Sustainability of Electrocoagulation Process in the Removal of Dicofol Pesticide using Aluminium Electrodes

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Abstract. Because of its different concentrations, sewage can cause serious water pollution problems for aquatic organisms. Dicofol is a pesticide derived from organic chlorine that effectively targets ticks, though it is a subject of controversy because of its negative effects on the environment. In this study, an aluminium electrode was employed for the elimination of dicofol from a water-based solution through the process of electrocoagulation. Aluminium electrodes are characterized by SEM and FTIR techniques. The optimization process for several parameters such as the maximum removal percentage of Dicofol by APE was determined under the optimized conditions: electrolysis time, NaCl concentration, voltage, pH, initial Dicofol concentration. The pseudo-first-order rate constant of the aluminium plate electrodes was determined according to the results of the kinetic study. The electrical energy used to remove dicofol in the electrocoagulation process was determined on aluminium plate electrodes.

1 Introduction

Dicofol is a pesticide with organochlorine properties [1,2] that effectively targets mites, but is debated due to its impact on the environment [3,4]. Dicofol, with the chemical formula C14H9Cl5O, is also referred to as 2,2,2-trichloro-1,1-bis(4-chlorophenyl) ethanol [5,6,7]. Dicofol, also known as kelthane, is a white crystalline substance that can be dissolved in a liquid carrier, typically water, and is in the form of a wettable powder. Dicofol is often combined with other pesticides like organophosphates, parathion-methyl, and dimethoate in numerous countries. Dicofol was the original pesticide registered in the U.S. in 1957 [8,9], although there are other commercial options available for this miticide as well. EPA released a registration standard for dicofol on December 30, 1983. During the 1980s, Oregon state university stated that dicofol was banned temporarily due to its production method and high DDT usage. At first, the development of dicofol would result in a

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significant production of DDT, causing serious harm to the environment. Dicofol, with the same chemical composition as DTT (C14H9Cl5), does not contain the oxygen element found in its miticide.
family member Research in both the lab and in natural settings indicates that dicofol does not move easily, and that leaching and volatility are not likely to significantly impact dicofol's breakdown.
Consumption of dicofol residues through the consumption of various crops like apples and citrus can lead to exposure to pesticides. Pesticide workers are exposed to dicofol in their jobs through its use in agriculture. Because of its liquid state, it has the ability to easily seep into the soil and pollute underground water sources and nearby streams. It can lead to sickness through breathing, skin absorption, or swallowing.
Dicofol is mainly utilized for Agricultural Pest Control, Protecting Ornamental Plants, Public Health Applications, and Vector Control.

2 Methods and materials used

2.1 Necessary chemicals included:

Chemicals such as Dicofol pesticide, Sodium hydroxide (NaOH), Hydrochloric Acid (HCl), Pyridine, 4-Aminoacetanilide, Glacial acetic acid, 1-propanol, and Anhydrous sodium sulphate are included.

2.2 Types of glass containers:

2L beaker, Graduated cylinder, Pipette, Separatory funnel, and more.

2.3 Equipment

pH meter, UV Spectrophotometer, DC regulated power supply, Electrolytic cell and Glass Ware etc.

2.3.1 pH meter

Device used for measuring the acidity or alkalinity of a solution by detecting the concentration of hydrogen ions present.

A pH meter is a tool used in science to determine the level of hydrogen ions in water solutions, which shows whether the solution is acidic or alkaline through its pH value. The pH meter is referred to as a "Potentiometric pH meter" due to its ability to measure the electrical potential difference between a pH electrode and a reference electrode. The variance in electrical potential is connected to the acidity or pH level of the solution. The pH meter is utilized in various settings including laboratory experiments and quality assurance.

2.3.2 Ultraviolet–visible spectroscopy

UV-Vis spectroscopy, also known as UV-Vis spectrophotometry, involves absorption or reflectance spectroscopy across the ultraviolet and visible spectral regions. This indicates that it utilizes light within the visible spectrum and nearby wavelengths. The way substances absorb or reflect light in the visible spectrum influences how we perceive their colour. Fluorescence spectroscopy and absorption spectroscopy work together, with fluorescence studying transitions from the excited state to the ground state and absorption focusing on transitions from the ground state to the excited state.

2.4 Preparation of Dicofol solution

Made a Dicofol solution by dissolving 10ml of dicofol in 1000ml of distilled water.

