

# Decolorization of Acid Yellow 23 by Photo-Electro Fenton Process using Used Alkaline Batteries

Clinton Brinyang Dris, Nurhafizah Mohd Selihin\*

Faculty of Applied Sciences, Universiti Teknologi MARA,  
Cawangan Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia

\*Corresponding Author's E-mail: [nurhafizah729@uitm.edu.my](mailto:nurhafizah729@uitm.edu.my)

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## ABSTRACT

Wastewater, a pressing environmental issue worldwide, is exacerbated by pollutants such as azo dyes (e.g., Acid Yellow 23) and the growing volume of electronic waste, including alkaline battery waste. Alkaline batteries contain harmful substances like lead, cadmium, and lithium, and the use of novel materials in batteries raises additional environmental safety concerns. Reusing materials from used batteries for wastewater treatment is gaining interest, as this approach can create a closed-loop system, converting e-waste into useful products and reducing the need for new raw materials. This study aims to determine the removal efficiency of Acid Yellow 23 using graphite electrodes derived from used alkaline batteries in the photo-electro Fenton (PEF) process and to identify the optimal conditions for maximizing this efficiency. PEF is a variant of the Fenton reaction that uses light to generate hydroxyl radicals ( $\text{OH}\cdot$ ) instead of relying solely on electrochemical methods. In this process, hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and a photosensitizer, which absorbs light and initiates chemical reactions, are added to a solution containing the target pollutant. The combination of  $\text{H}_2\text{O}_2$ , ultraviolet (UV) radiation, and ferrous ( $\text{Fe}^{2+}$ ) or ferric ( $\text{Fe}^{3+}$ ) ions (as oxalate ions) produces more  $\text{OH}\cdot$  radicals than the traditional Fenton method. The results indicated that the system achieved the highest removal rate of 88.2% under optimal conditions: an initial Acid Yellow 23 concentration of 40 mg/L, a catalyst concentration ( $\text{FeSO}_4$ ) of 6 mM, and an applied voltage of 4.5 V. Under these conditions, the kinetic study of dye removal followed a second-order reaction model. These findings demonstrate a significant improvement in dye removal efficiency with the introduction of light in



*the PEF process. This research highlights the potential of repurposing electronic waste materials for effective wastewater treatment, contributing to sustainable environmental practices and resource-efficient technologies.*

*Keywords: Acid Yellow 23; Photo-Electro Fenton; Alkaline Batteries; Graphite; UV-Visible*

## INTRODUCTION

Wastewater is a critical environmental concern affecting every corner of the globe. Defined as any water impacted by human activities, it becomes unsuitable for direct use or consumption, containing various contaminants, including organic and inorganic substances, chemicals, pathogens, and nutrients. Sources of wastewater include municipal, industrial, and agricultural activities. Processed wastewater is commonly reused for non-drinking applications such as agriculture, irrigation, groundwater replenishment, golf course watering, vehicle cleaning, toilet flushing, firefighting, and construction activities.

Alkaline batteries are among the most widely used household batteries, with billions consumed annually, generating significant waste. These batteries contain pollutants like lead, cadmium, and lithium, and the introduction of novel materials raises additional environmental safety concerns. Recycling alkaline batteries can help conserve natural resources by reducing the need for primary materials extraction and processing [1]. Graphite, a valuable material within these batteries, can be recycled and repurposed, helping to conserve natural resources and reduce the environmental impact of new graphite production [2].

In this study, we explore the use of graphite electrodes extracted from used alkaline batteries in the photo-electro Fenton (PEF) process for treating wastewater. The PEF process, an advanced oxidation process (AOP), leverages light to generate hydroxyl radicals ( $\text{OH}\cdot$ ) instead of relying solely on electrochemical methods. This involves adding hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and a photosensitizer to a solution containing the target pollutant, enhancing the production of  $\text{OH}\cdot$  radicals and improving pollutant degradation efficiency.

The system achieved the highest removal rate of 88.2% for Acid Yellow 23 under optimal conditions: an initial concentration of 40 mg/L, a catalyst concentration ( $\text{FeSO}_4$ ) of 6 mM, and an applied voltage of 4.5 V. The kinetic study indicated a second-order reaction model for dye removal.

