# APPLICATION OF COAGULATION AND ELECTRO-COAGULATION METHODS FOR REMOVAL OF PHOSPHATE FROM WASTEWATER

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## Abstract

In this study, two methods for phosphate ions removal from synthetic wastewater were conducted. In the first method, chemical coagulation such as alum, PAC and $FeSO_4$ were used. The three main studied variables were pH, coagulant dosage, and phosphate initial concentrations. In the second method, the electro-coagulation using aluminium plates with the main running factors (pH, phosphate initial concentration, electrolyte concentration, voltage, and electrolysis time) were assessed. The results have shown that the electro-coagulation treatment gave the best results (removal efficiencies of 78%, 91%, 88% and 95% and 98%) for pH, phosphate concentration, NaCl concentration, voltage and run duration factors, while the coagulation method results in efficiencies of 75%, 86% and 89% for alum. However, the two other coagulants gave lower results.

Keywords: Chemical coagulation, Electro-coagulation, Phosphate, Wastewater.

## 1. Introduction

The phosphate ions that currently already in use in numerous businesses such as mining, agriculture, fertilizing, laundries, and household washings, may cause contamination of water recourses [1]. A special concern is given to the wet processing of these industries, which consumes large volumes of water and chemicals. Wastewater from such industries is considered as a source of severe environmental contamination. Actually, the balanced phosphate ions in water are accepted for the ecological system, but when the concentration becomes higher than the requirements, the concentration needs to be reduced to acceptable levels that are useful to the ecological system [2]. The national and international wastewater standards required that the maximum concentration for phosphate ion should be between 0.1 to 0.2 mg/l [3].

Multiple techniques are used to remove phosphate from wastewater. For example, by conventional physical-chemical technique the reagent ions are added to react with soluble salts of phosphorus in order to make a colloid phosphate sediment. This sediment was effective with the aerobic and or anaerobic treatment simultaneously, but it removed only 20-40% of phosphorous [4]. Another technique is called the electrochemical methods which, includes electro-coagulation [5], and bio-electro-chemical [6].

Chemical precipitation method by the use of coagulants is typically effective in phosphorus removal like poly aluminium chloride which is the preferred coagulant among the aluminium salts [7]. The factors affecting the phosphate removal efficiency such as the mixing ratio for aluminium salts have been also studied [8]. Poly Aluminium chloride is effective in precipitate phosphate in a lake depending on puffer capacity [9]. Alum is successfully used to remove phosphate from low alkalinity wastewater [10]. Normal pH, alum and ferrous sulphate are coagulants that are available to remove TDS, turbidity, COD and BOD from dairy wastewater treatment process [11]. Phosphorus and nitrogen could be removed by coagulation through the use of ferrous sulphate [12]. The principle of coagulation process is destabilized electric charge of colloid particles by adding coagulant leading to formation of aggregates bigger than the initial colloids and result in sedimentation [13].

The other method to remove phosphate from wastewater is by using the electro-chemical technique, many research were used this method to remove phosphate by using a cribriform baffle to mix the solution instead of magnetic stirrers [14]. Phosphate ratio could be reduced by using two-electrode, air cathode and graphite plate with aluminium mesh. This method was done by connecting the power supply from anode to cathode, then by connecting aluminium mesh with air cathode [15]. The proposed mechanism of chemical reactions occurring in the electro-coagulation process is shown by the following main reactions [16]. From the reactions, $Al(OH)_3$ is formed as an effective coagulant.

$$Al \rightarrow Al^{3+} + 3e^- \qquad \text{Anode} \qquad (1)$$

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \qquad \text{Cathode} \qquad (2)$$
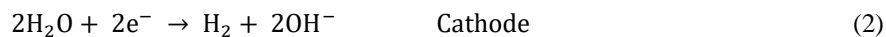
The main objective of this study is to investigate the efficiency of removing phosphate from wastewater by methods of coagulation and electro-coagulation, this will be as a comparison study between these two methods using different coagulants and different running factors.

## 2. Materials and Methods

### 2.1. Preparation of phosphate solution

Synthetic wastewater polluted with 1000 ppm concentration of phosphate was prepared by dissolving a certain amount of potassium dihydrogen phosphate $KH_2PO_4$ (with a molecular weight of 136.086 g/mol) in distilled water, thereafter, the stock solution was mixed with distilled water to the desired concentrations.