2.5 Procedure

In order to study the treated water sample, 50ml of solution was poured into a conical flask, followed by the addition of 1ml of pyridine, and finally 2ml of 5M NaOH was introduced and thoroughly mixed. The objects were immersed in a water bath at a temperature of 70℃ for a period of 3 minutes and were stirred occasionally. Following cooling in ice water, slowly incorporate 1ml of glacial acetic acid. A mixture of 2ml of 4-aminoacetanilide in a 1% solution and 1ml of hydrochloric acid in a 10N solution was prepared and allowed to stand for 10 minutes for color development. The orange-red compound was obtained by extracting it in 2.5*2ml of 3-methyl-1-butanol. The extract was measured at 525nm using a spectrophotometer in comparison to 3-methyl-1-butanol.

2.6 Determination and Calibration Curve of Dicofol:

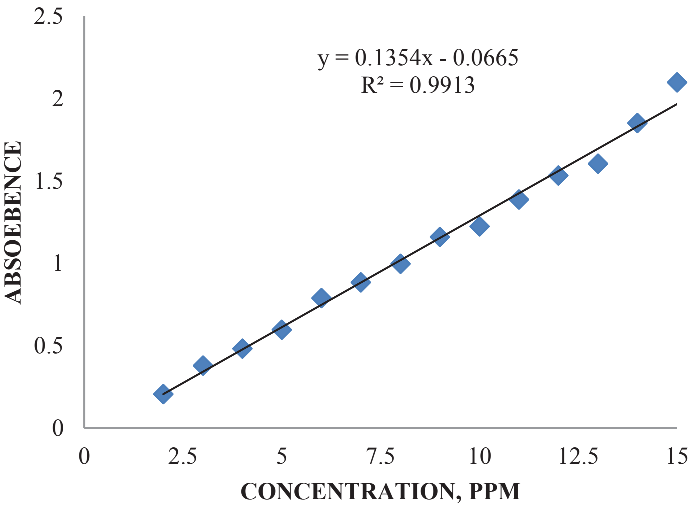


Fig. 1. Calibration curve for concentration of Dicofol

A calibration curve is essential for determining the relationship between the absorbance and concentration of Dicofol through spectrophotometric analysis. Below is the displayed Calibration curve for Dicofol analysis. The graph was created by plotting the Absorbance against the Concentration of Dicofol (in ppm) at a wavelength of 550 nm. The plot we acquired was linear and adheres to Lambert-Beer's law, allowing for the determination of Dicofol concentration within this range. The sample must be diluted if the concentration is above this range in order to make it measurable. Figure 1 displays the calibration curve for Dicofol.

2.6.1 Reaction mechanisms:

When Aluminium is chosen for electrode material, the reactions happen in the following way:

- At the anode:

$Al(s) \rightarrow Al^{3+} + 3e^{-}$

- At the cathode:

$3H_2O + 3e^{-} \rightarrow \frac{3}{2} H_2(g) + 3OH^{-}$

- In the solution:

$Al^{3+}(aq) + 3H_2O(l) \rightarrow Al(OH)_3 + 3H^{+}(aq)$

2.7 Experimental setup

A rectangular tank made of 1.5 litres of Plexiglas, measuring 11.5 × 11.5 cm at the base and 11 cm in height, consisted of four plate electrodes, specifically two anodes and two cathodes with an area of 10 × 22 cm2 as depicted in figure 3.3. The plates were positioned in a horizontal manner inside the tank with a 2.5 cm gap between each plate. Aluminium sheets were employed. Interchanging electrodes were linked to a digital DC power source (incorporating an ampere meter and a voltmeter for measuring current intensity and potential difference respectively), with a current range of 0-2 A and a voltage range of 0-32 V. Dicofol solution dissolved in water was utilized. Prior to each experiment, the Dicofol solution was added to the cell at a particular concentration, along with NaCl as an electrolyte to enhance the solution's conductivity. The timing of each run began once the DC power supply was turned on.

2.7.1 Electrolytic cell and plate electrodes

- Dimensions of electrolytic cell – 11.5cm x 11.5cm x 11cm
- Capacity of electrolytic cell – 1.5L
- Electrodes between the space – 2.5cm
- The dimension of plate electrode – 10cm x 6cm x 0.01cm
- Total area immersed plate electrode in electrolytic cell – 45cm^2

