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Research Article

Monitoring the Electrochemical Breakdown of Acetamiprid by Differential Pulse Polarography

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ABSTRACT

This study investigates the electrochemical mineralization of the neonicotinoid insecticide acetamiprid in aqueous environments using indirect electro-oxidation. Differential pulse polarography (DPP) with a dropping mercury electrode was used to characterize the electroanalytical behavior of acetamiprid, showing optimal reduction at pH 2.2 with a peak potential of -1.064 V. The DPP method demonstrated a linear detection range of 1.0 to 16.0 µg mL^{-1} with an LOD of 0.940 µg mL^{-1}. Electrochemical degradation experiments were conducted in NH_4Cl electrolyte using platinum electrodes at current densities ranging from 50 to 200 mA. Mineralization efficiency increased with current density and initial insecticide concentration, reaching up to 96.40%. The degradation followed first-order kinetics, and electrical energy consumption was evaluated. The study highlights the significant influence of current density and electrolyte concentration on degradation rates, confirming the efficacy of indirect electrochemical oxidation for pesticide removal. These findings contribute to optimizing reactor conditions for efficient aquatic pollutant remediation.

Keywords*:* Electrochemical mineralization, Acetamiprid, Differential pulse polarography, Indirect electro-oxidation, Degradation, Environmental remediation.

INTRODUCTION

The growing need for food security has led to the extensive use of synthetic pesticides in agriculture. Neonicotinoids, a class of insecticides resembling nicotine, are widely used due to their systemic action and specificity for insect nicotinic acetylcholine receptors [1,2]. Among them, acetamiprid is commonly applied to vegetables, fruits, and cotton [3,4]. Its high-water solubility (2950 mg/L), chemical stability, and resistance to biodegradation make it a persistent pollutant in surface and groundwater [5,6]. Studies report the occurrence of neonicotinoids in rivers, lakes, and drinking water, posing ecological and human health risks [7–9]. Conventional water treatments, such as chlorination and biological processes, are often ineffective due to the recalcitrant nature of these compounds [10–12]. Hence, advanced oxidation processes (AOPs) that generate reactive radicals like hydroxyl and sulfate radicals are being explored [13–15]. Electrochemical oxidation, a promising AOP, offers environmentally friendly degradation under ambient conditions and precise redox control [16–19]. Degradation occurs via direct electron transfer or indirect oxidation using electro-generated agents like hydrogen peroxide or active chlorine [20–22]. Research on various electrode materials, including boron-doped diamond (BDD), platinum, graphite, and titanium, has shown promising pesticide degradation results [23–25]. BDD generates powerful hydroxyl radicals, while platinum offers moderate efficiency and cost-effectiveness [26, 27]. Factors such as pH, current density, electrode material, and temperature greatly influence degradation efficiency [28–30]. Though less studied than other neonicotinoids, electrochemical degradation of acetamiprid has shown significant mineralization and reductions in TOC and COD under optimized conditions [31–33]. However, knowledge gaps remain regarding degradation kinetics and by-product identification [34, 35]. This study investigates the electrochemical degradation of acetamiprid using a platinum electrode under varying current densities and pH. The goal is to evaluate degradation efficiency, identify intermediates, and support green electrochemical water treatment.

MATERIALS AND METHODS

Chemicals and Reagents

Analytical grade reagents were used without further purification. Acetamiprid, supporting electrolytes (NH_4Cl, CH_3COONa, NaCl, NH_4NO_3, and KCl), acids (HCl, CH_3COOH, H_3PO_4, and H_3BO_3), bases (NaOH), and organic solvents (dichloromethane, hexane, and ether) were sourced locally. Distilled water was used throughout. Britton-Robinson Buffer (0.04 M)**:** Prepared by dissolving boric acid, glacial acetic acid, and orthophosphoric acid in water; pH adjusted with 1.0 M NaOH or HCl (buffer range pH 2.0–11).

Instrumentation

Differential Pulse Polarographic measurements were performed using a CL-362 polarographic analyzer (Elico Ltd., Hyderabad) connected to a PC via RS-232C and operated with ELICO's Windows-based software. A dropping mercury electrode (working), Hg/Hg_2Cl_2. KCl as the reference electrode, and a platinum wire (auxiliary) was used. pH was measured using an Elico pH meter. UV-vis absorbance of neonicotinoids in BRB solution was recorded using a PerkinElmer Lambda 25 spectrophotometer.

