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Electro-Fenton degradation of Methylene Blue using Graphite/Magnetite cathode

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Abstract: Methylene blue in aqueous solution was effectively discolored using an electro-Fenton (EF) system with a magnetite fixed on graphite by resin epoxy as cathode and graphite as anode. Experimental study was done to analyze the effect of pH, current density (J), supporting electrolyte concentration (electC), inner electrode spacing (IES), catalyst concentration (CatC), and BM concentration (CBM) on dye removal rate. The optimal conditions are pH= 3, J= 10 mA/cm^2, electC= 0.05 M, CBM= 10 ppm, IES= 3 cm, and CatC= 0.2 g/l, as shown in the results. After 30 and 100 minutes of reaction, respectively, the removing rate for 10 ppm and 50 ppm is 99.8 % and 99.5 %. Furthermore, with a 30-minute purification in a 1M HCl solution in between cycles, those cathodes may be used at least three more times without experiencing performance loss. In order to remove dyes from wastewater processes and applications, Magnetite fixed on Graphite can work well as an electro-Fenton system's cathode.

Keywords: *Electrolysis; Electro-Fenton systems; water treatment; electrochemistry; electrochemical reactor; Heterogeneous catalysis*

1. Introduction

The wide impacts of natural and man-made pollutants and wastes on the ecosystem necessitate advanced treatment plans based on multiple physical, biological, and chemical principles that can convert pollutants into less dangerous compounds. Many researches has studied a numerous of physical treatment of waste water as Absorption, flotation, coagulation, precipitation and a lot more all of the method aim to eliminate pollution from water (Agarwal et al., 2016; Anton et al., 2016; Bennajah, 2007; Borges et al., 2016; Boumaza et al., 2016; Btatkeu-K. et al., 2016; Buscio, Crespi, et al., 2015; Buscio, Marín, et al., 2015; el Bekkali et al., 2016; el Messaoudi et al., 2016; Foo & Hameed, 2010; Gupta et al., 2015; Kankou *et al.,* 2021; Majdy et al., 2015; Miyajima & Noubactep, 2012; Rodríguez-Chueca et al., 2015; Xiao et al., 2015), on the other hand some researches were done by biological treatment that aim of use biofilm consume wastewater (Dellamatrice et al., 2017).

The removal of methylene blue (BM), a toxic dye substance found in wastewater, has been the subject of numerous studies, (Ratiki et al., 2023) worked on the removal of Methylene Blue using Bassorin hydrogel (extracted from plant) as adsorption agent, (Badraoui et al., 2019) are using local clay from a natural basin as adsorbent, the results was very interesting and efficient on removal of BM, other research aim to eliminate the toxicity of Methylene Blue by valorization of natural biomass by using Moringa oleifera pods and kernels (Ahmadou et al., 2023), some research's aims to eliminate Methylene blue dye from drinking water using silico-aluminous clays with the dominance of mineralogical phases kaolinite of muscovite as an effective absorbent agent (Elmontassir et al., 2019). In recent years, innovative wastewater treatment strategies for removing stubborn chemicals have been researched (de Boer et al., n.d.). Although Fenton's reaction has existed and been evaluated for performance for about three centuries, the viability of large-scale environmental applications is currently a very hot research topic. The electro-Fenton (EF) (Bennajah, 2007; de Boer et al., n.d.; Do et al., 2017; Dong et al., 2016; Duarte et al., 2013; Espinoza et al., 2016; Foo & Hameed, 2010; Gomes et al., 2015; Mansour et al., 2011; Miyajima & Noubactep, 2012; Pajootan et al., 2014; Pereira et al., 2016; Sbai & Loukili, 2015; Zhou et al., 2015) process has emerged as one of the most successful among the wide range of existing processes whose reactivity is primarily dictated by the catalyzed transformation of a mild oxidizing reagent like H_2O_2 into the second-strongest oxidant known OH radical. Advanced oxidation techniques are one of them (AOPs), The advanced oxidation process (AOP) known as "Electro-Fenton" (EF) offers in situ electro-generation of H_2O_2 in the presence of the Fenton reaction catalyst to form OH radicals indirectly. The EF process can be very successful method of mineralization of any organic contaminant in aqueous media to carbon dioxide, water, and inorganic species at room temperature and pressure.

2. Experimentations

2.1. Materials & chemicals

The Graphite Electrodes 9.5 x 0.3 x 3 cm, the Magnetite powder (Fe_3O_4), Na_2SO_4, Methylene Blue (MB), NaOH, H_2SO_4 and HCl. The electrodes of Graphite with Magnetite fixed using epoxy resin are prepared in laboratory, spectrophotometer UV-VIS is from Analytic Jena used for sample analysis. A SARTORIUS 0.1 mg precision balance was used in all measurement procedures. The Figures 1 and 2 Shows using MEB images the difference between graphite surface and graphite with magnetite layer respectively. We can see that a portion of the graphite cathode surface is covered in a layer of magnetite in Fig. 2.; as can be seen, the surface is more regular and smoother than the portion of the graphite cathode that isn't (Figure 1.) The use of epoxy resin as a fixing agent and a coating of magnetite nanoparticles is primarily responsible for these findings. The Chemicals mentioned above were used without further purification.

2.2. Cathode preparation

Three different masses (0.05g, 0.2g, and 0.4g) of Magnetite were fixed by epoxy resin on the surface of three Graphite electrodes (0.5cm, 1cm and 2cm) x 3cm, respectively. All the three electrodes have the same dimension 3x9.5 cm. before and after the fixation of Magnetite, the electrodes must be washed with distilled water and dried at temperature of 120°C for 1h.

