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Dispersion of Ni Particles on SiO_2 by an Improved Incipient Wetness Method and Full Characterization by TEM

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In heterogeneous catalysis, the chemical reactions are carried out on the catalyst surface. Commonly, this catalyst is composed of small metal particles (active phase) distributed on an inert material (support). For this reason, the distribution and particle size of the active phase play a very important role on the overall efficiency of the reaction. Metals such as Au, Ag, Pd and Pt are commonly loaded on supports with high surface area (Al_2O_3 and TiO_2). Although, these catalysts have shown high conversion efficiencies in different chemical reactions, are costs to synthesize due to the availability of those precious metals. So it is necessary to use alternative materials that meet the same restrictions at a lower cost. An example of this type of material is metallic nickel (Ni) supported on silicon oxide (SiO_2). These materials together have been used as a model catalyst in various chemical reactions, including the partial oxidation of methane (POM). This model catalyst has been studied extensively by Crozier et al [1-2]. He proposed that the Ni particles undergo several phase transformations during the POM, reaching an efficiency close to that established by the thermodynamics. Obviously, these advances were achieved after having synthesized a well dispersed catalyst. Therefore, in this work we propose and explain a method for preparing such catalysts. The goal is to impregnate the metal particles on the support and distribute them the best.

Experimentally, the support was synthesized in the form of spheres. This was by following the Stober's method using tetraethylorthosilicate (TEOS) as the SiO_2 precursor [3]. These spheres have a well-defined size and a narrow size distribution. Spheres were impregnated using an improved incipient wetness technique. A fixed amount of support was impregnated with a solution containing the Ni precursor. Usually, nickel nitrate ($Ni(NO_3)_2.6H_2O$) was dissolved in water or ethyl alcohol. A known volume of this solution (200 or 500 μL) was dropped slowly onto the support. Then, these were mixed thoroughly on a mortar with the pestle. This procedure was then repeated inside a glove chamber with the atmosphere saturated with water vapor.
The full characterization of the catalysts synthesized in this way, was carried out using a transmission electron microscope JEOL JEM-2200FS. Techniques employed were: scanning transmission (STEM), conventional transmission (TEM), energy disperse spectroscopy (EDS) and selected area electron diffraction (SAD).

Figure 1 shows STEM images from several spheres impregnated with the nickel nitrate. The Ni precursor is located on the surface of the spheres and between these. After the heat treatment, nitrate is converted into oxide; this treatment disperses and distributes Ni particles in a more homogenous way. Ni dispersion was quantified by means of EDS; figure 2A shows these results. One may note that the concentration of Ni on the sphere increased when taking an impregnation volume of 500 μL. Moreover, when the impregnation is carried out in a controlled atmosphere, the dispersion is

improved. On the other hand, figure 2B shows the relative amount of spheres loaded with Ni. That is, the number of spheres that had at least one particle of Ni deposited on them. For an impregnation volume of 200 μL, this value (90%) is kept constant regardless the control of the atmosphere. However, for the impregnation volume of 500 μL, the percentage drops to values less than 75%. Overall, these results indicate that it is better to use an impregnation volume of 200 μL in a controlled atmosphere. This is because most of the spheres will be covered with Ni particles with a more homogeneous distribution.
These and other results concerning the particle sizes distribution and the effect of the controlled atmosphere will be discussed in more detail.

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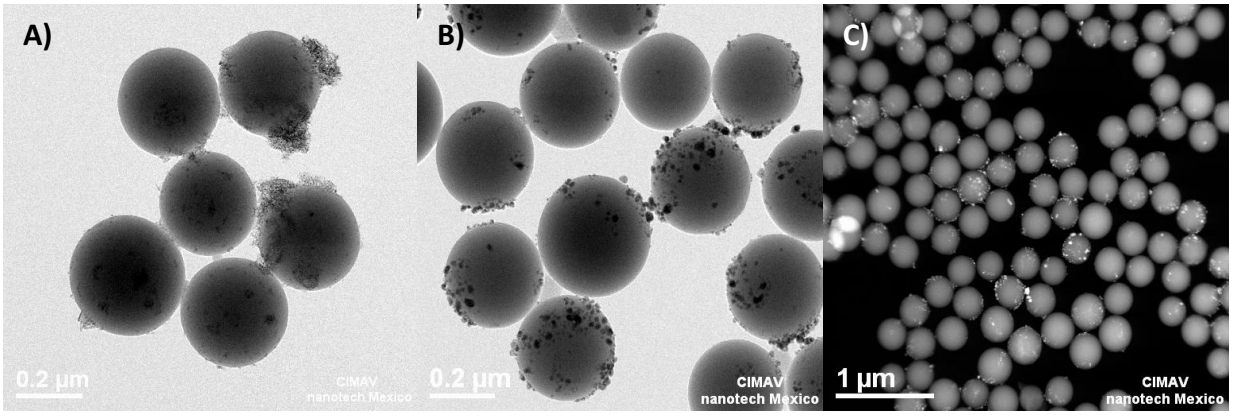

Figure 1: STEM images. A) Nickel nitrate supported on the spheres. B) Nickel oxide particles produced after heat treatment. C) Low magnification Z-contrast image showing the overall distribution of Ni particles.

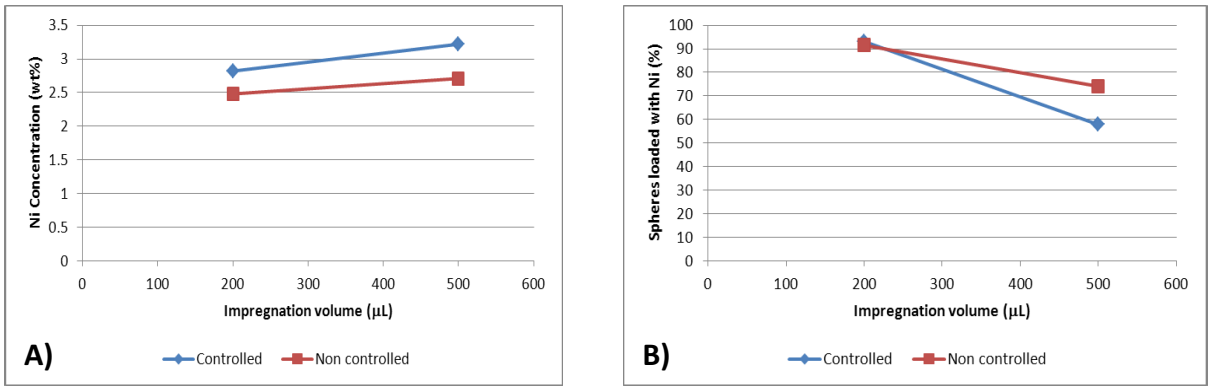

Figure 2: A) Average Ni concentration on the spheres; this was calculated from the EDS measurements. B) Relative amount of spheres impregnated with Ni particles.