The PEF process offers several advantages, including enhanced degradation efficiency and lower energy consumption due to the additional energy provided by UV light [3]. This study aims to:

- (i) Determine the removal efficiency of Acid Yellow 23 using graphite electrodes from used batteries in the PEF process via Ultraviolet Visible Spectroscopy (UV-Vis).
- (ii) Investigate the optimal conditions for using graphite electrodes from used batteries in the PEF process for Acid Yellow 23 removal.

## METHODOLOGY

### Photo-electro fenton set up

In this study, we are using artificial light as a source of light. The oxidation process can be improved in the presence of light as there are more hydroxyl radicals ( $\cdot\text{OH}$ ) present [4]. For electrode selection, platinum and boron doped diamond electrodes are expensive. So, for this study, we chose graphite which is easily obtained from e-waste (alkaline batteries).

### Graphite electrodes

Carbon rods from used alkaline batteries were carefully extracted using pliers, with necessary safety precautions taken by wearing gloves. Following a modified procedure based on Cruz-González *et al.* [5] with a slight modification, the extracted carbon rods were modified to fit an area of  $5\text{ cm}^2$ , serving as electrodes. Both electrodes were immersed in a beaker containing an electrolyte solution, maintaining a distance of approximately 1 cm from each electrode with an exposed area of  $5\text{ cm}^2$ .

## Electrolysis setup

To ensure a steady supply of hydrogen peroxide ( $H_2O_2$ ) a commercially available fish pump was utilized which also generated additional oxygen ( $O_2$ ) to support the production of hydrogen peroxide [6]. Prior to initiating the current flow, the pump was operated for 30 minutes to establish a consistent bubble stream. Subsequently, a magnetic stirrer was put into the beaker to produce a rotating effect, promoting the precipitation of ferric hydroxides [7]. A solution of 50 mg/L acid yellow 23 was prepared, followed by adjusting the pH to 3 by adding a few drops of sulfuric acid ( $1M H_2SO_4$ ). Once the catalyst, iron sulphate ( $FeSO_4$ ) was added at the specified concentrations,  $0.05 M Na_2SO_4$  was introduced as a background electrolyte to facilitate conductivity. Table 1 shows the summary of operating conditions for each parameter.

**Table 1: Experimental Design with a slight modification from Anil *et al.* [8]**

No.	Parameter	Operating Conditions
1.	Concentration (mg/L)	50, 40, 30
2.	Catalyst (mM)	2, 4, 6
3.	Voltage (V)	1.5, 3.0, 4.5

The whole setup was placed under visible light (table lamp with a 100-W tungsten bulb) whose distance from the top of the lamp to the bottom surface of the beaker is 30 cm. The setup was equilibrated in the darkness for 30 min. Before turning on the UV lamp, adsorption occurs when the pollutants in water are attracted onto the surface of the electrode material to increase the concentration of the pollutants near the electrode surface. After the addition of  $H_2O_2$ , the UV lamp was turned on to start the reaction [9]. Following the activation of the UV lamp, desorption occurs, where pollutants are released from the electrode surface back into the water solution, allowing them to come into contact with  $\cdot OH$  radicals [10]. Finally, the photo-electro-Fenton reaction commenced with the start of the current flow. Figure 1 shows the schematic PEF experimental setup, while Figure 2 shows the actual PEF setup.