Preparation of Solutions

A 1.0 mg/mL stock solution of acetamiprid insecticide was prepared in suitable solvents. Working solutions were diluted with distilled water. Britton–Robinson (BRB) buffers were prepared using AR-grade chemicals for pH adjustments.

Experimental Setup

Experiments were carried out in a 100 mL undivided batch cell using vertically placed platinum electrodes (1.0 cm^2 area, 1.0 cm gap). The setup included three acetamiprid solutions with supporting electrolytes (NH_4Cl, CH_3COONH_4, and CH_3COONa) to enhance conductivity and reduce electrolysis time. The cell was immersed in a water-filled glass bowl to maintain constant temperature, preventing volatilization losses. The solution was stirred at 200 rpm with a magnetic stirrer for uniform mixing (Figure 1). A DC power supply provided a stable current. Conditions for optimal electrocatalytic degradation were systematically investigated.

RESULTS AND DISCUSSION

Choice of Electrolytes

Since acetamiprid are not inherently electroactive, supporting electrolytes were necessary to facilitate degradation. Experiments were conducted using NH_4Cl, CH_3COONH_4, and CH_3COONa (2 g/L), under constant conditions: 200 mA current, 25 °C temperature, 6-hour electrolysis, and 0.01 mg/cm^3 insecticide concentration. Ammonium chloride (NH_4Cl) showed the highest degradation efficiency with over 85% degradation. In contrast, CH_3COONa showed the lowest efficiency. Based on these results, NH_4Cl was selected as the optimal electrolyte.

Choice of Electrode

Electrode material critically influences the efficiency and stability of electrochemical mineralization. Various electrodes were tested in an undivided cell using 0.01 mg/mL neonicotinoid acetamiprid and 100 mA current in NH_4Cl electrolyte. Platinum electrodes showed the best results, offering high mineralization efficiency and excellent stability. Carbon electrodes gave good degradation but suffered from anode swelling. Copper, steel, and silver electrodes showed poor stability due to rapid erosion or sol formation. Thus, platinum was selected as the optimal electrode material.

Method development and Validation

Instrumental and experimental parameters were optimized during method development to achieve maximum electrochemical response of acetamiprid, with emphasis on peak current at its corresponding peak potential. Differential pulse polarography (DPP) was utilized to study the electrochemical behavior and quantify acetamiprid using a dropping mercury electrode. The validated method was successfully applied for the analysis of acetamiprid in various samples. Acetamiprid contains a cyanoimine functional group, which plays a crucial role in its redox behavior and stability in aqueous solutions. Britton-Robinson (BR) buffer was used as the supporting electrolyte to evaluate the compound's electrochemical response over a broad pH range. Well-defined reduction peaks were observed in the acidic to near-neutral range (pH 2.0–7.0), corresponding to the reduction of nitro or cyano groups (Figure 2). The method showed good linearity in the concentration range of 4.49×10^{-7} to 1.07×10^{-5} M, with a correlation coefficient (R^2) of 0.990. The limit of detection (LoD) was found to be 0.9408 μg/mL (4.224×10^{-6} M), and the limit of quantification (LoQ) was 3.136 μg/mL (1.40×10^{-5} M). The regression equation obtained was $Ip = 0.099\ CAc + 0.0489$. Differential pulse polarographic (DPP) curves of acetamiprid at concentrations ranging from 1.0 to 10.0 μg/mL recorded in Britton-Robinson buffer (BRB) at pH 2.2, along with the corresponding linear calibration plot of peak current (Ip) versus acetamiprid concentration, are shown in Figure 3.

