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Synthesis of Rhodium Nano-Particles by Sol−Gel Processing

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Noble metals nanoparticles have a great scientific and industrial interest due to their wide range of possible applications in nanotechnology and catalysis [1]. The chemical synthesis methods like sol-gel, seem to offer some advantages over other physical, being the size control of the nanoparticles one of the most important. Normally, the chemical methods start for the reduction of the metallic precursor in the presence of the solvent; this helps to disperse the particles, and finally, the addition of any polymer such as acrylamide, in order to control the growth of the metal particles. [2]

The synthesis of rhodium nano-crystals was carried out by sol-gel method with the addition of acrylamide [3]. The rhodium metal was dissolved in sulfuric acid and distilled water at ~ 70 ° C, forming a yellowish solution. Once the solution reached room temperature, the pH is adjusted at ~ 7 by adding NH_4OH. Immediately after a pH of ~ 7 was obtained, Ethylenediaminetetraacetic acid (EDTA), acrylamide N-N' metilenebisacrylamide and α-α' azodiisobutyramidine dihydrochloride was added using a stoichiometric ratio of 1:1:5:0.2. Then, the polymerization was done at 80 ° C for 3 s, under constant agitation. Once obtaining the gel, this was placed in a microwave system for its decomposition. In order to produce the xerogel, thermal decomposition was carried out under an argon flow for 30 minutes from room temperature to 170 ° C. Thereafter, the resultant material was pulverized using an agate mortar and finally heated up to 1000 ± 4 ° C for 2 h in air.

The powder X-ray diffraction of the material is shown Figure 1. The three specific characteristic peaks of pure Rhodium, in angles 2θ=41.02, 47.7 y 69.8º, corresponding to its cubic structure (PDF file 089-7383), were observed. The SEM images showed in Figure 2 (a) and (b) display a morphology of the material resembling to type pores and agglomerates formed by nano-crystals in size between 200 nm and 1μm. Figures 3 (a) and (b), show the TEM micrographs of the material. The large particles detected in SEM observations are actually clusters, probably produced after sintering, from agglomerated Rh crystals (of ~ 15 nm). Thus, pure Rh nano-crystals were obtained easily with the proposed method in this work.

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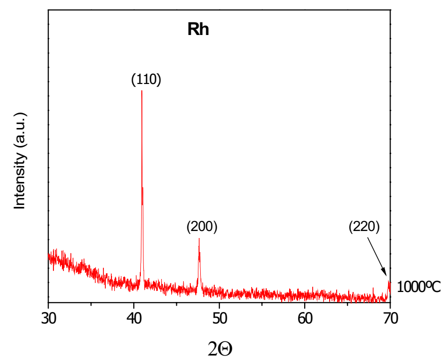

Figure 1. XRD pattern from nano-crystalline pure Rh, after heating at 1000 °C for 2 h.

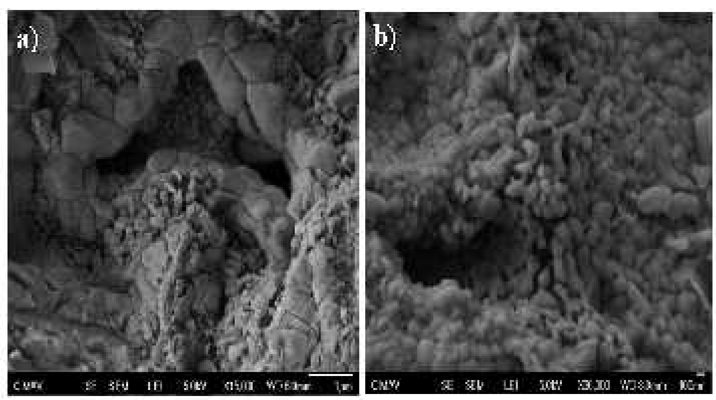

Figure 2. SEM images show the morphology of Rh clusters.

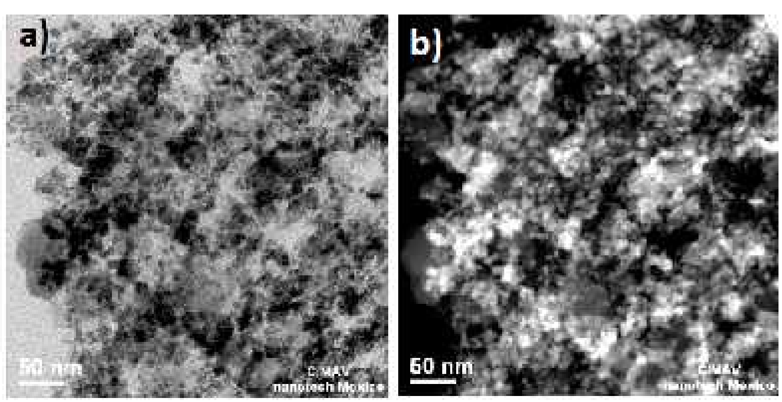

Figure 3. The Rh Micrographs of HRTEM by (a) Bright field and b) Dark field. These images clearly show clusters formed by small particles (~20 nm).