2.3. Experimental procedure

The degradation of various concertation of methylene blue (MB) solution is studied in experiments using a 250 ml Batch glass reactor fig.3. The working volume for the electrolysis tests was determined to be 200 ml of the solution. In order to adjust the pH, 0.5N H_2SO_4 and 0.5N NaOH were used. The anode was made of graphite with a 3 x 5 cm immerged area. As an alternative, a cathode with magnetite fixed on it using epoxy resin is functioning as a catalyst for the process. The removal efficiency of MB was measured at three different CatC 0.05 g/l, 0.2 g/l, and 0.4 g/l. The two electrodes are immerged in cylindrical beaker vertically and various inner electrode spacing was studied. The stirring was carried using magnetic stirrer at 240 rpm.

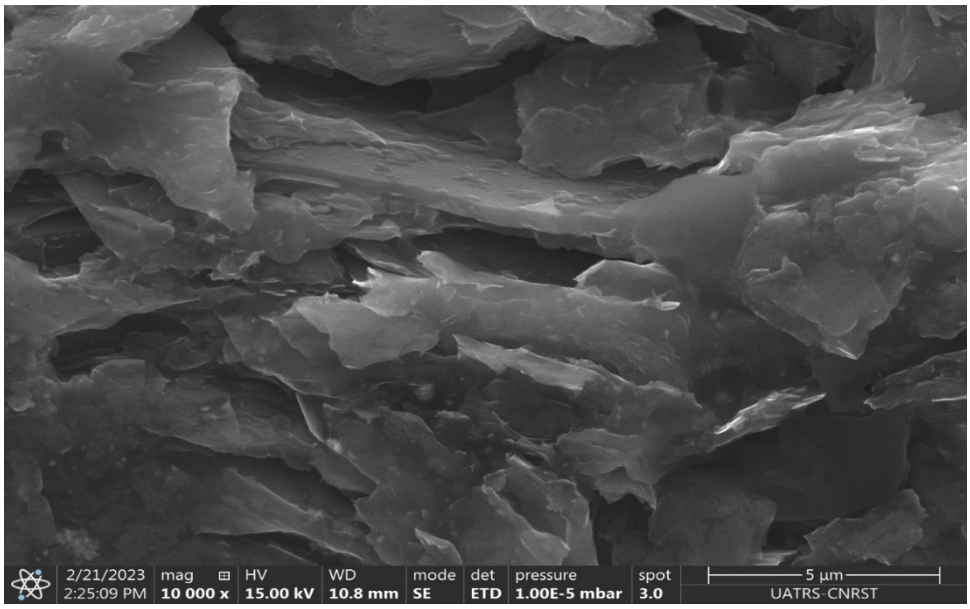

Figure 1. Images by MEB of graphite electrode surface 10.000 X

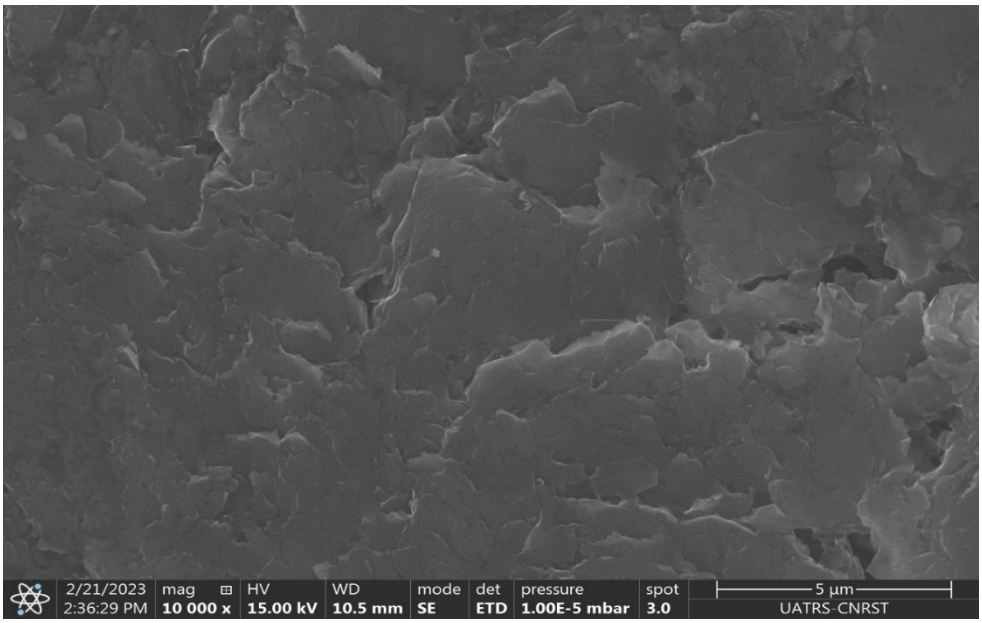

Figure 2. Images by MEB of graphite electrode surface covered with Magnetite 10.000 X

Na_2SO_4 was used as the supporting electrolyte with different electC. Samples were collected at different reaction time and then analyzed using Analytic Jenna UV-visible spectrophotometry, the rate de degradation of methylene blue is evaluated using the following equation (1)

$$\text{BM Rate degradation \%} = 100 \times \frac{(C_0 - C_i)}{C_0} \quad (1)$$

C_o and C_i are the initial and at instant of time MB concentrations of EF reaction, respectively.

The cathode pulled out from the solution, washed with distilled water and immersed in 1M HCl for 30 min for the removal of absorbed MB. Then used in another oxidation cycle under the same conditions described above. Yet there is a limit of use of anode due to the degradation of graphite after a few cycles, as a result a regular change of anode is necessary.