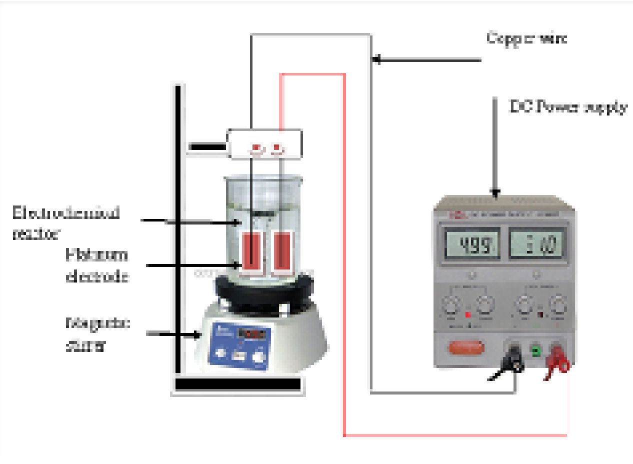

Figure 1: Electrochemical setup for mineralization of acetamiprid insecticide

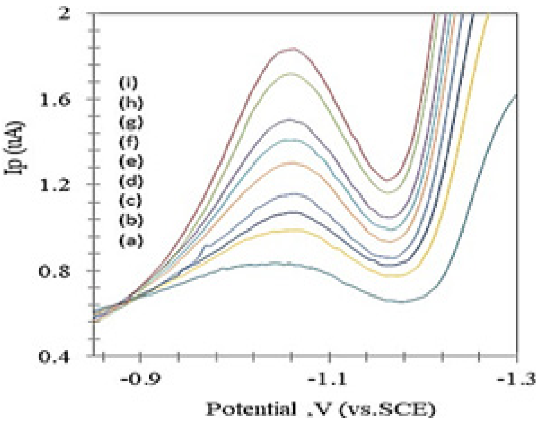

Figure 2: DPP curves of acetamiprid (1.0–10.0 μg/mL) at pH 2.2 in BR buffer

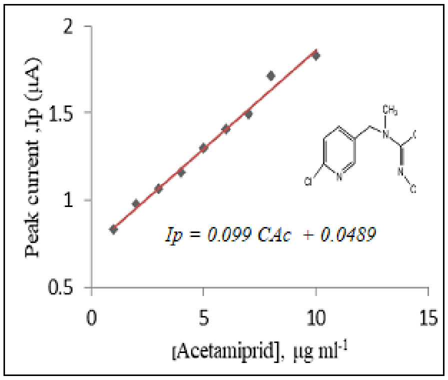

Figure 3: Linear plot of Ip versus concentration of acetamiprid in BRB at pH 2.2

Electrochemical Degradation Mechanism

During the electrolysis of ammonium chloride (NH_4Cl) solutions in pesticide-contaminated water, chloride ions (Cl^-) at the anode are oxidized to chlorine gas (Cl_2), which then hydrolyzes to form hypochlorous acid (HOCl) and hypochlorite ions (OCl^-) potent oxidizing agents known to degrade various organic pollutants, including pesticides [39]. Simultaneously, the anodic oxidation of water generates hydroxyl radicals (•OH), which are highly reactive and non-selective species capable of breaking strong chemical bonds within acetamiprid molecules, leading to their mineralization [37]. Furthermore, ammonium ions (NH_4^+) present in the electrolyte react with chlorine to produce chloramines (NH_2Cl, $NHCl_2$), which act as secondary oxidants, extending the degradation process [38]. At the cathode, water reduction produces hydrogen gas (H_2) and hydroxide ions (OH^-), increasing solution alkalinity and influencing the speciation of chlorine and chloramines [40]. The key electrode and solution-phase mechanisms involved in the electrochemical degradation of acetamiprid are summarized in Table 1. This combination of reactive chlorine species, chloramines, and hydroxyl radicals leads to effective acetamiprid oxidation and breakdown into less harmful substances or complete mineralization to carbon dioxide, water, and inorganic salts, making the electrochemical process a promising approach for insecticide removal in water treatment [36].

Table 1: Electrode and solution-phase mechanisms in the electrochemical degradation of acetamiprid

Electrode	*Reaction*	*Role in acetamiprid degradation*
Anode	$2\ Cl^- \rightarrow Cl_2 + 2e^-$	Produces chlorine, HOCl, OCl^- (oxidants)
Anode	$H_2O \rightarrow {}^{\bullet}OH + H^+ + e^-$	Produces hydroxyl radicals (strong oxidants)
Cathode	$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$	Generates an alkaline environment
In Solution	$NH_4^+ + Cl_2 \rightarrow$ Chloramines	Additional oxidants for degradation

