

Adsorption of Fenamiphos Pesticide from Aqueous Solutions by Electrocoagulation Using Sacrificial Anodes

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Abstract

The present study deals with removal efficiency of the pesticide fenamiphos and chemical oxygen demand (COD) from aqueous solution by electrocoagulation method. The effects of initial pH, current density, initial pesticide concentration, salt concentration, usage of different electrolytes and temperature on the removal efficiency and COD have been investigated. The obtained results showed that fenamiphos and COD removal were 97.4% and 96.4% by using C with SS (Carbon as the anode and stainless steel as the cathode) at 60 min and were 90.3% and 88.2% by using SS with Ti (stainless steel as the anode and titanium as the cathode) at the same time. These electrodes provided a higher oxidation rate, higher current efficiency, high over potential and consume less electrical energy. The experimental data were fitted with several adsorption isotherm models to describe the electrocoagulation process. This study has been carried out to determine the feasibility of fenamiphos adsorption on carbon and stainless steel species by EC process using the Langmuir and Freundlich Isotherm. In addition, results for the fenamiphos removal kinetics at various effects show that the kinetic rates conformed to the pseudo first order kinetic model with good correlation using both electrodes.

Keywords: Electrocoagulation; Adsorption; Stainless steel; Titanium; Carbon; Fenamiphos; Pesticide; Water treatment

Introduction

Organophosphorus pesticides (OPPs) are one group of chemical pesticides now widely used in agriculture, and account for more than 36% of the total world market of chemical pesticides [1]. Fenamiphos an organophosphorus pesticide is primarily used to control nematodes in a wide range of horticultural crops and in turf [2]. This pesticide is applied at various stages of plant growth such as pre-planting, at-planting, pre and post-harvest on a variety of plants including tobacco, turf, bananas, pineapples, citrus and other fruit vines, vegetables, and grains [3]. Fenamiphos is highly toxic via the oral route, with reported LD50 values of 2 to 19 mg/kg in the rat and 56 to 100 mg/kg in guinea pigs. The inhalation toxicity of the compound is also high, with reported inhalation LC50 values in rats of 0.11 to 0.17 mg/L [4,5]. Various treatment procedures have been reported for the removal of fenamiphos from water e.g. Photocatalytic [6,7], photolysis and photodegradation [8] and biodegradation [9]. In the recent years, there is an increasing interest in the development of environmentally friendly electrochemical methods to treat toxic organic pollutants in water [10]. The electrocoagulation is a simple and efficient method for the treatment of many water and wastewaters. In recent years, many investigations have been especially focused on the use of electrocoagulation owing to the increase in environmental restrictions on effluent wastewater [11,12]. EC has been successfully tested to treat various industrial water and wastewater such as potable water [13,14], food and protein wastewater [15], yeast wastewater [16], urban wastewater [17], restaurant wastewater [18,19], tar sand and oil shale wastewater [20], nitrate containing wastewater solutions [21] and arsenic containing smelter wastewater [22]. In recent studies, many pesticides such as Malathion [23], methyl parathion, atrazine and triazophos [24], malathion, imidacloprid and chlorpyrifos [25], and abamectin [26] have been treated successfully by the electrochemical methods.

The purpose of the present work is to study the effectiveness of electrocoagulation process on removing investigated fenamiphos pesticide and chemical oxygen demand (COD) in aqueous solution using different electrodes and investigate the effects of various operating parameters on the removal efficiency such as initial pesticides concentration, initial pH, current density, electrolyte concentration,

type of electrolyte, temperature and kinetic studies on the removal efficiency.

Experiment

Chemicals

The pesticide used in the present work was fenamiphos, Pesticide Solutions. Fenamiphos solutions were prepared from the commercially available pesticide, in distilled water. Fenamiphos also known as (Nemacur), at a concentration of 400 g L⁻¹. This concentration is the same as that used by farmers during strawberry cultivation. The properties of the fenamiphos are given in Table 1.

Sodium chloride, sodium sulfate, sodium carbonate, potassium chloride, sodium fluoride, sodium hydroxide, sulfuric acid, potassium dichromate, was of analytical grade and purchased from Merck. Distilled water was used for the preparation of solutions. Standard solutions of potassium dichromate (K₂Cr₂O₇), sulfuric acid (H₂SO₄) reagent with silver sulfate (Ag₂SO₄), Mercury sulfate (HgSO₄) and were prepared to measure the COD. A stock solution of pesticide (1000 mg/L) was prepared by dissolving an accurate quantity of the pesticide in distilled water and suitably diluted to the required initial concentrations. Different standard solutions of pesticide with concentration from 30-150 mg L⁻¹ were prepared to measure its removal at different conditions. The pH of the working solution was adjusted to the desired values with 0.1N HCl or 0.1N NaOH.