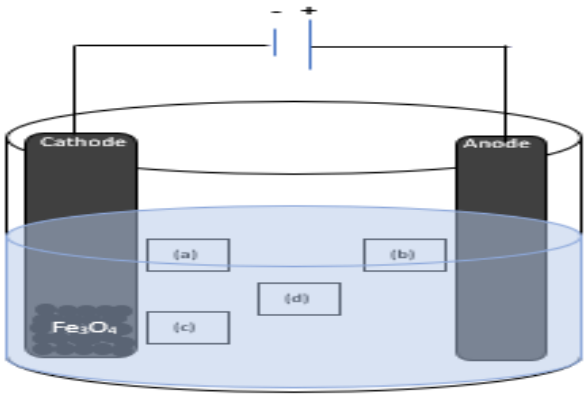

Figure 3. Conceptual representation of the Electro-Fenton setup

Figure 3 shows multiple reactions happening on our system. The anode generates O_2 by H_2O oxidation (Rea.1.) (a). In-situ regenerated Fe^{2+} in the system maintains the catalytic activation of H_2O_2 to create the OH radical (Rea. 2. and Rea. 3.) (b) and (c). Fe^{3+} can be reduced with H_2O_2 in water (Rea. 4. and Rea. 5.) (d), intermediate organic radicals, or directly on the cathode surface (Rea. 6) to regenerate Fe^{2+}. According to reaction 3., active OH radicals are produced, which convert pollutants R-H into less hazardous molecules and even non-toxic substances like CO_2 and H_2O (Rea. 7) (d) (de Boer *et al.*, n.d.).

$H_2O \rightarrow H^+ + e^- + O_2$	Rea.1.	**(a)**
$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2$	Rea.2.	**(b)**
$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^-$	Rea.3.	**(c)**
$OH^{\bullet} + H_2O_2 \rightarrow H_2O + HO_2^{\bullet}$	Rea.4.	**(d)**
$Fe^{3+} + HO_2^{\cdot} \rightarrow Fe^{2+} + O_2 + H^+$	Rea.5.	**(d)**
$Fe^{3+} + e^- \rightarrow Fe^{2+}$	Rea.6.	**(d)**
$OH^{\bullet} + R\text{-}H \rightarrow R^{\bullet} + H_2O \rightarrow CO_2 + H_2O$	Rea.7.	**(d)**

3. Results & discussion

3.1.Electro-Fenton reaction

3.1.1. Effect of pH solution

Four different initial pH values (2.5, 3, 3.5 and 4.0) on our electro-Fenton system was tested. Figure 4 shows clearly The MB degradation efficiency of EF process after 20 min of electrolysis were 93.17%, 99.26%, 87.31% and 77.22% at pH 2.0, 3.0, 3.5 and 4, respectively. In fact, the Fenton process is favorized in acidic condition due to high productivity and stability of H_2O_2 (Dong et al., 2016), yet in acidic condition below pH=3, the rate of H_2O_2 decreases by reacting with H^+ forming H_3O^+. Moreover, the increase of pH value above 3 can destabilize H_2O_2 by decomposing to H_2O and O_2, also at this pH condition the exitance of Fe^{2+} as catalyze less available due to transformation to Fe^{3+}. As result there is less iron ion to react with H_2O_2 to form OH radical (Sbai & Loukili, 2015). The figure 4 shows the removal efficiency decreases at higher pH value. pH=3 was selected as the optimal pH for further study. The decolorization of methylene blue (MB) by the electro-Fenton process using stainless steel (SS) mesh electrodes was investigated to reach the highest dye removal (99%) was observed at a Fe^{2+}:H_2O_2 ratio of 1:4 at 20 minutes (Loloei & Rezaee, 2016).

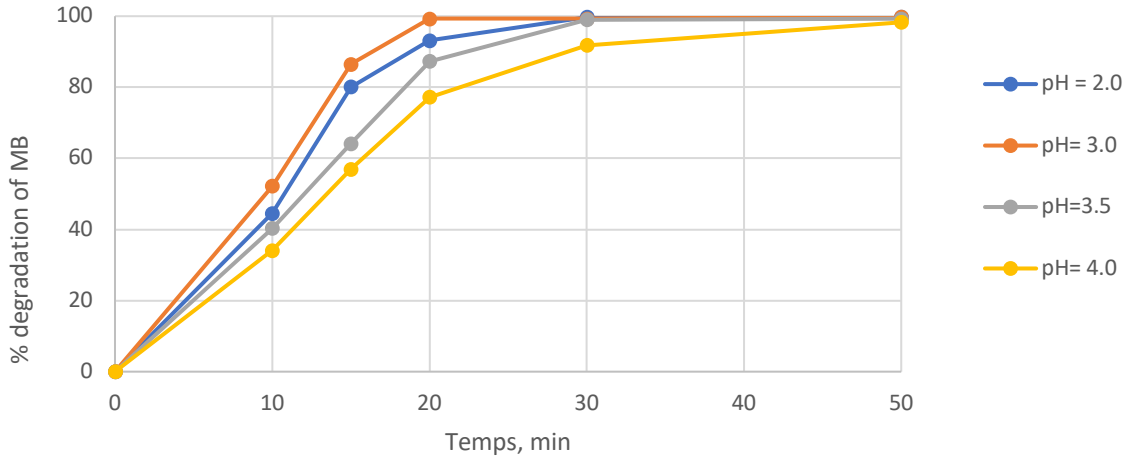

Figure 4. Effect of pH of solution on MB degradation. 0.2 g/l Fe_3O_4, 10mA/cm2, 10 mg/l MB, 240 rpm, 0.05 M Na_2SO_4.

