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Original Article

Electrochemical sensor based on low silica X zeolite modified carbon paste for carbaryl determination



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ABSTRACT

A new and simple approach for carbaryl determination in natural sample was proposed using Low Silica X (LSX) zeolite modified carbon paste electrode. LSX zeolite with a porous structure was incorporated into carbon paste electrode in the appropriate portion. The prepared electrode was then characterized using scanning electron microscopy, cyclic voltammetry and electrochemical impedance spectroscopy. Various experimental parameters as the zeolite amounts, pH, accumulation time, and differential pulse voltammetric parameters were optimized. Under optimal conditions, a linear response was obtained in the range of $1-100 \mu$ M of carbaryl using differential pulse voltammetry with detection limit of 0.3μ M (*S*/N=3). The sensors showed good selectivity, stability, and reproducibility and has been successfully applied for detection of carbaryl in tomato samples with good recoveries.

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Introduction

Pesticides and their degradation products residue are a major pollutant and represent a potential menace to the ecosystem and

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human health. The non-rational uses of these agricultural inputs have many negative consequences and lead to the pollution of soil, fruits, vegetables, surface water, and groundwater. Moreover, these compounds are characterized by their persistence, their toxicity and known to bioaccumulate in the environment [1,2]. The use and impact of pesticides are an increasingly noticeable concern of the community. Consequently, there is an urgent need to

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develop new procedures for the determination of low amounts of these pollutants in different matrices. Carbaryl (CBR) is the widespread name for a compound known as 1-naphthyl methylcarbamate. It is a type of most frequently carbamate insecticide used to control a wide variety of pests, such as moths, beetles, cockroaches, ants, ticks, and mosquitoes by the inhibition of cholinesterase, one of the most important enzymes in the nervous systems of pests, vertebrates, and humans (WHO, 1994); [3]. The excessive and indiscriminate use of this pesticide is a major preoccupation because of the potential damage that this compound could cause to the environment and human. For instance, the harm caused to the major systems of the body, immune, nervous, and endocrine system [4,5]. During the last decades, several analytical methods were employed to analyze pesticides such as gas chromatography-mass spectrometry, Electro-Fenton technology, amperometric detection, Raman spectroscopy, acoustic technologies, and fluorescence methods [6–11]. Electrochemical techniques known as rapid and inexpensive methods are a good alternative to the classical methods used to determine trace level of pesticides and organic compounds [12-16]. Electrochemical studies concerning detection of carbamate pesticide, mainly carbaryl, were reported in the literature based on amperometric or voltammetric methods using non-enzymatic and enzymatic sensors [17-23]. During the last few years, detection of this pesticide with electrochemical biosensors based on enzymes was considered as a promising tool [24]. However, the enzymatic ways need long analysis time and pre-treatment steps. Non-enzymatic methods remain a very attractive option, capable to provide quantitative detection of carbaryl with a low cost and short analysis time. Zeolites, identified as microporous crystalline aluminosilicate materials [25], widely used for their high surface area, ion exchange capability, adsorptive capacity, and molecular sieving ability, are considered as interesting materials, which can be exploited in the development of modified electrodes. Zeolite modified electrodes were used as sensors for different reasons explained in the paper of Walcarius [26]. In fact, zeolite modified electrodes combine the advantage of ion exchange voltammetry with the molecular sieving property of zeolites. The properties of zeolites. such as size selectivity, high chemical and thermal stability could be coupled with the high sensitivity of voltammetric techniques [27-29]. Indeed, zeolite modified electrodes were previously used to detect some kind of pesticides like linuron and paraquat as reported by Siara et al., and Walcarius et al. [30,31]. In the literature, only the photodecomposition of carbaryl was investigated using zeolite and silver as a catalyst [33,34]. The potential of both modified and unmodified carbon paste electrodes in electrochemical applications and in modern electroanalysis of inorganic ions has been studied by many authors [35,36]. Zeolite modified carbon paste electrodes are a kind of modified carbon paste electrodes used to detect several molecules. However, to our best knowledge, the use of zeolite doped carbon paste electrode was not studied for the detection of carbaryl. In this work, new sensor was made from LSX zeolite modified carbon paste for the indirect detection of carbaryl. The electrode was characterized using microscopic, voltammetric and electrochemical spectroscopic techniques. The analytical performances of the resulting sensor were investigated after the optimization of experimental parameters. The obtained electrochemical electrode was used to determinate carbaryl in tomato sample using differential pulse voltammetry.