Current density plays a key role in electrochemical degradation. While Murphy *et al.* (1992) found no significant effect of current density in direct oxidation, Chiang *et al.* (1995) reported improved chlorine/hypochlorite production and thus better pollutant removal with increased current density in indirect oxidation. In this study, acetamiprid was degraded in an ammonium chloride electrolyte using platinum electrodes over 7. In the present study, the indirect electro-oxidation of acetamiprid insecticides from aqueous solutions at various initial concentrations was investigated using platinum electrodes (as both anode and cathode) in an NH_4Cl electrolyte. The process was carried out for 7.5 hours under varying current densities of 50, 100, 150, and 200 mA. The results, illustrated in Figures 4-6, indicate that at an initial acetamiprid concentration of 0.01 mg mL^{-1}, the degradation efficiency of acetamiprid increased from 74.59% at 50 mA to 90.76% at 200 mA.

Although higher current densities led to increased degradation across different initial concentrations of neonicotinoids, the improvement was not directly proportional to the increase in current density. The electrical energy consumption associated with the degradation of 0.01 mg mL^{-1} neonicotinoids at current densities of 50, 100, 150, and 200 mA was calculated to be 0.07812×10^{-3}, 0.333×10^{-3}, 0.3125×10^{-3}, and 0.2916×10^{-3} kWh m^{-3}, respectively, for a reaction duration of 7.5 hours.

Electrochemical mineralization of neonicotinoids

The electrochemical mineralization of three neonicotinoids acetamiprid, was studied using differential pulse polarography (DPP).

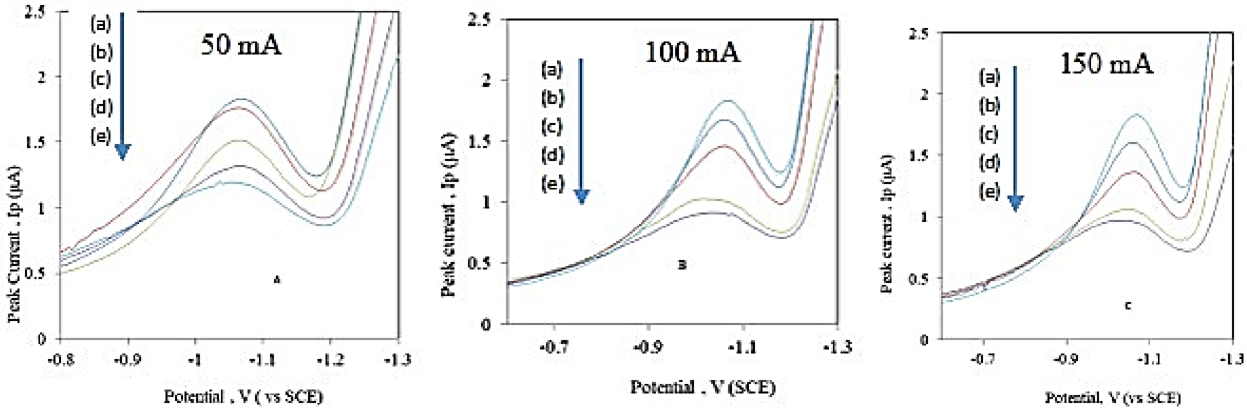

Figure 4: Differential pulse polarogram of electrochemical mineralization of 0.010 mg$mL^{-1}$of acetamiprid at different current densities. [A] 50 mA, [B] 100 mA, [C] 150 mA and [D] 200 mA.Time of electrolysis a) 0.00 hours (b) 1.0 hours (c) 2.5 hours (d) 5.0 hours and (e) 7.5 hrs