### 2.2. Chemicals

Poly aluminium chloride or PAC, aluminium sulphate or alum $(Al_2(SO_4)_3.14H_2O)$ and ferrous sulphate $(FeSO_4)$ was used in different dosages as coagulants. The stock was prepared previously with a concentration of 1000 mg/L, also, electrolyte solution was prepared using NaCl salt. For acidic and alkaline conditions, hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used respectively. The chemicals were supplied from local venders at the Baghdad City, Iraq.

### 2.3. Jar Tests

The coagulation tests were conducted to find the optimum pH solution which was adjusted by adding acid 0.1N HCl or alkali 0.1N NaOH, in the jar test (model: Lovibond, Germany) in which six 1000 ml size bakers and paddle stirrer were used to mix the phosphate solution (which was prepared previously) with the coagulants at a desired concentration. First, the rapid mix was used with 200 rpm for 2 min, there after; slow mix with 50 rpm was used for a period of 30 min. The settling time for each test was 30 min, so the period of each of chemical coagulations experiments was 62 min, after settling, the phosphate samples were taken from a depth approximately 2 cm below the solution surface and the samples were analysed by using UV-spectrophotometer (Model SP-3000 plus, Optima Co., 2003, Japan). All tests were done at room temperature.

### 2.4. Electro-coagulation cell

The experiments were conducted in a batch reactor with an effective volume of 2L. For each test, 1.5L of phosphate solution was used and NaCl was added as an electrolyte. Cathode and anode were aluminium plates with dimensions of 7 x 4 x 0.3 cm positioned vertically with 3.5 cm distance in between the two plates. For the electric source, direct current (DC) power supply was used. For each test, the solution was mixed at a speed of 200 rpm by using stirrer Model LMS-1003, Korea. Electrode's ware washed in distilled water after each test in addition they cleaned well in some kind of metal sponge and dilute acid $(H_2SO_4)$. All experiments were performed at room temperature. Figure 1 shows the diagram of the setup.

### 2.5. Analytical methods

For each test, the samples of treated phosphate solutions were analysed using yellow vanodomolybdo phosphoric acid method [17], with a spectrophotometer, maximum wavelength of 490 nm. The phosphate removal efficiencies were calculated by:

$$R\% = \left(\frac{c_r - c_t}{c_r}\right) x \, 100^{(1)} \tag{3}$$

where $R\%$ is the percentage removal efficiency, $C_r$ is the phosphate concentration before the treatment and $C_t$ is the phosphate concentration after the treatment (mg/L). The values and ranges of the operational variables are listed in a Table 1.
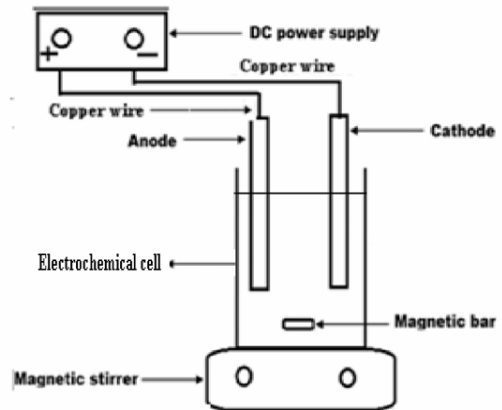