3.1.2. Effect of current density

In this part of study. The effect of current density on MB removal rate is shown in Figure 5. An increase in MB removal rate increases with current density J from 5 to 10 mA/cm^2, this increase proportionality is due to high production of hydroxyl radical as consequences high MB degradation rate. Because the degradation efficiency did not increase significantly at applied current density higher than 10mA/cm^2, 10mA/cm^2 was selected as the optimal current density for further study.

3.1.3. Effect of BM initial concentration

A Magnetite concentration of 0.1g/l fixed on graphite cathode was used in this study and effect of CBM was investigated. Figure 6. shows that when CBM increase, MB degradation Rate (RBM) decrease. For example, in t= 80 min, RBM decreased from 99.63% to 85.73% with the increase of CBM from 10 to 50 mg/l. Although, at the same electrolysis time, 80 min, the quantity of MB removed increased from 9.40 to 42.86 mg/l.

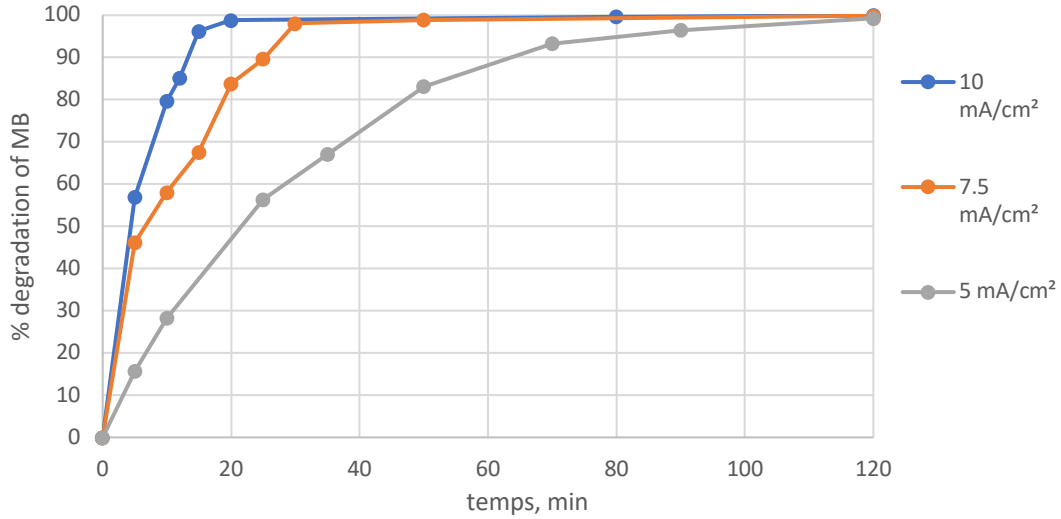

Figure 5. Effect of current density at pH=3, 0,2g/l Fe_3O_4, 10 ppm de BM, 0,05M de Na_2SO_4 and d=3cm

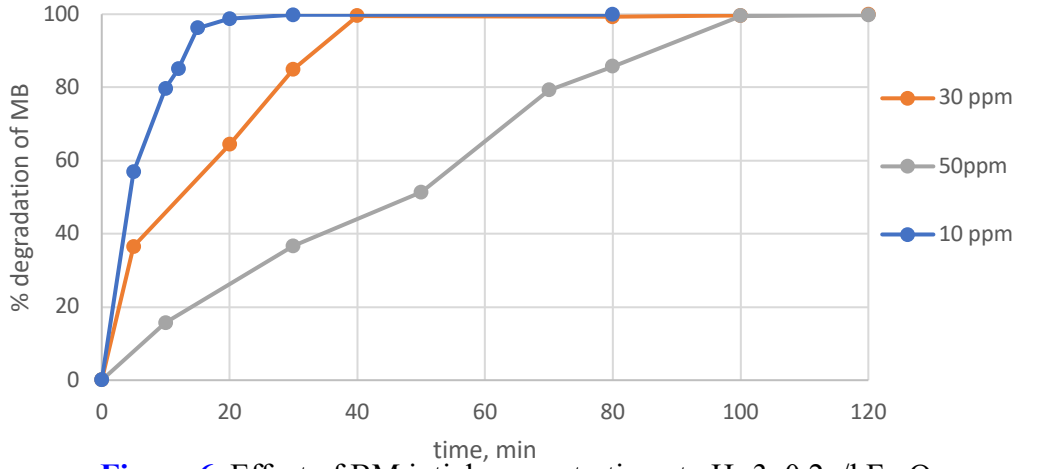

Figure 6. Effect of BM intial concentration at pH=3, 0,2g/l Fe_3O_4, 10 mA/cm² ,0,05M Na_2SO_4 and d=3cm

To explain what happening we can simply relay the increase of the amount of BM on the increase of radical hydroxyl that's mean there is enough amount of OH radical due to operational catalyzer that has enough active site to decompose H_20_2 generated by the same cathode, as result a higher amount of BM was degraded at CBM= 50 mg/l and this is happening under our experimental conditions.

4. *Effect of Fe_3O_4 concentration (catC)*

The effect of catC (Fe_3O_4) is essential to our EF system study, any luck of magnetite efficiency could decrease significantly MB removal Rate. catC was investigated by four concentrations of 0.05 g/l, 0.2 g/l, and 0.4 g/l of Magnetite fixed on Graphite for 10 ppm MB solution (Figure 7). As result, 0.1 g/l of Magnetite is the optimal catC with RBM of 99.10% after 30 min of treatment. And we can observe a less RBM at catC = 4g/l, because the excess of ferric substance could react with hydroxyl radial therefore degradation rate of BM. 0.2 g/l of Magnetite was chosen as optimum for this EF reaction.

