Magnetic Field Enhanced Electrocoagulation Using Iron Electrode in Removing Glyphosate from Aqueous Solution

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The widespread use and high solubility nature of glyphosate posed a significant threat to water contamination. Glyphosate is a non-selective herbicide for weed control in various agricultural applications. Electrocoagulation method was thus proposed to coagulate this pollutant, and a magnetic treatment was introduced to shorten settling time and to assist sedimentation of suspended solids. The effects of operational variables such as initial glyphosate concentration, electrolysis time and applied voltage towards removal of glyphosate, chemical oxygen demand (COD) and total suspended solids (TSS) were explored to broaden the core understanding of settling velocity and ferromagnetic effects of magnetic fields. The combination of electrocoagulation and the magnetic field was designed and setup into batch laboratory experiment and static mode with two parallel iron (Fe) plates for both anode and cathode. This design used permanent magnets namely NdFeB of 0.55 T and the magnetic exposure time was 6 h. Results showed that the increment in both applied voltage and treatment time and the reduction in initial glyphosate concentration were beneficial for improving glyphosate, COD, and TSS removal efficiencies. An initial glyphosate concentration of 25 mg/l, applied voltage of 30 V and treatment time of 40 min, were obtained as optimum experimental conditions. Respective glyphosate, COD and TSS removal efficiencies of 95.84%, 71.43%, and 79.08% were observed in experiments conducted in optimum conditions. As a conclusion, magnetic field strongly encouraged electrocoagulation process in obtaining better results due to a ferromagnetic mechanism in aqueous solutions.

Key words: Glyphosate aqueous solution; electrocoagulation; permanent magnet; iron electrode; suspended solid; sedimentation

The herbicide has been widely used since it was first marketed and application of herbicide in agriculture has further extended with increment usage for crops. In Malaysia, there are 172 herbicide products containing glyphosate and the glyphosate concentration range for a technical grade of herbicide is usually between 70%–95% [1]. For instance, the amount of glyphosate in a bottle of RoundUp® herbicide is 30.5% w/w or 360 g/l. Glyphosate, N-(phosphonomethyl)-glycine, which is a non-selective herbicide, has a function to control plants, including grasses, broad-leaved weeds, sedges, and woody plants. It's broad-spectrum and low toxicity compared to other herbicides make it quickly to be adopted by farmers [2]. Additionally, glyphosate is a phosphonomethyl derivative of the amino acid glycine. It is a white and odourless crystalline solid comprised one basic amino function and three ionisable acidic sites. Figure 1 below shows chemical structure of glyphosate.

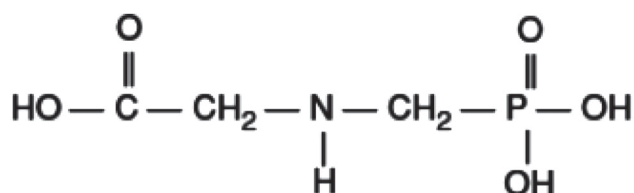Department of Environment reported that in Malaysia, pesticides worth more than RM500

Figure 1. Chemical structure of glyphosate (N-(phosphonomethyl)-glycine).

million were sold since 2007 to improve the quantity and quality of agricultural production. Many methods of treatment have been employed to remove or isolate herbicide from agricultural wastewater. Several treatments have been implemented. The widespread and enormous usage of herbicide can create a potential source of pollution and poses a significant threat to contaminate both surface water and underground water. It is due to discharge of herbicide wastes from a point or diffused sources such as agricultural runoff. Additionally, consumption of water containing glyphosate can result in adverse effects to human health in the short and long-term due to carcinogenicity [3]. Therefore, treatment needs to be introduced to remove glyphosate from the water.

Electrocoagulation process is susceptible to produce flocs of higher size and density which facilitates pollutant removal by sedimentation in an aqueous solution [4]. Nonetheless, settling rate is low due to the slow movement of aggregated colloids in treated solution which results in the longer time needed and low efficiency to completely remove soluble aggregated colloids from the solution. It is because of the settling rate of aggregated colloids which is depend on settling velocity, due to gravitational attraction. The settling velocity is also affected by the density of aggregated colloids. The colloids have different densities which depend on their size. Thus, a new consecutive process which can overcome an existing inherent problem needs to be developed. A magnetic field is proposed to be used along with the electrocoagulation process to reduce settling time and facilitate the removal of aggregated colloids more efficiently by assisting

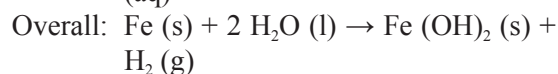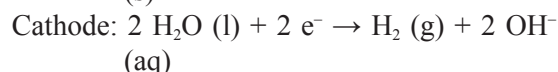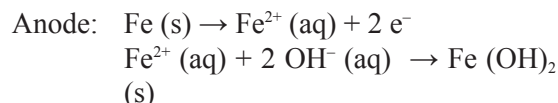sedimentation of suspended solids towards magnet attraction.