**Fig. 1. Experimental setup.**

**Table 1. Variables of operating conditions
and ranges for chemical coagulation and electro-coagulation processes.**

| | **Variables** | **Ranges** |
|---|---|---|
| **Chemical Coagulation** | Dosage of coagulants = 200 mg/L<br>Phosphate concentration = 30 mg/L<br>Temperature = 30°C | pH = 2 – 12 |
| | pH of alum = 8, pH of PAC = 10<br>pH of FeSO$_4$ = 4,<br>phosphate concentration = 30 mg/l<br>Temperature = 30°C | Coagulant dosage =<br>50 – 500 mg/L |
| | Dosage of coagulants = 400 mg/L<br>pH of alum = 8, pH of PAC = 10<br>pH of FeSO$_4$ = 4, Temperature = 30°C | Initial concentration of phosphate =<br>5 – 80 mg/L |
| **Electro-coagulation** | Concentration of phosphate = 30 mg/L<br>Concentration of NaCl = 1000 g/L<br>Voltage = 5 V, Time = 60 minutes<br>Temperature = 30°C | pH = 2 – 12 |
| | Concentration of phosphate = 30 mg/L<br>pH = 6, Voltage = 5V, Time = 60 minutes<br>Temperature = 30°C | Concentration of NaCl = 150 – 5000 mg/L |
| | Concentration of phosphate = 30 mg/L<br>pH = 6, Voltage = 5V<br>Time = 60 minutes, Temperature = 30°C | Initial concentration of phosphate =<br>5 – 80 mg/L |
| | Concentration of phosphate = 5 mg/L<br>Concentration of NaCl = 5000 g/L<br>pH = 6, Time = 60 minutes<br>Temperature = 30°C | Voltage = 1 – 10V |
| | Concentration of phosphate = 5 mg/L<br>Concentration of NaCl = 5000 g/L<br>pH = 6, Voltage = 10V<br>Temperature = 30°C | Time = 10 – 150 minutes |

## 3. Results and Discussions

### 3.1. Chemical coagulation results

#### 3.1.1. The pH results

The pH of the aqueous solution plays as an important role in the coagulation process. The optimum conditions could be achieved by controlling the pH. The amount of hydroxide and hydrolysis precipitate depends on the increase or decrease in pH [18]. The effect of pH on phosphate removal has been studied. The dosage of 200 mg/L of each coagulant was considered, while the varying values of pH samples were taken from 2 to 12 by using 0.1N HCl and 0.1N NaOH. The phosphate concentration was fixed at 30 mg/L [19], during the experiments. Figure 2 shows that the maximum removal of phosphate, were 75%, 68%, and 20% at pH values of 8, 10, and 4 for alum, PAC and $FeSO_4$, respectively. It is clear that when alum was used, the percentage of phosphate removal was increased from 2% to 75% while the pH was increased from 2 to 8, at the same time, it decreased when pH exceeds 8. When PAC was added to the untreated water, the removal efficiency was increased from 5% to 68% while the pH was increased from 2 to 10 and afterward it decreases. When $FeSO_4$ was used, the removal efficiency increased from 0 to 20% while the pH values increased from 2 to 4 and then decreased as shown in Fig. 2. These results are clear because the coagulant is charged with positive sign at a specific range of pH (4 to 10). The coagulant is attracted to the negative sign ions (phosphate ions), however, above and below this range, the coagulant charge was negative, so, it would not be attracted to the negative sign ions. The theory for that, there are several changes of coagulants composition which occurred and lead to form other compounds of coagulants having different charges [7, 8], studied the removal of phosphate from river water using poly aluminium chloride and the experiments in the present study has obtained similar results.
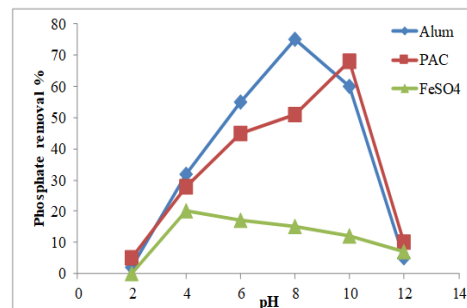


**Fig. 2. Phosphate removal as a function of pH.**

#### 3.1.2. Coagulant dosage results

The dosage effect was studied and after many tests, the pH optimum values of 8, 10, and 4 were obtained for alum, PAC and $FeSO_4$, respectively for a phosphate concentration of 30 mg/L. The dosages of the coagulants were selected to be as 50, 100, 200, 300, 400 and 500 mg/L. The best result was obtained with a dosage of 400 mg/L, thereafter; no more increase in removal was detected. As the coagulant dosage increases, the number of binding sites between the phosphate ions also increases [20]. After a concentration of 400 mg/L, the colloidal suspension would be stabilized, and coagulant active area would be prohibited [10]. The maximum

removal efficiencies were 86%, 83% and 33% for alum, PAC and FeSO₄, respectively. Figure 3 shows the dosages of the coagulant.
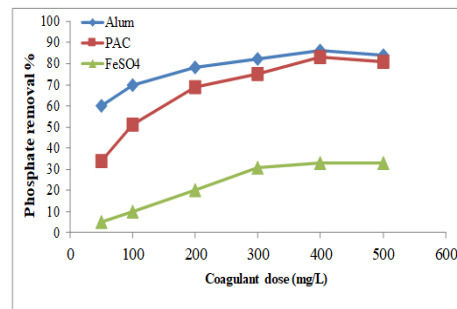