2.7.2 DC power supply inbuilt range

- Voltage – 0 to 32V
- Current – 0 to 2A

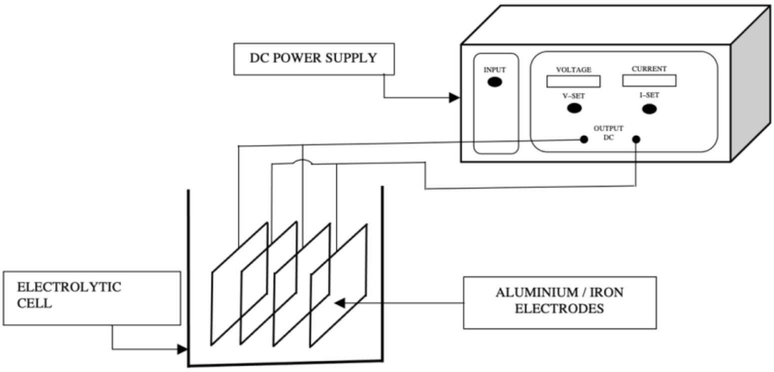


Fig 2: Diagram showing the setup used in the experiment

2.8 Experimental procedure

In each trial, a specified amount of sodium chloride, acting as a conductor, was mixed with a 1L of Dicofol solution. The remedies were introduced into the electrolytic cell. The pH was modified using 0.1N HCl / 0.1N NaOH solutions. In the electrolysis procedure, the solution received direct current from the D.C power supply via the four electrodes. A 10 ml sample was taken every 5 minutes during the cycle. The position of the gathered samples remained consistent for every iteration. Samples were filtered and subsequently measured for absorbance at a wavelength of 660nm.

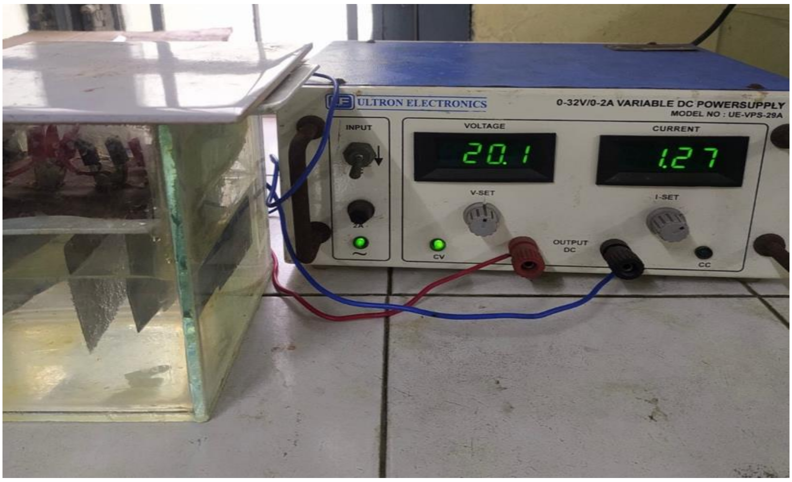


Fig 3: Electrolytic cell with DC machine

2.9 Experimental Parameters:

In the present study, Dicofol was removed by Aluminium plate electrodes, electrodes by Electrocoagulation process. Samples were gathered at consistent time intervals. The UV-

Spectrophotometer was employed to assess the concentration of Dicofol in the supernatant. The research concentrated on enhancing process variables like electrolysis duration, NaCl level, acidity level, electrical power, and initial content.

2.10 Effect of Electrolysis time:

From the stock solution, a 20ppm Dicofol solution was made and 1000 ml of this solution was poured into the electrolytic cell along with a NaCl concentration of 1.5gm/L.. The electrolytic cell has connected to Aluminium /, electrodes. After 5min, the sample was collected, filtered, and analysed for Dicofol present in the solution. In the same way, additional samples were gathered from the electrolytic cell at different contact durations of 10, 15, 20, 25, 30, 35, 40, 45 minutes in order to determine the best time.

2.11 Effect of NaCl concentration:

1000 ml of Dicofol solution with a concentration of 20 ppm was added to an electrolytic cell. The concentration of NaCl in the electrolytic cell was increased to 0.5 gm/L. Aluminium electrodes were placed in an electrocoagulation experimental apparatus until reaching the optimal time. The sample underwent collection, filtration, and analysis to determine the remaining amount of Dicofol in the solution. The procedure outlined above was replicated with varying NaCl concentrations of 1.0, 1.5, 2.0, 2.5, and 3.0gm/L in order to determine the best NaCl concentration.

2.12 Impact of Voltage:

By conducting the experiment, the impact of voltage was evaluated by using a 1000ml sample and keeping all other factors such as electrolysis time and NaCl concentration at their optimal levels [11,12]. The voltage varies from 5V to 30V with an interval of 5V. Aluminium electrodes were kept in electrocoagulation experimental setup to reach optimum time [13]. The samples are collected, filtered, and analysed for remaining amount of Dicofol present in the solution [14,15].

2.13 Effect of pH:

An electrolytic cell was filled with 1000 ml of Dicofol solution containing 20 ppm. The pH of the Dicofol solution was adjusted by adding the required amounts of 0.1N HCL and 0.1N NaOH. The pH levels of the Dicofol solution were adjusted to 4, 5, 6, 7, 8, 9, 10 and 11. Optimum NaCl concentration was added to the electrolytic cell [16,17,18]. Aluminium electrodes were kept in electrocoagulation experimental setup to reach optimum time. The samples are collected, filtered, and analysed for remaining amount of Dicofol present in the solution [19,20].