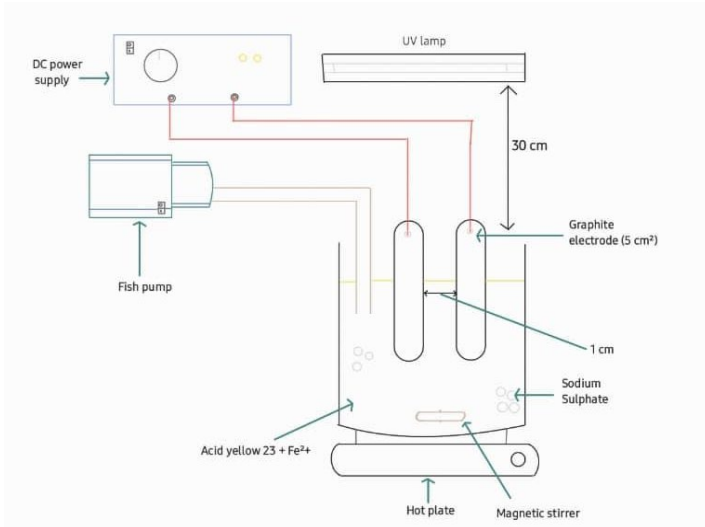


Figure 1: The schematic PEF experimental set up

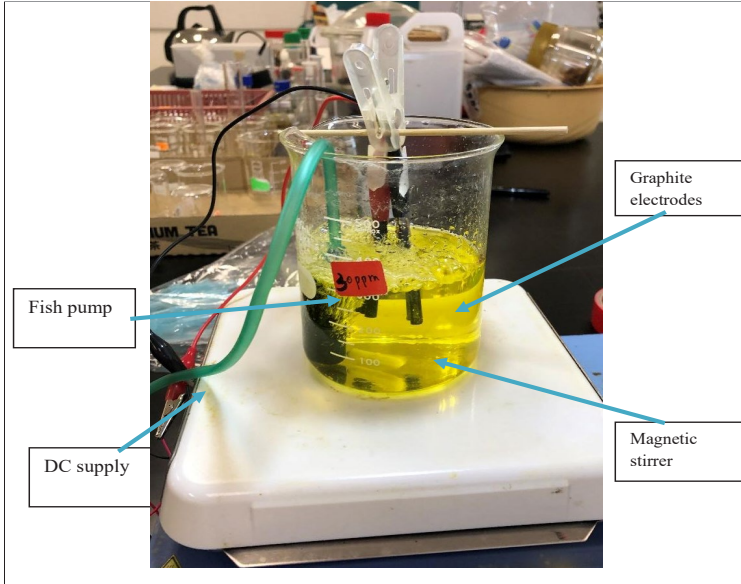


Figure 2: The actual PEF experimental set up

## Analysis of optimal conditions:

It should be noted that the selection of parameters below was based on a study conducted by Anil *et al.* [8] with a slight modification. The samples were taken at hourly intervals and analyzed using ultraviolet-visible (UV-Vis) spectroscopy at 428 nm maximum wavelengths. All samples were done in triplicate and standard calibration was conducted using standard concentration of acid yellow 23 (10, 15, 20, 25, 30 mg/L) prior to sample analysis as quality control. The  $R^2$  values were initially determined to be 0.995. The study was conducted in a laboratory with proper ventilation. All hazardous materials are carefully handled.

## Concentration

The PEF setup was initiated using optimal conditions, with a contact time set at 3 hours and a pH of 3. Fixed parameters included a voltage of 1.5 V, room temperature, a catalyst concentration of  $\text{Fe}^{2+}$  at 2 mM, and a light intensity of 1000 lx. To enhance the electrolysis process, a supporting electrolyte, 0.05 M  $\text{Na}_2\text{SO}_4$ , was added in all experiments.

In the first step, we study the effect of initial concentration using 30, 40, 50 mg/L concentration of acid yellow 23. The samples were taken at hourly intervals. The optimal concentration was chosen from 30, 40, 50 mg/L based on the highest removal efficiency (%) calculated from the maximum absorbance of 428 nm in the ultraviolet-visible (UV-Vis) spectroscopy.

## Catalyst

Once the optimum initial concentration of acid yellow 23 was obtained, the study then proceeded to find the optimum catalyst ( $\text{FeSO}_4$ ) concentration. Catalyst concentration of 2 mM, 4 mM, and 6 mM was prepared to determine which concentration of catalyst is the most optimal. Here, PEF setup was fixed at conditions of optimum initial concentration of acid yellow 23, pH 3, contact time of 3 hours, 0.05 M  $\text{Na}_2\text{SO}_4$ , 1.5 V voltage, room temperature and light intensity of 1000 lx.

## Voltage

Once the optimum catalyst concentration was obtained, the study then proceeded to find the optimum voltage. Here, PEF setup was fixed at conditions of optimum initial concentration of acid yellow 23, optimum concentration of catalyst at pH 3 with 3 hours contact time, 0.05 M Na<sub>2</sub>SO<sub>4</sub>, room temperature and light intensity of 1000 lx. The voltage analyzed were 1.5 V, 3.0 V and 4.5 V. One criterion of a good cathode is that it must have a high overvoltage for hydrogen evolution [11]. Therefore, we chose this range for voltage selection similar to the study conducted by Anil *et al.* [8].

At the contact time of 3 hours, samples were collected to determine the absorbance values. The optimized voltage was achieved at the maximum removal efficiency.

