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Research Article

Optimizing Electro-Fenton process for removal of atrazine from aqueous solutions using Taguchi method

Huynh Ngoc Loan[1], Duong Dinh Hoan[2], Bui Manh Ha[3,*]

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ABSTRACT

The purpose of this study is centered on the removal of atrazine, one of the popular organochlorines in Vietnam, from an aqueous solution, using an electro Fenton process with iron and carbon plated steel electrodes at a batch electro reactor. This study had applied the Taguchi method, one of the most uncomplicated cases of design of experiments involving the minimum number of experiments to be performed within the permissible limit of factors and levels through the Signal to Noise ratio. This study design was conducted with five independent factors: initial pH, current density, Fe^{2+} concentration, sodium sulfate and reaction time, at a fixed atrazine concentration of 10 mg/L to find the best condition to eliminate atrazine from the solution. The Signal to Noise ratio results illustrates that the initial pH is the most important factor, followed by the reaction time and Fe^{2+} concentration, while sodium sulfate and current density seem neglectable to the removal of atrazine using electro Fenton process. The optimal Taguchi condition shows that the electro Fenton process reached the best efficiency, approximately 76% atrazine eliminated after 180 min of reaction time at initial pH 3.5, sodium sulfate of 990 mg/L, Fe^{2+} concentration of 2 mM and current density of 2.22 mA/cm^2. Three confirm experiments at optimal test conditions also indicated good agreement with predicted results with small error variation (1.21 - 3.54%). Thus, the relationship between the removal efficiency and operating parameters could be understood. These obtained results highlight the potential of using the electro Fenton process to eradicate or reduce pesticide contaminants. Electron beam also could be one of the pre-treatment techniques to eliminate persistent organic pollutants before biological treatment systems.

Key words: Atrazine, herbicide, electro-fenton, Taguchi design

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INTRODUCTION

Agrochemicals (pesticides) has become an essential aspect of modern agriculture, helping to increase yields and crop growth while also ensuring agricultural output stability[1]. Pests, diseases, and weeds are estimated to account for roughly 25% of global crop output losses that traditional pesticides could control. However, the pesticide contamination of water sources has recently become a serious environmental issue[2].

Organochlorines are among the most widely used pesticides to control pests and diseases carriers. In particular, atrazine stands out as high production and worldwide distribution[3]. Atrazine was first registered in the United States in 1959 and was used as herbicides to control broadleaf and grassy weeds pre-and post-emergence phases. Therefore, possibly these compounds can be found in the environment. In a previous study, the atrazine concentration detected in the surface water is up to 2.1 μg/L that is higher than the limited standard of 0.1 μg/L[4]. Phyu, Warne[5] found that atrazine was moderately toxic to tropical freshwater *daphnia* species (48-h-LC_{50} 24.6 mg/L). Lerro, Beane Freeman[6] also reported that atrazine could be the main responsible for human disease in Iowa, the USA, which required a lot of time for recovery. Therefore, research on appropriate techniques is necessary to preserve the environment from contaminated water.

To degrade pesticides, diverse techniques, i.e., adsorption[7], membrane filtration[8,9], coagulation[10], biological methods[11], and other methods[12], have been successfully used to eliminate pesticide contaminants from wastewater. However, the majority of these techniques are constrained by costs or operational difficulties. As a result, they could not be used for true agrochemical wastewater treatment for real agrochemical wastewater treatment. Recently, the Advanced Oxidation Processes (AOPs) studied focuses on its organic pollutant removal.

AOPs have successfully been applied to remove persistent organic pollutants through reaction with hydroxyl radicals ($^{\bullet}OH$). Among the AOPs, Fenton

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technology is broadly used in practice because of its cheap and high efficiency with the low toxicity of its reagent (Fe^{2+} ions and hydrogen peroxide-H_2O_2)[13]. In the Fenton process, iron (Fe^{2+}) catalyzes H_2O_2 into $^{\bullet}OH$, a powerful oxidant capable of decomposing organic pollutants[14]. The Fenton reagent effectively mineralizes contaminants using cheap materials. Moreover, both Fe^{2+} and Fe^{3+} can act as flocculants that further remove the organic pollutants. Because of its high efficiency and simplicity, the Fenton process is regarded as one of the most appealing AOPs used in wastewater treatment.