In this study, iron plates were used as electrodes to generate ferromagnetic coagulants, and magnetic field of permanent magnet was applied to the process to shorten settling time. Several previous studies reported that iron plates as electrodes has been used in electrocoagulation process to treat wastewater contains orange II azo-dye [5], methylene blue [6], polyvinyl alcohol [7], hardness [8], pesticide [9], natural organic matter (NOM) [10,11] and suspended solid [12]. Electrocoagulation process using iron plates as electrodes also removed heavy metals such as lead and zinc [13], arsenic [14], indium ions [15] and chromium ions (Cr^{6+} and Cr^{3+}) [16].

The aim of this study was to remove and reduce the amount of glyphosate in aqueous solution. Three important electrocoagulation factors were investigated. These are initial glyphosate concentration, applied voltage and treatment time. The objectives of this study are to optimize the operating parameters in order to determine optimum percentage removal of glyphosate, total suspended solids (TSS) and chemical oxygen demand (COD), and to investigate the effect of initial glyphosate concentration, treatment time, and voltage applied towards glyphosate removal, COD removal, and TSS removal. Besides, the aim of this study is also to prove that electrocoagulation process with magnetic field application results in efficient removal than electrocoagulation process without magnetic field application. This aim broadens basic understanding of settling velocity and ferromagnetic mechanism.
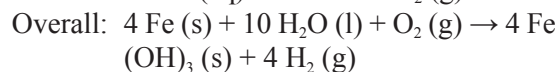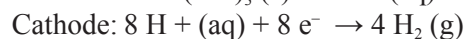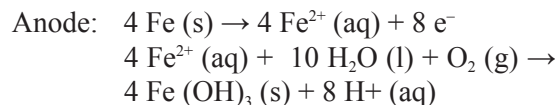
MECHANISM

Oxidation and reduction process mechanisms at both iron plates, anode and cathode are represented by Equation (1) and (2) below:

(a) Mechanism 1:

Anode: $Fe (s) \rightarrow Fe^{2+} (aq) + 2 e^-$
$Fe^{2+} (aq) + 2 OH^- (aq) \rightarrow Fe (OH)_2 (s)$

Cathode: $2 H_2O (l) + 2 e^- \rightarrow H_2 (g) + 2 OH^- (aq)$

Overall: $Fe (s) + 2 H_2O (l) \rightarrow Fe (OH)_2 (s) + H_2 (g)$

(b) Mechanism 2:

Anode: $4 Fe (s) \rightarrow 4 Fe^{2+} (aq) + 8 e^-$
$4 Fe^{2+} (aq) + 10 H_2O (l) + O_2 (g) \rightarrow 4 Fe (OH)_3 (s) + 8 H+ (aq)$

Cathode: $8 H+ (aq) + 8 e^- \rightarrow 4 H_2 (g)$

Overall: $4 Fe (s) + 10 H_2O (l) + O_2 (g) \rightarrow 4 Fe (OH)_3 (s) + 4 H_2 (g)$

Normally, during the electrocoagulation process, three main processes occurres, these are: electrolytic reactions at electrode surfaces, the formation of coagulants in the aqueous phase, adsorption of soluble or colloidal pollutants on coagulants, and removal by sedimentation or floatation [17,18]. The success of electrocoagulation process is determined by the production of the bubbles. Electrocoagulation process is intrinsically associated with electro-flotation since bubbles of hydrogen gas are produced at the cathode electrode. Some coagulated aggregates interact with the bubbles that float to the surface of the aqueous solution and the coagulated aggregates which are denser settle at the bottom due to gravity attraction.

Ferromagnetism is a basic mechanism by which certain materials such as iron form permanent magnet, or are attracted to a magnet. This electrocoagulation process generates iron hydroxide, $Fe(OH)_3$ by anode dissolution to be a coagulant to coagulate glyphosate molecule in the aqueous solution. The $Fe(OH)_3$ is ferromagnetic materials and has functioned as ferromagnetic coagulants in this process. Therefore, flocs of coagulated aggregates which are combinations of $Fe(OH)_3$ and glyphosate molecules has a significant potential to attract to magnet due to the ferromagnetic mechanism. Magnetic technology has the potential to be a promising treatment process that can enhance the separation of suspended particles from the sewage. Colloidal suspended particles carry a surface charge, and this charge is affected by the imposed magnetic fields [19]. Exposure to magnetic field would lead to a higher electrokinetic movement among the colloid. Particles are then attracted and cloaked together. This phenomenon intensifies coagulation that enables them to flocculate and precipitate when becoming heavier. The advantages of the magnetic separation process include high elimination performance, no power input for magnet operation, compact process, small space requirements, and no clogging because of an open structure.

