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The stability of magnetic colloid based from copaiba oil

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Abstract. The magnetite (Fe₃O₄) nanoparticles (7 nm average diameter) have been synthesized and stably-suspended in a natural copaiba oil. The morphological and structural characteristics of the nanosized magnetite and the colloidal stability of the as-produced magnetic fluid sample were investigated using transmission electron microscopy, X-ray diffraction, photoacoustic spectroscopy and Mössbauer spectroscopy.

1. Introduction
Magnetic fluids (MFs) are typical colloids with a suspended solid phase consisting of i) an ananosized magnetic nanoparticle surface-coated with a stabilizing molecular layer and ii) either organic or inorganic carrier fluid [1]. The manufacture of magnetic fluids using biocompatible natural oil represents a novelty, and therefore novel strategies to achieve a high colloidal stability as well as the new characterization approaches are required. Long-term colloidal stability, one of the key issues in the magnetic fluid research and development, depends on the solid-liquid interface interaction, particle-particle interaction, applied magnetic field gradients, and temperature variations. In this study magnetite (Fe₃O₄) nanoparticles have been synthesized and stably-dispersed in natural copaiba oil.

1. Experimental
Magnetic fluid samples were manufactured using magnetite nanoparticles and natural copaiba oil, the latter providing the stabilizing nanoparticle coating as well as the carrier fluid. Nanosized magnetite was synthesized by the co-precipitating chemical reaction in alkaline medium using a mixture of aqueous solution containing ferrous chloride (FeCl₂·4H₂O, c.a. 0.5 molar) and ferric chloride (FeCl₃·6H₂O, c.a. 1.0 molar). Precipitation of the nanosized solid phase took place at temperatures close to 100 ºC (under vigorous stirring) by adding 50 mL of concentrated ammonia aqueous solution (25% v.v.) to 90 mL of the above described iron ion aqueous solution. The as precipitated nanoparticulated sample was separated from the supernatant using a strong magnet and washed using mili-Q water. The washing and the supernatant separation procedure was repeated five times while the pH was set between 8 and 9. Copaiba oil (CO) was then added to the washed, freshly-precipitated nanosized powder under vigorous stirring to produce the stock magnetic fluid sample (MFCO) containing about 6×10¹⁷ particle/mL. The nanosized magnetite particles were investigated using
transmission electron microscopy (TEM), X-ray diffraction (XRD), photoacoustic spectroscopy (PAS), and Mössbauer spectroscopy (MS).

3 Results and discussion
A representative TEM micrograph of the nanosized magnetite is shown in Figure 1; the average diameter is about 7 nm.

![Figure 1](image1.png)

**Figure 1** Typical TEM micrograph of the synthesized nanoparticles.

Figure 2 shows the X-ray diffractogram (XRD) of the synthesized magnetite nanoparticle, indicating the characteristic diffraction lines (220, 311, 400, 511, and 440) with peak positions and relative intensities in good agreement with the ASTM data. Analysis of the full width at a half height of the (311) XRD line indicated an average nanoparticle diameter very close to 7 nm.

![Figure 2](image2.png)

**Figure 2** X-ray diffractogram of the nanosized particles.
The photoacoustic spectrum (Figure 3) of the copaiba oil (CO) in the 300-1000 nm wavelength range exhibits an intense and sharp absorption in the ultraviolet region and a relatively weak absorption in the near infrared range (750-900 nm). However, the photoacoustic spectrum of the copaiba oil-based magnetic fluid (MFCO) containing magnetite nanoparticles shows two strong absorption bands, already described in the literature as the band-S and band-L at the lower and higher wavelengths of the spectrum, respectively [2]. The strong photoacoustic band in the lower wavelength region (band-S) has been related to the complex polyoxyhydroxi layer surface-coating freshly-precipitated nanosized magnetite particle [2,3]. On the other hand, as shown in Figure 3, the strong photoacoustic band in the higher wavelength region (band-L) has been related to molecules attached to the nanoparticle surface, shifting natural hydroxyl groups from the complex polyoxyhidroxi layer to copaiba oil components. The relative enhancement of the band-L photoacoustic signal, as observed in sample MFCO (see Figure 3) is due to the effective attachment of copaiba oil molecular components to a nanosized magnetite surface [3]. At this point it should be emphasized that PAS is a typical surface probing experimental technique whereas the nanosized magnetite-based sample provides a huge effective surface area. Nevertheless, the intense and wide band-S characteristics associated to sample MFCO (see Figure 3) indicates that molecules from the copaiba oil are not strongly attached to the magnetite nanoparticle surface, but instead more likely bound to the natural poloyoxyhydroxy layer via the hydrogen bonds.

![Figure 3](image-url) Photoacoustic spectra of the magnetic fluid (MFCO) and the copaiba oil (CO) at room temperature.

Figure 4 shows the Mössbauer spectra of the synthesized magnetite nanoparticle at 77 and 300 K. The 77 K hyperfine parameters obtained from the fitting of the Mössbauer spectrum confirm the magnetite structure with a typical isomer shift (0.32 mm/s), quadrupole splitting (0.02 mm/s) and internal field (50 T).
Figure 4 Mössbauer spectra of the magnetite nanoparticles at 77 K (upper panel) and at 300 K (lower panel).

4 Conclusions
This study reports on the successful preparation of a magnetic fluid sample consisting of magnetite nanoparticles (7 nm average diameter) suspended in natural copaiba oil. Freshly-precipitated nanosized magnetite was peptized in copaiba oil as a stable colloid. While X-ray diffraction data and Mössbauer spectra confirm the solid magnetic phase as a nanosized magnetite, the PA spectroscopy indicates that copaiba oil molecules are attached to the nanoparticle surface, providing colloidal stability to the manufactured magnetic fluid sample. However, the PA data also indicates that copaiba oil molecules are more likely attached to the nanoparticle surface via hydrogen bonding with the natural polyoxyhydroxi layer.

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