concentration was then becoming the constant variable in the next stage of the experiment.

All runs were performed at room temperature. In each run, 1 L of the dye wastewater was decanted into the 1 L beaker of an EC cell. At the end of the EC treatment, all samples were allowed to settle for 20 min. The calculation of color removal efficiency (CR) was calculated using the following equation (Niza et al., 2021):

$$CR\ (\%) = \frac{C_o - C}{C_o} \times 100 \qquad (10)$$

where C_o and C are concentrations of color before and after electrocoagulation in mg/L, respectively.

2.3 Kinetic Study

To investigate the kinetic study of EC, the first and second orders were constructed by using the data obtained from the electrocoagulation study. To determine the appropriate EC kinetic, a graph of color removal efficiency versus time was plotted and modelled as first and second order reaction kinetic. Below is the rate law (R) equation:

$$R = \frac{dC}{dt} = -kC^{\alpha} \quad (11)$$

where R is the rate of decrease in the concentration of particles, C is the total particle concentration at time t, k is the rate constant, α is the reaction order of coagulation. The experimental data was fitted into the linearized form and was inspected for the line of best-fitting when $\alpha = 1$ (first-order reaction) and $\alpha = 2$ (second-order reaction). For the first-order reaction, integration of Equation 3 gives the following equation:

$$\ln [C]_t = -k_1 t + ln[C]_0 \quad (12)$$

with C_t and C_0 denoting the concentration at time t and initial concentration in mg/L, respectively, and the first order rate constant k_1 in min^{-1}. Similarly, the linearized form of the second order reaction can be described by Equation 5:

$$\frac{1}{C_t} = k_2 t + \frac{1}{C_0} \quad (13)$$

where k_2 is the second order rate constant in L/mg.min

3. RESULTS AND DISCUSSION

3.1 Optimum initial concentration of MB

To determine the optimum dye concentration for the color removal efficiency by EC, five different dye concentrations of 5 to 25 ppm were treated in an EC cell for 25 min with constant current intensity of 1.5 A and vibration intensity of 1.5 V between pH 6.5 and 7. The results in Figure 2 indicates that the color removal was not significantly affected by the changes in dye concentrations, especially at low concentration of 5 to 20 ppm. This is because at constant current intensity and operating time, the same amount aluminum hydroxide complexes were produced in all dye solutions, resulting in comparable removal efficiency in the dye concentration between 5 and 20 ppm. This result was in agreement with the findings by Sadri Moghaddam et al. (2010) on the effect of initial dye concentration on dye removal efficiency. However, when the dye concentration was raised from 20 to 25 ppm, the color removal was reduced from 47.19% to 31.52%. This is because when the flocs were produced at high dye concentrations, the capacity to adsorb all the solution's dye molecules became low (Nandi and Patel, 2017). Moreover, at a pH of 6.5–7, the efficiency of color removal seemed to be lowered compared to the study by El-Hosiny et al. (2018). The research revealed that the removal efficiency of cationic dye was high in the pH range of 7–11 due to dye breakdown at high pH (Jiang et al., 2002). Nevertheless, due to the sheer differences in inter electrode distance, area of electrodes submerged, and type of dye utilized, the results gained from this experiment are different from the study. From the results, a dye concentration of 15 ppm was chosen as the optimum dye concentration and was used as a constant dye concentration value for other variables studied.

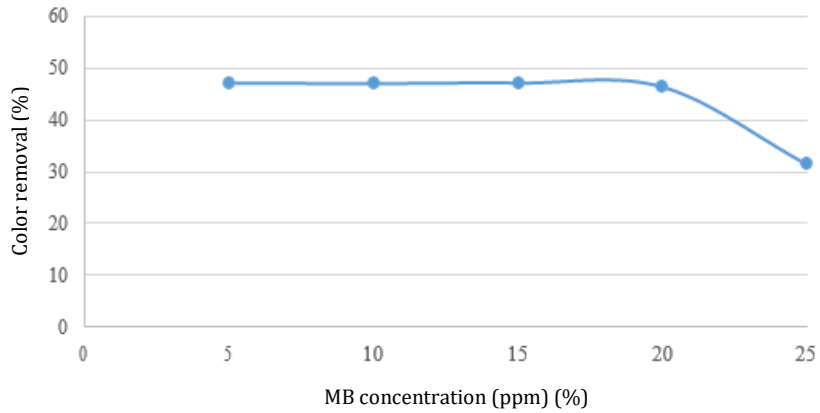

Figure 2. MB dye concentration (ppm) as a function of percent color removal (at operating time of 25 min, current intensity of 1.5 A and vibration intensity of 1.5 V)