MATERIALS AND METHODOLOGY

Glyphosate Aqueous Solution

Glyphosate aqueous solution was prepared by dissolving a certain amount of glyphosate powder within an electrolyte. The electrolyte chosen was sodium chloride solution. The presence of the electrolyte is necessary to allow movement of free ions in the aqueous solution; thus, enabling the raw sample solution to conduct electricity. The aqueous solution was prepared by dissolving 400 mg of sodium chloride, NaCl powder in 400 ml of distilled water to have an amount of conductivity approximately 4.5 µS/cm. The conductivity was kept constant for every experiment by adjusting the amount of NaCl added to the aqueous solution. Different amount of glyphosate powder was then added to the aqueous solution, depending on required initial glyphosate concentration. For instance, 40 mg of glyphosate powder was added to the solution to get an initial glyphosate concentration of 100 mg/l.

Experimental Setup

A magnetic field has the potential to enhance suspended solids removal by accelerating and

assisting settling sludge at the bottom of beaker when allocated a magnet below the beaker. The magnetic field also has potential to direct and to help all sludge towards magnet location, therefore increase sludge density. The magnet that used in this study was a permanent magnet bar, NdFeB 0.55 T (material type Neodymium-Ferrum-Boron with the strength of 0.55 T). Figures 2 and 3 showed that the power of the permanent magnet was reduced approximately 1/3 when it was tested in between a glass beaker. Thus, it was revealed that only 0.36 T of magnet power dealt in the aqueous solution. The magnet bar was not allocated in the glass beaker to avoid secure attachment between all the settled colloidal aggregates and magnet which might be difficult to remove or to scrap off all the sludge from the magnet.

A laboratory scale made up of electrocoagulation cell, electrodes, and other equipment were set up as illustrated in the schematic diagram, Figure 4 below. The electrocoagulation unit comprised a 500 ml beaker (Pyrex, England) electrochemical cell and two iron plates with a size of 50 mm × 50 mm as electrodes. The separation between the anode electrode and the cathode electrode was kept at a constant distance of 80 mm for all experiments. 400 ml of the glyphosate solution was prepared in the beaker according to the different initial glyphosate concentration required. Meanwhile, electrocoagulation treatment time and voltage supplied were varied. DC power supply (Diginess Concept Series, model: HY 3003-2) was then switched on to start the electrocoagulation process. During the electrolysis process, the positive connection (anode electrode) underwent anodic reactions, while the cathodic reactions were encountered at the negative contact (cathode electrode). Free ions released from the electrodes which neutralized the charges of particles and therefore initiated the coagulation process. The glyphosate aqueous solution was stirred during the process at 100 rpm using magnetic stir rod of 3 cm length on a hot plate magnetic stirrer (Favorit) to give fast process. The treated aqueous solution was then transferred to another beaker, and the beaker was put above a 0.55 T NdFeB permanent magnet bar for 6 h sedimentation. The treated solution before and after 6 h sedimentation above the magnetic bar was analyzed to determine the percentage of glyphosate removal, COD removal, TSS removal. All the processes were handled at room temperature, 25ºC.

Analytical Procedure

Determination of total suspended solids. A glass fiber filter paper was prepared by wetting with 20 ml of distilled water. It was dried in an oven for 24 h at a temperature between 103ºC to 105ºC; the filter paper was then cooled in a desiccator to stabilize the temperature. Its weight was recorded as A mg. The steps were repeated by filtering a volume of 50 ml of uniformly mixed sample of glyphosate solution through a standard GF/F glass fiber filter paper (Whatman GF/C,47 MM diameter, 0.45µm pore size) under a vacuum
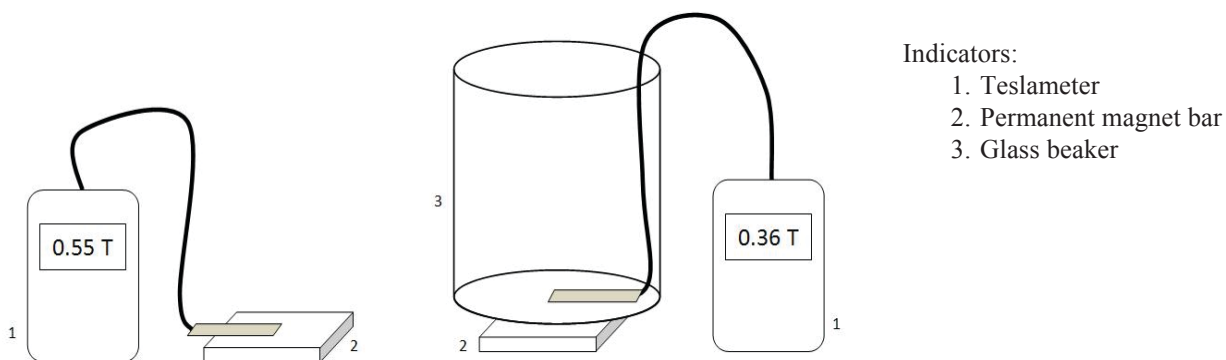

