

Área: INO

Preparation of silicon carbide ceramic by polymer pyrolysis route and further use as electrochemical sensor for phenolic compound determination

Karen L.B. Santos (PG),¹ Cauan C. Lemes (IC),¹ César R.T. Tarley (PQ),^{1,2} Mariana G. Segatelli (PQ)^{1*}mariana@uel.br

¹ Departamento de Química, Universidade Estadual de Londrina (UEL), 86055-900 Londrina-PR, Brasil; ²Instituto Nacional de Ciência e Tecnologia de Bioanalítica (INCTbio), Universidade Estadual de Campinas (UNICAMP), 13083-970 Campinas-SP, Brasil.

Keywords: Polymer-derived ceramics, C-rich polymer, hydrosilylation reaction, pyrolysis, sensing

Highlights

SiC was obtained by pyrolysis of C-rich precursor. SiC sensor, prepared without any modification, provided satisfactory analytical parameters. The proposed method demonstrated accuracy.

Abstract

Silicon carbide (SiC) is a versatile non-oxide ceramic featuring high hardness, low density, corrosion/oxidation resistance and thermal/electrical conductivity. Regarding electrochemical applications, SiC has been used in the modified electrodes production. The polymer-derived ceramic (PDC) approach to SiC is advantageous due to enhanced processability of polymers over powders and possibility of adding compounds to modify its structure. This study involved the preparation of SiC ceramic material, by the PDC route, and its further use as electrochemical sensor for determining guaiacol, an emerging contaminant derived from lignin and found in the food sector. Initially, polymer precursor was prepared by Pt-catalysed hydrosilylation reaction between diphenylsilane and divinylbenzene at 1:1 molar ratio. Following, the C-rich polymer was heated at 5 °C min⁻¹ from room temperature up to 1500 °C, under argon atmosphere, maintaining 5 h at final temperature, giving rise to SiC ceramic. The produced ceramic material was characterized by FT-IR, XRD, Raman and XPS spectroscopies, SEM and cyclic voltammetry. Electrochemical measurements were carried out using a potentiostat and a conventional electrochemical cell containing a Pt wire as a counter electrode, a silver chloride as reference electrode (Ag/AgCl 3.0 mol L⁻¹ KCl) and a paste electrode composed of 80 wt.% ceramic and 20 wt.% Nujol as working electrode. Inorganic ceramic network was confirmed by the presence of typical absorption bands assigned to SiC materials and absence of those related to organic groups attached to polymer structure. Ceramic network was composed of β-SiC phase and graphitic and turbostratic carbon domains, together with amorphous regions. D- and G-bands, exhibited in the Raman spectrum, indicated the presence of Cp² (32.74%) and Csp³ (40.57%) sites within residual carbon (C_{free}) phase, attesting for the conductive and semiconductive properties of ceramic. Initially, the electrochemical behavior of guaiacol was assessed by cyclic voltammetry at ceramic sensor and compared with glassy carbon sensor. Cyclic voltammograms showed improved anodic current for ceramic sensor regarding glassy carbon. Further assays revealed improvement of analytical signal using 0.1 mol L⁻¹ Britton-Robinson electrolyte and pH 5.00 as the best medium conditions. The analytical method was carried out by square wave voltammetry and the respective frequency, potential step and amplitude values of 25 Hz, 10 mV and 75 mV achieved the highest currents. The range of analytical curve (from 10 to 100 μmol L⁻¹) and limits of detection (2.62 μmol L⁻¹) and quantification (8.75 μmol L⁻¹) demonstrated sensibility and comparable values to those found in the literature. The method accuracy involving reproducibility and repeatability assays, in different guaiacol concentrations, was attested by the relative standard deviations (RSD) results lower than 4.54%. The developed method will be applied in saw powder samples, whose assays are in progress.

Acknowledgments

The authors are grateful for the collaboration of the CNPq, CAPES, INCT-Bio, INCT-SP, UEL, LADEMA, GMPC, and SBQ-SUL.