



Trade Science Inc.

ISSN : 0974-7419

Volume 11 Issue 4

Analytical CHEMISTRY

An Indian Journal

Full Paper

ACAIJ, 11(4) 2012 [146-148]

Polarographic behavior of crotamiton and determination of its residues in water samples

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Received: 2nd January, 2012 ; Accepted: 28th January, 2012

ABSTRACT

This effort deals with the electrochemical behaviour of crotamiton the C=C group containing pesticide and its relics in water samples. Differential pulse polarographic method was used to study the reduction behaviour of crotamiton. Dropping mercury electrode was used as working electrode and universal buffer with pH range 2 to 6 as supporting electrolyte reduction mechanism evolved by cyclic voltammetry. The optimum pH to catch well distinct peak for the discovery is originate to be 6.0. The peak current is found to differ linearly with the concentration of the pesticide over the range $1.06 \times 10^{-3} \text{M}$ to $1.05 \times 10^{-5} \text{M}$. The lower detection was limit found to be $1.06 \times 10^{-5} \text{M}$. © 2012 Trade Science Inc. - INDIA

KEYWORDS

Crotamiton;
Differential pulse Polarographic method;
Dropping mercury electrode;
Universal buffer;
Water samples.

INTRODUCTION

Crotamiton ($\text{C}_{13}\text{H}_{17}\text{NO}$) (*N*-ethylcrotono-*o*-toluidide) is an acaricide widely used as bird repellent.

For the determination of crotamiton residues in environmental matrices and biological samples different analytical techniques were reported. A Sioufi; N Sandrenan J P Dubois^[1]; Determined crotamiton in plasma and urine by high-performance liquid chromatography. Anton K, Bach M, Geiser A^[2]. Performed Supercritical fluid chromatography in the routine stability control of antipruritic preparations. Economical (reduction of analysis time, fewer experimental steps and less sample pre-separation) and ecological (carbon dioxide of organic solvents) advantages make SFC an attractive alternative to liquid chromatography in the determination of crotamiton. H. Nishi^[3] explained Pharmaceutical applications of micelles in chromatography and electrophoresis. Wolfgang schulz and andre

Schreiber^[4] subjected combined liquid chromatography time of flight mass spectrometry for water screening with soft ware tools to identify pesticides and their metabolites.

So far we have not come across any such study in the literature on the electrochemical reduction behaviour of crotamiton. There fore, we have under taken the present work to establish the experimental conditions for under standing the electro chemical behaviour of crotamiton from the measurement of cyclic voltammetry, differential pulse polarography, controlled potential electrolysis and milli coulometry. It is also aimed to develop DPP method for analysis of crotamiton in its pure form and in spiked water samples.

EXPERIMENTAL

Apparatus and electrodes

Polarographic assays were performed using a model

364 polarographic analyzer supplied by Princeton applied research corporation, (Princeton, NJ USA) coupled with a kipp and zonen BD8x-t recorder. A dropping mercury electrode (DME) was used as working electrode and a saturated calomel electrode (SCE) as the reference electrode differential pulse polarograms were recorded with a unit supplied by metrohm (herisau, Switzerland) coupled with E 506 polarocard and E 612 VA scanner cyclic voltammograms obtained by a digital electronics model 2000x-y/t recorder (Mumbai, India) in connection with the above unit. The DME used had an area of 0.223cm^2 at a drop time of 2s. A hanging mercury drop electrode (HMDE) used had an area of 0.223cm^2 in cyclic voltammetry. In all the above experiments platinum wire was used as auxiliary electrode all the experiments were performed at 25°C . pH measurements were carried out with an elico digital pH meter (Hyderabad, India). The millicoulometric apparatus used was supplied by radelkis (Budapest, Hungary) controlled potential electrolysis was carried out using a techno potentiostat (tech. ini electronics, Lucknow, India) in a modified cell with a mercury pool cathode saturated calomel reference electrode.

Reagents and solutions

Pure samples obtained from RANKEM India limited. The purity of sample was tested with tin layer chromatography and melting point determinations. A stock solution of pesticides under investigation were prepared in dimethylformamide. Universal buffer containing 0.2M boric acid, 0.05M citric acid and 0.1M trisodium orthophosphate were used as supporting electrolyte.

RESULT AND DISCUSSION

The electrode reduction of crotamiton was studied in universal buffer and consisted of a single step process in the pH range 2.0-6.0. The effect of pH on the polarogram has been investigated by recording the current voltage curves of crotamiton at a concentration of $1.0 \times 10^{-5}\text{M}$. For experimental results obtained by CV and DPP, the facile reduction of C=C group in crotamiton in the present study found to proceed with a 2 electron addition. Typical voltammograms are shown in Figures 1-2.