Indicators:
1. Teslameter
2. Permanent magnet bar
3. Glass beaker

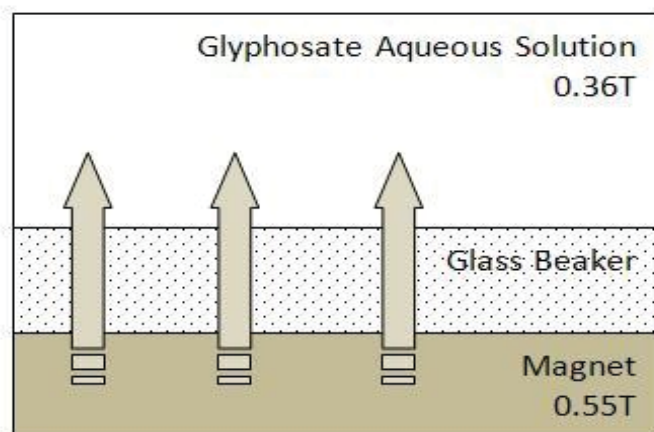**Figure 2**. Detection of magnetic power by teslameter.

Figure 3. An illustration of magnetic power transfer from magnet through glass beaker into solution.

pump (GAST Model DOA-P504-BN 220 V, AMP 8: 1.9 Hz: 50). The filter paper was then transferred carefully to a sheet of aluminium foil on a petri dish. Residual retained on the filter paper, together with the aluminium foil was dried in an oven (Memmert) and were cooled in the same manner as the preparation earlier. It was then weighed by using an analytical balance (Precisa XT220A/ AND GR-200); the data were recorded as B mg. Total suspended solids obtained were calculated by using the following formula:

Total suspended solids (mg/l) = [(B − A) × 1000] / [sample volume (ml)]   (3)
where, B = Weight of filter paper + solids (mg); and

        A = Weight of filter paper (mg)

Determination of chemical oxygen demand. A sample of 2.5 ml was placed in a culture tube. Then, 1.5 ml of potassium dichromate solution was added to the sample, followed by 3.5 ml of sulphuric acid reagent and therefore an acid layer was formed under the layer of sample digestion solution. After that, a cap of culture tube was tightened and inverted several times each to ensure a complete mixing. The culture tube which was filled with the sample was then placed in a block digester, COD block heater (Stuart SBH 200D) which was preheated to 150ºC, and the sample was left to reflux for 2 h behind a protective shield. After the reflux, the tube was

left to cool to room temperature. Then, the culture tube cap was removed, and 0.05 to 0.10 ml (1 to 2 drops) of Ferroin indicator was added to the solution. The sample was stirred rapidly while titrating with standardized 0.10 M FAS. The end point of the titration was reached when there was a sharp colour change being observed, from blue-green to reddish brown (although the blue-green may reappear within a few minutes). In the same manner, a blank containing all reagents and a volume of distilled water equal to that of the sample was refluxed and titrated. The COD calculation was made by using the following formula:

COD (mg/l) = [(A − B) × M × 8000] / [sample volume (ml)]   (4)
where, A = Amount of FAS used for blank (ml);
        B = Amount of FAS used for sample (ml); M = Molarity of FAS; 8000 = Miliequivalent weight of oxygen × 1000 ml/l.

Determination of glyphosate concentration. According to glyphosate molecular structure in Figure 1, glyphosate has a phosphate functional group. The phosphate functional group helps determination of the existence of glyphosate in aqueous solution. Therefore, chemical reaction procedure was employed in order to detect phosphate in aqueous solution. Firstly, 50 ml of sample was prepared in a beaker. One ml of hydrogen peroxide, H_2O_2 was added to the sample