However, several challenges remain in conventional Fenton-based technologies, including materials for storing corrosive chemicals, i.e., H_2O_2, H_2SO_4, NaOH, etc. and massive production of sludge formation. As a result, many efforts have been made to develop new technologies that address these issues while still exploiting the Fenton process's powerful oxidation efficiency.

The electrochemical Fenton technique developed lately from the Fenton process is highly attracted among wastewater treatment to remove toxic organic pollutants. In the electrochemical Fenton process, pollutants are eliminated with the Fenton reagent and cathode oxidation across the anode surface. Anode oxidation is unsuccessful at mineralizing most organic contaminants due to the persistent production of organic acids. On the other hand, the electrochemical Fenton might accomplish a spectacular degrading effect of organic pollutants by producing • OH, as shown in equation 1[15].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \cdot OH + OH^- \quad (1)$$

Compared to the traditional Fenton, the electrochemical Fenton process could *in-situ* H_2O_2 generation that decreased the chemical usage. It has successfully been applied to eliminating organic contamination from wastewaters like dyeing and textile[16,17], pharmaceutical[18], coke[19], and pesticides[20–22]. Even though EF can reduce the organic compounds in aqueous solutions, studies on pesticide contaminants removal are limited.

Aside from deciding on removal strategies, experimental design plays a vital role in reducing both the time and cost associated with wastewater treatment. Taguchi method, a type of experimental design that based on orthogonal arrays and signal to noise ratio (S/N) qualification[23,24], has succeeded in improving, optimizing, and interpreting the factor effects in many treatment processes from wastewater of textile[17], pulp and paper mill[25], oily[23], etc. However, the research literature of Taguchi design on the elimination of pesticides using EF method is still insufficient.

Thus, this study aims to investigate the atrazine elimination of EF technology from aqueous solutions. This EF process study was investigated using a variety of parameters, including electrolysis time, current density, initial pH, sodium sulfate (Na_2SO_4) and Fe^{2+} concentration.

MATERIALS AND METHODS

Reagents

Sigma-Aldrich provided atrazine (2-Chloro-4-ethylamino-6-isopropylamino-1,3,5-triazine) with a purity of >98%, which was used without further purification. Biochem in France provided analytical quality ferrous sulfate heptahydrate ($FeSO_4.7H_2O$) and sodium sulfate (Na_2SO_4). 10 mg/L of atrazine solution was adjusted to the desired value in experiments using H_2SO_4 (0.5N) and NaOH (0.1N). The 100 mg/L of atrazine stock solution for EF process was described in our previous procedure[12] using deionized water and stored at 5°C; atrazine concentrations of 10 mg/L were obtained by diluting this stock.

Experimental setup and analysis

All experiments were conducted in 4 L electro-Fenton batch reactor, as illustrated in Figure 1. The reactor was involved two iron electrodes anode (Fe: 99.25% - CT_2) and carbon plated iron cathode, with 146 × 150 × 4 mm dimensions linked in parallel. At the same time, the DC power supply (QJ3003XE, 30V - 3A) was linked to the outer electrodes.

The L27 Taguchi design was conducted with signal-to-noise ratio (S/N) to determine the efficiency of the EF treatment. Five independent parameters, e.g., Fe^{2+} concentration, Na_2SO_4 concentration, pH, current density and reaction time with three levels, were evaluated as in Table 1. S/N ratio plots have been used to explore the effect of variables on the response by describing the relationship between the response and the variables. The EF process was subjected to multiple response adjustments to establish the best parameters for maximum atrazine removal efficiency. Minitab 18 was used as a statistical program to conduct the study.