Equipments and procedures

The electrocoagulation unit consisted of a 100 ml electrochemical reactor with carbon with stainless steel and stainless steel with titanium

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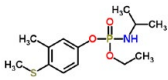
Pesticide	Fenamiphos
IUPAC Name	((<i>RS</i>)- <i>N</i> -[Etoxy-(3-methyl 4methylsulfanylphenoxy)phosphoryl]propan-2-amine
Chemical structure	
Solubility in water	400 mg/L
Chemical formula	C ₁₃ H ₂₂ NO ₃ PS
Molecular weight (g/mol)	303.40 g/mole
λ_{max}	249 nm

Table 1: Properties of Fenamiphos.

electrodes (anode and cathode) with an effective surface area of 5.4 cm². The electrodes were 17 mm×10 mm and inter electrodes distance was 1 cm. The electrodes were positioned vertically and parallel to each other. The current density was maintained constant by means of a precision DC power supply, model (DZ040019) EZ Digital CO. Ltd. (Korea). The pesticide concentration was determined using a double - beam UV-Vis spectrophotometer, model UV 1601 is from Shimadzu (Japan) at 249 nm. Hot Plate, model (HB502), BIBBY STERILIN LTD. (U.K.). A pH meter model AC28, TOA electronics Ltd., (Japan). Water bath model SB-650, Tokyo Kikakai CO. Ltd., (Japan). A closed reflux titrimetric unit was used for the COD determination. Chemical Oxygen Demand (COD), HANNA instruments, Thermo reactor, model C9800 Reactor in Hungary – Europe.

Analysis

Two main parameters were measured to evaluate the electrochemical treatment efficiency, the remaining pollutant concentration and the COD. Remaining pollutants fenamiphos concentration was measured with the double-beam UV-visible spectrophotometer at λ_{max}=249 nm using calibration curve with standard error ±0.5%. The COD was determined using a closed reflux colorimetric method. The equation used to calculate the pesticide removal efficiency in the treatment experiments was:

$$\% E = \left[\frac{A_o - A}{A_o} \right] \times 100 \quad (1)$$

Where A_o and A are absorbance values of pesticide solutions before and after treatment with respect to their λ_{max} [11].

The calculation of COD removal efficiencies after electrocoagulation treatment was performed using the following formula [27].

$$C_R \% = \left[\frac{C_o - C}{C_o} \right] \times 100 \quad (2)$$

Where C_o and C are concentrations of wastewater before and after electrocoagulation

Result and Discussion

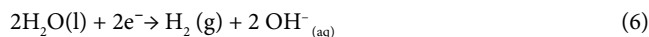
Mechanism of electrocoagulation process

In electrocoagulation process we can use variety of electrodes for the treatment of aqueous solutions, which are iron or steel, aluminum, magnesium or combination of them [28]. The possible reactions which occur during the electrocoagulation process are [29,30].

At the anode:



At the cathode:



Where M is the material used as electrode and n is the number of electrons. During the electrocoagulation process metal hydroxides, polyhydroxides and/or polyhydroxymetallic compounds of the electrode material will be generated. These materials contain strong affinity for dispersed particles and counter ions, which results in coagulation [29].

Effect of current density

The current density is the current per unit surface; it is a parameter that controls the anode dissolution speed on the one hand, and that of hydrogen formation on the other hand [14,31]. The influence of the variation of this parameter between 9-55 mA/cm² has been examined on the removal efficiency of fenamiphos and COD at 60 min using (C with SS) and (SS with Ti) electrodes at initial concentration of 50 mg/L, NaCl concentration of 1 g/L, pH of 6.7 and temperature of 20°C. Figure 1 and Table 2 indicate that a current of 18.5 mA/cm² gives fenamiphos removal efficiency of 97.4% and COD 96.4% using (C with SS) electrodes. While by using (SS with Ti) electrodes fenamiphos removal efficiency reached 90.3% and COD 88.2%. From Figure 1 and Table 2 when the current density increases, the efficiency of ion

