University of Novi Sad
INSTITUTE OF FOOD TECHNOLOGY IN NOVI SAD

FINS

food congress

28-30. 10. 2014.

Novi Sad, Serbia

II International Congress Food Technology, Quality and Safety

ISEKI - food Association

EFFoST

PROCEEDINGS

ISBN 978-86-7994-043-8

II INTERNATIONAL CONGRESS "FOOD TECHNOLOGY, QUALITY AND SAFETY", NOVI SAD 2014, SERBIA

Publisher

University of Novi Sad
Institute of Food Technology
Bulevar cara Lazara 1.
21000 Novi Sad

Main editor
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All abstracts and papers are reviewed by International Scientific Committee and competent researchers

Technical editor
Bojana Kokić

Cover
Boris Bartula, BIS, Novi Sad, Serbia

Printed by
"Futura" – Novi Sad, Serbia

Number of copies
350 copies

Organization of Symposium:

- INSTITUTE OF FOOD TECHNOLOGY, University of Novi Sad, Serbia

Symposium is supported by:

- Ministry of Education, Science and Technological Development of the Republic of Serbia - Belgrade
- Provincial Secretariat for Science and Technological Development - Novi Sad
- Ministry of Agriculture and Environmental Protection of the Republic of Serbia - Belgrade
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CONTENT

DEAERATION METHOD FOR IMIDACLOPRID DETERMINATION ON GLASSY CARBON ELECTRODE

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ABSTRACT

In this work deaeration methods were investigated for chronopotentiometric determination of pesticide imidacloprid [1-(6-chloro-3-pyridylmethyl)-N-nitroimidazolidin-2-ylideneamine] on glassy carbon electrode. As a supporting electrolyte, a Britton-Robinson buffer was used. All investigations were carried out in model systems. Cathodic peak of imidacloprid could not be registered only in Britton-Robinson buffer, due to electrochemical reduction of dissolved oxygen. For this reason, it was necessary to investigate different deaeration methods for removing oxygen from the solution. Within this, deaerations by various reductants and by passing a nitrogen stream were compared. Investigated reductants were sodium sulfite, ascorbic acid, oxalic acid, potassium bromide and sodium bromide. Combinations of a certain reductant and nitrogen were compared as well. Addition of a saturated solution of sodium sulfite directly in the tested solution proved as the best deaeration method. This method provided fast deaeration step as it took less than a minute for completely removing the oxygen. An optimal concentration of the solution of sodium sulfite was also investigated. Concentrations of sodium sulfite were tested in the range of 0.6 g/dm^3 to 230 g/dm^3. Considering the height of the analytical signal and its reproducibility, concentration of 8.8 g/dm^3 showed as optimal. This method could be applied for the determination of imidacloprid in commercial formulations and some real samples.
Keywords: *imidacloprid, glassy carbon, deaeration method*

INTRODUCTION

Imidacloprid belongs to group of neonicotinoids, relatively new group of insecticides, characterized by low toxicity to warm-blooded animals, and good efficiency against potato beetle, thrips, aphids, as well as many other insects (Šovljanski and Lazić, 2007). Its specific mode of action interrupts nervous functions of insects, which brings to paralysis and death (Šovljanski and Lazić, 2007). Wider application of imidacloprid leads to its more frequent occurance in food and environment, which imposes development of rapid and simple methods for its detection.

In this work, chronopotentiometric determination of imidacloprid on a glassy carbon electrode as a working electrode was proposed. This electrochemical method is based on oxidation/reduction of the analyte on the electrode surface, in steady solution with the constant current. Qualitative and quantitative characteristics of the analyte were obtained by measuring the oxidative/reductive potential and oxidative/reductive time. Given that imidacloprid in its structure has a nitro group, it reduces in two steps. The first step is based on irreversible reduction of nitro group to hydroxylamine and then to the corresponding amine in the second step (Guiberteau *et al.*, 2001). In previous research on reduction of imidacloprid on glassy carbon electrode, when voltammetry was used, only one reduction peak could be registered at -1,2 V (*vs. saturated calomel electrode*) (Guzsvány *et al.*, 2005). Electrochemical detection of imidacloprid was not possible without previous deaeration of the tested solution, due to reduction of dissolved oxygen on the working electrode, resulting in high residual current, which interfered with the measurement of many reducible analytes (Walace, 1985; Wang, 2006). In addition, species that were formed during this reaction (H_2O_2 and OH^-) might affect the electrochemical process being studied (Walace, 1985).