Before experimenting, compressed air was forced into the cathode at a rate of 2.5 L/min for 15 min and maintained throughout the electrolysis. In each run, atrazine solutions were delivered to the feed tank and

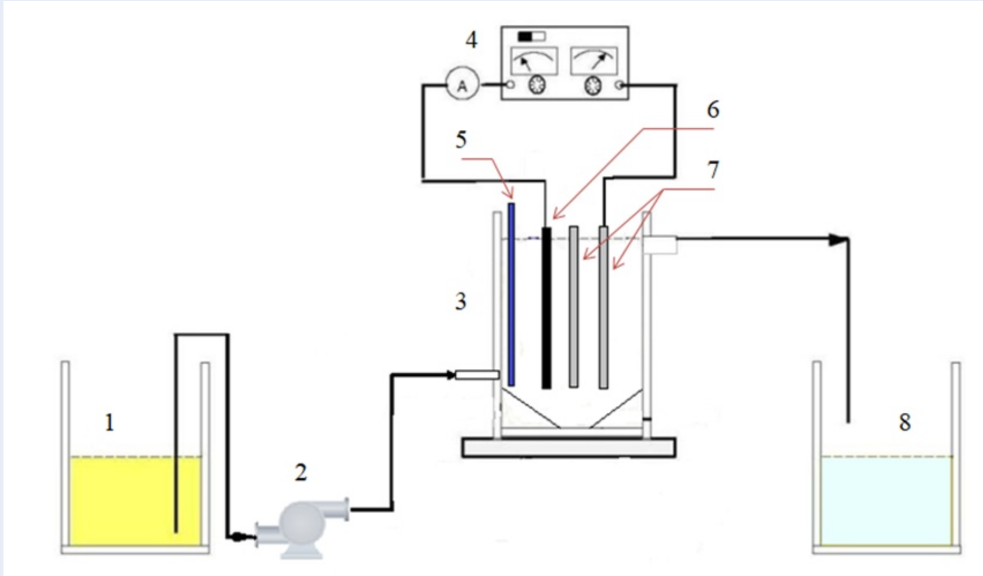

Figure 1: Schematic of the experimental setup for EF treatment. (1) Storage tank, (2) Pump, (3) EF tank, (4) DC power supply, (5) Feed air, (6) carbon plated iron, (7) iron-plated, (8) Product tank

Table 1: Taguchi design for EF treatment

Independent factors	Level 1	Level 2	Level 3
pH	3	3.5	4
Current density (mA/cm^2)	1.11	2.22	3.33
Fe^{2+} concentration (mM)	0.05	0.2	1
Sodium sulfate (mg/L)	330	660	990
Reaction time (min)	60	120	180

reaction reactor. The feed pump controlled the appropriate flow rate while H_2SO_4 changed the initial pH values.

At various time intervals, samples were obtained from the reactor and instantly examined with a syringe equipped with a 0.2 μm filter. The atrazine concentration was determined using High-Performance Liquid Chromatography (HPLC) with the following parameters: wavelength 224 nm, C18 column, length and diameter of column 4.6 × 250 mm, and injection volume of 20 μL. The percentage of atrazine removal was calculated as follows:

$$Atrazine\ removal\ efficiency = \frac{C_0 - C_t}{C_0} \times 100\% \quad (2)$$

Herein, C_0 is the initial atrazine concentration and C_t is the atrazine concentration at t reaction time.

RESULTS AND DISCUSSION

Experimental Design Analysis

According to the Taguchi approach, twenty-seven experiment results with 3 levels and 5 factors are indicated in Table 2. The output signal–noise (S/N) ratio from the Taguchi analysis would be evaluated for each test run to determine the distinguishing characteristics between control and signal factors to optimize the pesticide removal procedure. The higher the S/N ratio, the sufficient information there is compared to noisy erroneous data. The "large, the better" of S/N was also used to evaluate the maximize pesticide removal efficiency of the EF process.