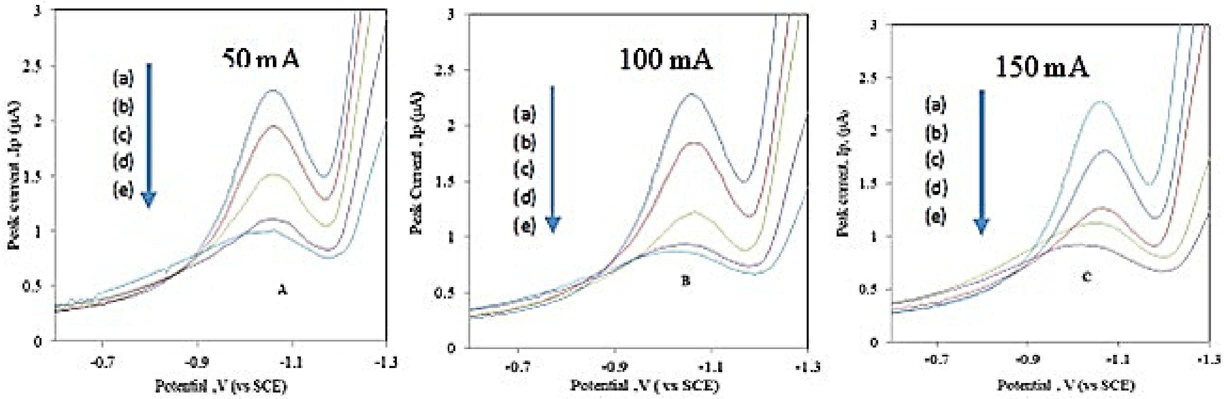

Figure 5: Differential pulse polarogram of electrochemical mineralization of 0.015 mgmL^{-1} of acetamiprid at different current densities. [A] 50 mA, [B] 100 mA and [C] 150 mA. Time of electrolysis (a) 0.00 hours (b) 1.0 hours (c) 2.5 hours (d) 5.0 hours and (e) 7.5 hours

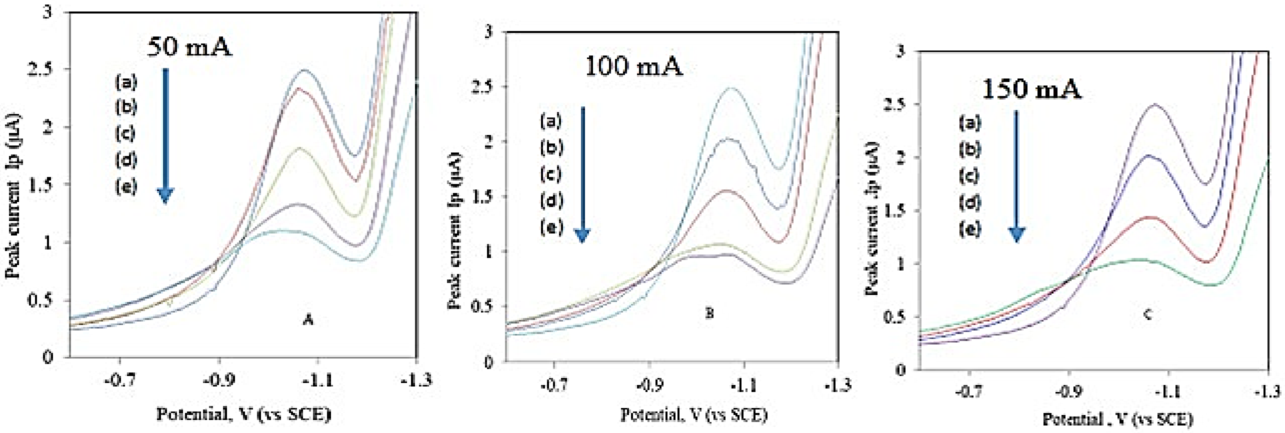

Figure 6: Differential pulse polarogram of electrochemical mineralization of 0.020 mgmL^{-1} of acetamiprid at different current densities. [A] 50 mA, [B] 100 mA and [C] 150 mA. Time of electrolysis (a) 0.00 hours (b) 1.0 hours (c) 2.5 hours (d) 5.0 hours and (e) 7.5 hours