Experimental

Regents and materials

The chemical reagents used in the preparation of all solutions were analytical reagent grade. Graphite was supplied from Sigma Aldrich, St. Louis, Mo., USA. Chemical products used in synthesis of low silica X zeolite are: sodium aluminate (NaAlO₂, 50–56 wt %, Sigma Aldrich, Saint-Quentin Fallavier, France) as source of Al, sodium metasilicate nonahydrate (Na₂SiO₃·9H₂O, \geq 98 wt% Merck, Fontenay Sous Bois, France) as source of Si, sodium hydroxide (NaOH, 98 wt%, Sigma Aldrich, Saint-Quentin Fallavier, France), potassium hydroxide (KOH, 85 wt%, Merck, Fontenay Sous Bois, France) and bidistilled water. Carbaryl (CBR, C₁₂H₁₁NO₂, 99.8% purity) was purchased from Sigma-Aldrich, St. Louis, Mo., USA.

Synthesis of low silica X zeolite (LSXZ)

The low silica X zeolite powder was hydrothermally synthesized. Based on Kühl method, the molar composition of synthesis batch was Al_2O_3 :2.2 SiO_2 :5.5 Na_2O :1.65 K_2O :400 H_2O . Solution of sodium aluminate (SA) was prepared by dissolving sodium hydroxide and sodium aluminate in water. A sodium silicate (SB) solution was obtained by adding potassium and sodium hydroxide, and sodium silicate to water. Solutions SA and SB were carefully mixed under strong stirring at room temperature for at least 1 h to form aluminosilicate hydrogel. The mixture was poured into Teflon autoclave and then put in the stove for hydrothermal crystallization of zeolite that was carried out at a temperature of 90 °C for 24 h. After crystallization, synthesized powder was cooled down to 20 °C and washed with bidistilled water until the pH value of washing water became neutral. Then, the obtained powder was finally dried at 100 °C in the stove during 6 h.

Instrumentation

Cyclic voltammetric, electrochemical impedance spectroscopy and differential pulse voltammetric experiments were performed using AUTOLAB PGSTAT302N (Metrohm Autolab B.V., Utrecht, The Netherlands) Potentiostat/Galvanostat controlled by GPES 4.9 software. The three-electrode system consisted of a saturated calomel electrode (SCE) as reference electrode (which can be replaced by Ag/AgCl/KCl_{sat}), a platinum disk as auxiliary electrode, and a modified carbon paste as working electrode. The pH was measured using pH meter Fisher Scientific Accumet AB15 Basic, Waltham, MA USA. All the potentials reported in this work were given against SCE 3M KCl reference electrode at a laboratory temperature. X-ray diffraction (XRD) patterns were measured by using a Philips X'Pert PRO (PANalytical B.V, Limeil-Brevannes, France) with Cu_{Kα1} radiation source ($\alpha = 1.5406$ Å). Scanning Electron Microscopy (SEM) measurements were carried out by using a FEI Company model (Mérignac, France), Quanta 200, 10 kV.

Preparation of working electrode

The powder of graphite and zeolite were hand mixed with different proportions ($w_{(LSXZ)}/w_{(G)}$). Then, the paste was packed vigorously into the cavity (2 mm, Φ = 3 mm) of cylindrical Teflon-PTFE tube electrode and electrical contact was established with a copper rod. The resultant electrode is hereby denoted as zeolite X modified carbon paste electrode (ZXCPE). The unmodified electrode (carbon paste-CPE) was prepared with similar way without adding zeolite. The electrodes were renewed by simple extrusion of a small quantity of the paste from the electrode surface.

Procedure

Standard solution of carbaryl was daily prepared in acetonitrile. Aliquots of this solution were mixed with 0.5 mol L^{-1} sodium hydroxide solution in a 20 mL volumetric flask to hydrolyze the pesticide. The supporting electrolyte solution was added to the mixture for the voltammetric experiments. The solution of carbaryl Download English Version:

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