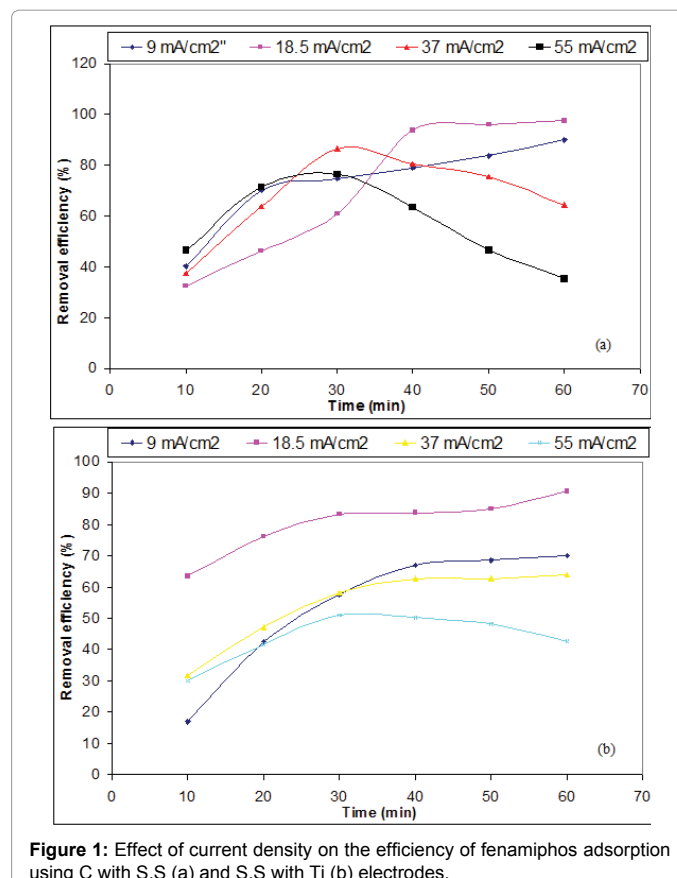


Figure 1: Effect of current density on the efficiency of fenamiphos adsorption using C with S.S (a) and S.S with Ti (b) electrodes.

Current density (mA/cm ²)	C with S.S(a)					S.S with Ti(b)				
	9	18.5	37	55	-	9	18.5	37	55	-
COD (%)	88.2	96.4	63.5	58.8	-	82.3	88.2	80	63.5	-
pH	2	4.5	6.7	8	10	2	4.5	6.7	8	10
COD (%)	76.4	88.2	96.4	82.3	72.9	47	67.5	88.2	89.4	88.2
Electrolyte	KCl	Na ₂ CO ₃	NaCl	Na ₂ SO ₄	NaF	KCl	Na ₂ CO ₃	NaCl	Na ₂ SO ₄	-
COD (%)	80.5	53.5	96.4	68	32.1	84.7	70.5	88.2	70.5	-
[NaCl] (g/L)	0.5	1	1.5	2	-	0.5	1	1.5	2	-
COD (%)	82.3	96.4	74.1	74.1	-	72.9	88.2	70.5	43.5	-
[PESTICIDE] (mg/L)	30	50	100	150	-	30	50	100	150	-
COD (%)	88.2	96.4	61.1	49.4	-	91.7	88.5	35.2	20	-
Temperature (°C)	10	20	30	40	-	10	20	30	40	-
COD (%)	94.1	96.4	75.2	74.1	-	83.5	88.2	88.2	64.7	-

Table 2: Effect of current density, pH, type of electrolyte, concentration and temperature on the efficiency of COD removal for fenamiphos electrolyte, pesticide using C with S.S (a) concentration and S.S with Ti (b) electrodes.

production on the anode and cathode increases. Therefore, there is an increase in floc production in the solution and hence an improvement in the efficiency of pesticides removal. The increase of coagulant and bubbles generation rate lead to the increase number of H₂ bubbles and decrease their size with increasing current density resulting in a faster adsorption of pesticides [32,33].

Effect of electrolyte Concentration

The supporting electrolyte increases solution conductivity; hence it influences the generated current and energy consumption of the process. Many researchers have found NaCl as the best supporting electrolyte [34]. The effect of NaCl on removal efficiency of fenamiphos and COD was examined at 60 min using (C with SS) electrodes and (SS with Ti) electrodes at initial concentration of 50 mg/L, current density of 18.5, pH of 6.7 and temperature of 20°C. Figure 2 and Table 2 shows the maximum removal efficiency of fenamiphos and COD was obtained at 1g/L NaCl concentrations. However, with the increasing in NaCl concentration > 1 g/L the removal efficiency decreased due to when the increase in NaCl concentration greater than 1 g/L, the removal efficiency decreased. It can be attributed that at a constant voltage with increasing of electrolyte concentration, conductivity of pesticide solution increases and resistance decreases, so the passed current increases and the produced amount of metallic hydroxide. This leads to a reduction of the oxide layer and an enhancement of the anodic dissolution of the electrode material and the pesticide adsorption increases [35,36].

Effect of pH

The influent pH is one of the important factors in affecting the performance of electrochemical process [14]. Experiments were carried out to evaluate the effect of pH, using solutions containing a sample with an initial pH varying in the range (2-10) at a current density of 18.5 mA / cm², initial concentration of 50 mg L⁻¹, inter electrode distance of 1 cm, NaCl concentration of 1 g L⁻¹ and temperature of 20°C. Figure 3 and Table 2 show the pesticide removal efficiency and COD as a function of pH. The removal efficiency of the pesticide and COD are high in neutral medium (pH 6.7), meanwhile, in acidic and basic medium are low. The decrease of adsorption when the pH is higher than 9, and more acidic was observed by many investigators [37,38] and was attributed to an amphoteric behavior of M(OH)₃ which leads to soluble metal cations (at acidic pH) and to monomeric

anions (at alkaline pH).