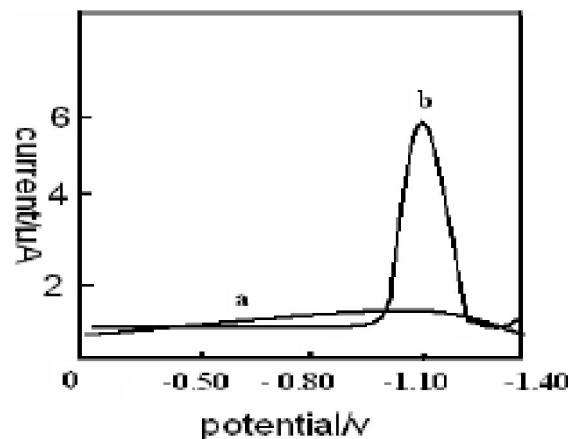


Figure 1 : Differential pulse polarogram of crotamiton at pH 6.0 Concentration: $1.0 \times 10^{-5}\text{M}$; pulse amplitude: 50 mV droptime: 2sec. a=blank, b=sample

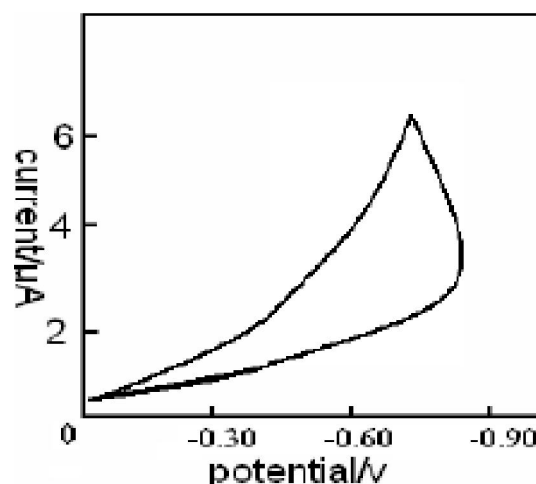


Figure 2 : Typical cyclic voltammogram of crotamiton for an accumulation time of 80 sec at HMDE, scan rate: 45 mV s^{-1} ; concentration: $1.0 \times 10^{-5}\text{M}$; pH: 6.0.

Figure 1 exhibits differential pulse polarogram for $1 \times 10^{-5}\text{M}$ crotamiton with DME. The systematic studies of the various experimental and instrumental parameters that affect the polarograms response were carried out in order to establish the optimum conditions. Typical cyclic voltammograms are shown in Figure 2. No reduction peak is observed in basic medium ($8 \leq \text{pH} \leq 12$) for C=C group due to the precipitation of electroactive species.

Effect of pH

The pH of a solution is a critical factor affecting both the rate and equilibrium state of the reduction process and the rate of the electrode reaction. The influence of the pH on the DPP response was studied at DME of the $1 \times 10^{-5}\text{M}$ crotamiton between the pH ranges 2.0 to 6.0. It can be

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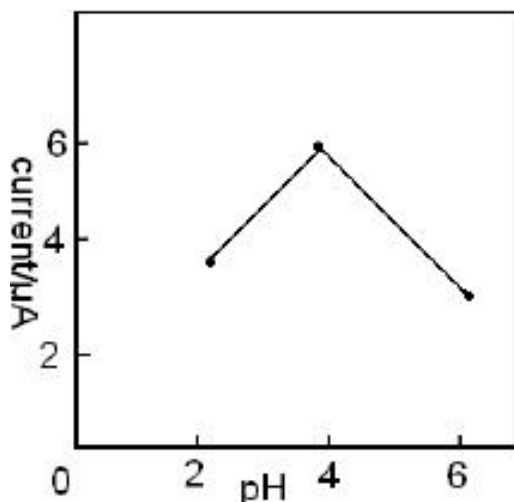
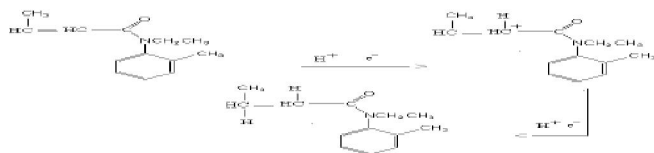


Figure 3 : Effect of pH on peak current of crotamiton.

observed from Figure 3 that the maximum peak currents are obtained with pH 6.0.

Electrode mechanism

On the basis of the results obtained as well as from the literature, the reduction mechanism as per Scheme 1 for crotamiton was proposed at pH 6.



Scheme 1 : Electrode mechanism of crotamiton

Analysis

Well defined peak obtained for crotamiton is helpful for the investigation of in crotamiton spiked water samples. The optimum pH to get well defined peak for the detection is found to be 6.0. The peak current is found to vary linearly with the concentration of the pesticide over the range 1.06×10^{-3} M to 1.05×10^{-5} M. The lower detection was limit found to be 1.06×10^{-5} M.

Recovery experiments

Recommended analytical procedure

A stock solution (1.0×10^{-3} M) of crotamiton is prepared in DMF. In voltammetric cell, 1 mL of standard solution of crotamiton is taken and 9 mL of the supporting electrolyte (pH 6.0) is added to it. Then the solution is deaerated with nitrogen gas for 10 min. after obtaining the polarograms, small additions of standard solution are added and the pollerograms are recorded

under similar experimental conditions.

Determination of crotamiton in spiked water samples

Water samples, were collected form swarnamukhi river belt, Vakadu, Nellore district, A.P., India. These samples were filtered through a Whatman No.41 filter paper and known amount of crotamiton were added. Aliquots of water samples were taken in a 25mL graduated tube, to it buffer solution was added and analysed as described above. The recoveries are summarized in TABLE 1.

TABLE 1 : Recoveries of crotamiton in spiked water samples

Sample	Amount added ($\mu\text{g/mL}$)	Amount found ($\mu\text{g/mL}$)	Recovery (%)	Standard deviation
Tap water	2	1.95	97.5	0.012
	4	3.92	98.0	0.011
River water	2	1.97	98.5	0.032
	4	3.98	99.5	0.021

CONCLUSION

The method shows a good reproducibility and high accuracy compared with spectrophotometric, spectrofluorimetric and chromatographic methods of analysis.

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