## Analysis Using UV-Vis Spectrometer

In this methodology, UV-Visible (Perkin Elmer Lambda 25 UV/Vis) was used to record the absorbance of treated acid yellow 23. The magnitude of the absorbance was proportional to its concentration and follows the Beer-Lambert Eq. (1) [12].

$$A = \epsilon bC \quad (1)$$

Where, A = absorbance,  $\epsilon$  = molar absorptivity (L mol<sup>-1</sup> cm<sup>-1</sup>), b = length of light path (cm) and c = concentration (mol L<sup>-1</sup>).

The absorbance value of solution was measured at a wavelength of 428 nm. The removal efficiency of acid yellow was calculated according to Eq. (2) [13].

$$\text{Removal efficiency} = (C_0 - C_t) / C_0 \times 100\% \quad (2)$$

Where C<sub>0</sub> is the initial concentration of acid yellow 23 (mg/L), C<sub>t</sub> is final concentration of acid yellow 23 (mg/L).

## Kinetic study

To assess the catalytic performance of different systems more accurately, zero, first and second order kinetic analysis were implemented based on the Eq. (3-5) [14].

$$\text{Zero order kinetic model: } C_0 - C_t = kt \quad (3)$$

$$\text{First order kinetic model: } \ln (C_t / C_0) = -kt \quad (4)$$

$$\text{Second order kinetic model: } 1 / C_t - 1 / C_0 = kt \quad (5)$$

## RESULTS AND DISCUSSION

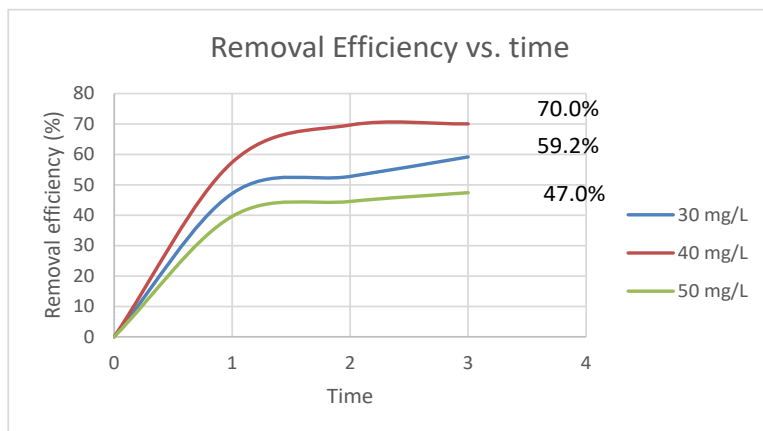
### Effect of initial dye concentrations

Before we continue with the first parameter, a UV lamp with light intensity of 1000 lux was put under the experiment set up with a distance of 30 cm from the top of the lamp to the bottom surface of the beaker. This PEF process is an advanced oxidation process that combines electrochemical and photochemical methods to produce hydroxyl radicals ( $\cdot\text{OH}$ ). These radicals are highly reactive and can effectively break down a wide range of organic contaminants based on Eq. (6) to (10).



Each variable in the system was prepared according to the proposed initial measurements. The removal of acid yellow 23 was determined using a UV-Visible spectrometer at 428 nm. The removal efficiency was determined using standard concentrations of acid yellow 23 with 10, 15, 20, 25, 30 mg/L (where the obtained  $R^2 = 0.995$ ). It was found that the removal efficiency increased when initial concentration of acid yellow 23 increased from 30 mg/L (59.2%) to 40 mg/L (70%) but decreased to 47% when the concentration increased at 50 mg/L (Figure 3).