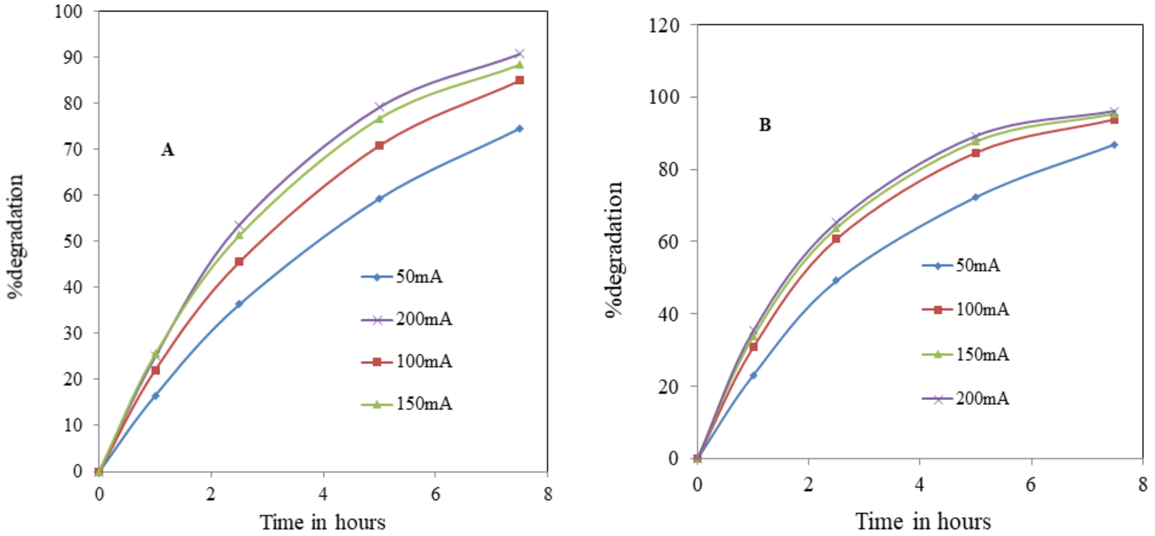

Figure 7: Electrochemical mineralization of acetamiprid insecticide at different current densities and concentrations [A] 0.010 mgmL^{-1}and [B] 0.015 mgmL^{-1}.

This technique was used to measure peak currents at optimized parameters before and after electrolysis, following the extraction of the compounds in dichloromethane. Mineralization efficiency was calculated using the relation (Figure 7):

$$\%E = \frac{C_0 - C_t}{C_0} \times 100$$

Where C_0 is the initial concentration and C_t is the final concentration value at a time (t) of the neonicotinoids solution, respectively. The indirect electro-oxidation was carried out in NH_4Cl electrolyte using platinum electrodes (anode and cathode) at a current density of 50 mA for 7.5 hours. Initial insecticide concentrations of 0.01, 0.015, and 0.020 mg mL⁻¹ were tested. As the initial concentration increased, mineralization efficiency also improved. For acetamiprid, the efficiency rose from 74.59% at 0.01 mg mL⁻¹ to 86.90% at 0.020 mg mL⁻¹, as presented in Table 2. These results confirm the effectiveness of indirect electrochemical

Electrochemical kinetics study of neonicotinoids

The electrochemical kinetics of the acetamiprid insecticide were investigated through the electrolysis of ammonium chloride (NH_4Cl) in pesticide-contaminated water. The combination of chlorine-based oxidants, chloramines, and hydroxyl radicals facilitates effective oxidation and mineralization of neonicotinoids into less harmful substances, such as carbon dioxide, water, and inorganic salts, making this electrochemical process a promising method for pesticide removal in water treatment. The kinetics of this degradation process follows a pseudo-first-order reaction model. Although the overall reaction is second-order, depending on both the pesticide concentration and the concentration of chlorine species, the latter is considered constant during electrolysis. This assumption simplifies the rate law to a pseudo-first-order form, where the degradation rate is directly proportional to the concentration of the pesticide. The kinetic expression is given as:

$$-d\,[PEST] / dt = k\,[PEST]$$

Table 2: %Removal of acetamiprid insecticide at different current densities and concentrations after 7.5 hours

Current density (mA)	[0.010 $mgmL^{-1}$.]	[0.015 $mgmL^{-1}$].
50	74.59	86.90
100	85.00	93.84
150	88.45	95.38
200	90.76	96.14

Table 3: Electrochemical degradation kinetic study of acetamiprid.

Current density (mA)	*Regression equation*	*k ($h^{-1)}$*	*$T_{1/2}$(hrs)*	*R^2*
	[Acetamiprid]= 0.010 $mgmL^{-1}$ ($4.490x10^{-6}$M)			
50	y=0.079x-0.001	0.1809	3.830	0.999
100	y=0.109x-0.003	0.2480	2.794	0.999
150	y=0.128x+0.001	0.2957	2.346	0.999
200	y=0.138x-0.008	0.3098	2.228	0.999
[Acetamiprid]= 0.015 $mgmL^{-1}$ ($6.735x10^{-6}$M)				
50	y=0.116x-0.003	0.2641	2.624	0.998
100	y=0.161x+0.00	0.3707	1.869	1.00
150	y=0.179x+0.00	0.4122	1.681	0.999
200	y=0.189x-0.001	0.4342	1.596	0.999

The experimental data supporting this kinetic model, showing percentage removal of acetamiprid at different current densities and concentrations after 7.5 hours, are presented in Table 2.