There are many different methods for removing dissolved oxygen. Currently, the most popular method is purging samples with an inert gas, usually nitrogen. Time of purging depends on the volume of the solution, flow rate and bubble size of deaeration gas, and technique that is used. Deaeration step by inert gas usually lasts 10-15 minutes.
Another method of oxygen removal is chemical deaeration, by adding reductant in the tested solution, which will react with dissolved oxygen from the solution. Addition of sulfite ion represents the oldest method for oxygen removal. Due to electroactivity of the sulfite ion at pH ≥ 7, this procedure is not commonly used in analytical practice (Walace, 1985).
According to literature, standard deaeration method that is used within different electroanalytical methods for pesticides determination is passing a nitrogen or argon stream (El-Shahawi and Kamal, 1998., Farzinnejad *et al.*, 2005; Guibertau *et al.*, 2001; Guzsvány *et al.*, 2005; Pushpalatha *et al.*, 2011; Pushpalatha *et al.*, 2013; Navalón *et al.*, 1999; Nigović *et al.*, 2011; Papp *et al.*, 2010; Papp *et al.*, 2011; Papp *et al.*, 2009; Zuman *et al.*, 2000). This deaeration method is time consuming, as it lasts up to 15 minutes, much longer than the analysis. Nowadays, the development of rapid and simple deaeration method is necessary. Therefore, in this study, different deaeration methods for chronopotentiometric determination of imidacloprid were compared. Investigation included comparison of passing a nitrogen stream, addition of different reductants, combinations of certain reductants, and combination of nitrogen and reductant.

MATERIAL AND METHODS

Apparatus

All chronopotentiometric experiments were carried out by automatic system for potentiometric and chronopotentiometric stripping analysis, constructed by our laboratory. Electrochemical cell consisted of three electrodes and electrical stick stirrer. Glassy carbon disc electrode was used as a working electrode (total surface area of 7.07 mm^2), a platinum wire (φ=0.7 mm, l=7mm) served as a counter electrode, and Ag/AgCl (KCl, 3.5 mol/dm^3) was used as the reference electrode. The three-electrode system and electrochemical stick stirrer were placed in a process glass. It was a glass vessel of 50 cm^3, with tapered bottom. Before each measurement, the surface of the glassy carbon was washed with acetone and doubly distilled water. All measurements were carried out at room temperature (23 ± 2 °C). All values of the potential were shown versus Ag/AgCl (KCl, 3.5 mol/dm^3) reference electrode.

Reagents and solutions

All chemicals used were of analytical reagent grade purity. For all dilutions and dissolutions doubly distilled water was used. Stock solution of imidacloprid (0.4 g/dm^3), was prepared by exact weighing of the reagent (Bayer AG, Leverkusen, Germany) and dissolution in doubly distilled water. This solution was stable for a three-week period when stored in the dark at 4°C. Britton-Robinson buffer was used as a supported electrolyte. It was prepared from equimolar 0.04 mol/dm^3 stock solutions of orthophosphoric (Zorka, Šabac, Serbia), boric (Zorka, Šabac, Serbia) and acetic (Lach-Ner, Brno, Czech Republic) acids. Required pH value of the buffer (pH 7.5) was set by adding 0.2 mol/dm^3 sodium hydroxide (Donau Chemie, Wien, Austria).
General procedure for investigation of deaeration method implied introducing of a certain volume of analyzed solution (usually 20 cm^3) into the process glass, followed by adding certain mass of reductants, stirring the solution, and after a brake of 10 seconds, the analytical step was performed from –0.91 V to –1.42 V. The tested reductants were: sodium sulfite (Centrohem, Stara Pazova, Serbia), oxalic acid (Lachema, Brno, Czech Republic), ascorbic acid (Lach-Ner, Brno, Czech Republic), potassium bromide (Merck, Darmstadt, Germany) and sodium bromide (Carlo Erba, Milano, Italy). Combinations of certain reductants were also investigated. When deaeration was performed by passing a nitrogen stream, the procedure was similar with the blank (20 cm^3 of Britton-Robinson buffer). Deaeration usually lasted for 10 minutes. Any additional measurements in the same solution implied additional deaeration for 2 minutes.