3.2 Performance of electrocoagulation treatment

3.2.1 Effect of vibration intensity

The effect of vibration intensity on the removal of color was investigated at vibration intensities of 1.5, 2.5, and 3.5 V at a constant initial pH of 9, an operating time of 60 min, and current intensity of 1.5 A. Figure 3 shows that when vibration intensity was set to 2.5 V, it produced the maximum percentage of color removal, which was 61.86%. The percentage of color removal, however, slightly decreased to 55.13% as the vibration intensity increased to 3.5 V. This is because the electrode plates had already achieved their maximum vibration intensity of 2.5 V (Niza et al., 2021) where at this stage the bubbles were fully intensified in a way that detachment of bubbles from the electrode plate surface was completely achieved and the bubbles were well-dispersed into the solution. At a low vibration intensity of 1.5 V, the bubbles adhered and accumulated on the electrode plate surface, and only partially detachment occurred at the electrodes. Meanwhile, a higher vibration intensity of 3.5 V resulted in vigorous mixing of the solution.

The synergistic effect of vibration intensity with the EC process could be observed where it created a swirling effect around the electrode plates' surface, improving the mixing of the pollutant particles, which ended up being a better separation to the top surface of reactor by flotation. Contradictorily, the swirling or stirring became excessively vigorous when plates vibrated at 3.5 V, consequently lowering the amount of color removed. Thus, the optimum performance of vibration-induced plates was at 2.5 V of vibration intensity.

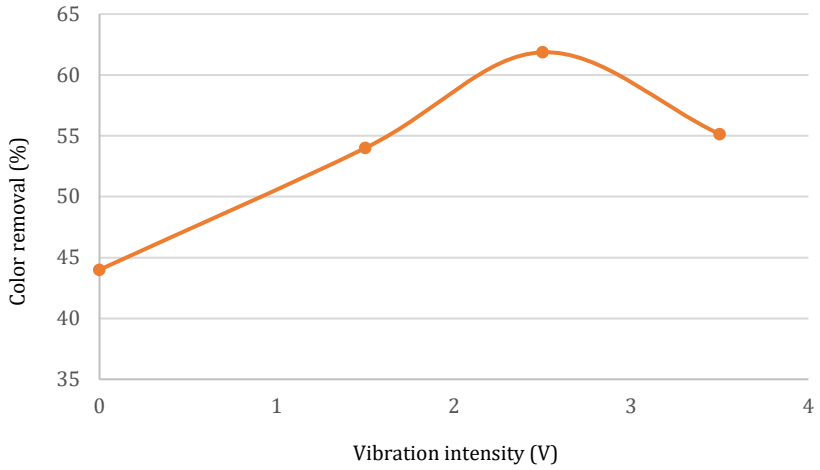

Figure 3. The percent color removal at different vibration intensity of 0 – 3.5 V at constant pH of 9, current intensity of 1.5 A and operating time of 60 minutes

3.2.2 Effect of initial pH and treatment time

The initial pH and operating time are important parameters in the EC process, for they influence the solubility of the dissolvable electrode and metal speciation in the aqueous solution, affecting dye removal efficiency. To further determine the synergistic effect of the vibration-induced plate on the flocculation-flotation of electrocoagulation treatment, the effects of initial pH and treatment time were investigated at varied vibration intensities. In comparing the color removal percent between Figure 4 and Figure 5, when the treatment time increased, the removal percent increased, and it was observed that the color of dye had better removal efficiency at the basic solution or at pH 9. This result is in agreement with other reported findings that the removal efficiency of color was efficient when the pH was alkaline (Daneshvar et al., 2006; Naje et al., 2017).

The synergistic effect of vibration intensity was significant when it could be seen that the color removal percentages were higher at the optimum vibration intensity of 2.5 V for both pHs. Other than induced stirring, the vibration of the plates allowed bubbles to disperse evenly through the solution, hence enhancing the flocculation-flotation process. During the experiment, it was also observed that the pH shifted and increased during the reactive phase, and it stabilized at final pH close to 9 to 10. This is similar observations reported by other researchers (Nandi and Patel, 2017), where it was stated that the electrolysis process in EC results in hydrogen evolution and the generation of OH^- ion, and the relative stability of pH afterwards is most likely due to the production of insoluble $Al(OH)_3$ flocs (Nandi and Patel, 2017).