**Figure 3: Removal efficiency for 30,40,50 ppm of initial acid yellow 23 concentrations**

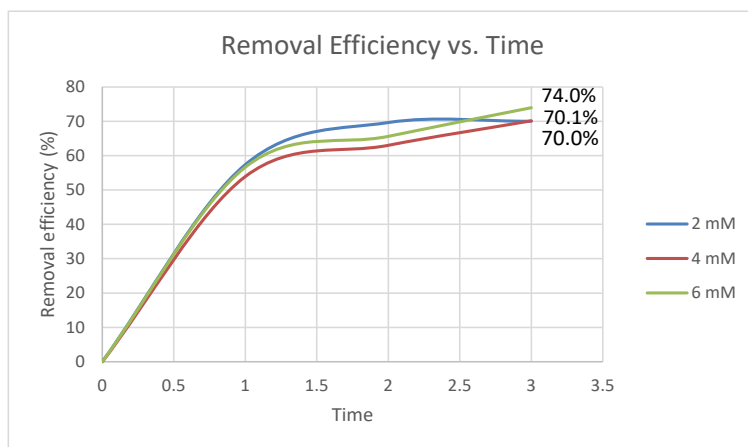
It shows that with increasing initial dye concentration from 30 mg/L to 40 mg/L causes the removal efficiency to increase. Higher initial dye concentrations enhance the interactions between the  $\cdot\text{OH}$  and dye molecules and the higher driving forces involved, resulting in increased dye removal efficiency [15]. The increase in initial concentration also boosts the interaction between  $\cdot\text{OH}$  and the dye, leading to a greater degradation of the dye [16]. However, if  $\cdot\text{OH}$  becomes saturated, additional dye molecules will not be degraded, and the efficiency per unit of dye will decrease in which is why removal efficiency for 50 mg/L is decreases. There is a point at which  $\cdot\text{OH}$  becomes saturated with dye molecules, beyond which no further degradation can occur. This saturation point limits the maximum removal capacity of the dye [17]. Moreover, higher initial concentrations result in less light entering the PEF system cause the reduction of the amount of  $h\nu$  reacting with  $\text{H}_2\text{O}_2$  and generating  $\cdot\text{OH}$  [18]. Therefore, its resulted in low removal efficiency of 50 mg/L (47%).

Using a similar AOPs technique, the removal of eosin yellow dye from industrial wastewater using photoelectron-Fenton techniques was conducted by [18]. A low-pressure mercury lamp (UV-C tube, 45 cm/15 W, Philips) with steel sacrificial anode and graphite cathode of 7 mm diameter were used in the electrochemical setup. Their study found that 100 ppm showed

the highest removal efficiency with 98% after 30 minutes contact time compared to 150 ppm (91%) and 200 ppm (27%) respectively. Similarly, the study done by Elton *et al.* [19] also used similar AOPs techniques to degrade methylene blue from industrial wastewater using PEF technique. Mixed iron and copper were synthesized by the modified Pechini method ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/CuO). Their study found that 10 mg/L initial dye concentrations showed 100% dye removal in 30 minutes of reaction compared to 2.28% for 50 mg/L.

### Effect of catalyst

Catalyst of Fe<sup>2+</sup> were prepared according to different concentrations which are 2 mM, 4 mM and 6 mM and removal efficiencies obtained were 70.0%, 70.1% and 74.0% respectively (Figure 4).



**Figure 4: Removal efficiency of acid yellow 23 with 2,4,6 mM catalyst concentration**

The appropriate concentration of Fe<sup>2+</sup> ions in the PEF process is a critical factor. Generally, the efficiency of the PEF process improves with an increased concentration of Fe<sup>2+</sup> ions, as Fe<sup>2+</sup> serves as a potent oxidizing agent capable of breaking down large molecules in wastewater, such as dyes from dyeing processes [20].

Additionally, the study of tetracycline (TC) solution degradation from ground waters was done by Ma *et al.* [21] using the same AOPs technique which is PEF technique. The study was carried out in a 200 mL glass vessel (internal diameter 5.3 cm) covered with foil, and a 300 W Xe lamp (PLS-SXE300c,  $\lambda \geq 420$  nm). They found that the  $\alpha\text{-Fe}_2\text{O}_3$  catalyst can activate  $\text{H}_2\text{O}_2$  and achieved a 85-95% removal of 30-100 mg/L within 60 minutes. Similarly, study conducted by Wang *et al.* [22] in the removal of the colored wastewater using EF technique found that, the removal efficiency was poor (9%) when no  $\text{Fe}^{2+}$  ions were added to the solution, but the color removal efficiency was notably increased from 9% to 46% by externally adding 5 mM  $\text{Fe}^{2+}$  ions.

### Effect of voltage

The decolorization efficiency was significantly affected by voltage in the range of 1.5 V to 4.5 V. In three hours, the removal efficiencies were found to be 73.0 %, 77.8% and 88.2% for 1.5 V, 3 V and 4.5 V respectively. It can be concluded that higher voltage led to higher removal efficiency. Figure 5 below shows the removal efficiency of acid yellow 23 under different voltage.