Indicators:
1. Thermometer
2. Iron plates electrodes
3. 500ml beaker (electrochemical cell)
4. Magnetic bar stirrer
5. Hot plate magnetic stirrer
6. Copper wire
7. Digital DC power supply
8. Permanent magnet bar
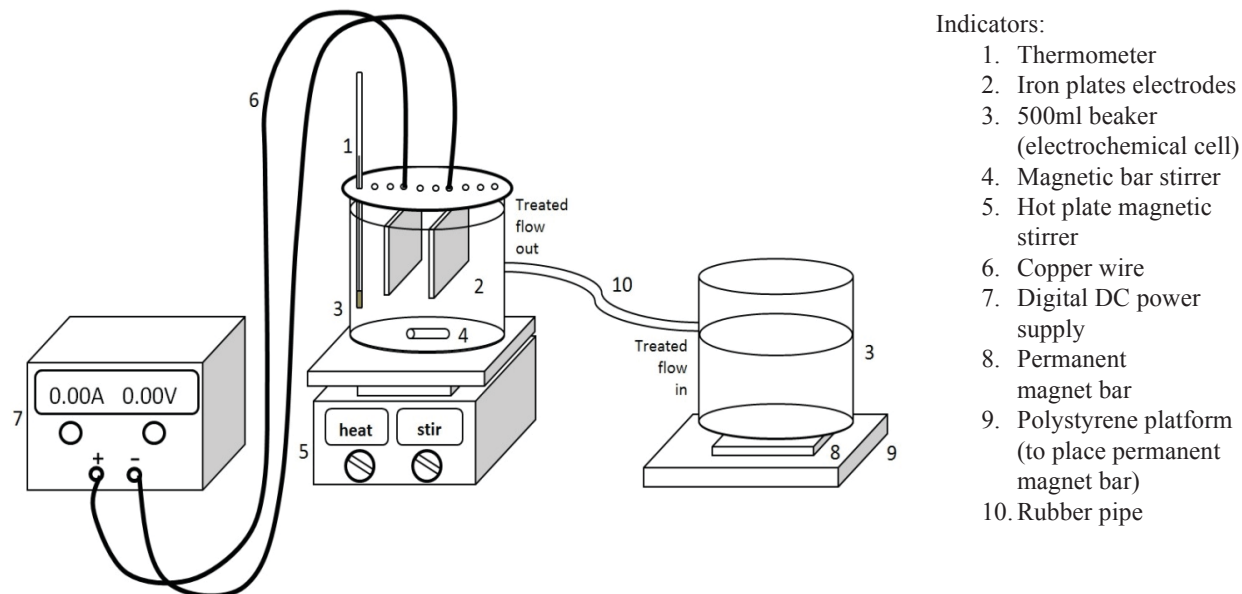9. Polystyrene platform (to place permanent magnet bar)
10. Rubber pipe

Figure 4. Static experimental setup for combined electrocoagulation and magnetic field.

and then heated up by using a hot plate stirrer until all water was vaporized. The sample was then cooled to room temperature. Next, 30 ml of 0.25 M hydrochloric acid, HCl was added to the sample, followed by adding of one drop of phenolphthalein indicator. After that, a volume of 8 ml combined reagent was added to the solution and the sample was shaken to ensure even mixing. The combined reagent was a mixture of 50 ml 5 N H_2SO_4, 5 ml of potassium antimonyl tartrate solution, 15 ml of ammonium molybdate solution and 30 ml of ascorbic acid. The sample was then placed in a cuvette for analysis by using a spectrophotometer (Dynamica, Halo DB-20) to obtain the absorbance readings.

Determination of percentage removal. The percentage removal for glyphosate, TSS and COD after electrocoagulation and sedimentation processes were accessed using formula below:

$$\text{Percentage removal (\%)} = [(C - C_0) / C_0] \times 100 \qquad (5)$$

Where, C_0 = Concentration of sample before treatment (mg/l); C = concentration of sample after treatment (mg/l).

RESULTS AND DISCUSSION

Experimental Observation

Colourless glyphosate aqueous solution treated with iron electrodes appeared green after a few minutes DC power supply was turned on and then transformed into brown and appeared turbid as treatment proceeded. The green colour was due to the presence of Fe^{2+} ions while the brown colour which indicated formation of Fe^{3+} ions during the electrocoagulation process. Fe^{2+} has a relatively high solubility at acidic or neutral conditions, and it could be easily oxidized to Fe^{3+} ions by existing of dissolved oxygen in water. It was also observed that the dissolution of anode took place throughout the process, while gas bubbles were generated at the cathode. The generation of gas bubbles indicated the presence of hydrogen gas. The treated samples were then placed above permanent magnet for 6 h sedimentation to enhance the settling rate of metal hydroxides. It was found that three layers were formed in the beaker. There were a very thin layer of flocs formed near the surface of the solution, clear supernatant at the middle and a thick layer of precipitated sludge at the bottom part as showed in Figure 5.
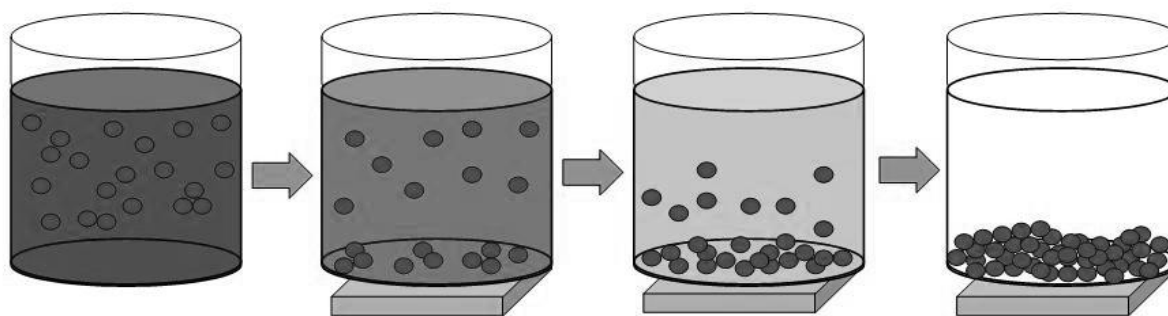
Figure 5. An illustration of colloidal settlement using a permanent magnet bar NdFeB 0.55 T.

