EXPERIMENTS REGARDING THE ROLE OF MAGNETIC/NON-MAGNETIC STIRRING IN THE PROCESS OF FERROPHASE PREPARATION FOR STABLE MAGNETIC FLUIDS

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This study is focused on the optimization of oil-based magnetic fluid preparation. Soft magnetic materials in the form of colloidal suspensions of iron oxides have been prepared based on magnetite nanoparticles and hydrocarbons aiming to obtain raw materials suitable for wide range technical uses, as well as for peculiar biological applications. The ferrophase was dispersed in kerosene and sterically stabilized with oleic acid surfactant, magnetic and non magnetic stirring being applied in the frame of several preparation protocols.

Small particle size, as well as narrow size distribution represent a "sine qua non" condition of magnetic fluid utilization [1-2]. The stability of magnetic colloidal suspensions is strongly dependent on the solid phase granularity, as summarized by Rosensweig [3]. Stable magnetic suspensions can be produced consisting in magnetite nanoparticles coated with surfactant layer, when the diameter of the ferrophase core is lower than about 10 nm. Several synthesis methods of preparation and stabilization of nanoparticles of appropriate size have been developed, the most classical methods being the co-precipitation of iron salts, and the mechanical grinding of larger, micron sized particles. Size reduction by "wet" milling allows one to obtain small and rather uniform ferrophase particles [4-6], however the long time duration of this procedure may appear as a significant disadvantage.

Ferrophase particles were synthesized by the Massart method [7], consisting in the co-precipitation of ferric and ferrous oxides in alkali medium - magnetite and possibly maghemite being precipitated from auto-catalysis reactions between ferric and ferrous salts, namely $FeCl_3x6H_2O$ and $FeSO_4x7H_2O$. Magnetic stirring was applied to favor chemical processes during ferrophase precipitation and subsequent coating. After the solid phase separation by filtration and rinsing with distilled water, the water traces were removed by repeated rinsing with acetone and ethanol. Ferrophase particles were coated with oleic acid at high temperature (over 80^0C) in hexane under continuous, vigorous stirring kerosene being further added as carrier liquid after hexane vaporization. Magnetic stirring was applied to favor chemical processes during ferrophase precipitation and respectively ferrophase coating for equal durations of sixty minutes (S1 sample).

Second type of nanoparticles was obtained by non magnetic processing (wet milling) of micron size magnetite particles of industrial provenance at room temperature, in the presence of oleic acid and kerosene (for thousands of hours). Consequently, two types of samples resulted:

Sample	Stirring during ferrophase precipitation	Stirring during ferrophase dispersion
S1	Magnetic	Magnetic
S2	-	Non-magnetic

Structural properties of these magnetic fluids were investigated by transmission electron microscopy (TEM – Fig. 1), atomic force microscopy (AFM – Fig. 2), X-ray diffraction and small-angle neutron scattering and magnetization measurements (Fig. 3). The influences of the two types of mixing (magnetic and non-magnetic) on the microstructural and magnetic features

are compared and discussed. Ferrophase particles with an average diameter of about 10 nm were identified as preponderant in the prepared magnetic fluid samples, the narrowest histogram being revealed in the case of totally non-magnetic stirring (S2). Rare aggregates having quasi-spherical shape were also observed. Also, it was shown that short chains of particles were predominant among the particle aggregates.

These results showed the feasibility of both routes of preparing magnetite nanoparticles of comparable sizes and size distributions. Under the applied conditions, wet milling resulted in a ferrofluid batch with better granularity – smaller average physical diameter, smaller crystallite size and lower frequency of particle aggregates – evident advantages overrunning the disadvantage of long time processing. Further improvement of preparation protocol is planned to include combined methods of final fluid extraction from the bulk sample by magnetic filtration and controlled concentration in order to optimize the practical procedure. Variations in certain parameters of preparation (e.g. milling time, liquid medium) will be explored aiming to control the properties and quality of the final product.

References:

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Figures:

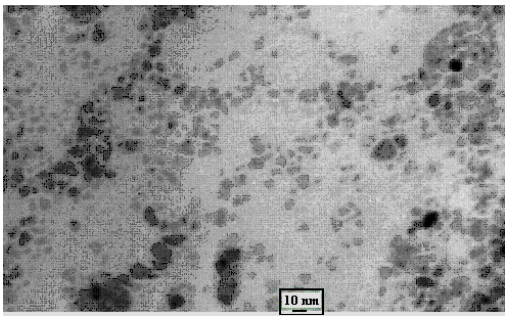

Fig. 1. Ferrophase particles revealed by TEM investigation

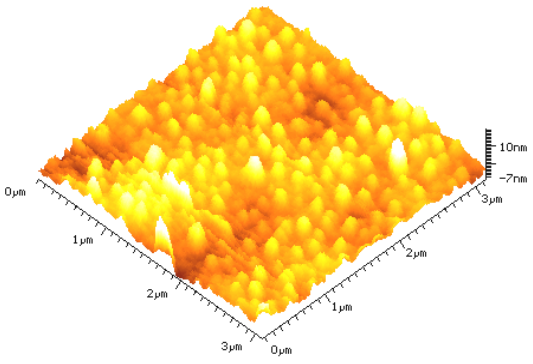

Fig. 2. AFM 3-D image recorded for S2 magnetic fluid sample (3x3 nm)

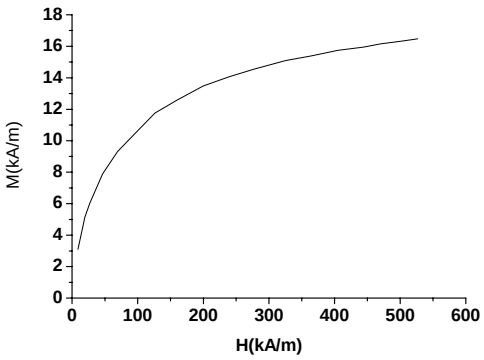

Fig. 3. Magnetization curve of magnetic fluid