Effect of initial pesticide concentration

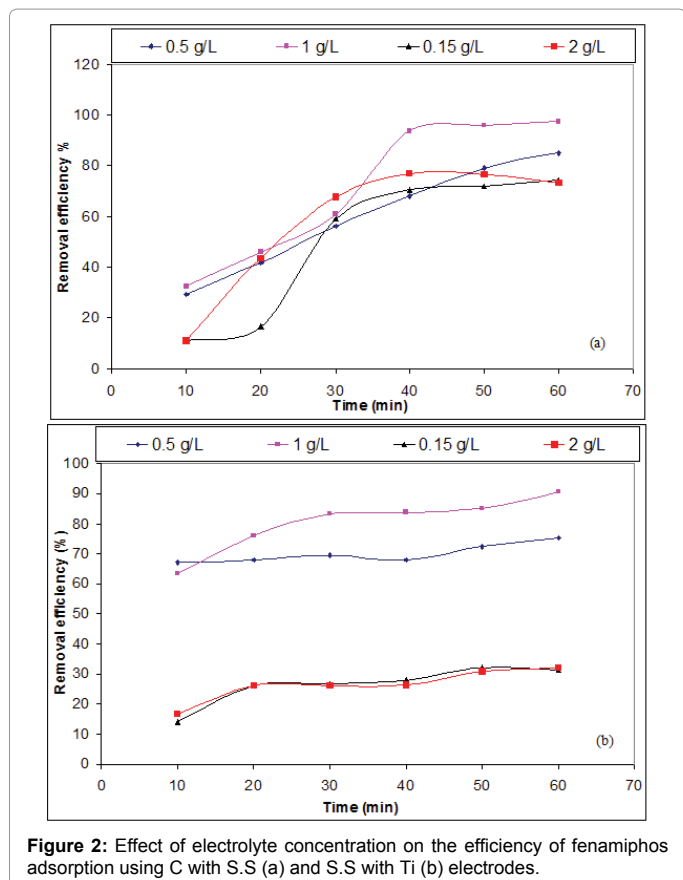
Effect of the initial concentration of fenamiphos in EC cell was investigated in the range of 30 to 150 mg/L using (C with SS) and (SS with Ti) at a current density of 18.5 mA / cm², pH of 6.7, inter electrode distance of 1 cm, NaCl concentration of 1 g L⁻¹ and temperature of 20°C. From Figure 4 and Table 2 it may be seen that increasing initial pesticide concentration results in decreasing the adsorption because the amount of produced flocs is insufficient to adsorb all pesticide molecules, therefore pesticide and COD removal decreases. The maximum removal was obtained at initial concentration 50 mg/L.

Effect of temperature

In the present work, effect of temperature from 10 to 40°C has been studied for the removal efficiency of fenamiphos and COD as shown Figure 5 and Table 2 at initial concentration of 50 mg/L, current density of 18.5, pH of 6.7 and NaCl concentration of 1 g/L. Figure 5 indicate that the efficiency removal and COD were optimum at 20°C. However, with increasing temperature > 20°C the removal efficiency and COD decrease. Higher temperature value (40°C) the pesticide removal and COD % dropped to lower values. More dropping occur at 50°C using the whole electrodes. However, it should be noted that the operation of electrocoagulation process at higher temperature significantly reduced electrical energy consumption [39]. When the temperature was over than 60°C, the current efficiency began to decrease. In this case, the volume of colloid M(OH) will decrease and pore production on the metal anode well be closed [40].

Effect of the electrolyte type

Figure 6 and Table 2 explain the effect of electrolyte type on the removal efficiency of fenamiphos and COD at 60 min using (C with SS) and (SS with Ti) at a current density of 18.5 mA / cm², pH of 6.7; inter electrode distance of 1 cm and temperature of 20°C. In the presence of different supporting electrolytes including NaCl, KCl, Na₂SO₄ and Na₂CO₃, According to Figure 6, the pesticide removal at NaCl, is better than KCl, Na₂CO₃ and Na₂SO₄. These results may be Due to formation of hypochlorite (OCl⁻) and hypochlorous acid (HOCl). This behavior may be due to the small ionic size of Na⁺ which increases the ion mobilities and the loss ability of Cl⁻ ion. But in the other electrolytes such as Na₂CO₃, Na₂SO₄ and NaF which do not contain chloride ions, the removal pesticide and COD efficiency dropped. Na₂SO₄ and NaF



electrolytes showed the least efficiency in the degradation of pollutant. This may be attributed to the formation of an adherent film on the anode surface which poisons the electrode surface. Also, these electrolytes do not contain chloride ions (Cl⁻) in their structures and may form stable intermediate species that could not be oxidized by direct electrolysis. These observations were also confirmed in other study [41].

Adsorption data

Kinetic studies: Figure 7 display the removal of fenamiphos exhibited pseudo first order with good correlation coefficients (>0.96) using C with SS electrodes and (>0.95) using SS with Ti electrodes according to equation (7).

$$\ln \frac{A}{A_0} = -kt \quad (7)$$

The values of rate constants at optimum condition and reaction time were 0.0295 and 0.01 mol⁻¹ using C with SS and SS with Ti electrodes respectively. Results show that the removal rate using C with SS electrode was higher than the removal rate using SS with Ti electrodes.