Minitab analysis of the Taguchi design

The effect of the independent factors, i.e., pH, current density, Fe^{2+} concentration, sodium sulfate, reaction time on atrazine removal, had been investigated. Minitab used the signal-to-noise ratios and means in each level to rank process factors for effec-

Table 2: Experimental design, the obtained responses

			Factors				
Run	pH	Current density (mA/cm^2)	Fe^{2+} concentration (mg/L)	Sodium sulfate (mg/L)	Reaction time (min)	Atrazine removal (%)	
						Experime	Predicted
1	3.0	1.11	0.05	330	60	36.67	34.88
2	3.0	1.11	0.05	330	120	40.91	40.19
3	3.0	1.11	0.05	330	180	43.73	46.23
4	3.0	2.22	0.20	660	60	48.57	51.23
5	3.0	2.22	0.20	660	120	55.92	56.54
6	3.0	2.22	0.20	660	180	65.87	62.58
7	3.0	3.33	1.00	990	60	41.91	42.47
8	3.0	3.33	1.00	990	120	50.14	47.80
9	3.0	3.33	1.00	990	180	52.05	53.82
10	3.5	1.11	0.20	990	60	33.35	34.00
11	3.5	1.11	0.20	990	120	38.26	39.32
12	3.5	1.11	0.20	990	180	47.05	45.35
13	3.5	2.22	1.00	330	60	22.62	22.29
14	3.5	2.22	1.00	330	120	25.71	27.61
15	3.5	2.22	1.00	330	180	35.20	33.64
16	3.5	3.33	0.05	660	60	25.39	25.49
17	3.5	3.33	0.05	660	120	32.46	30.81
18	3.5	3.33	0.05	660	180	35.30	36.84
19	4.0	1.11	1.00	660	60	16.29	16.13
20	4.0	1.11	1.00	660	120	21.11	21.45
21	4.0	1.11	1.00	660	180	27.67	27.48
22	4.0	2.22	0.05	990	60	17.96	17.00
23	4.0	2.22	0.05	990	120	21.19	22.31
24	4.0	2.22	0.05	990	180	28.49	28.34
25	4.0	3.33	0.20	330	60	19.77	19.04
26	4.0	3.33	0.20	330	120	24.70	24.36
27	4.0	3.33	0.20	330	180	29.32	30.39

Table 3: Response table for S/N ratio

Level	pH	Current density	Fe^{2+} concentration	Sodium sulfate	Reaction time
1	33.57	30.16	29.59	29.52	28.71
2	30.12	30.22	31.53	30.43	30.24
3	27.04	30.35	29.62	30.78	31.79
Delta	6.53	0.19	1.94	1.26	3.09
Rank	1	5	3	4	2

