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PRELIMINARY AFM INVESTIGATION ON MAGNETIC FLUID DIMENSIONAL ANALYSIS

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Microstructural insight in some new ferrofluids was obtained using ferrophase size scanning. Two ferrofluids, were prepared: the oil-ferrofluid and kerosene-ferrofluid (the ferrophase being stabilized with oleic acid). The Atomic Force Microscopy was used to visualize and to measure the nanoparticle diameter and height – the 3-D imaging representing one of the main advantages of this scanning technique. Both 3-D and phase representations have been used. The transmission Electron Microscopy images have been also analyzed. The diameter distribution has been obtained. The chain and aggregates particles are related to the ferrofluid stability.

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1. Introduction

The applications of ferrofluids in biology and medicine are considered more and more as one of the most prolific multidisciplinary research domain [1-5]. Due to the importance of the stability of ferrofluids, the ferrophase size distribution needs to be established by various techniques: transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffractometry, atomic force microscopy (AFM) etc. The possibility of the applications in the field of biology of oil-ferrofluids supply has been already revealed in the frame of several experiments where the vigorous shaking of small aliquots of diluted ferrofluids could be homogeneously mixed in the viscous agarized culture media [6-7].

2. Material and method

The AFM device in our laboratory works in the taping mode with commercial standard silicon nitride cantilever (NSC21) having a force constant of 17.5 Nm^{-1}, 210 kHz resonance frequency and tips with between 10 nm radius. The AFM images cover a range of areas, from 50 × 50 to 3 × 3 µm. The image results from multiple scans, the surface being sampled with 256×256 pixels. Phase images were recorded simultaneously with the three-dimensional topography images. AFM measurements have been carried out on ferrofluid samples deposited on mica substrate. They have been repeated on different sites of the sample, prepared in the same conditions of room temperature and ambient atmosphere. Alternatively Transmission Electron Microscopy (TEM) was used. TESLA device (40.000×) having a resolution of 1.0 nm, the ferrofluid being diluted 10^{-4} in toluen and deposited on collodion sheet. The oil-ferrofluid was prepared by dispersing the ferrophase, coated with oleic acid in natural hydrocarbons (kerosene). The ferrophase has been prepared accordingly to Cotae, 1986 [8], being based on magnetite and maghemite co-precipited from self-catalysis reaction between ferric and ferrous salts.

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3. Results and discussion

The 2-d images obtained using AFM scanning are mostly as that presented in Fig. 1 a, while alternatively phase sight is given in Fig. 1 b.

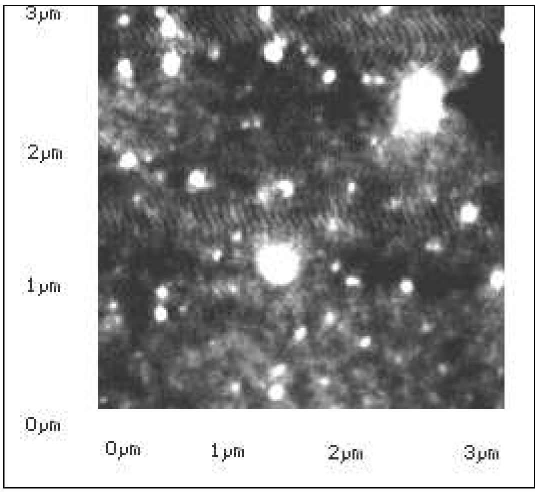

Fig. 1 a.

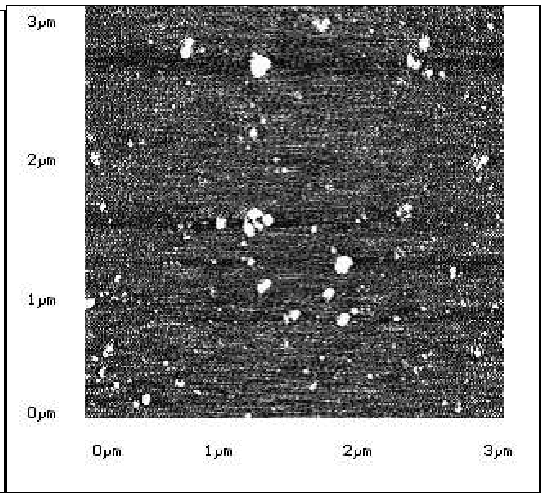

Fig. 1 b.

The best accuracy measurements have been carried out on images similar to that presented in Fig. 1.b those where the particle contour is better shaped.

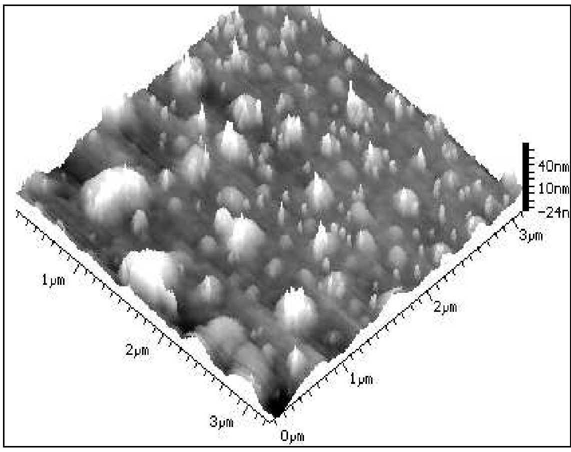

Fig. 2 a.