Isotherm modeling: The purpose of the sorption isotherms is to reveal the specific relation between the equilibrium concentration of adsorbate in the bulk and the adsorbed amount at the surface. The isotherm results fenamiphos at a constant temperature of 20°C were analyzed using four important isotherms including the Langmuir and Freundlich, isotherm models (Figures 8 and 9).

Langmuir isotherm: The Langmuir isotherm model was developed

to represent chemisorptions at a set of well-defined localized adsorption sites with the same adsorption energy, independent of the surface coverage, and with no interaction between adsorbed molecules. This model assumes a monolayer deposition on a surface with a finite number of identical sites. The linearized form of Langmuir adsorption isotherm model is [42].

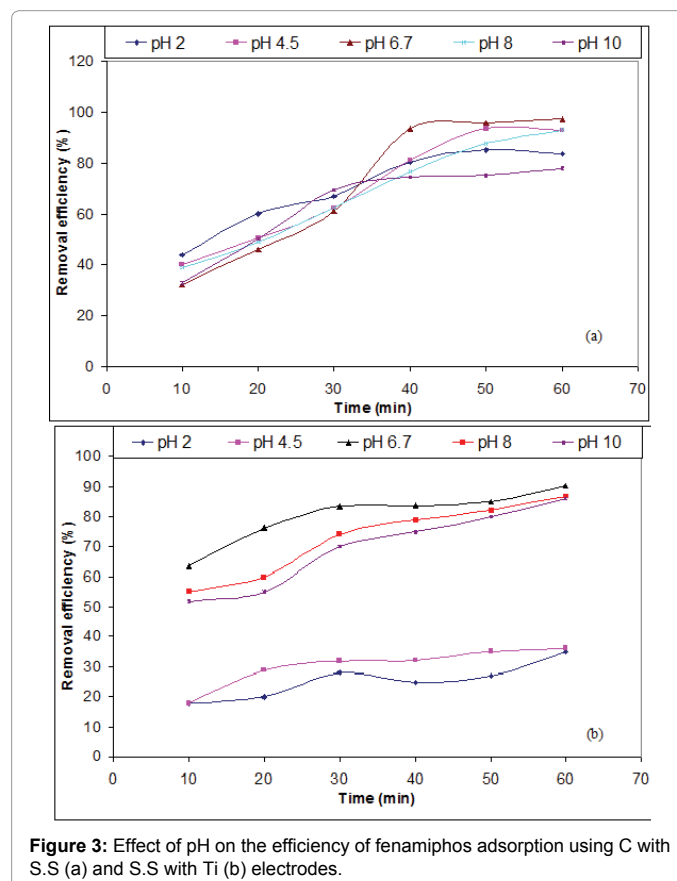
$$C_e/q_e = 1/q_m b + C_e/q_m \quad (8)$$

Where q_e (mg/g) is amount adsorbed at equilibrium, C_e (mg/L) equilibrium concentration, q_m is the Langmuir constant representing maximum monolayer adsorption capacity, and b is the Langmuir constant related to energy of adsorption

Freundlich isotherm: The Freundlich adsorption isotherm model includes considerations of surface heterogeneity and exponential distribution of the active sites and their energies. The isotherm is adopted to describe reversible adsorption and is not restricted to monolayer formation. This isotherm typically fits the experimental data over a wide range of concentrations. The linearized and logarithmic expression of the Freundlich model is [43]

$$\log q_e = \log k_f + n \log C_e \quad (9)$$

where k_f (mg/g) and n (dimensionless) are constants that account for all factors affecting the adsorption process, such as the adsorption capacity and intensity. The Freundlich constants K_f and n are determined from the intercept and slope, respectively, of the linear plot of log q_e versus log C_e. Adsorption isotherms were obtained in terms of equations (8) and (9) by using experimental adsorption results in these equations. The values q_m, b, K_f, R_L and n are summarized in Table 3.



