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Electrochemical Determination of Propiconazole Residues in Grain Samples by Voltammetry at Carbon Nanotubes Paste Electrodes

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Abstract- In this study the residues of propiconazole in grains samples determined on the basis of its reduction behavior at carbon nanotubes paste electrodes. The reduction product is identified by using cyclic voltammetry and the universal buffer with pH range 2.0-6.0 is used as supporting electrolyte. The best curve and highest peak current are obtained in universal buffer at pH 4.0. Accumulation potential, Accumulation time and Scan rate were optimized. Calculations made of by standard addition method. The peak heights are in linear trend over the concentration range of 1.02×10^{-9} to 1.05×10^{-10} M. The relative standard deviation and correlation coefficient for propinacazole is 0.97% and 0.998 respectively.

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I. INTRODUCTION

Pest is any species that competes with human being for food, invades lawns and gardens, destroys wood in houses, and spreads disease or simply a nuisance. Worldwide about 100 species of plants, (which one calls weeds); animals (mostly insects), fungi and microbes (which can infect crop plants and livestock animals) cause about 90% of the damage to the crops. Insects cause much of the damage to the crops. Pesticides are designed to target specific pests, such as weeds, fungi, insects and parasites, but they can kill other species if they enter the wider environment. The manufacture, sale and use of pesticides are highly regulated, but, even so, pesticides can be detected widely at low levels in many parts of the environment.

Propiconazole ($C_{15}H_{17}Cl_2N_3O_2$) is broad spectrum fungicide (conazole fungicides). Defferent methods are employed for determination of propiconazole residues in environmental samples Lopez et al. [1] determined fungicides carbendazim, metalaxyl, folpet, and propiconazole in must and wine by using HPLC method. Karina et al. [2] determined Propiconazole Residue in Boronia Extract by using gass chromatography/mass spectroscopy. But there is no literature fond for the determination of propiconazole residues in environmental samples by using electro

chemical methods such as polarography, voltammetry. Rasekhar reddy et al. [3-7] employed voltametry for the determination of oxabetrinil residues in environ mental samples. In this work deferential pulse adsorptive stripping voltammetry employed [8-12]. for the determination of propiconazole residues in vegetables.

II. INSTRUMENTS AND REAGENTS

This investigation performed by using a model meterohm Auto Lab 101 PG stat (Netherlands). CNTPE was used as working electrode for differential pulse adsorptive stripping voltammetry and cyclic voltammetry. pH measurements were carried out with an Eutech PC_510 cyber scan. Meltzer Toledo (Japan) Xp26 delta range micro balancer were used to weigh the samples during the preparation of standard solutions. All the experiments were performed at 250C.

All reagents used are analytical reagent grade. Double distilled water was used throughout the analysis. In the present investigation universal buffers of pH 4.0 are used as supporting electrolytes and are prepared by using 0.2 M boric acid, 0.05M citric acid and 0.1M trisodium orthophosphate solutions.

III. RESULT AND DISCUSSION

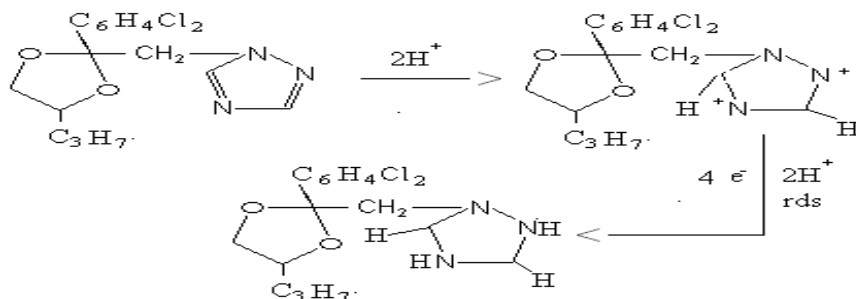
Propiconazole exhibits a single well-defined peak in the pH range 2.0 to 6.0 (table 2.0) when potential was scanned from -1.05 V and so on at CNTPE (Fig.1.0) which is due to the reduction of the azomethine groups by taking 4 electrons. The experimental parameters that affect the AdSV signal carried out by considering stripping peak in order to establish the optimum conditions. Both standard addition and calibration methods are employed for the determination of propiconazole in grains.

