SYNTHESIS AND CHARACTERIZATION OF Mn-Zn FERRITE MAGNETIC NANOPARTICLES BY THE MICROEMULSION REACTION METHOD

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The development of novel and more efficient methods of nanoparticle preparation generates a great interest in areas such as chemistry, biology, physics and medicine. Conventional methods for the synthesis of nanoparticles, such as co-precipitation, sol-gel and impregnation processes, are often not capable of resulting in the very small and controlled size required in catalysis and other applications, in spite of modern refining which is present in such methods [1]. Other methods, such laser evaporation, sputtering, ionized beam deposition, laser or flame spray pyrolysis, chemical vapour deposition, sonochemical processing, etc. involve the use of complicated and expensive equipment. Hence, in recent years there has been a growing interest in the synthesis of nanoparticles by the microemulsion reaction method (MRM), which consists in promoting reactions of formation of species in small droplets of nanometric size, each of which could be considered as an individual nano-reactor [2]. Such technique has been used recently in the synthesis of nanoparticles (5-7 nm) formed by mixtures of oxides of Zr-Ce [3], as well as in the fabrication of oxide nanocatalysts active in reforming reactions [4]. It has also been used for the synthesis of spinels and perovskites [5, 6], and in some cases pure spinel phases have been obtained directly in the microemulsion [7].

Recently, Kosăk et al have shown that it is possible to synthesize nanostructured spinels [7] at relatively low temperature (50ºC), obtaining the pure crystalline phase directly in the microemulsion, without the need of calcination. In those studies, as well as in most of the investigations cited above, the surfactants used for microemulsion formation have been of the ionic type, such as single chain surfactants, alkyl sulphates or quaternary ammonium salts (which require a cosurfactant, a medium chain alcohol), as well as double-chain alkyl sulfosuccinate or Aerosol OT. Nonionic surfactants of the nonylphenol ethoxylated type have been used as well. The use of ionic surfactants has some drawbacks, as complex functional species could be absorbed at the particle surface and interfere with its growth as well as with the reaction itself. If ceramic materials whose properties are highly dependant on metallic dopant species are to be synthesized, the use of ionic surfactants (especially of the anionic type) should be avoided in order to prevent contamination with surfactant counterions. The possibility of using commercial linear nonionic surfactants with the appropriate HLB could help to overcome these drawbacks.

The aim of the present study is to explore the potential of nonionic microemulsion systems for the synthesis of mixed ceramic oxides, namely Mn-Zn ferrites. The mixed oxide chosen, Mn-Zn ferrite, is of interest in the biomedical field due to potential super paramagnetic properties and hence possible applications as a Magnetic Resonance Imaging (MRI) contrast agent. In addition, it could be useful as a starting point or model for the future synthesis of a variety of nanostructured spinels and perovskites, which could have potential applications in the catalytic production of hydrogen from alcohol and hydrocarbon fuels, thereby promoting a fundamental advance in the preparation of active and economical catalysts for the reforming processes.

The phase behavior of a ternary aqueous solution/nonionic surfactant/hydrocarbon system was investigated in order to identify w/o microemulsions, in the presence of the metallic precursors, or precipitant agent. Addition of cosurfactant was also investigated, in order to optimize the formulations in terms of surfactant content. The microemulsions were characterized by diverse techniques (conductimetry, Pulse-Field Gradient NMR, Dynamic Light Scattering, Small Angle Neutron Scattering), in order to determine their structure and size. Selected microemulsions with different compositions were used in order to determine its influence on the characteristics of the nanoparticles. Characterization of the obtained materials was performed by transmission electron microscopy, X-Ray Diffraction, and magnetic properties. Nanoparticles in the same size range of the microemulsions (3-10 nm), and with spinel-type crystalline structure were obtained (Figure 1), demonstrating the microemulsion template effect. The results suggest that these environmentally friendly microemulsion systems could be useful for the preparation of nanostructured mixed oxides and other materials, which could have potential applications in the catalytic production of hydrogen from alcohols and hydrocarbon fuels, as well as for the preparation of superparamagnetic nanoparticles with potential application as MRI agents.

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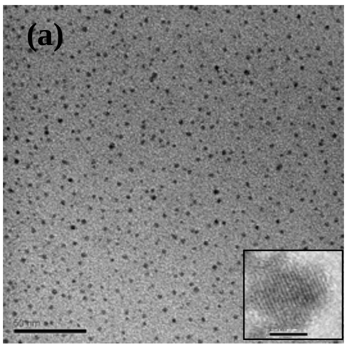
Figures:



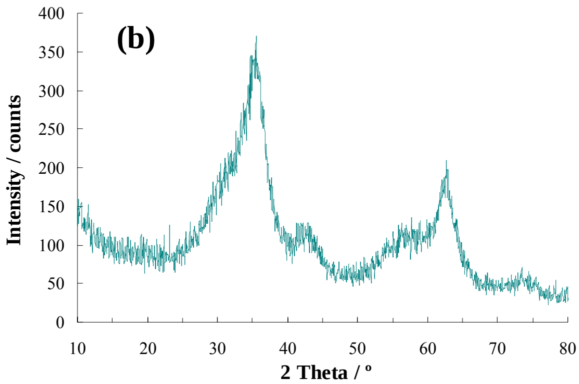


Figure 1. (a) High Resolution Transmission Electron Microscopy (HRTEM) of Mn-Zn ferrite nanoparticles obtained by MRM. Scale bar 50 nm (scale bar in inset 20 nm). (b) X-Ray Difraction (XRD) pattern for Mn-Zn ferrite nanoparticles obtained by MRM.