Morphological and Structural Analysis of Magnetic Support Produced from Magnetite (Fe₃O₄) Nanoparticles and Recycled Polyamide

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In recent years, the number of systems that involves the use of magnetic nanoparticles has exponentially increased. Magnetic nanoparticles have been a viable alternative as catalyst support. Its high surface area results in high catalyst loading capacity, high dispersion, outstanding and convenient catalyst recycling. Hybrid nanoparticles consisting of polymers and magnetic nanoparticles are based on a polymeric layer either coating the surface of magnetic nanoparticles or the magnetic nanoparticles are embedded on a polymer network [1].

The aim of this work is to analyze the morphology and structure of a magnetic support produced from nanoparticles of Fe₃O₄ and a polyamide of recycled origin for a possible use as catalyst support. The recycled polyamide was prepared according to a patent filed by State University of Londrina-Brazil (n° BR 10 2013 032153 2). The preparation of Fe₃O₄ nanoparticles (NPM) and magnetic support (RPC) was carried out according to the methodology adapted proposed by Xie and Ma [2]. Transmission electron microscopy (TEM) measurements were conducted using a JEOL 2100F instrument operating at 200 kV under diffraction and phase contrast modes. In addition, the instrument was operated in STEM mode alloying analytical information through XEDS spectroscopy and elemental mapping.

TEM image shown in Figure 1A presents NPM and its corresponding diffraction pattern. Through the diffraction pattern the sample was identified as Fe₃O₄ (JCPDS PDF 01-075-0449). It is noticed that the nanoparticles are rather agglomerated with spheroidal shape an average size of 15 nm. This agglomeration observed in Fe₃O₄ is due dipole interactions between the nanoparticles [3]. Figure 1B presents a high resolution (HRTEM) image showing in more detail an NPM where the distance between the atomic planes is measured as 0.25 nm, which corresponds to the (311) planes.

Figure 2A shows a bright field TEM image of RPC. The composite polymer nanoparticle sample does not exhibit a specific morphology. Figure 2B shows a dark field TEM image of RPC putting in evidence the spheroidal shape of the nanoparticles, eventually with some faceting. XEDS microanalysis (Figure 3) shows that the nanoparticles are rich on Fe and O, thereby confirming are Fe₃O₄. Current effort focusses on the structure the magnetic properties relationships of the magnetic support [4].

- [1] E. M. Reyes-Gallardo et al, Journal of Chromatography A 1345 (2014), p. 43.
- [2] W. Xie and N. Ma, Energy & Fuels **23** (2009), p. 1347.
- [3] J. Liu et at, Small 7 (2011), p. 425.
- [4] The authors acknowledge are grateful to CBPF for the access to the LabNano Electron Microscopy facilities and to the Brazilian Funding agencies CAPES and CNPq.

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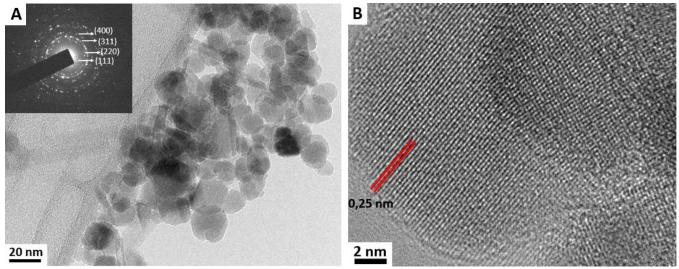


Figure 1. (A) TEM image of NPM and its respective diffraction pattern and (B) HRTEM image of NPM.

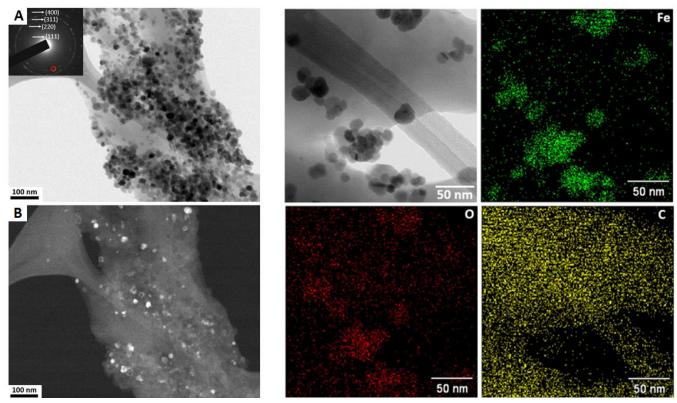


Figure 2. (A,B) TEM bright field/dark field pair with the diffraction beam marked red on the diffraction pattern.

Figure 3. STEM bright field of RPC and correspondent X-ray elemental mapping showing Fe, O and C elements.