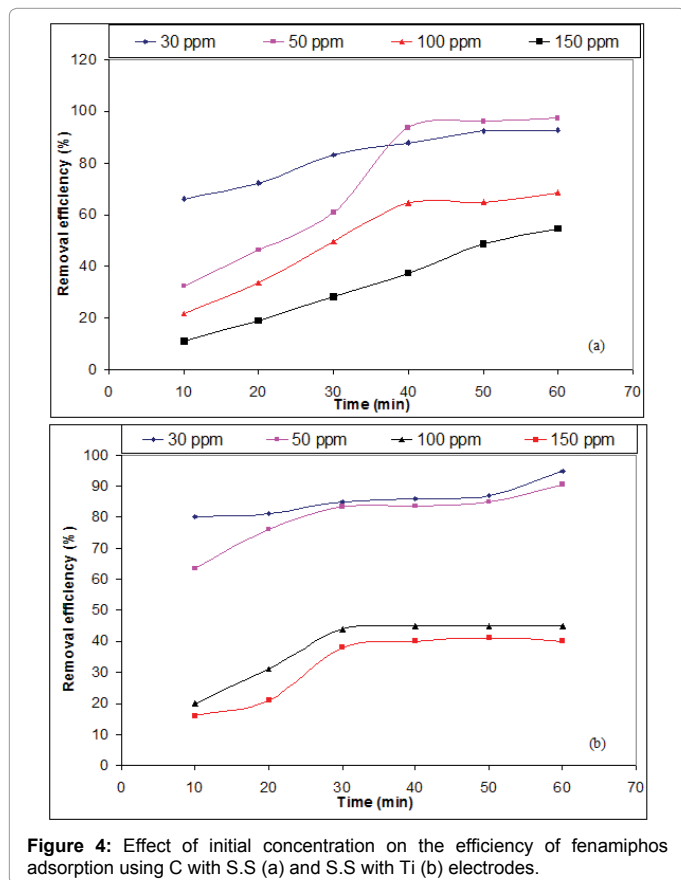


Figure 4: Effect of initial concentration on the efficiency of fenamiphos adsorption using C with S.S (a) and S.S with Ti (b) electrodes.

The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter R_L , which is a dimensionless constant referred to as separation factor or equilibrium parameter [44].

$$R_L = 1 / 1 + bC_e \quad (10)$$

where R_L is the equilibrium constant and indicates the type of adsorption, b is the Langmuir constant. C_e is initial concentration of fenamiphos solution. The R_L values between 0 and 1 indicate the favorable adsorption.

It is obtained from R^2 values that the equilibrium data of the system was well explained by the Langmuir model when compared to the Freundlich model. By comparing results listed in Table 3, it is clear that equilibrium data fit Langmuir and Freundlich models. Correlation coefficient of Langmuir model is higher than that of Freundlich, which means that Langmuir sorption isotherm more accurately describe the sorption of fenamiphos pesticide adsorption on the coagulant formed.

Electrical Energy Consumption and Electrode Consumption

In order to assist in assessing the economic feasibility of electrocoagulation in comparison with other techniques, the energy consumption and metal consumption were calculated as follows.

a) In an electrochemical process, the most important economical parameter is energy consumption E (kWh/m^3) [45,46]. This parameter is calculated from the following expression:

$$E = I V_t / \text{volume} \times 1000 \quad (11)$$

where V , I and t stand for average voltage of the EC system (V),

electrical current intensity (A) and reaction time (h), respectively.

$$\text{(For C with SS electrodes)} E = 1.93 \times 0.0185 \times 1 / 1 \times 10^{-4} \times 1000 = 0.356 \text{ kWh/m}^3$$

$$\text{(For SS with Ti electrodes)} E = 1.1851 \times .01851 \times 1 / 1 \times 10^{-4} \times 1000 = 0.2194 \text{ kWh/m}^3$$

b) The electrode consumption can be estimated according to Faraday's law and the amount of coagulant generated can be estimated stoichiometrically [47].

$$W = I T M / Z \times F \quad (12)$$

Where W : the amount of the electrode dissolved (g), I : the current intensity (A), t : the time (seconds), M : the relative molar mass of the electrode, z : the number of electrons in the redox reaction, and F : Faraday's constant (96500 coulombs).

Comparison with Other Method

Isotherm	R^2	Constnt	Value
Langmuir	0.98	q_m (mg /g)	74.6
		b (L/mg)	67
		R_L	0.0022
Freundlich	0.96	K_F (mg /g)	50.1
		n	1.115
		$1/n$	0.896

Table 3: Parameters of Langmuir and Freundlich isotherm constants and correlation coefficients using C with SS electrodes.