**Fig. 3. Phosphate removal as a function of coagulants dosage.**

### 3.1.3. Initial concentration of phosphate results

The removal efficiency of phosphate was studied with the consideration of the initial concentration of phosphate. The initial concentrations were 5, 15, 30, 40, 50 and 80 mg/L with pH values of 8, 10 and 4 for alum, PAC and FeSO₄, respectively (the coagulants concentration was 400 mg/L). The curve in Fig. 4 represents the relation between the change in initial concentration and the removal efficiency. For all coagulants, the best removal efficiency was noted when the phosphate concentration was 50 mg/L, hence, the removal efficiencies were 89% for alum, 82% for PAC and 41% for FeSO₄. As a result, the maximum efficiency was found with the phosphate concentration of 50 mg/L. However, phosphate concentration is more than 50 mg/L, the efficiency was lowered. For phosphate concentration of 80 mg/L, the efficiencies were 81%, 77% and 33% for alum, PAC and FeSO₄, respectively, higher removal efficiencies were obtained with the three types of coagulants. However, when the phosphate concentration was more than 50 mg/L, the curve went down, but the differences were not significant (because the ration of phosphate particles to the coagulant particles increased). This can be attributed to fact that the reaction was active between the coagulant and phosphate salt, as a result, phosphate metal salts would be released in the solution as colloidal particles, and would precipitate, then would be removed. For this reaction, many types of metal salts are used as coagulants, and sufficient amount of coagulants (metal salt ions) should be added to sediment and hold phosphate ions. If the amount of phosphate salt is not proper, the removal efficiency decreases [4].
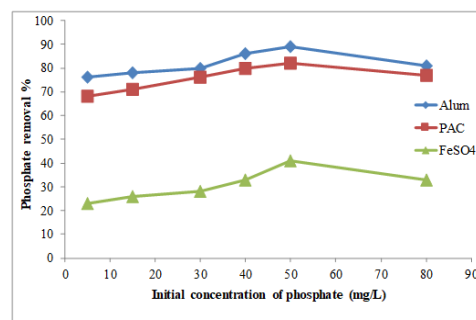


**Fig. 4. Phosphate removal as a function of the initial concentration of phosphate.**

## 3.2. Electro-coagulation results

### 3.2.1. The pH results

The best result of pH for all tests was 6. It is clear in Fig. 5 that pH values that have been used are 2, 4, 6, 8, 10 and 12. The maximum removal efficiency was found to be 78% in acidity solution (pH = 6). This could lead the oxide surfaces to exhibit a net positive charge at acidic pH and columbic attraction to enhance adsorption of $PO_4^{-3}$. The oxide surfaces had a net negative charge and would tend to repulse the $PO_4^{-3}$ in solution at higher pH values [21-23] have studied the removal of phosphate by electro-coagulation using aluminium plates, and the results were found in line with the results of the present study.
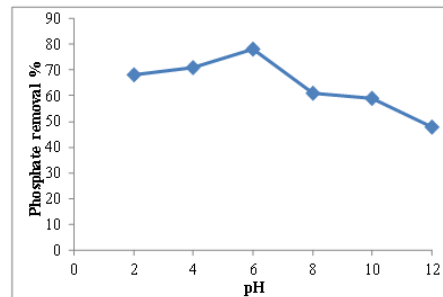