The degradation rate constant k can be determined by using the following first-order rate equation

$$\log C_0/C_t = k\,t$$

The slope of the plot (not shown) of log (C_t/C_0) versus time (t) represents the rate constant *k* (in min⁻¹). Here, *C₀* denotes the initial concentration of neonicotinoids (mg/L), while *Ct* is the concentration remaining at time *t*. Table 3 presents the calculated rate constants and corresponding half-lives for acetamiprid. The high correlation coefficients (greater than 0.98) strongly support the assumption

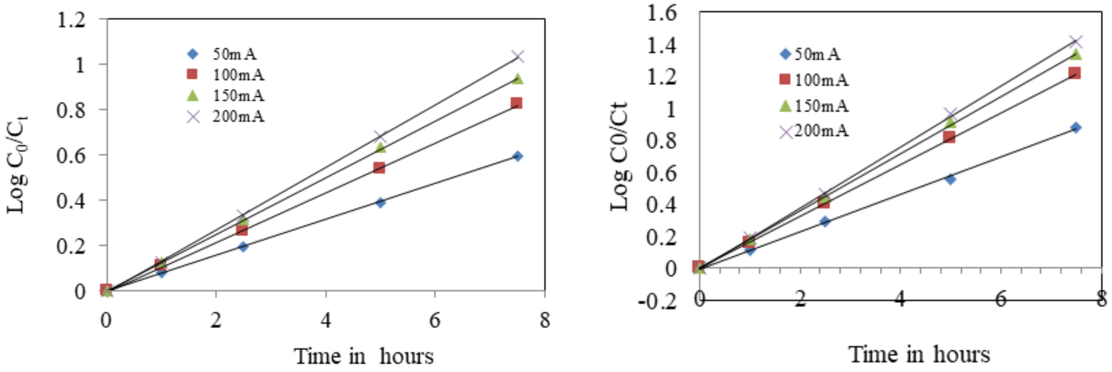

Figure 8: Electrochemical degradation kinetics of acetamiprid insecticide at various current densities at (A) [Acetamiprid] = 0.010 $mgmL^{-1}$ ($4.490x10^{-6}$M) and (B) [Acetamiprid] = 0.015 $mgmL^{-1}$ ($6.735x10^{-6}$M)

that the degradation of neonicotinoids follows a pseudo-first-order kinetic model.

Two kinetic parameters, the effect of current density and electrolyte concentration, were studied in relation to acetamiprid insecticides to determine the optimal operating conditions for the electrochemical reactor to be developed in the next phase. The influence of current density and electrolyte concentration on the degradation rates of three neonicotinoids was investigated individually graphically illustrated in Figure 8. Additionally, degradation behavior under varying conditions was examined to gain a deeper understanding of the reaction dynamics within the reactor. Efforts were also made to identify any mass transfer limitations present in the system.

CONCLUSION

This study successfully developed and validated an electrochemical approach for the mineralization of the neonicotinoid insecticide acetamiprid in aqueous solutions. Differential pulse polarography (DPP) was employed to track the degradation process. The electrochemical behavior of acetamiprid was initially characterized using a dropping mercury electrode (DME) with a calomel reference electrode in Britton–Robinson buffer, revealing an optimal reduction peak at pH 2.2 and a peak potential of -1.064 V. Indirect electro-oxidation was performed using platinum electrodes in an NH_4Cl electrolyte across various current densities. Results showed that degradation efficiency increased with both current density and initial acetamiprid concentration, though the relationship was not strictly linear. Kinetic analysis indicated a first-order reaction mechanism. Mineralization was confirmed by comparing DPP peak currents before and after electrolysis, following dichloromethane extraction. Additionally, the effects of current density and electrolyte concentration were separately investigated to optimize reactor conditions and assess potential mass transfer limitations.

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