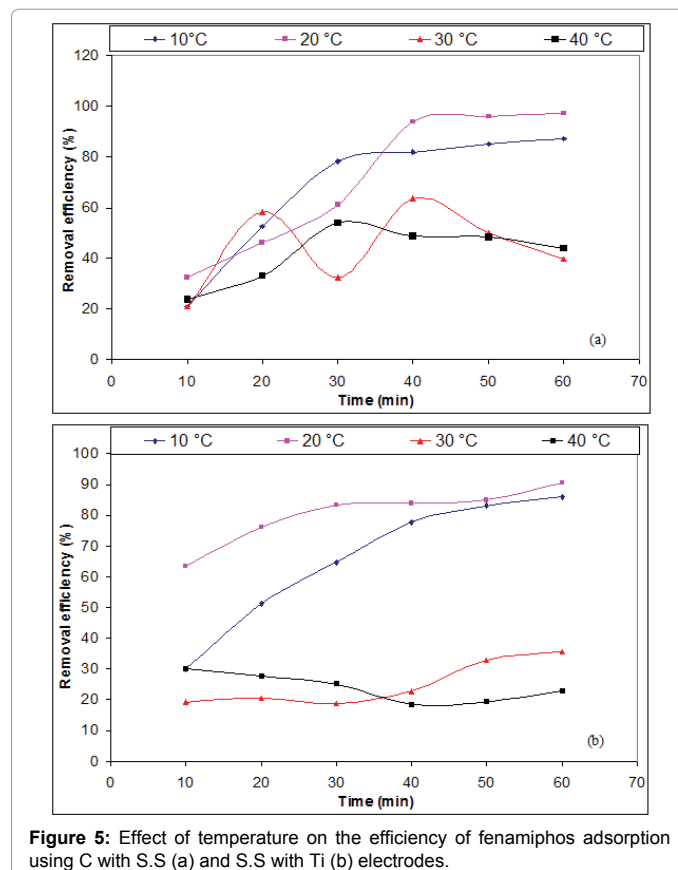


Figure 5: Effect of temperature on the efficiency of fenamiphos adsorption using C with S.S (a) and S.S with Ti (b) electrodes.

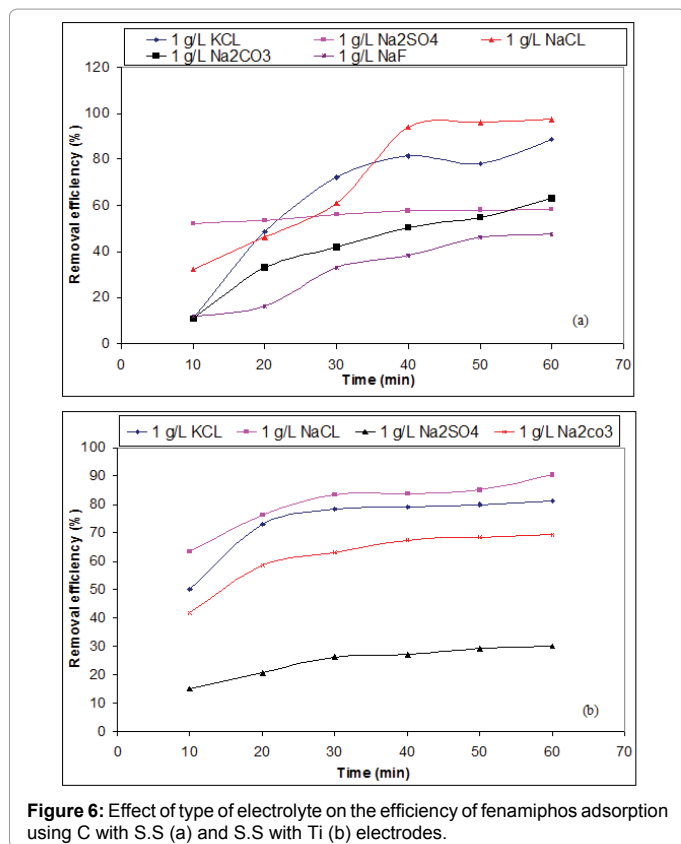


Figure 6: Effect of type of electrolyte on the efficiency of fenamiphos adsorption using C with S.S (a) and S.S with Ti (b) electrodes.

Type of degradation	Reference	Removal %	Time
Photocatalytic	Fenoll et al. [6]	100%	240 min
Photolysis and photodegradation	El Yadini et al. [8]	100%	180 min
photocatalytic	El Yadini et al. [7]	100%	180 min
Electrocoagulation	This study using (C with S.S)	97%	60 min
Electrocoagulation	This study using (S.S with Ti electrodes)	90.30%	60 min

Table 4: Comparison between the Electrocoagulation methods for removal of fenamiphos with other methods.

The percentages of degradation for each method using in literature and the electrochemical method in this work were represented in the Table 4. It is clear that the electrochemical degradation is the best.

Conclusion

The present study attempted to investigate the applicability of an electrocoagulation method in the treatment of fenamiphos pesticide in aqueous solutions. The effects of initial pH, initial pesticides concentration, current density, type of electrolyte, salt concentration, and temperature were investigated on removal efficiency and COD. The following conclusions are drawn up based on the results:

- Electrocoagulation is a fast, effective, and clean process to remove of pesticides from aqueous solution.
- The equilibrium adsorption behavior is analyzed by fitting the isotherm models of Langmuir and Freundlich. It was noted that the Langmuir model (0.98) is higher than that of Freundlich (0.96) using C with SS electrodes.

- Adsorption kinetics of electrocoagulants is analyzed using first kinetic models with value (>0.96) using C with SS electrodes and (>0.95) using SS with Ti electrodes
- The electrodes arrangement of C with SS and SS with Ti as the anode-cathode achieved that the best efficacy in fenamiphos and COD removal during the EC to reach 97.4% and 96.4% by using (C with SS) electrodes at 60 min and were 90.3% and 88.2% by using (SS with Ti) electrodes at the same time at typical operating conditions: an initial pH of 6.7, initial pesticide concentration of 50 mg/L, current density 18.5 mA/cm², salt concentration of 1 g/L and temperature of 20°C.
- It is clear that our results were better than other method in literature.