Experimental Results

The effect of initial glyphosate concentration, treatment time and applied voltage towards percentage removal of glyphosate, COD and TSS has been investigated. Electrocoagulation process with magnetic field application improved electrocoagulation performance.

Effect of initial glyphosate concentration. All three percentage removal of glyphosate, COD and TSS decreased serially with increased initial glyphosate concentration. Figure 6 showed clearly that the highest percentage removal was achieved at a condition where there was a low initial glyphosate concentration used in the process. Approximately 80%, 76% and 78% were given by glyphosate, COD and TSS removals, respectively which is due to the limitation amount of glyphosate dosage in initial glyphosate concentration. At 50 mg/l of initial glyphosate concentration, percentages of all three removals started to decrease exponentially. The 50 mg/l concentration of initial glyphosate indicated the smallest concentration had the amount of glyphosate that was adequate to be coagulated with the amount of $Fe(OH)_3$ produced. It is because, at a higher concentration, the amount of $Fe(OH)_3$ were not adequate to coagulate with glyphosate to isolate glyphosate from aqueous solution. The amount of $Fe(OH)_3$ as coagulant produced was constant for all samples regarding constant treatment time; thus, samples with lower initial glyphosate concentration resulted in more effective treatment as the availability of iron hydroxide coagulant were greater than the amount of glyphosate to be removed. Therefore, all glyphosate molecules had

the potential to coagulate with the coagulant. In short, a better percentage removal was achieved with less initial glyphosate concentration. Besides, it was noted that a high amount of initial glyphosate concentration in the process resulted in a reduction of percentage removal for all three removals, glyphosate, COD and TSS.

Effect of treatment time. The effects of treatment time of glyphosate, COD and TSS removal were shown in Figure 7. As indicated in the figure, all three removals increased by an increment of treatment time. It was due to the increase of the amount of $Fe(OH)_3$ as coagulant was adequate to coagulate glyphosate molecules. Therefore, flocs of colloidal aggregates increased with the time and the treated aqueous solution became clearer. Besides, increment at treatment time also contributed to a high rate of hydrogen gas bubble generation, which in turn helped to float light flocs. The maximum percentage removal could be found in condition with longest treatment time ranging 30–40 min, with 96%, 80% and 86%, which were given by glyphosate, COD and TSS removal, respectively.

Effect of applied voltage. The effects of applied voltage on glyphosate, COD and TSS removal percent were shown respectively in Figure 8. From the figure, it could be summarized that the highest percentage removals were achieved at condition with greatest applied voltage, with 80%, 70% and 75%, which were given by glyphosate, COD and TSS removal, respectively. Percentage of all three removals increased with an amount of applied voltage ranging from 10–30 V. The voltage ranged
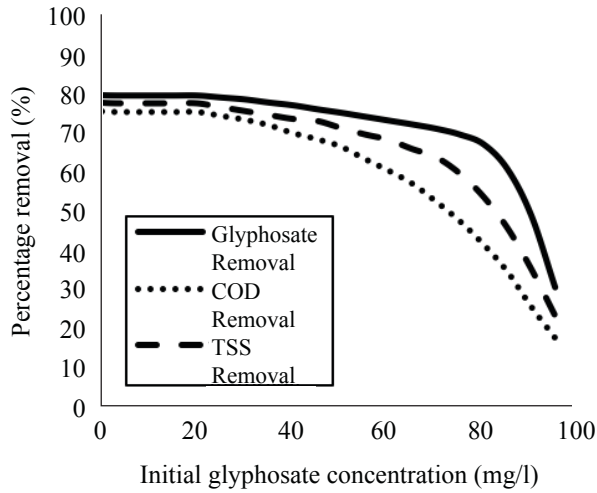
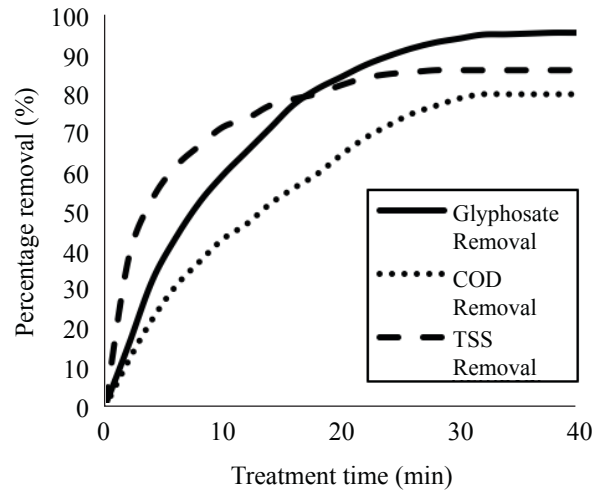**Figure 6**. Effect of the amount of initial glyphosate concentration on percentage removal of glyphosate, COD and TSS.