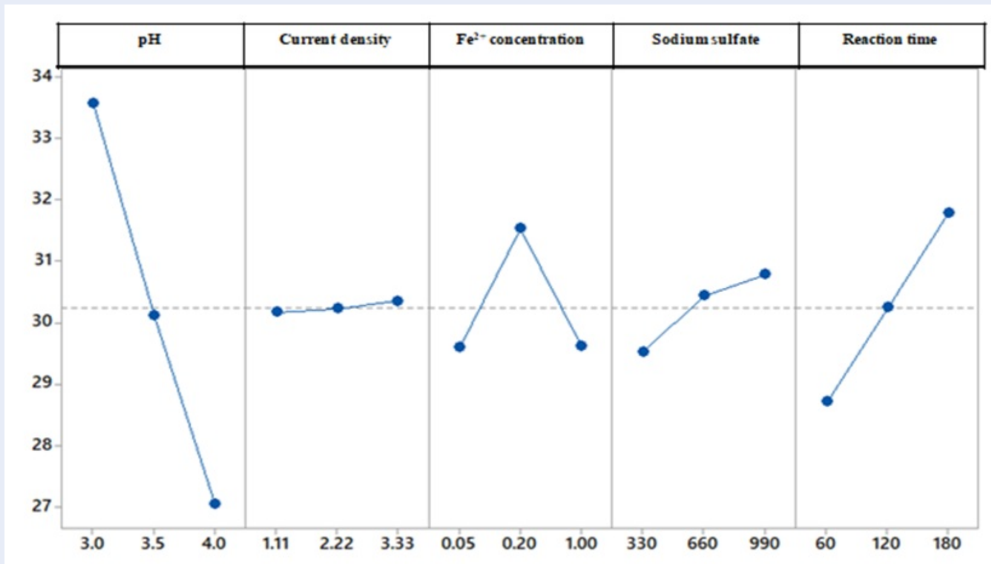

Figure 2: The primary influence of parameters on the S/N for atrazine removal capacity

tive atrazine removal (Table 3).

Besides, the S/N ratio figure also shows how important the influence parameters are in determining the response. Figure 2 shows the mean of S/N ratios for each parameter category corresponding to its' level. The response with the highest S/N ratio consistently produces the best results. As a result, pH level 1 (pH 3), current density level 3 (2.22 mA/cm^2), Fe^{2+} concentration level 2 (0.2 mM), sodium sulfate level 3 (990 mg/L), and reaction time level 3 (180 min) was the optimal combination of parameters for obtaining the maximum value for S/N for pesticide removal effectiveness during EF process. In a previous study of Zhang, Zhang[26] EF process mainly influence by pH and reaction time, who stated that the decomposition of 5000 mg/L of COD from landfill leachate to around 2500 mg/L only within 30 min and obtained 1000 mg/L around 75 min at pH approximately 3.5.

Experiments confirm optimal atrazine treatment efficiency

According to the S/N ratio and predicted regression model, the optimal operation treatment using EF to atrazine removal efficiency could be selected as 1st level of pH value (3), 2nd level of current density (2.22 mA/cm^2), 2nd level of Fe^{2+} concentration (0.2 mM), 3rd level of sodium sulfate (990 mg/L) and 3rd level of reaction time (180 min). The serial confirmation experiments of the four factors with these values on various reaction times (30 to 180 min) were also conducted. The results of the confirmation experiments in Figure 3 proved to be consistent with predicted optimal results with the highest atrazine removal efficiency around 76.52% and minimal error variation (1.21-3.54%). The obtained results again demonstrated the excellent accuracy of the proposed model or Taguchi approach.

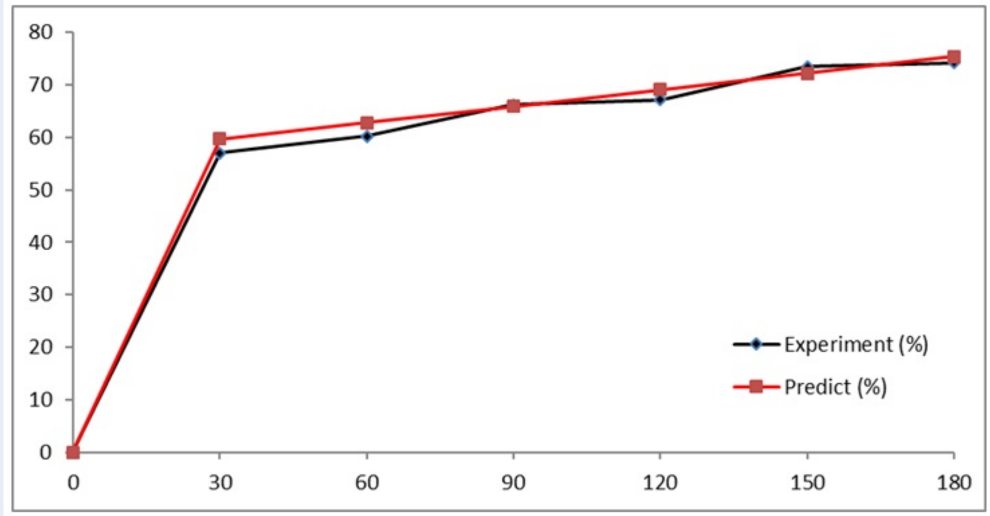

Figure 3: Variation of atrazine removal efficiency on reaction time using EF treatment at optimal conditions: current density = 2.22 mA/cm^2; [Fe^{2+}] = 0.2 mM; sodium sulfate = 990 mg/L, V = 3L.