2.14 Effect of Initial concentration:

Taking a 1000ml sample was done to observe the impact of initial concentration. During the experiments, the electrolysis time, NaCl concentration, pH, and voltage were maintained at optimal levels. Dicofol solution available in concentrations of 10, 20, 40, 60, 80, and 100ppm [21,22]. Aluminium electrodes were kept in electrocoagulation experimental setup to reach optimum time. The samples are collected, filtered, and analysed for remaining amount of Dicofol present in the solution.

2.15 Kinetic models:

The elimination of Dicofol can be demonstrated by the equations provided.

2.15.1 Model of pseudo-first order:

The chances of data adhering to pseudo-first order kinetics are determined by.:

$$-\ln\left(\frac{C_t}{C_o}\right) = K_1 t \qquad (3.1)$$

The original concentration at time t (ppm) is symbolized as 'Ct', with 'Co' standing for the initial concentration (ppm), and K1 representing the rate constant (min-1) with t being the electrolysis time in (min). To calculate the rate constant, create a linear graph with the formula '-ln (Ct/Co)' plotted against 't'. Rewrite the text while using the same language and maintaining the word count: (3.1) [23,24].

Electrical Energy Consumption: The amount of electrical energy used and the efficiency of the current are crucial economic factors in the process of electrocoagulation. The following formula can be used to calculate electrical energy consumption.

$$E = \frac{U*i*t}{V} \quad (\text{kWh/m}^3) \qquad (3.2)$$

E represents the electrical energy consumed per cubic meter in kilowatt-hours, with U as the voltage applied in Volts, I as the current in Ampere, and t as the duration of electro coagulation in hours. V stands for the amount of liquid measured in litres.

3 Characterization of Aluminium electrodes

3.1 Scanning Electron Microscope (SEM):

SEM is an instrument that uses electron beam to generate high resolution images of the surface of samples.

An SEM is an electron microscope that produces images of a specimen by scanning its surface with a focused electron beam. The electrons interact with atoms in the sample, producing various signals that provide information about the surface topography and composition of the sample. The electron beam is oscillated in a grid-like pattern, combining its position and signal strength to create an image. Secondary electrons produced by atoms being excited by the electron beam are detected using a secondary electron detector, known as the Everhart-Thornley detector, in the standard SEM mode. Specimen topography affects both signal intensity and the detectable number of secondary electrons. Various factors contribute to these influences [25].

3.2 Fourier Transform Infra-Red Spectroscopy, also known as FTIR:

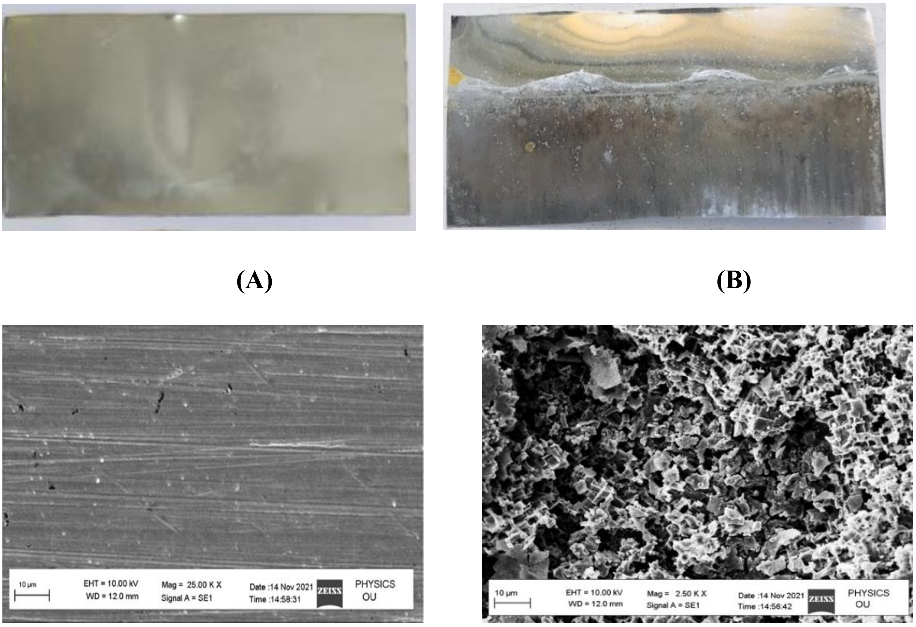
FTIR is a technique used to obtain an infrared spectrum of absorption or emission from a solid, liquid, or gas. An FTIR spectrometer gathers detailed data across a broad spectral range all at once. This gives a big benefit compared to a spectrometer that measures intensity within a limited set of wavelengths.