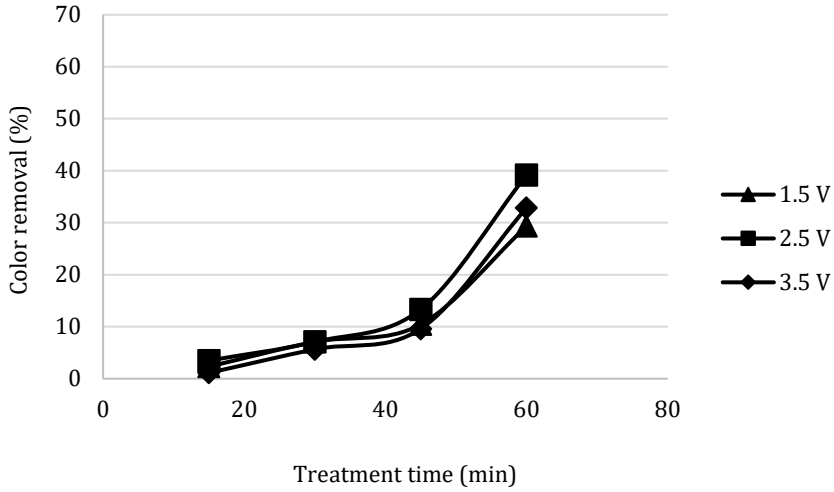

Figure 4. Color removal percent at different vibration intensity at pH 5 at current intensity of 1.5 A

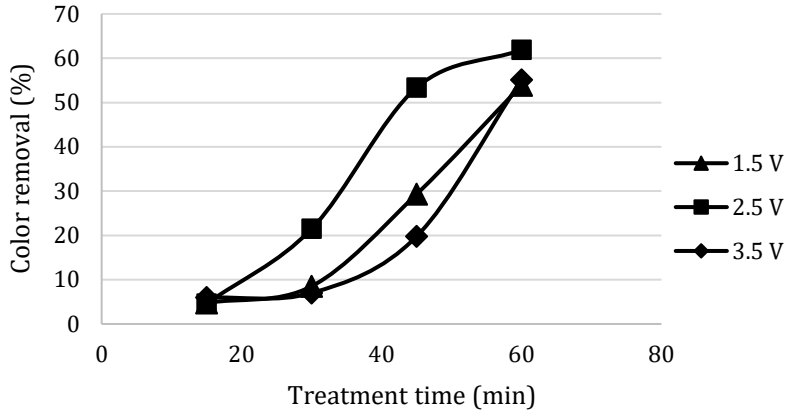

Figure 5. Color removal percent at different vibration intensities at the pH 9 at current intensity of 1.5 A

3.2.3 Effect of current intensity

The current intensity also significantly impacts the reaction rate in the EC process as it influences the amount of the coagulant, the rate and size of bubble formation, the development of the floc, and the color removal efficiency. According to Ghanbari et al. (2014), this would be attributed to the fact that the amount of Al^{3+} produced during the treatment depends on electrical current. Faraday's Law states that with an increase in the current intensity, the removal efficiency would increase. Thus, a range of current intensity was varied within 0.5 – 2.5 A, investigated at both pH 5 and 9 and at constant vibration intensity of 2.5 V (taken as the optimum vibration intensity). The results are depicted in Figures 6 and 7. From the figure, it illustrates that when the current intensity was raised along with the increase in treatment time, the effectiveness of color removal also increased, reaching its maximum removal of 74.76% at 2.5 A, pH 9, and 60 min of treatment time. The high current intensity supplied to the electrode caused more oxidation at the anodes, which increased the efficiency of color removal (Ghanbari et al., 2014).

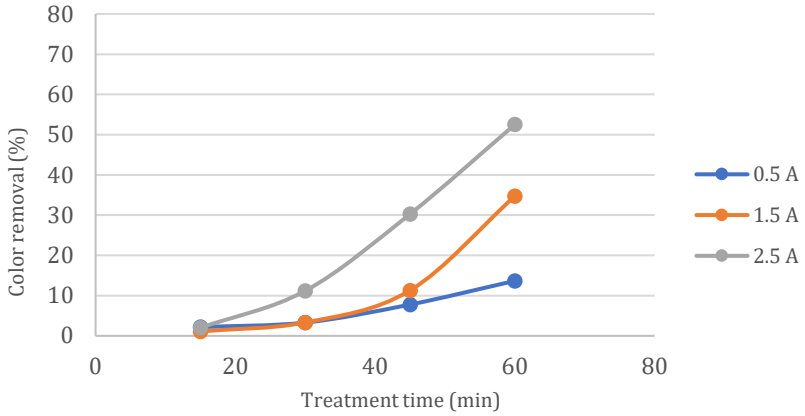

Figure 6. Color removal percent at different current intensities at pH 5 at the constant vibration intensity of 2.5 V