Figure 7. Effect of the treatment time on percentage removal of glyphosate, COD and TSS.
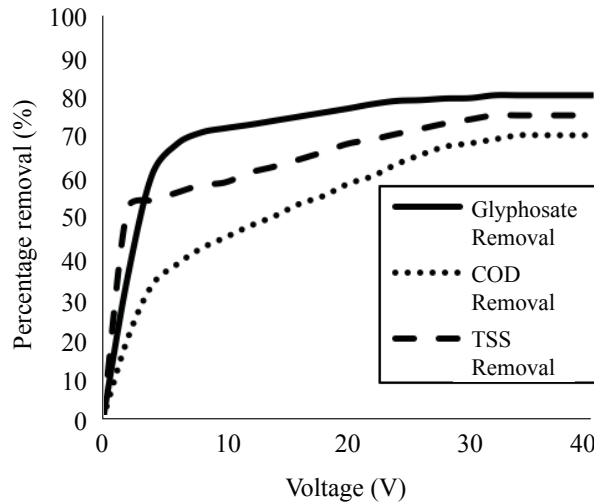
Figure 8. Effect of the amount of voltage on percentage removal of glyphosate, COD and TSS.

from 10–30 V was adequate for the design. This range was flexible and easy to be verified and suitable with the size of iron plates used in this study. When the high amount of voltage applied to the iron plates, the rate of hydrogen gas bubble generation increased, and the size of the bubbles decreased. It results in a greater upwards flux and a high flocs removal by hydrogen gas flotation [20]. There is a major driving force existing during the

electrocoagulation process. According to Faraday's law, the amount of coagulant or dissolved anodic metal is theoretically and directly proportional to the applied current onto an electrolytic cell at a certain time [21]. Thus, increasing applied voltage resulted in a greater extent of anodic dissolution of iron plates and contributed to the increment of $Fe(OH)_3$ coagulants available for the removal of colloidal aggregates. In short, this situation

enhanced applied voltage to increase the amount of current usage simultaneously which directly amplified the rate of electrochemical reactions. Therefore, production of Fe(OH)$_3$ as coagulant increased and it induced a high percentage of removals.

Comparison with and without Using Magnet

According to previous results, electrocoagulation process with magnetic field application improved electrocoagulation performance by indicating high removals of glyphosate, COD and TSS. The experimental outcomes were optimized according to the best high percentage removals of glyphosate, COD and TSS given previous data which from the electrocoagulation process with the magnetic field application. Optimum condition at 25 mg/l initial glyphosate concentration, 40 min treatment time and 30 V applied voltage was then selected to set up experiments to compare the effect of electrocoagulation process performance with or without using a magnet. The results indicated that treated aqueous solution after 6 h sedimentation using magnet were more efficient as it provided better removal performance for all three replacements; the percentage removal

of glyphosate, COD and TSS were by 97.34%, 71.43%, and 79.08%, respectively. Meanwhile, for treated solution without using a magnet, the percentage removal for glyphosate and TSS were 75.5% and 56% which were lower than that removed by using the magnet. The percentage removal of COD was relatively low, in which it only offered a small removal rate of 50%. This was shown in Figures 9–11. In short, the performance of the electrocoagulation process with the magnet had approximately doubled the efficiency of electrocoagulation process without using the magnet.

Observation obviously determined that treated aqueous solution by electrocoagulation process with the magnet after 6 h sedimentation appeared clearer and less turbid rather than treated solution treated by electrocoagulation process without the magnet. This was also proven by a lower turbidity value possessed by the treated sample using magnet sedimentation. Electrocoagulation process without using a magnetic field application had a low sedimentation rate which is the sedimentation which was only affected by the gravity effect which then resulted in longer settling time and low rate of settling velocity. Consequently, the treated aqueous solution was hard to separate thoroughly,
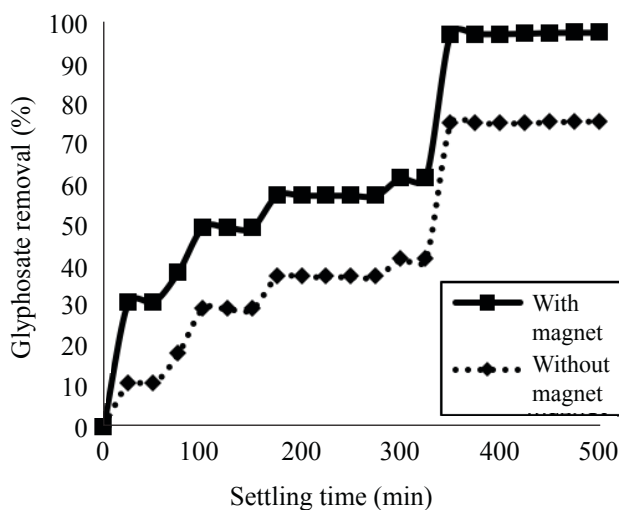