RESULTS AND DISCUSSION

At the beginning of the study, preliminary experiments were performed by comparing chronopotentiogram recorded in supporting electrolyte with a certain amount of various reductant. After a period of stirring (30-60 s), the analytical step was performed. Afterwards, certain amounts of standard solution of imidacloprid were added, and analytical signals of analyte were compared. The first tested reductant was sodium sulfite. After addition of sodium sulfite, deaeration took only 30 seconds, analysis was fast (1-2 s), and concentration of 15 mg/dm^3 of imidacloprid was detected. Ascorbic acid (Figure 1.a), oxalic acid, potassium bromide (Figure 2.b) and sodium bromide were not able to completely deaerate the solution, so analytical signal of imidacloprid could not be registered, even at higher concentrations (40 mg/dm^3 and 60 mg/dm^3). Increasing the amounts of ascorbic acid, oxalic acid and potassium bromide did not lead to improvements. By increasing the amount of oxalic acid, the pH of the tested solution was very low (pH 2). At this pH value imidacloprid could not be detected at all. When sodium sulfite was added to the solution containing ascorbic acid, sodium bromide or potassium bromide, the final potential was reached, and concentration of 20 mg/dm^3 was detected. Addition of higher concentrations of sodium bromide in blank was enough for achieving the final potential of -1.35 V, but the concentration of 20 mg/dm^3 of imidacloprid could not be detected.

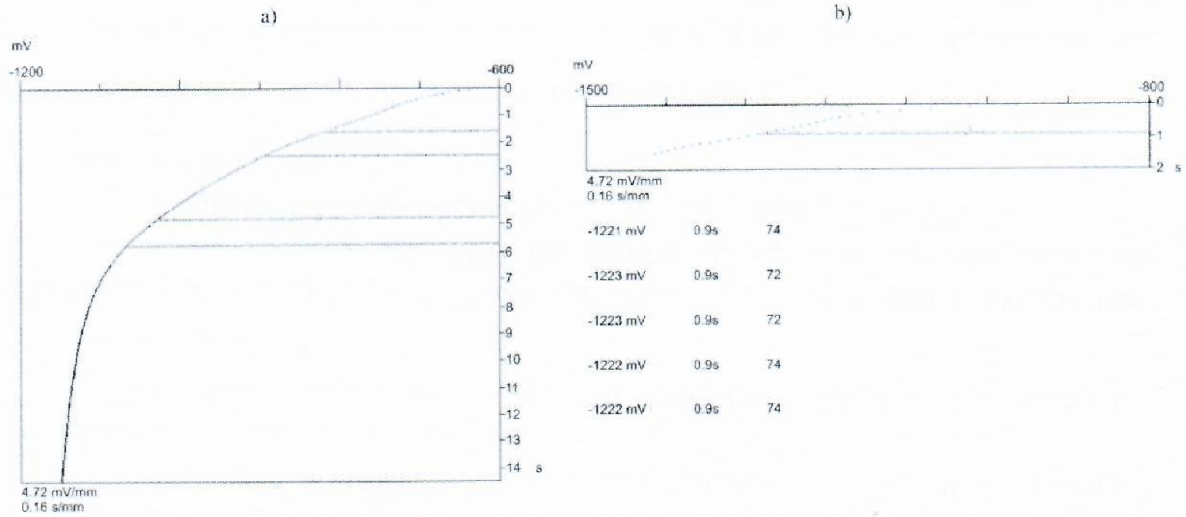

Figure 1. Original chronopotentiograms obtained by deaeration with (a) ascorbic acid, and (b) 8.8 g/dm^3 sodium sulfite

When nitrogen was used for deaeration, usual time for deaeration was 10 minutes. Analysis of the blank was fast (2-3 s), and there were no signals. After addition of standard solution of imidacloprid, in this case, the analysit had to be very agile, because in a short time when an airtight apparatus was opened, oxygen re-enetered the system, so additional deaeration for 2 minutes between adding a standard solution was performed. When nitrogen was used for deaeration, concentration of imidacloprid that was detected was 10 mg/dm^3. Sometimes, analysis of the blank and different concentrations of imidacloprid could not be performed at all, due to blocking the glassy carbon surface with bubbles of nitrogen (Figure 2.a).
This behavior was similar to the situation when oxygen was present in solution, which led to blocking of the analyzer at the potential of -1.3 V. After wiping of the glassy carbon surface with acetone and doubly distilled water, the analysis could be performed, but conditions were not reproductive, as demonstrated by slightly lower analytical signal. The same thing happened when the combination of nitrogen stream and certain concentration of sodium sulfite were studied, but in this case, deaeration lasted only 5 minutes. Considering all these results, as well as difficulties when nitrogen was used, all reductants, except sodium sulfite,

