Characterization of nanocomposite xerogel systems based on carbon, bismuth and iron

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Abstract

Nanocomposite systems based on porous carbon structures with diverse systems of metal / oxide nanoparticles are sought as promising solutions in various domains of applications including catalysts, energy storage, energy production and novel sensors.[1] The quest for sol-gel derived nanocomposite materials based on carbon, bismuth and iron optimized for electrochemical sensing applications represents the main focus of the present research. Complex systems of porous carbon matrices with integrated nanoparticles based on Bi and Fe are obtained through a modified resorcinol-formaldehyde (RF) sol-gel synthesis route.[2] After gel formation and maturation, drying and pyrolysis steps are performed in order to yield carbon based nanocomposites. The controlled variation of different synthesis parameters such as type of drying procedure,[3] Fe concentrations, pyrolysis temperatures lead to different morphological and structural parameters, were analysed using several techniques including X-ray diffraction, Raman spectroscopy, N_2 adsorption/desorption, scanning and transmission electron microscopies (SEM and TEM). During the characterization stage, different phenomena such as Bi-O-Fe interactions and the activation of Fe assisted growth of carbon nanostructures are highlighted. Relations are traced between the investigated material parameters and their notable electrochemical performances[3-4] in heavy metal and/or hydrogen peroxide (as biomarker) detection. Advances were made in the understanding of C-Bi-Fe xerogel systems, simultaneously adding contributions to impact applications such as decentralized analysis of water quality and inorganic biosensors.

Keywords: carbon; nanocomposite; electron microscopy; bismuth; iron; heavy metal detection; hydrogen peroxide;
Domain: Physics
Section: Elaboration of the doctoral thesis

Motivation: The detection of aqueous pollutants such as heavy metal ions represents a global necessity. From a technological development point of view, new techniques are investigated so that parameters such as good sensitivity and selectivity are addressed simultaneously with other requirements such as fast, cost-effective and on sight measurements, while establishing new merging points with other technologies such as bio-sensors. Novel nanocomposite materials are explored as electrode modifiers that detect heavy metal ions (i.e. Pb^{2+}, Cd^{2+}) and also bio-markers (i.e. H_2O_2) through electrochemical techniques such as square wave anodic stripping voltammetry (SWASV).

Methodology of Research: Considering their inherent properties such as enhanced sensitivity to heavy metals (for Bi), H_2O_2 sensing ability (Fe oxide) and good electro-chemical properties and structural tunnability of sp^2 like carbons, modified carbon xerogels and aerogels with embedded nanoparticles based on Bi and Fe metal/oxides are obtained. To find optimal conditions, different synthesis parameters are changed i.e drying procedure, Fe concentration and pyrolysis conditions. The impact of each parameter variation is investigated through a morphological and structural characterization using advanced electron microscopy in complement with X-ray diffraction, Raman spectroscopy, N_2 adsorption/ desorption. Then, the changes in the morphological and structural parameters are correlated with synthesis and application related parameters.

Results and Comparison with State-of-the-art: Results indicate that the obtained carbon-bismuth xerogels are highly compatible with other systems, mentioning here their enhanced sensitivity and limit of detection values.[3] The C-Bi-Fe systems revealed an interesting Bi-Fe-O interaction which could result in decreases in heavy metal detection performances, nevertheless, through this ternary design, both heavy metal and H_2O_2 species could be detected using the same type of material. The catalytic graphitization mechanism of Fe based nanoparticles, that could lead to enhanced electrically conductive properties, was also observed and investigated in detail.

Conclusions: C-Bi and C-Bi-Fe nanocomposites were synthesized through a modified xerogel synthesis route. The morphological and structural characterization reveal complex nanostructured systems with notable Bi-Fe-O interactions and graphitizing activities of Fe nanoparticles. The modified electrodes indicated promising results in terms of sensitivity, limit of detection and the dual use of the nanocomposites for heavy metal and bio-marker detection applications using xerogels based on Bi and Bi-Fe respectively.

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