**Fig. 5. Phosphate removal as a function of pH.**

### 3.2.2. NaCl concentration results

Variable concentrations of NaCl (150, 500, 1000, 3000, 5000 mg/L) were prepared to study the solution conductivity. The experiments were done with a fixed pH value of 6, phosphate concentration of 30 mg/L [24] voltage of 5V and electrolysis time of 60 min. Figure 6 displays the results of NaCl concentrations versus removal efficiency. The curve shows a direct relationship between phosphate removal and the NaCl concentration. The increasing in NaCl concentration caused an increase in the removal efficiency, thus, the optimum removal was 88% at NaCl 5000 mg/L. For each run, NaCl was added more than the previous run, so, the concentration was increased, and the conductivity was increased too to allow for more current, another reason increasing water conductivity reducing ohmic drop [25], hence, more Cl⁻ ions were produced to limit any oxide formation on the anode. This action has enhanced Al(OH)₃ to remove the phosphate from wastewater [5]. Almost, similar results were reported when the phosphate compounds were treated using aluminium rings in the electro-chemical cell [26].
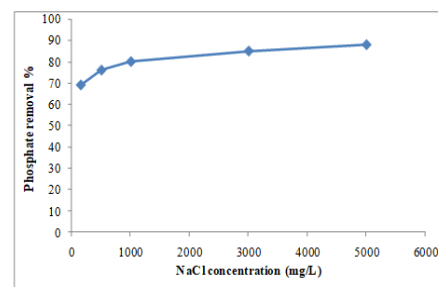


**Fig. 6. Phosphate removal as a function of NaCl concentration.**

### 3.2.3. Initial phosphate concentration results

Phosphate solution with variable concentrations (5, 15, 30, 40, 50, 80 mg/L) were prepared to examine the effect of initial phosphate concentration on the treatment efficiency. Throughout the examination, a fixed values were used, and these values were pH = 6, NaCl concentration = 5000 mg/L, voltage = 5V, and electrolysis time = 60 min. As a result, the maximum removal efficiency was 91% at a phosphate lowest concentration of 5 mg/L as illustrated in Fig. 7, hence, the curve went down and the relation between the initial concentration and removal efficiency was of inverse nature. This can be attributed to the limited amount of $Al(OH)_3$ ions that were produced at anode. On the other hand, the concentration of phosphate was increased while there were no sufficient $Al(OH)_3$ to treat phosphate ions. The results of the phosphate initial concentration influence on the treatment were very close to the results of [20, 21].
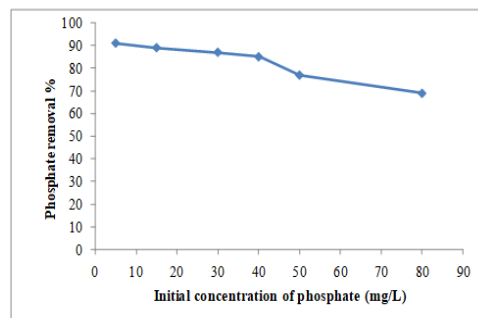


**Fig. 7. Phosphate removal as a function of Initial phosphate concentration.**

### 3.2.4. Voltage results

The voltage effect was assessed by taking a variable amount of electric cell voltages (1, 3, 5, 10 Volt) at pH 6, phosphate concentration 5 mg/L, NaCl concentration 5000 mg/L and electrolysis time 60 min. It is found that the percentage of removal was 78%, 85%, 92% and 95%, respectively (Fig. 8). All the previous results to acceptable, however, the maximum voltage value was the best (95%). When the voltage increases, $Al^{+3}$ dissolution increases from anode which undergo hydrolysis to produce $Al(OH)_3$, therefore, phosphate removal increases. [25], studied the effect of voltage variation to treat wastewater from organic components by electro-coagulation, the study found that when the voltage increases the removal efficiency increases too.
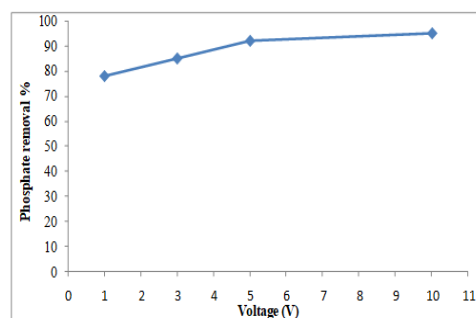