4 Results and Discussion

The data from experiments were generated in groups to study the impact of various factors such as Electrolysis time, NaCl concentration, Voltage, pH, and Initial concentration on the removal of Dicofol from a lab-made Dicofol solution. The study also involved examining the characteristics of the Aluminium electrode using SEM and FTIR. Dicofol removal was initially examined through Figical analysis, followed by an attempt to theoretically explain the findings from the same analysis. The study also included an analysis of Kinetics and Electrical energy usage [26].

4.1 Characterization studies:

SEM of Aluminium plate electrodes: SEM technology was utilized to investigate how the electrochemical Dicofol removal process affects the morphology of the Aluminium surface. Figure 4.1 displays the SEM images of the Aluminium plate electrode prior to and following the treatment process.



(A) **(B)**

(C) **(D)**

Fig 4: Aluminium plate electrode, (A) Aluminium sheet before EC process (B) Aluminium sheet after EC process (C) SEM image of before EC process and (D) SEM image of after EC process.

4.2 FTIR spectrum of Aluminium plate electrodes

The findings indicated the presence of numerous peaks, indicating the complex nature of the material. Figure 4.3 displays the FTIR spectra before and after analysis. The different functional groups are represented by peaks at various points. The spectra obtained for APE before EC showed appearance spectral peaks at 3472.72cm^{-1}, 3514.55cm^{-1}, 3736.07cm^{-1}, 3665.82cm^{-1} appearance has that correspond to O–H groups stretching in alcohols. The spectral peaks observed at 1639.08cm-1 are associated with C=C stretching and the type of compound is alkene. Additional peaks were detected at 1074.56cm-1, showing moderate intensity and indicating C-O stretching related to primary alcohol compound class. Peak also observed at 982.01cm^{-1} appearance has medium that can be attributed to C=C bending and compound class is alkene [27]. Peak also observed at 777.69cm^{-1}, appearance has medium that can be attributed to C–H bending and compound class is 1,2,3-trisubstituted.

The spectra for APE after EC revealed peaks at 3589.86 cm-1 that correspond to the stretching of O-H groups in alcohols. Spectral peaks at 3500.53cm^{-1} appearance has that correspond to N–H stretching in primary amine group. Spectral peaks which occurred at 1653.73cm^{-1} correspond to C=C stretching and compound class is alkene. Additional peaks were also noticed at 1070.38cm-1, with a medium intensity indicating C – O stretching which suggests the compound belongs to the primary alcohol class.

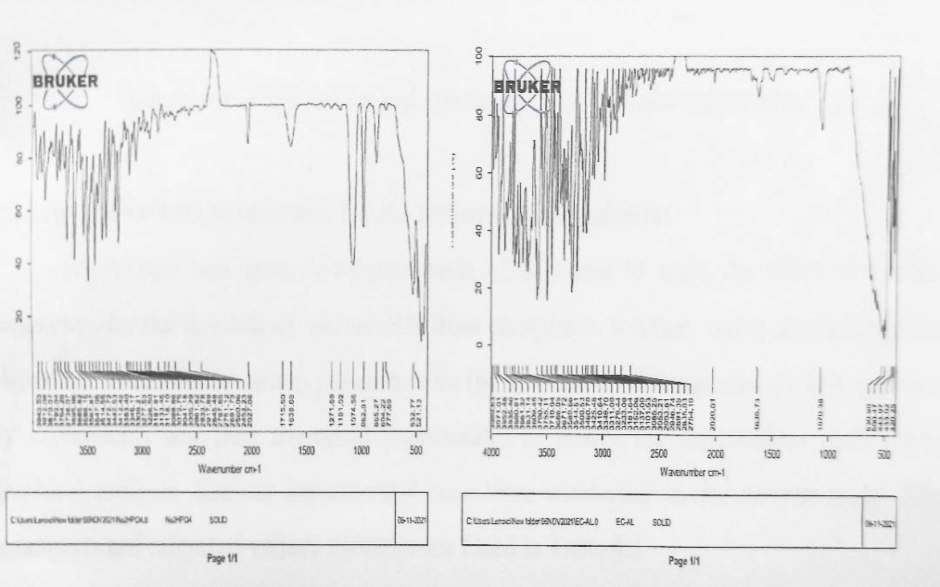


Fig 5: FTIR before and after analysis of Aluminium plate electrodes

4.3 Impact of different factors on Dicofol Elimination:

An investigation was conducted in batch operation mode to analyse the impact of different factors on eliminating Dicofol from a solution containing Dicofol by utilizing aluminium plate electrodes. Experiments are initially used to analyse the impact of different parameters on the removal of Dicofol, with attempts made to theoretically explain the observations obtained from Figical analysis. Different trials were carried out in the current investigation. Table 1 lists the parameters and ranges of different variables.

