Shiraz University

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A NOVEL SILVER NANOCOMPOSITE ELECTRODE FOR AMPEROMETRIC DETERMINATION OF HYDROXYLAMINE AND SEMICARBAZIDE

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September 2011

In The Name Of GOD

In the name of GOD

Declaration

I, Zahra Shojaeifard, a chemistry student, majored on analytical chemistry from the faculty of science declare that this thesis is the result of my research and I had written the exact reference and full indication wherever I used others sources. I also declare that the research and the topic of my thesis are not reduplicative and guarantee that I will not disseminate its accomplishments and not make them accessible to others without the permission of the university. According to the regulations of the mental and spiritual ownership, all rights of this belong to Shiraz University.

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IN THE NAME OF GOD

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BY

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Dedicated to:

My Parents,

My Dear Sister and Brothers

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ABSTRACT

A novel silver nanocomposite electrode for amperometric determination of hydroxylamine and semicarbazide

by

Zahra Shojaeifard

A novel silver (Ag) nanocomposite electrode was proposed that fabricated from metal nanostructures and ionic liquids. The combined application of unique properties of nanomaterials and ionic liquids in the design of this Ag nanocomposite results in electrodes with interesting advantages compared to the conventional metal disk electrode.
In the first part of this thesis, the elecrocatalytic effect of Ag paste nanocomposite electrode toward the oxidation of hydroxylamine in alkaline media was investigated.
Hydroxylamine was detected over a linear concentration range of 1µM- 5mM with double potential step chronoamperometry. The limit of detection was obtained as 0.30 µM. This new method of hydroxylamine determination is reproducible, selective, and sensitive, and can be used for real sample analysis. The independency of the system from most of the interferences and its ability of easily removing the effect of most cationic interferences are the important features of this work.

In the second part, the Ag paste nanocomposite electrode was employed as an efficient electrocatalyst for semicarbazide oxidation in alkalin media.
The results confirm that this nanocomposite electrode has high sensivity, low detection limit (0.6 µM), wide linear range (1.3 µM-0.5 mM and 0.5 mM- 2.3 mM), good long term stability and reproducibility for determination of semicarbazide.
Moreover, the electrode surface can be cleaned and renewed very easily by polishing on the smooth paper.
Therefore, this proposed electrode can have many applications as electrochemical sensors.

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CHAPTER ONE

INTRODUCTION

1.1 Nanostructured materials

The world of nanotechnology is opening many new opportunities and challenges for science, technology, and society. Nanomaterials are designed at the molecular (nanometer) level to take advantage of their small size and have novel properties that are typically not observed in their conventional, bulk counterparts. Nanomaterials have a much larger surface area to volume ratio than their bulk counterparts, which is the basis of their novel physical and chemical properties exhibited by them.[1] Controlling the size, shape, monodispersity, yield of the desired size, as well as the shape and the various useful properties of nanomaterials with different chemistry have become a challenge for material chemists.[2] The ability to control the size, shape, and distribution of the metal nanoparticles and nanostructures provides great opportunities to investigate them for catalytic properties and to discover new applications in form of novel research techniques or consumer oriented medical devices and many things in between. [2, 3, 4]

1.1.1 Synthesis of nanostructured materials

Nanoparticles can be prepared by physical and chemical methods. The physical methods, which frequently involve vapor deposition, depend on the principle of subdividing bulk precursors into nanoparticles. The chemical approaches involve reducing metal ions to metal atoms in the presence of stabilizers, followed by the controlled aggregation of atoms.[5]

Physico-chemical properties of metal nanoparticles for various applications strongly depend on their surface properties, in addition to the more commonly known size and shape effects. These surface properties of metal nanoparticles may vary significantly from one synthesis protocol to another, predominantly because concentration and type of stabilizing agents used in these synthesis methods can be quite different.[6]

In this approach, typically in the presence of a capping/stabilizing agent, metal ions are controllably reduced, wherein individual metal atoms combine to form metal nanoparticles stabilized by capping agents. Although in few instances reducing agents themselves act as stabilizers, capping agents are generally considered essential to minimize the surface free energy of metal nanoparticles, which would otherwise aggregate in the absence of these capping agents. Nanoscale particles of any material with a wide range of properties can be synthesized by employing various precursors such as polymers (poly vinylpyrolydone), Br^- , ionic liquid (IL) and etc.[7]

In most of the well established wet-chemistry routes of metal nanoparticles synthesis, aqueous or organic solvents are generally employed, while the use of ionic liquids (ILs) for synthesis of metal nanoparticles is a relatively recent phenomenon.[8]