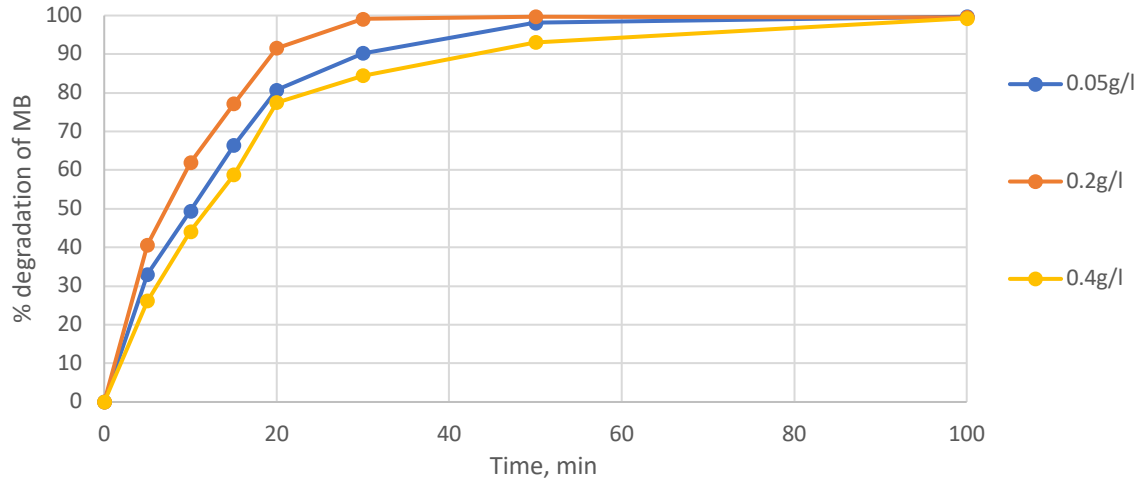

Figure 7. Effect of Fe_3O_4 concentration at pH=3, 10mA/cm², 10 ppm BM, 0,05M Na_2SO_4 and d= 3cm

5. *Effect of inner electrode spacing (IES)*

The effect of inner electrode spacing on the degradation of BM is shown in Figure 8. with same optimal experimental condition maintained as mentioned above. We can observe a Low RBM in IES=4cm because there is decrease in the mass transfer rate of Fe^{3+}. A slightly low RBM was seen when the spacing was reduced from 3 to 2 cm. Given that the cathode is too close to the anode, this could be the electro-regenerated Fe^{2+} being oxidized to Fe^{3+} at the anode. To ensure better EF system we must maintain the in-situ reactive generation in best way possible by making a good amount of H_2O_2 in cathode and ensuring more of Fe^{2+} and that's possible by optimizing the IES, we chose 3 cm as the optimal electrode spacing for this EF reaction.

6. *Effect of Na_2SO_4 concentration (electC)*

The effect of Na_2SO_4 concentration (electC) shown in Figure 9. Was investigated, the result of the study shows that the increase of RBM is observed from 5mM to 50mM, this is mainly due to increase of support transportation of ions, Therefore, the best Na_2SO_4 concentration for MB degradation was found to be 50 mM. The use of a combined Fenton-photocatalytic system in which TiO2 nanotube arrays (TNA) were used as photoanode and Pt-black/Pt as cathode for the degradation of MB conducted at pH 1.5, 0.2 mM $FeSO_4$ as the source of ferrous ions, and 0.10 M

Na_2SO_4 supporting electrolyte, under UV irradiation (2.0 mW cm^{-2} light intensity) targeted at the degradation of 95%, after 60 min of electrolysis (Zhao *et al.*, 2017).

7. inhibition of hydroxyl radical

To make sure the primary reaction for BM's degradation is identified. The formation of hydroxyl radicals in the system was inhibited by the addition of hydroquinone (HyQ) (Do *et al.*, 2017). As shown in Fig. 10, the presence of HyQ significantly inhibits the degradation of MB. In the presence of HyQ 4.10^{-4} M, MB degradation dropped from 99.78% to 26.13% after 30 minutes of electrolysis time. Additionally, BM removal efficiency was reduced by raising the concentrations of HyQ. These findings show that the attack of the OH radical is what primarily decomposes MB.

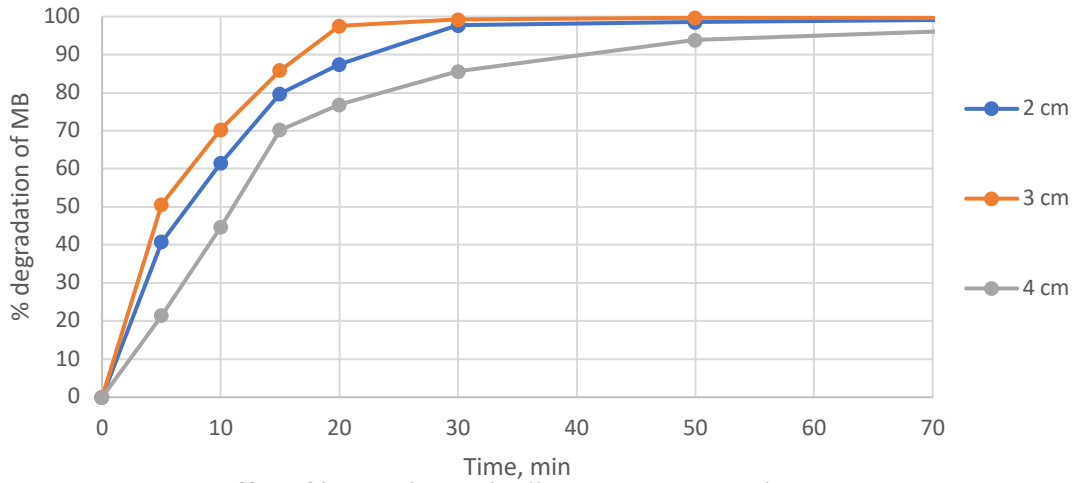

Figure 8. Effet of inner electrode distance at 0.05M de Na_2SO_4 , 10mA/cm², 10 ppm of BM, pH = 3 and 0.2 g/l de Fe_3O_4