Table 1. Experimental parameters

Variables	Symbols	Units	Range	
			From	**To**
Electrolysis time	t	min	1	45
NaCl concentration	-	g/L	0.5	3.0
Voltage	V	Volt	5	30
pH	pH	-	4	11
Initial concentration of Dicofol	C_o	ppm	10	100

4.4 Effect of Electrolysis time:

The amount of time needed to decrease concentration of Dicofol sample was estimated by taking 1000ml of sample in an electrolytic cell and Aluminium, electrodes were kept in electrocoagulation experimental setup.

The study focused on eliminating Dicofol using Aluminium plate electrodes within a time frame of 0 to 35 minutes. The initial Dicofol concentration was 20 mg/l, with a pH of 7, voltage of 10 volts, and NaCl concentration of 1.0gm/L. Dicofol concentration was measured every 5 minutes. There was no variation in the percentage of Dicofol removed with extended time beyond 25 minutes. Most of the Dicofol elimination occurred when the electrolysis time was at optimal levels (35 minutes), as indicated in Table 6. 66.41% was the rate of Dicofol removal achieved.

The % Removal of Dicofol are calculated as follows,

$$\%\ \text{Removal of Dicofol} = \frac{(C_O - C_t)}{C_O} * 100 \quad (4.1)$$

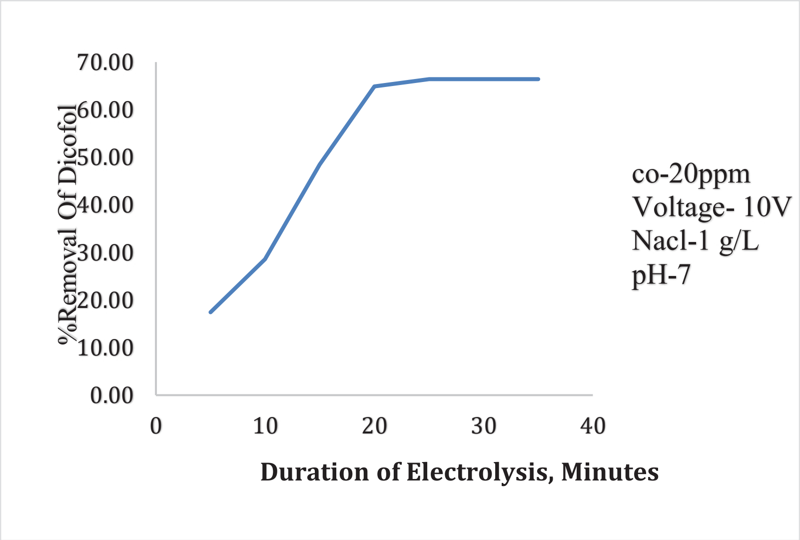


Fig 6. Impact of the duration of electrolysis on the percentage of Dicofol removed using APE.

4.5 Impact of sodium chloride levels:

Different concentrations of NaCl were added as a conductive electrolyte to enhance the solution's conductivity. The balance contact period was 35 minutes with 25 minutes of APE. Figures 4.3 and 4.4 showed that as the NaCl quantity rises, the removal efficiency also increases. The quantity of Aluminium produced rose quickly as the NaCl level changed from 0.5 to 3 g/L. This could be attributed to the higher chloride ion levels, resulting in a stronger ability of Cl^- to break down any passive oxide layer on the anode, leading to an increase in Al(OH)3 in solution and enhancing the efficacy of Dicofol elimination. The inclusion of NaCl would also result in a reduction in power consumption as a result of the rise in conductivity.

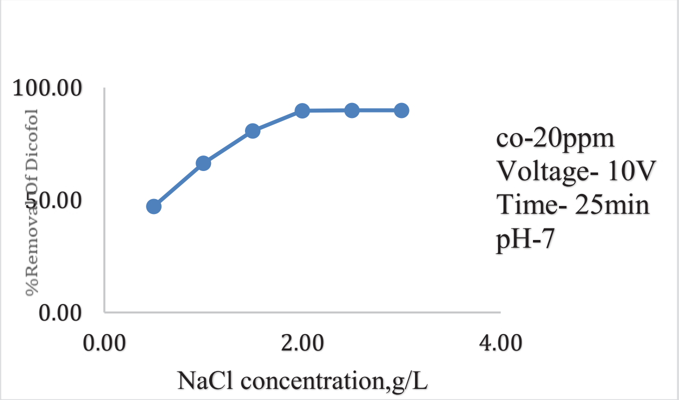


Fig 7. Impact of different NaCl concentrations on the percentage of Dicofol removal using APE

4.6 Voltage's impact:

The impact of voltage was assessed by using a 1000ml sample for each procedure at every voltage level within the 5V to 30V range with an interval of 5V and remaining parameters maintained at optimum time, optimum NaCl concentration, pH 7 and 20 ppm of Initial concentration. Voltage increases, the removal percentage of Dicofol also increases and becomes stable at 20V and 10V for APE. The maximum percentage removal of Dicofol is as shown in table 4.6.