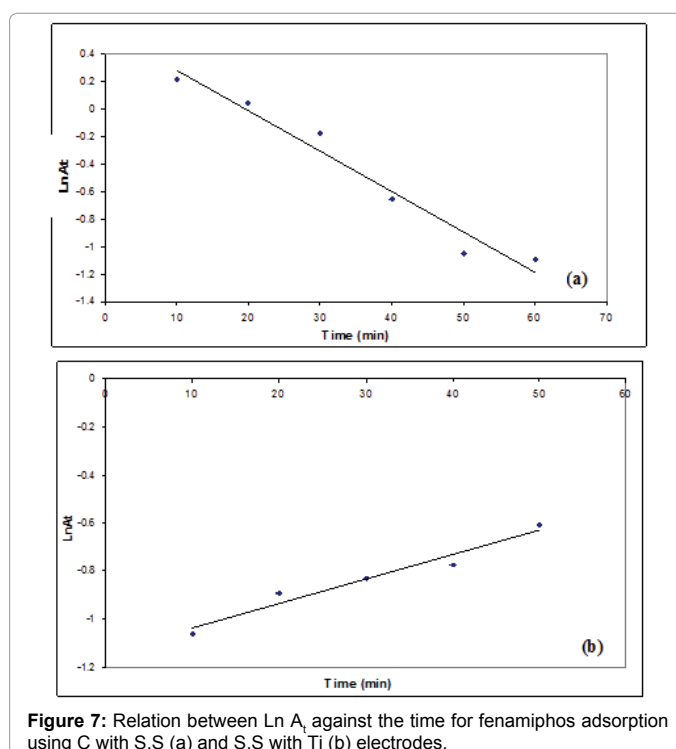


Figure 7: Relation between $\ln A_t$ against the time for fenamiphos adsorption using C with S.S (a) and S.S with Ti (b) electrodes.

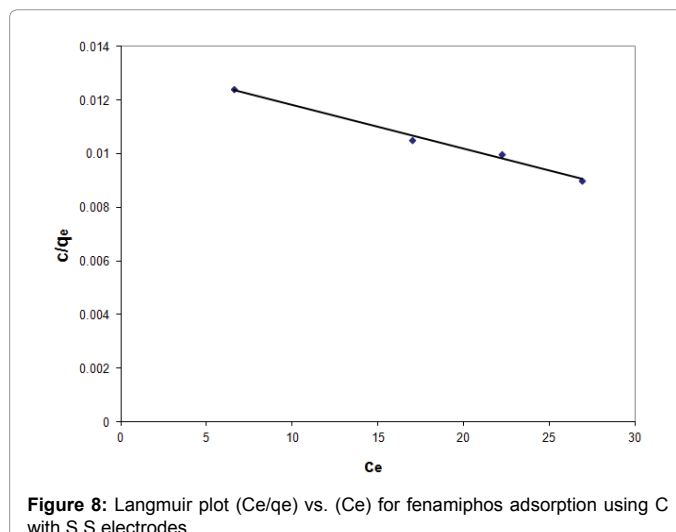


Figure 8: Langmuir plot (C_e/q_e) vs. (C_e) for fenamiphos adsorption using C with S.S electrodes.

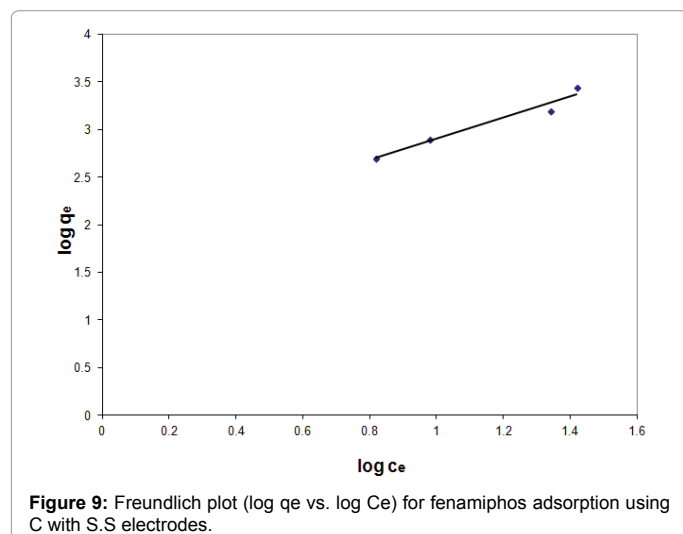


Figure 9: Freundlich plot (log q_e vs. log C_e) for fenamiphos adsorption using C with S.S electrodes.

The results were concludes that the electrode material play an important role in electrocoagulation method for treatment of pesticides in aqueous solution.

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