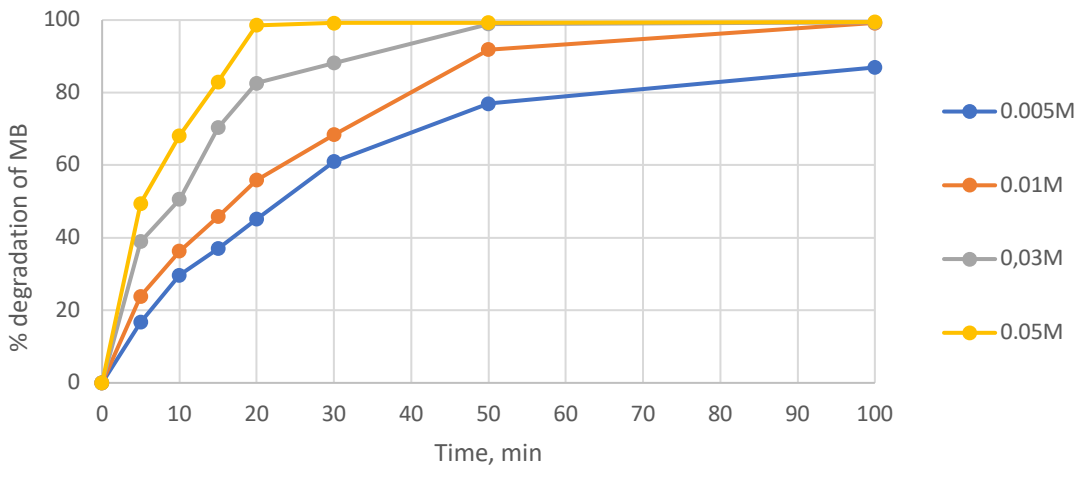

Figure 9. Effet of Na_2SO_4 at 10mA/cm², 10 ppm de BM pH = 3; 0.2 g/l of Fe_3O_4 and d=3cm

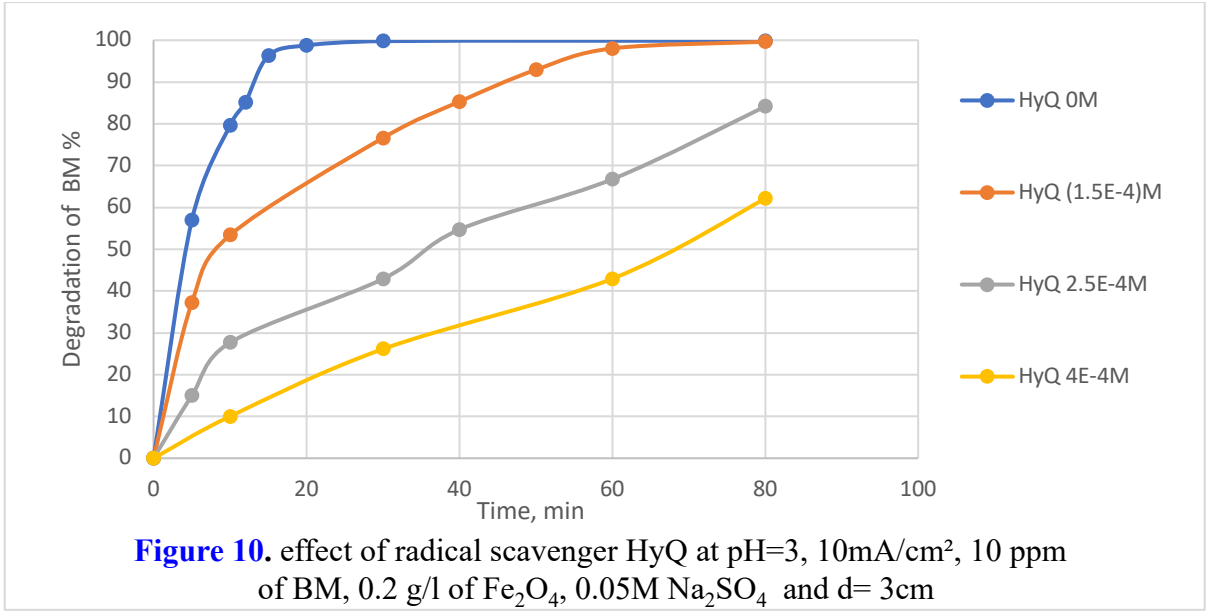

Figure 10. effect of radical scavenger HyQ at pH=3, 10mA/cm², 10 ppm of BM, 0.2 g/l of Fe_2O_4, 0.05M Na_2SO_4 and d= 3cm

8. Reusability of the Magnetite-Graphite Cathode

Reusability tests were conducted under the optimum condition yet identified in order to evaluate the magnetite-graphite cathode's reusability. The degradation efficiency of MB for multiple runs is shown in Figures 11 and 12. It was realized that the removal efficiency could only be maintained for three cycles and there would be a significant decrease in the degradation rate, implying that our Magnetite-Graphite cathode was relatively constant and reusable.