were excluded from further research. However, in order to achieve the best sensitivity with satisfactory reproducibility, it was necessary to investigate an optimal concentration of sodium sulfite. Saturated solution of sodium sulfite was prepared (230 g/dm^3), and different volumes of this solution were added to the Britton-Robinson buffer. Tested concentrations of sodium sulfite were in the range: 0.6 to 230 g/dm^3, in order to compare analytical signal of imidacloprid concentration of 15 mg/dm^3. The concentration of 0.6 g/dm^3 of sodium sulfite was sufficient for deaeration, but the concentration of 15 mg/dm^3 of imidacloprid could not be detected. When only saturated solution of sodium sulfite was used as supporting electrolyte, concentration of 15 mg/dm^3 of imidacloprid was detected, and this solution gave the highest analytical signal, but also outstretched chronopotentiograms, with very poor reproducibility (RSD = 19.63%). Results of investigations are shown in Table 1. It is evident that there is no significant difference in the height of the analytical signals when different concentrations of sodium sulfite were used. In terms of reproducibility, there was a great difference when different concentration of sulfite was used. Due to the satisfactory sensitivity and the best reproducibility of determination, concentration of 8.8 g/dm^3 of sodium sulfite was accepted as optimal (Figure 1.b).

Table 1. Overview of the reduction time in function of the concentration of sulfite

Concentration of sodium sulfite (g/dm^3)	**Reduction time (s)**	**RSD (%)**
0.6	/	/
1.1	0.78±0.08*	5.73
1.7	0.78±0.16	10.73
2.3	0.72±0.08	6.21
2.8	0.74±0.10	7.40
3.4	0.72±0.08	6.21
4.0	0.72±0.08	6.21
4.5	0.7±0.00	0.00
6.7	0.88±0.16	9.51
8.8	0.90±0.00	0.00
11.0	0.94±0.22	12.13
57.5	0.78±0.16	10.73
76.7	0.90±0.20	11.11
115.0	0.94±0.18	9.52
230.0	0.98±0.38	19.63

*Xmean±2SD
RSD – Relative standard deviation

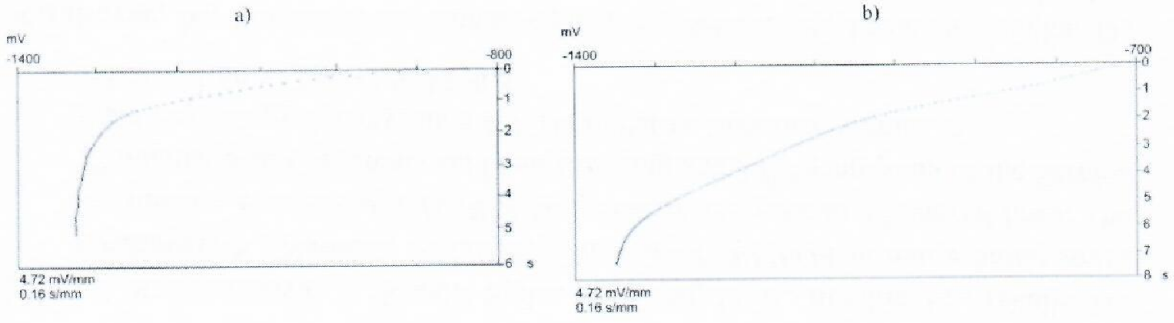

Figure 2. Original chronopotentiograms obtained by deaeration with (a) nitrogen, and (b) potassium bromide.

CONCLUSIONS

This study suggests the importance of chemical deaeration for electrochemical determination of imidacloprid, due to the fact that proper selection of deaeration method can lead to faster analysis and better reproducibility. When solution of sodium sulfite was used for deaeration, no blocking the glassy carbon surface was observed. This method is fast as it takes only 30 seconds to completely remove the oxygen from the solution. The proposed deaeration method can be used for development of chronopotentiometric determination of imidacloprid on glassy carbon electrode, or some other solid electrode, which can be applied to commercial formulations and real sample analyses.

ACKNOWLDGEMENTS

This investigation is financially supported by the Ministry of Education and Science of the Republic of Serbia (Grant III 46009).

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