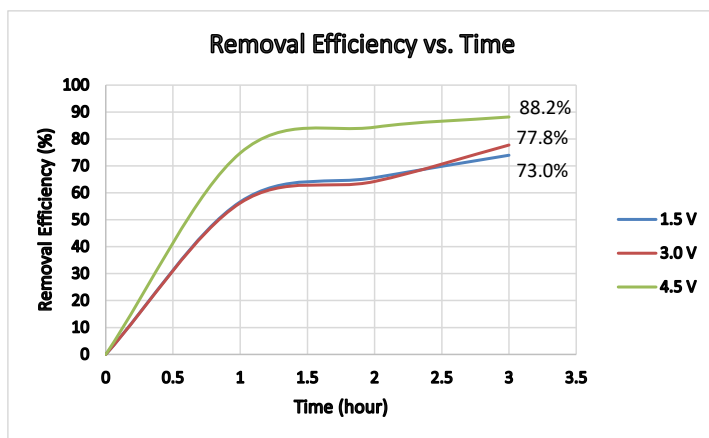


Figure 5: Removal efficiency of acid yellow 23 under 1.5, 3.0, 4.5 V

The enhancement in degradation with increasing voltage is due to the increase in the production of  $\bullet\text{OH}$ . In addition to increasing the concentration

of  $\bullet\text{OH}$  in the solution, applying high voltage also enhances the regeneration of iron ions, thereby improving the efficiency of the PEF process [20].

Similarly, according to a study by Anil *et al.* [8], they found that, when the applied voltage was 1 V, the removal percentage of 10 mg/L RhB dye was 22.15% then with the increasing of applied voltage to 2 V, degradation enhanced to 37.32% and upsurge degradation was achieved when the applied voltage was 3 V that is 97.52%. In another study done by Paz *et al.* [23] to remove Orange II (OII) dye using the similar PEF technique proved that using current density of  $200 \text{ mA cm}^{-2}$  caused fastest decolorization of OII in 50 minutes compared to using  $100 \text{ mA cm}^{-2}$  (120 minutes) and  $200 \text{ mA cm}^{-2}$  (75 minutes).

It has been identified that the parameters of initial dye concentrations, catalyst concentration and applied voltage are the key factors affecting both  $\text{H}_2\text{O}_2$  generation and the dye degradation. Moreover, PEF utilized light to generate hydroxyl radicals ( $\bullet\text{OH}$ ) instead of relying solely on electrochemical methods. The experimental results shown in Fig. 5 demonstrated that the increase of the applied current density up to 4.5 V in the PEF reaction could enhance the azo dye degradation significantly, since the higher rate of  $\text{H}_2\text{O}_2$  generation was achieved at the higher applied voltage.

## Kinetic study

The coefficient of determination ( $R^2$  values) exceeding 0.9 indicates that the dye removal efficiency using PEF adheres closely to second-order kinetics. A moderately high correlation coefficient indicates that there is a feasible relationship between the concentrations of radicals and organic matter. From the data presented in Table 2, the highest reaction rate constant,  $k$  of  $0.062 \text{ M}^{-1} \text{ h}^{-1}$  was observed at 4.5 V, achieving a removal efficiency of 88.2% in the photo-electro-Fenton process (PEF). A higher  $k$  value signifies a faster reaction rate in PEF. A comparison between initial and optimized conditions revealed a significant difference in the reaction rate constants, with  $k$  being  $0.0057 \text{ M}^{-1} \text{ h}^{-1}$  and a removal efficiency of 47.0% initially, while under optimized conditions, the highest  $k$  value was obtained. This suggests that employing optimized parameters in the experiment could effectively minimize contaminants in the sample.

**Table 2: Kinetic analysis table**

Parameter	Reaction orders	Zero order		First order		Second order	
		K (Mh <sup>-1</sup> )	R <sup>2</sup>	K (h <sup>-1</sup> )	R <sup>2</sup>	K (M <sup>-1</sup> h <sup>-1</sup> )	R <sup>2</sup>
1. Initial dye concentration	30 ppm	-5.4937	0.7689	-0.2799	0.8376	0.0152	0.9063
	40 ppm	-8.894	0.7401	-0.3954	0.8142	0.0199	0.8783
	50 ppm	-7.3558	0.7347	-0.2012	0.7762	0.0057	0.8215
2. Catalyst	2 mM	-8.894	0.7401	-0.3954	0.8142	0.0199	0.8783
	4 mM	-8.7821	0.7904	-0.3847	0.8877	0.019	0.9675
	6 mM	-9.2353	0.7934	-0.4269	0.9021	0.0228	0.9792
3. Voltage	1.5 V	-9.2353	0.7934	-0.4269	0.9021	0.0228	0.9792
	3.0 V	-9.6511	0.8292	-0.4711	0.9399	0.0275	0.9626
	4.5 V	-10.965	0.7236	-0.6883	0.8791	0.062	0.9934