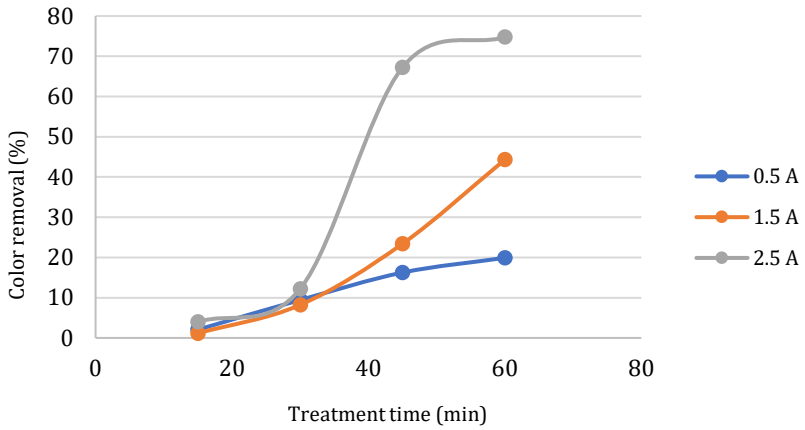

Figure 7. Color removal percent at different current intensities at pH 9 at the constant vibration intensity of 2.5 V

Other than the high current intensity, the higher removal of color at 2.5 A can also be attributed to the vibration of plates. When the electrode plates vibrate, enhances the flow of the moving gas bubbles on both vertical and horizontal directions, as opposed to only vertical direction as achieved by electrocoagulation without any bubble's intensification (Muhammad Niza et al., 2020). This could facilitate efficient mixing during flocculation and floatation in which anodic metal dissolution and gas bubbles can be hastened by higher water flow rates between the electrodes (Muhammad Niza et al., 2020). Furthermore, high water flow rates lower energy consumption and prevent the formation of a metal oxide film on the surface of the electrodes during electrocoagulation (Sillanpää and Shestakova, 2017).

3.3 Kinetic Study

The kinetic study of EC with vibration-induced plates was carried out to investigate the dependency of color removal efficiency on the current density. The rate of reaction is necessary to understand the rate constant and the order of reactions in the EC treatment process since it describes the rate of change in reactant concentration per unit of time. Tables 2 and 3 show the reaction rate constants, k_1 and k_2, as well as the regression coefficients (R^2) that were determined for the first- and second-order models, respectively. The rate constants of k_1 and k_2 were taken from the slope of the linear equation as shown in Equations 12 and 13, respectively.

From the results obtained, the reaction kinetics model that gave the best fit to the experimental data was the first-order reaction. This means the color removal rate is directly proportional to the current density. From Tables 2 and 3, it could be seen that the first-order reaction had the highest R^2 value of 0.973 with a reaction rate of 0.0037 min^{-1} at acidic pH and 0.0022 min^{-1} at alkaline pH. Since the reaction rate obtained for acidic pH was greater than alkaline pH, this suggests that electrocoagulation at acidic treatment requires a longer time to remove color from dye wastewater. The highest color removal was achieved at 43.48 mA/cm^2, which was 74.76% removal efficiency.

Table 2. Rate constants and R^2 of kinetic in batch EC with vibration-induced plates for the pH of 5

Current density, mA/cm^2	First order		Second order	
	k_1	R^2	k_2	R^2
8.69	0.0037	0.973	4.0×10^{-7}	0.9723
26.09	0.008	0.8757	0.0001	0.8417
43.48	0.0193	0.8341	0.0003	0.819

Table 3. Rate constants and R^2 of kinetic in batch EC with vibration-induced plates for the pH of 9

Current density, mA/cm^2	First order		Second order	
	k_1	R^2	k_2	R^2
8.69	0.0022	0.9171	3.0×10^{-7}	0.9075
26.09	0.0058	0.7418	8.0×10^{-7}	0.7115
43.48	0.0098	0.8991	0.0001	0.8621

4. CONCLUSION

This study investigated the feasibility of EC with vibration-induced plates to enhance the flocculation-flotation for color removal in synthetic dye. The results showed that with the vibration of electrode plates, it is efficient in removing color, especially at high pH compared to low pH. The highest total color removal percentage was 74.76% at pH of 9 and at vibration and current intensities of 2.5 V and 2.5 A, respectively. The higher percentage of color removal was attributed to the intensification of bubbles during plate vibration, which consequently helped to enhance the flocculation and flotation processes during treatment. For the kinetics model of electrocoagulation with vibration of plates, the EC kinetics follows the first-order reaction for color removal efficiency, which indicates that the rate of color removal is directly proportional to the current density. The maximum color removal efficiency was attained at a pH of 9 with a current density of 43.48 mA/cm^2.

For future studies, a comparison study can be explored between electrocoagulation with vibration of plates and other agitation mechanisms in electrocoagulation, such as using magnetic stirring or other types of mechanical stirring in the electrocoagulation cell. Performance can be distinguished with the vibration-induced plates in terms of the determination of overall resistance, removal efficiency, gas characteristics, and flow behavior using these agitators. Electrocoagulation with vibration-induced plates can be applied on a large scale as a preliminary treatment of wastewater. Therefore, the use of vibration-induced plates can be proposed as an alternative agitation mechanism in electrocoagulation since the vibration of the plates, which is able to intensify the gas bubbles roles during the flocculation and flotation stages, results in enhanced efficiency.

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