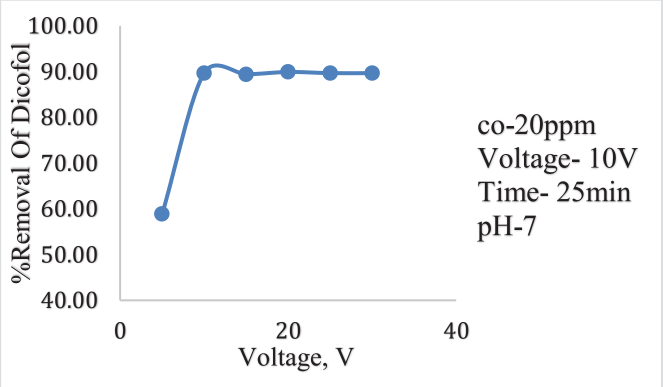


Fig 8: Impact of electrical potential on percentage of Dicofol elimination using APE

4.7 Effect of pH:

pH plays a critical role in determining the efficiency of electrochemical processes. Figures 4.7 display how the percentage of Dicofol removed with APE is influenced by pH.

The percentage of Dicofol removal by APE increased when pH increased from 4 to 6, but decreased when pH increased up to 11 as indicated in table 4.8. Therefore, the ideal pH was set at 6.

According to Figures 4.7, the greatest removal effectiveness for APE is reached at pH 6. The decreased Dicofol removal rates at more extreme acidic and alkaline pH levels are due to the amphoteric nature of Al(OH)3, preventing precipitation at very low pH levels. As the pH levels rise, the presence of OH- ions becomes more competitive, resulting in a lower percentage of Dicofol removal. Bridging of cations present at the electrode's surface can lead to high pH values. The percentage of Dicofol removed decreases significantly when the pH is above 8 due to the increased negative charges on the electrode's surface.

The rise in pH in acidic conditions was caused by H2 production at the cathodes, while the formation of Al(OH)3 near the anode would result in the release of H+ and ultimately lead to a decrease in pH. Moreover, the O2 evolution reaction also results in a decrease in pH.

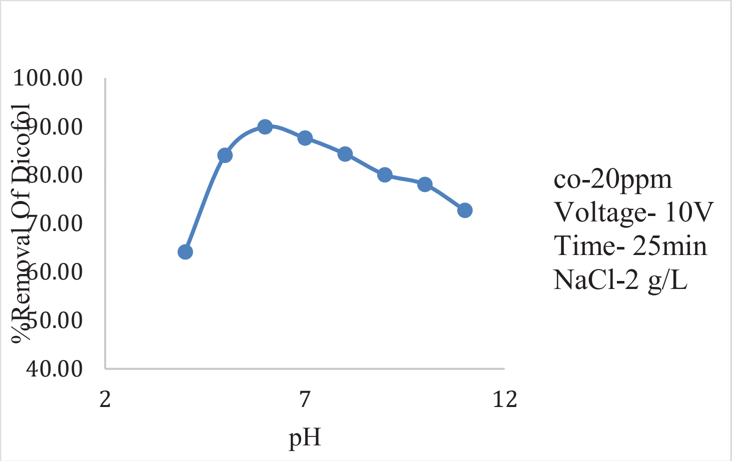


Fig 9: Effect of pH on % removal of Dicofol using APE

4.8 Effect of Initial concentration of Dicofol:

Tests were conducted to examine how the initial concentration impacts the elimination of Dicofol from the Dicofol solution. The obtained results are displayed in tables 4.10. Figures 4.9 display the relationship between the percentage removal of Dicofol and the initial concentration of Dicofol. It was noted in the graph that as the initial concentration of Dicofol increased, the percentage of Dicofol removed decreased. The removal percentage of Dicofol for APE decreased from 91.92% to 30.81%.

At a consistent current density, the solution allows the same amount of Aluminum ions to pass through at varying Dicofol concentrations [28]. As a result, there were not enough

Aluminium hydroxides formed to cause the coagulation of a larger amount of Dicofol molecules at higher Dicofol concentrations.