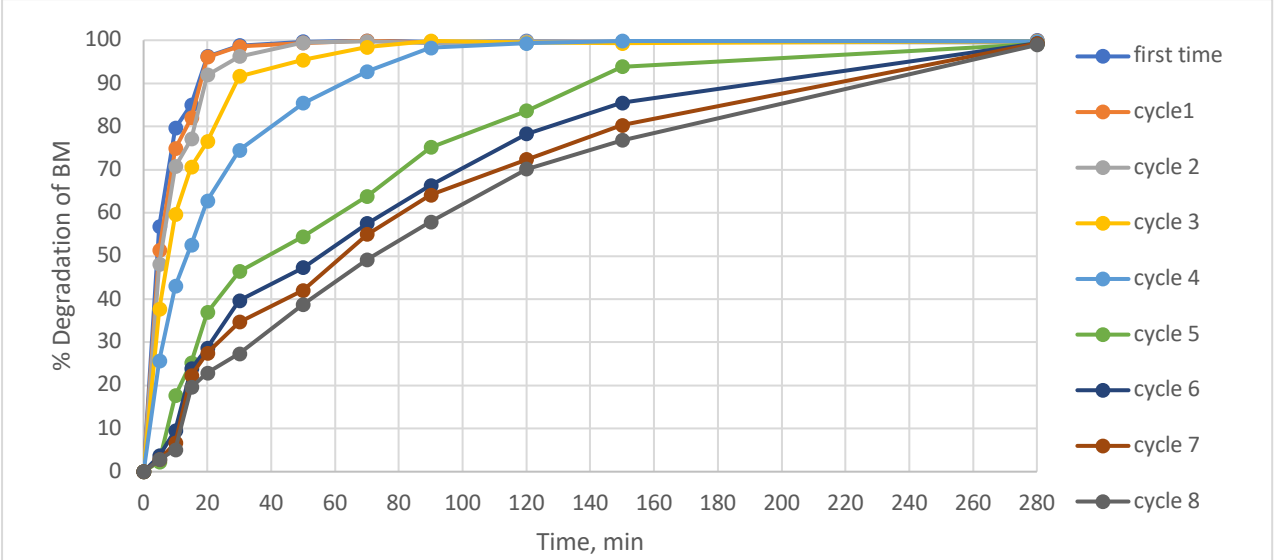

Figure 11, reuse of catalyse at 10mA/cm2, 10ppm BM, 3cm, Fe_3O_4 0.2g/l and 0.05M Na_2SO_4

Graphite anode appeared to be partially oxidized with a portion of the anode surface turning slightly gray after 7 to 8 repeated decomposition studies. In such circumstances, decomposition performance was restored after the anode was changed. We think that this degradation of Graphite

anode is not a serious issue for the actual application of our system because it is neither expensive nor difficult to replace Graphite anode. As previously stated, the Magnetite-Graphite electrode must be treated with 1M HCl solution for 30 min. between each cycle and for 24 hours to be reusable.