Figure 9. Comparison of percentage of glyphosate removal with and without magnetic treatment.
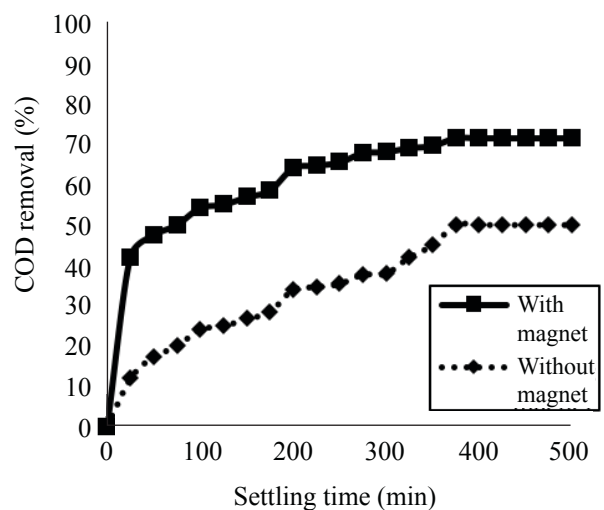
Figure 10. Comparison of percentage of COD removal with and without magnetic treatment.
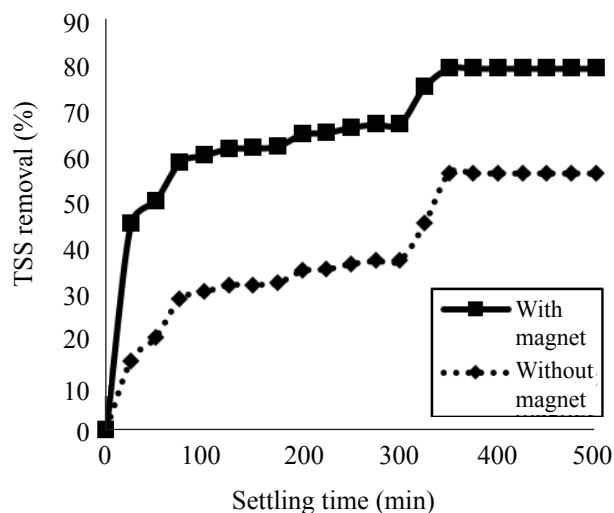
Figure 11. Comparison of percentage of TSS removal with and without magnet treatment.

since much time was needed to gain all suspended solid to settle down. Therefore, low amount of glyphosate molecules were removed from the aqueous solution and lower percentage removal of glyphosate, COD and TSS were indicated. On the other hand, electrocoagulation process by using magnetic field application had a high sedimentation rate which was due to gravity effect and magnet power. It resulted in shorter settling time and high rate of settling velocity. Consequently, the treated aqueous solution was easy to separate thoroughly; all suspended solid was then rapidly settled down. Therefore, a lot of glyphosate molecules removed from the aqueous solution and high percentage removal of glyphosate, COD and TSS were indicated. The magnet bar cannot be placed below a beaker of electrocoagulation during the electrocoagulation process because it may affect production process of $Fe(OH)_3$ coagulant and disturbed formation of flocs of colloidal aggregates. The magnetic field might attract all types of iron or ferromagnetic material, including $Fe(OH)_3$ coagulant to settle down before it coagulated with glyphosate molecules. Thus, it might disturb the coagulation process and results in a reduction of percentage removal of glyphosate.

CONCLUSION AND RECOMMENDATIONS

The influence of operating parameters such as applied voltage, initial glyphosate concentration and electrocoagulation time towards glyphosate, COD and TSS removal efficiencies were studied. The sedimentation of suspended solids was carried out for 6 h which was aided by a 0.55 T NdFeB permanent magnet. Based on the experimental results, it was found that the removal efficiencies obtained in the range of 42% to 96% for glyphosate, 30.77% to 80% for COD and 45% to 86.70% for TSS respectively, depending on experimental conditions of the treatment. Therefore, electrocoagulation process with the magnet was thus chosen as an appropriate method.

This study also revealed that highest removal efficiencies could be achieved at conditions with lower initial glyphosate concentration, longer treatment time, and greater applied voltage. All three removal efficiencies exhibited similar trends for interaction effects between the three operating variables. The increment in both treatment time and applied voltage contributed to increasing removal efficiencies due to the increment in the amount of

metal hydroxide coagulants and the rate of bubble generation during the electrocoagulation process. While for initial glyphosate concentration, the less value contributed to higher removal efficiencies as there was a lesser amount of glyphosate molecule to be removed compared to those with higher initial glyphosate concentration, which under the condition of the limited amount of Fe(OH)$_3$ coagulant available to coagulate with glyphosate molecules. The optimum conditions were obtained at 25 mg/l of initial glyphosate concentration, 40 min of treatment time and 30 V of applied voltage, which resulted in removal efficiencies of 97.34%, 71.43%, and 79.08% for glyphosate, COD and TSS, respectively.

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