CONCLUSION

In this study, the oxidation of atrazine was investigated by electro-Fenton (EF) under various operating conditions with Taguchi design. An atrazine degradation efficiency of 76.52% was obtained when the current density of 2.22 mA/cm^2, Fe^{2+} dosage of 0.2 mM, sodium sulfate of 990 mg/L with initial atrazine concentration of 10 mg/L during 180 min reaction. An increase in reaction time and sodium sulfate increase the degradation efficiency, whereas excessive Fe^{2+} concentration and current density can hamper the atrazine elimination. The initial pH is also an essential factor. A pH of 3 was shown to be the ideal pH for the EF process, resulting in a rapid degradation rate. This study found that the EF method can be utilized to pre-treat atrazine-contaminated wastewater before biological treatment.

ACKNOWLEDGMENTS

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ABBREVIATION

AOPs: Advanced Oxidation Processes
COD: Chemical Oxygen Demand
DC: Direct current
EF: Electro-Fenton
S/N: Signal/Noise

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this article

AUTHORS' CONTRIBUTIONS

Huynh Ngoc Loan, Hoan Dinh Duong and Bui Manh Ha have made substantial contributions to the work reported in the manuscript.

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Bài nghiên cứu

Tối ưu hóa xử lý atrazine trong nước bằng phương pháp Fenton điện hóa sử dụng thiết kế Taguchi

Huỳnh Ngọc Loan[1], Dương Đình Hoan[2], Bùi Mạnh Hà[3,*]

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TÓM TẮT

Atrazine là một trong những loại thuốc bảo vệ thực vật clo hữu cơ được sử dụng phổ biến tại Việt Nam, chất hữu cơ này thường rất bền và gây nhiều vấn đề nguy hại trong môi trường. Nghiên cứu này sử dụng phương pháp Fenton điện hóa với hai loại điện cực sắt và thép phủ carbon để loại trừ atrazine trong dung dịch. Nghiên cứu áp dụng phương pháp Taguchi, một phương pháp thiết kế thí nghiệm đơn giản, cần thực hiện ít thí nghiệm nhưng có thể xác định được các tương tác, mức độ ảnh hưởng của các yếu tố cũng như dự đoán được điểm tối ưu trong thí nghiệm. Trong thí nghiệm Taguchi này 5 yếu tố ảnh hưởng chính: mật độ dòng, lượng Fe^{2+} cũng như Na_2SO_4 thêm vào, pH ban đầu và thời gian phản ứng tại hàm lượng atrazine 10 mg/l được đưa vào mô hình thí nghiệm. Kết quả cho thấy pH, thời gian phản ứng và hàm lượng sắt là các yếu tố ảnh hưởng lớn đến hiệu quả loại trừ atrazine, trong khi hàm lượng muối Na_2SO_4 và mật độ dòng ít ảnh hưởng đến hiệu quả xử lý thuốc trừ sâu này. Hiệu quả loại trừ atrazine tốt nhất đạt đến 76% tại mật độ dòng 2,22 mA/cm^2, pH 3,5, hàm lượng Na_2SO_4 thêm vào là 990 mg/l, hàm lượng Fe^{2+} 2 mM trong 180 phút. Kết quả này cho thấy tiềm năng của việc loại trừ các hợp chất ô nhiễm hữu cơ bền như thuốc bảo vệ thực vật bằng Fenton điện hóa, nó có thể là một giải pháp tiền xử lý trước quá trình xử lý sinh học.

Từ khoá: Atrazine, thuốc trừ cỏ, Fenton điện hóa, thiết kế Taguchi

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