The best curve and highest peak current was obtained in universal buffer at pH 4.0. An enhanced adsorption peak obtained at potential -0.60V. The accumulation time of 70 sec. is used for further studies.. The reduction peak current increased linearly with the scan rate over the range from 25 mVs^{-1} to 75 mVs^{-1} . Better sensitivity was observed at 50 mVs^{-1} .

Electrode mechanism: Based on the results obtained from all the techniques, the electro-chemical reduction mechanism is as follows.

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Figure

IV. RECOVERY EXPERIMENTS

a) Analysis

Adsorptive stripping voltammetry is a suitable technique for the analysis of propiconazole due to its high sensitivity and resolution. The well resolved stripping voltammetric peak for the analytical purpose was obtained at pH 2.0 to 6.0. The peak heights are in linear trend over the concentration 1.02×10^{-9} to 1.05×10^{-10} M. The relative standard deviation and correlation coefficient for propinacazole is 0.97% and 0.998 respectively.

b) Determination of propiconazole in spiked grain samples

The developed analytical procedure has been applied to the quantitative estimation of propiconazole in grain samples. Grain (wheat, rice) samples sprayed with known amounts of propiconazole and left for 1-2 hours. Then the samples are weighed, crushed and homogenized. The extracts were prepared by treatment

of the above sample with two 50 mL portions of acetone and evaporated to dryness. The residue of dissolved in methanol and transferred to a 100 mL volumetric flask. 1.0 mL of standard solution is transferred in to cell and made up with 9 mL of supporting electrolyte and then purged with oxygen free N_2 gas for 10 min. prior to each run. After obtaining the voltammogram, small addition of standard solutions are added and voltammogram recorded after each addition under similar experimental parameters. Results obtained for the determination of propiconazole. The results are summarized in Table.2.0

V. CONCLUSIONS

In this approach for the determination of pesticide residues carbon nano tubes paste electrode is satisfactory applied to interpret the instrumental outputs without considerable errors and during the estimations pollution arises due to heavy metal electrodes such as mercury electrodes is avoided by using carbon electrodes.

Table 1.0: Typical cyclic voltammetric data of propiconazole, concentration: 1.0×10^{-5} M, scan rate: 50 mVs^{-1}

pH of the supporting electrolyte	$\frac{-E_p}{V}$	$\frac{i_p}{\mu A}$	αn_a	$\frac{D \times 10^6}{\text{cm}^2 \text{ s}^{-1}}$	$\frac{k^0_{f,h}}{\text{cm s}^{-1}}$
2.0	0.95	8.2	0.45	1.50	4.10×10^{-9}
4.0	0.99	8.8	0.36	1.48	8.92×10^{-10}
6.0	0.94	8.1	0.28	1.28	6.23×10^{-12}

Table 2.0: Recoveries of propiconazole in spiked grain samples

Sample	Amount added ($\mu\text{g/mL}$)	Amount found ($\mu\text{g/mL}$)	Recovery (%)	Standard deviation
wheat	2.0	1.98	99.00	0.0512
	6.0	5.99	99.83	0.0952
rice	3.0	2.98	99.33	0.004
	5.0	4.97	99.40	0.071

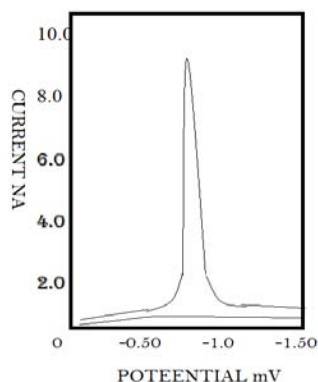


Fig. 1.0: Typical differential pulse adsorptive stripping voltammogram of propiconazole at CNTPE at pH 4.0.

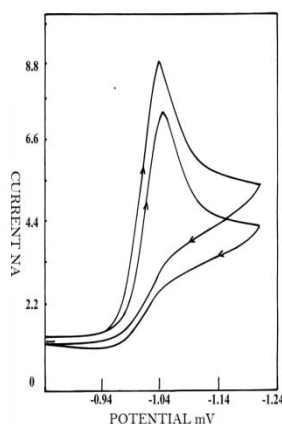


Fig. 2.0: Typical cyclic voltammogram of propiconazole at CNTPE at pH 4.0 concentration: 1.0×10^{-5} mM; scan rate: 50 mVs^{-1}

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