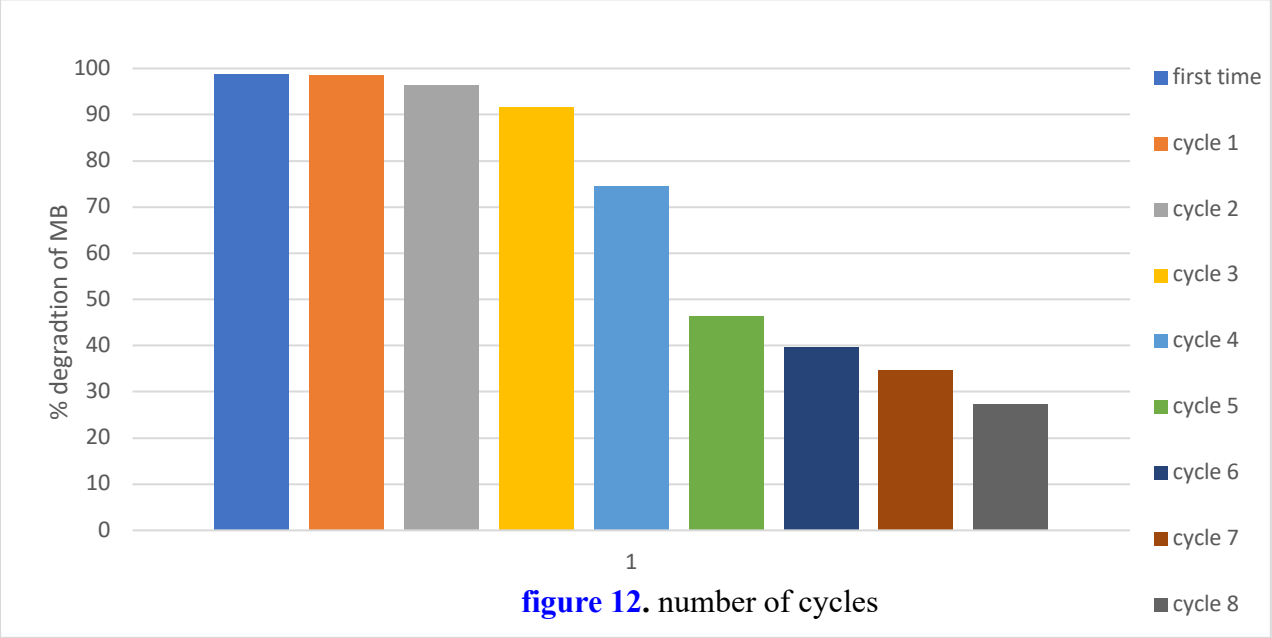

figure 12. number of cycles

9. Kinetic study of methylene blue degradation

With the objective to enhance an approach to comprehend the kinetics of the degradation of methylene blue, we have in our system the facts that the cathode itself is the source of our iron ions and also that H_2O_2 was created at the cathode by the reduction of oxygen in the solution. The degradation of BM should have been more effective at reducing created H_2O_2 since the generated iron ions are localized near the cathode (Espinoza *et al.,* 2016). Three reaction pseudo-orders (Figure 13) kinetics was further examined in the ways listed below. Assuming that the first pseudo-order reaction kinetics by hydroxyl radicals can adequately characterize the electro-Fenton oxidation for MB degradation (Espinoza *et al.,* 2016; Berhe *et al.,* 2022):

$$\ln \frac{C_t}{C_0} = k_{app} * t \qquad (2)$$

And reaction kinetics for second and third pseudo-orders can be described by the following equation with n equals 2 and 3 for second pseudo-order reaction kinetic and third pseudo-order reaction kinetic, respectively:

$$\frac{(-1)^n}{1-n} * \left(\frac{1}{C_t^{1-n}} - \frac{1}{C_0^{1-n}}\right) = k_{app} * t \qquad (3)$$

where Co and Ct, respectively, represent the MB concentrations at beginning and given time t, and $k_{app} = k\, C_{OH^{\cdot}}$.

For n=2 and n=3 we have R^2=0.8465 and R^2 = 0.7534, respectively, as coefficients of determination that shows our system could not be described as second pseudo-order reaction kinetic nor third pseudo-order reaction kinetic. Figure 13 depicts the curve of -ln (Ct/Co) against t for the equation mentioned above. R2 = 0.9551 for the determination coefficients (Figure 13). shows that our system is characterized as pseudo first-order reaction kinetic, with apparent pseudo first-order constant kapp = 0.227.

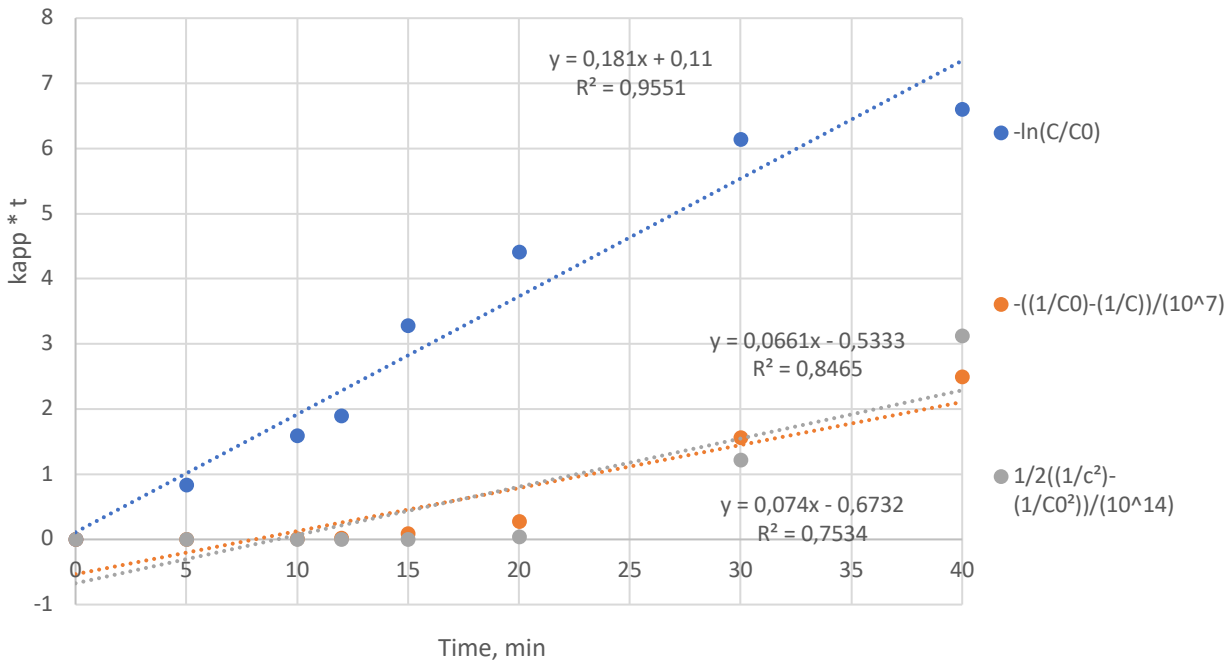

Figure 13 plot of kinetic reaction orders for the degradation of methylene blue

Conclusion

Methylene blue in aqueous solution was effectively discolored using an electro-Fenton system with magnetite fixed on graphite as the cathode and graphite as the anode. The system did not require the addition of H_2O_2 or Fe^{2+} ions. We studied the effects of pH, applied voltage, supporting electrolyte, electrode inner space, and catalyst levels. With the aid of the usage of this cathode, 10 ppm of methylene blue was neutralized with a 99.78% removal rate after 30 minutes and 99.5 % at 50 ppm after 100 minutes of reaction. These cathodes could also be utilized at least three more times without degrading significant performance. The results of the experiment were used to indicate potential reaction pathways. Magnetite fixed on Graphite has a high capacity for degradation, is easy to recover, and has a high capacity for reuse, making it a potential cathode for electro-Fenton systems to remove chemicals from wastewater.

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Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects

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