Similarly, a pseudo-second order model fitted a study conducted by Ramos *et al.* [24] in the degradation of indigo carmine (50 mg/L) for 120 minutes by photo-Fenton with R<sup>2</sup> 0.98 with a maximum k value of  $2.5 \times 10^{-3} \text{ Lmg}^{-1} \text{ min}^{-1}$  at  $9 \text{ mmolL}^{-1} \text{ Fe}^{2+}$  and a  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  ratio of 7:1. Also from the study done by Tavares *et al.* [25], they attempted to degrade 50 mg/L reactive blue 19 (RB19), reactive red 195 (RR195), and reactive yellow 145 (RY145) using electro-Fenton reactions that were conducted in an agitated batch reactor using 100 mL of dye solution. From the kinetic study, they found that for the RY145 dye best fitted the second-order model.

## CONCLUSION

Photo-Electro-Fenton (PEF) demonstrated superior efficacy in treating azo dye wastewater due to its integration of UV light into the Fenton reaction, which enhanced both the degradation rate and efficiency of azo dyes. The UV light provided supplementary energy that amplified the oxidizing power of hydroxyl radicals ( $\cdot\text{OH}$ ) and accelerated the decolorization process. In this study, we applied the PEF process using graphite electrodes extracted from used alkaline batteries. These batteries contained abundant graphite material that could be readily recovered and used for azo dye removal in synthetic wastewater. Utilizing graphite from alkaline batteries offered a

cost-effective and widely available option for wastewater treatment.

The efficiencies of various factors affecting dye removal were analyzed, considering initial dye concentrations, catalyst concentrations, and different applied voltages. The results indicated that the system achieved the highest removal rate of 88.2% when the optimum conditions of initial concentration of acid yellow 23 was 40 mg/L, the catalyst concentration ( $\text{FeSO}_4$ ) was 6 mM, and the applied voltage was 4.5 V. It was found that the contribution rate of hydroxyl radicals ( $\cdot\text{OH}$ ) to the removal of acid yellow 23 was proportional to the removal ratio of the dye. These findings also provide a theoretical basis for enhancing the current efficiency of the photo-electro-Fenton system.

In this study, several challenges were encountered while optimizing the Photo-Electro-Fenton (PEF) process for the decolorization of acid yellow 23. One significant challenge was the optimization of various parameters, such as initial dye concentration, catalyst concentration, and applied voltage, to achieve the highest dye removal efficiency, which required extensive experimentation and precise fine-tuning. Additionally, the extraction and preparation of graphite electrodes from used alkaline batteries presented practical difficulties, including ensuring the purity and suitability of the recovered graphite for the electro-Fenton process. The graphite electrodes also need to be changed for every set of parameters since graphite is very prone to rust.

Another critical challenge was ensuring the efficient generation and maintenance of hydroxyl radicals ( $\cdot\text{OH}$ ), as their contribution rate is directly proportional to the dye removal efficiency. Furthermore, maintaining the stability of the electro-Fenton system over prolonged operational periods and scaling up the process for potential industrial applications posed significant obstacles. While using graphite from alkaline batteries was identified as a cost-effective approach, ensuring a consistent and sufficient supply of used batteries for large-scale applications remained challenging. Additionally, the costs associated with the energy required for UV light and applied voltage had to be carefully considered. Addressing these multifaceted challenges is crucial for the practical and efficient application of the electro-Fenton process using recycled graphite electrodes, ultimately contributing to the sustainable treatment of wastewater.

Furthermore, the need for pilot research must be intensively studied among scientists and researchers. Further studies required higher removal efficiency for large amounts of wastewater by optimization of parameters. For example, the surface area of graphite extracted from alkaline batteries can be larger. So that it can absorb many photons and generate a large number of electron-hole pairs. Other than that, the concentration of the catalyst can be higher so more ferrous ions,  $\text{Fe}^{2+}$  can generate more  $\cdot\text{OH}$ . Effect applied voltage can also be studied to find the optimal voltage for large amounts of wastewater dye removal. Consequently, massive use of used alkaline batteries in degrading wastewater dye can help in decreasing the amount of e-waste on the landfill.

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