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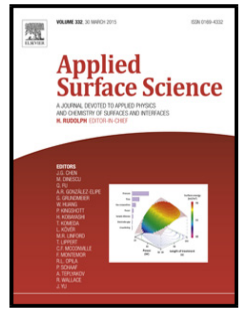

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Solventless synthesis of ruthenium nanoparticles

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Abstract

This paper presents a novel solventless method for the synthesis of zero-valent ruthenium nanoparticles Ru(0). The proposed method, although not entirely new in the nanomaterials world, was used for the first time to synthesize zero-valent ruthenium nanoparticles. This new approach has proved to be an environmentally friendly, clean, cheap, fast, and reproducible technique which employs low amounts of solvent. It was optimized through varying amounts of reducing salt on a determined quantity of precursor and measuring the effect of this variation on the average particle size obtained. The resulting products were fully characterized by powder XRD, TEM, HR-TEM, and XPS studies, all of which corroborated the purity of the nanoparticles achieved. In order to verify the advantages of our method over other techniques, we compared our nanoparticles with two common colloidal-synthesized ruthenium nanoparticles.

Keywords

Ruthenium nanoparticles, mechanochemistry, solventless synthesis, zero-valent nanoparticles

Introduction

The existence of nanoparticles has been known for some time[1-4] but interest in these structures has grown exponentially in the last few years. Particular characteristics of nanoparticles, such as high specific surface area, electronic confinement, or changes in thermodynamic properties[4-6] have led to their use in areas such as environmental remediation[2], chemical detection[4], catalysis[7,8], electronics[9], optics[10], ceramics[11], magnetic storage[12] and medicine[13].

In the catalytic field, ruthenium is one of the most important noble metals, as it is possible to use it in cataytic reactions as diverse as hydrogenation of unsaturated bonds[14-16], ammonia synthesis[17], Fischer-Tropsch synthesis[18], reduction of nitro compounds[19], methanol electro-oxidation[20], oxidation of alcohols and amines[21], di-hydroxilation[22], or C-C cross-coupling reactions[23].

Nevertheless, the method of synthesis used to obtain ruthenium nanoparticles has proven to be critical for a posterior application[24]. Different techniques have been developed to obtain them. There are usually two approaches: chemical methods[25,26], also known as bottom-up; and physical methods[27,28], known as top-down. Both approaches, with their respective advantages[29-31] and disadvantages[29,32,33], produce highly homogeneous nanoparticles of only a few nanometers in size[31].

A number of authors have argued that bottom-up colloidal methods have advantages over the top-down techniques, such as higher control of the dispersion or purity of the resulting nanoparticles[34]. This, however, is not always true. The synthesis of small, monodisperse nanoparticles is highly dependent on the type of solvent used and the concentrations of the precursors[35,36]. Therefore, in order to obtain high yields of very small nanoparticles, large quantities of solvent are needed. This high use of solvent translates into a more expensive and slower process. Moreover, by-products are frequently not eliminated[37], and their prescence may result in inconsistencies when nanoparticles are used. Of course, there are notable examples where nanoparticles have been synthesized from zero-valent organometallic precursors[32] with very little or nothing in the way of by-products, though this method increases the cost and complexity of the process.

For these reasons, we searched for an alternative technique that would enable the production of nanoparticles in less time and with lower amounts of solvents and by-products[38,39]. We found it in mechanochemical processes[40].

Mechanochemistry relates to physicochemical and chemical transformations induced by a mechanical force that changes the crystal structure of solids and creates fresh surfaces rich in active catalytic sites, which enhances the mass transfer required to initiate a chemical reaction[41]. The use of milling to produce new

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