**Fig. 8. Phosphate removal as a function of voltage.**

### 3.2.5. Electrolysis time results

To assess the time effect, for each test, samples were taken at different intervals (10, 30, 60, 100, 150 min), to measure the treatment efficiency. As it is shown in Fig. 9, the best removal was achieved at 100 min. Beyond this duration, no significant changes were noticed. After the electrolysis time, the production of flocs becomes higher, thereafter, the phosphate removal increases, after a certain time, flocs production decreases and the number of flocs were not able to remove the same amount of phosphate. For the electro-coagulation method, many researchers studied time effect and the treatment was successive during the first period, but after the electrolysis time, there was no progress in the process [5, 26, 27].
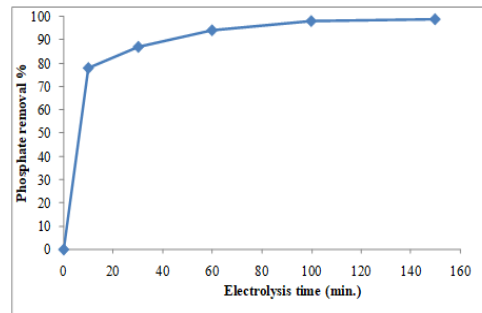


**Fig. 9. Phosphate removal as a function of electrolysis time.**

### 3.3. Comparison study

Table 2 reveals a comparison between present study and previous studies for removal of phosphate by using chemical coagulation and electro-coagulation techniques. Different parameters were present in this study. However, the present work has excellent results in comparison with other studies.

**Table 2. Reveals this study results in compare with pervious results**

| Parameter | Material used | Phosphate removal, % | Reference | Present study |
|---|---|---|---|---|
| **Chemical coagulation** | | | | |
| **Optimum pH** | Aluminium chloride | 74.9 % | [7] | - |
| | Aluminium sulphate | 62 % | | 75% |
| | PAC | 80 % | | 68% |
| **Optimum coagulant dosage** | Aluminium chloride | 74.9 % | [7] | - |
| | Aluminium sulphate | 62 % | | 86% |
| | PAC | 80 % | | 83% |
| **Optimum initial concentration** | Aluminium sulphate | 85 % | [19] | 89% |
| | Iron sulphate | 80 % | | - |
| **Electro-coagulation** | | | | |
| **Optimum pH** | Aluminium electrode | 89 % | [5] | 78% |
| **Optimum NaCl concentration** | Aluminium electrode | 100 % | [5] | 88% |
| **Optimum initial concentration** | Aluminium electrode | 90 % | [5] | 91% |
| **Optimum voltage** | Aluminium electrode | 100 % | [28] | 95% |
| | Iron electrode | 78 % | | - |
| **Optimum electrolysis time** | Aluminium electrode | 98 % | [26] | 98% |
| | Iron electrode | 93 % | | - |

## 4. Conclusions

The phosphate removal by electro-coagulation method gives better results than chemical coagulation method. The experimental results show that the phosphate removal efficiency by the coagulant is dependent on pH, initial concentration of phosphate and both coagulant dosage and type. Best removal efficiencies were achieved by using alum and as follows 75% at pH = 8, 86% at 400 mg/L dosage and 89% at 80 mg/L concentration of phosphate. Electro-coagulation is dependent on pH, initial concentration of phosphate, electrolyte concentration, voltage amount and electrolysis time. The removal efficiencies by aluminium plate were as follows 78% at pH = 6, 91% at 5 mg/L concentration of phosphate, 88% at 5000 mg/L NaCl concentration, 95% at voltage = 10 V and 98% at electrolysis time = 100 min.

## Future research

Studies to be carried out about the removal of phosphate from wastewater by coagulation method using another chemical coagulant and/or natural coagulants. Research may be conducted to study energy consumption in the study of phosphate removal by electro-coagulation method.

## Acknowledgment

| Nomenclatures | |
| --- | --- |
| $C_o$ | Phosphate concentration before the treatment, mg/L |
| $C_t$ | Phosphate concentration after the treatment, mg/L |
| $R$ | Percentage removal efficiency of phosphate |
| **Abbreviations** | |
| HCl | Hydrochloric acid |
| NaOH | Sodium hydroxide |
| PAC | Poly aluminium chloride |

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