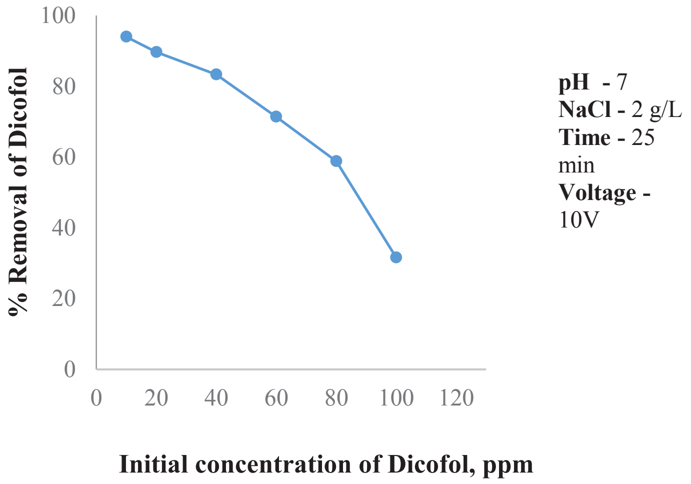


Fig 10: Effect of initial concentration of Dicofol on % removal of Dicofol using APE

4.9 Kinetic studies of pseudo first order:

Predicting the rate at which Dicofol is removed provides crucial data for designing a batch Electrocoagulation system. Knowledge of the kinetics is necessary to choose the best operational parameters for a complete batch process. In addition, the kinetic analysis showed that the elimination of Dicofol is in line with a pseudo first order model.

4.10 Kinetic studies on Aluminium plate electrodes

(Concentration = 20ppm, Voltage = 20V, pH = 6, NaCl = 2.5g/L)

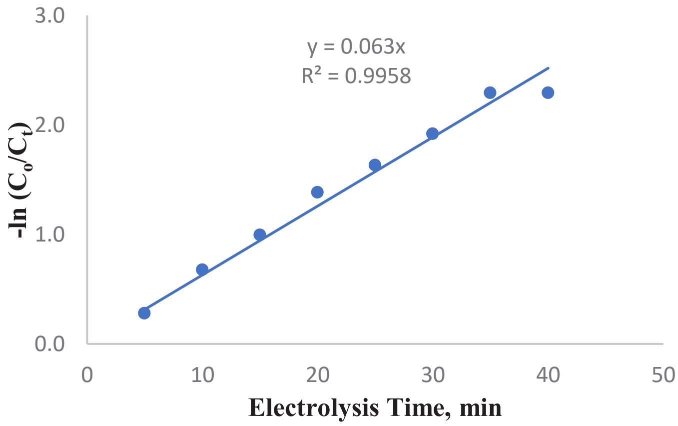


Fig 11: Pseudo first order kinetics in the elimination of Dicofol with the use of APE

- Equation $$-\,ln\left(\frac{C_t}{C_o}\right) = K_1 t$$
- A Fig is drawn between -ln (C_t/C_o)) vs t:
  slope, K_1 = 0.063 min^{-1}
  R^2 = 0.996
- The equation obtained:
  $$-\,ln\left(\frac{C_t}{C_o}\right) = 0.063t$$

4.11 Electrical energy consumption:

From equation 3.3, the relationship is given by

$$E = \frac{U*i*t}{V} \quad (\text{kWh/m}^3)$$

In the formula, E represents the electrical energy used per cubic meter in kilowatt-hours, U is the voltage in Volts, I is the current in Ampere, and t is the electro coagulation time in hours. V represents the amount of liquid in litres.

4.12 Electric energy consumption for removal of Dicofol using APE

The lowest energy usage recorded was 7.29 kWh/m3 during the process of eliminating Dicofol using 10V voltage, 1.75 A current within a one-litre volume with a 25-minute electrolysis duration. [29].

5 Conclusion

The current research explored the elimination of Dicofol through the Electrocoagulation technique. Electrocoagulation, involving extended residence time and elevated current, greatly enhances the percentage of Dicofol removal from water solution.

SEM analysis is used to examine the characterization of Aluminium electrodes both before and after the Electrocoagulation process. The FTIR analysis is used to study the characterization of Dicofol, both before and after the Electrocoagulation process.

APE achieved the highest removal percentage of phosphates at 93.94% by optimizing the electrolysis time to 25 minutes, NaCl concentration to 2 g/L, voltage to 10, pH to 7, and initial phosphate concentration to 10 ppm.

From the results of kinetic study, the pseudo first order rate constant was obtained as K1 = 0.082min^{-1} for Aluminium plate electrodes.

It was discovered that the amount of electrical energy needed for the elimination of Dicofol through electrocoagulation was 7.29kWh/m3 when using Aluminium plate electrodes.

Finally, it was concluded that the performance of Aluminium plate electrode shows more efficient for the removal of Dicofol.

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