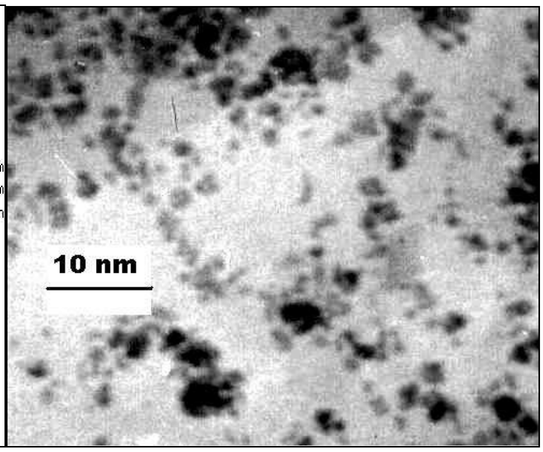

Fig. 2 b.

Repeated scanning was performed on numerous areas in the frame of the mica deposition preparation so that the final number of the analyzed particles was equal to about 1,000. In Fig. 2 a one 3-D image recorded using the AFM device are given while in Fig. 2 b one TEM image is presented. The histogram constructed using the AFM data is given in Fig. 3. The maximum at about 120 Å while the average diameter at 98 Å was evidenced.

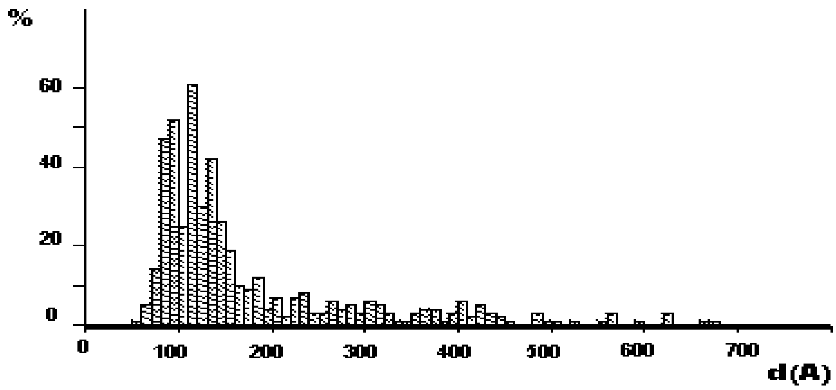

Fig. 3.

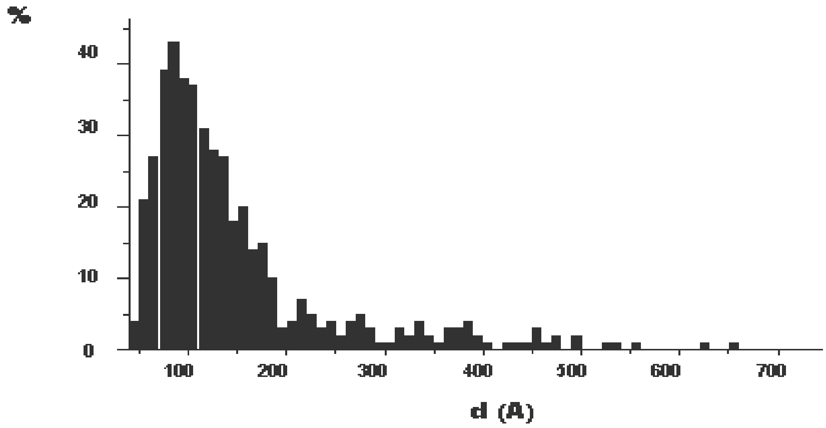

Fig. 4.

The TEM data have been provided by typical images as e.g. that presented in Fig. 4. The corresponding histogram in Fig. 5 can be seen. The maximum is at about 90 Å while the average value corresponds to the particle diameter of 87 Å. In both cases the histograms are rather asymmetric because of the presence of certain particle aggregates and chains that have diameters of 300 to 600 Å.

Details on the structure of these particle agglomerations from the careful examination of the 3-D images obtained by means of AFM scanning can be extracted. Indeed, unlikely the TEM images the AFM pictures can provide qualitative and quantitative information on the height of the scanned particles. In the present case the particle height was smaller that its diameter in most of the cases; however when we are dealing with particle agglomeration then the superposition of several particles may result in particle aggregates with relatively big height (Fig. 2 a).

The existence of such agglomerates and chains imposes the improvement of the preparation protocol since they suggest that the attractive electric and magnetic forces are not totally balanced by the steric repulsion conferred by the oleic acid coating molecules, so that the phenomenon of particle precipitation in weak magnetic field gradient can threaten the magnetic colloid stability. In the next step of our experimental research the conversion of the histogram toward a log-normal distribution need to be considered. The utilization of ferrofluid in the life sciences is tightly related to its stability as well as to its fine ferrophase.

4. Conclusions

The AFM investigation allowed to carry out measurements on the particle diameter and height. Similar histograms of diameters have been obtained from AFM and TEM investigation. The presence of some large aggregates and short chains of ferrophase particles in the investigated ferrofluid was also noticed. The ferrofluid stability is ensured by the dominance of small size nanoparticles well dispersed within the carrier fluid.

References

[1] A. Halbreich, J. Roger, J. N. Pons, D. Geldwerth, M. F. Da Silva, M. Rodier, J. C. Bacri, Biochimie **80**, 379 (1998).
[2] C. Groβ, K. Büscher, E. Romanus, C. A. Helm, W. Weitschies, European cells and Materials **3**, suppl. 2, 163 (2002).
[3] P. Frick, S. Khripchenko, S. Denisov, D. Sokoloff, J. F. Pinton, European Physical Journal B, **25**, 399 (2002).
[4] Y. Sahoo, A. Goodarzi, M. T. Swihart, et al., J. Phys. Chem. B **109**, 3879 (2005).
[5] M. Chastellain, A Petri, M. Hofmann, H. Hofmann, European cells and Materials **3**, suppl.1, (11-12), (2002).
[6] Al. Manoliu, et al., J. Magn. Magn. Mater. **201**, 89 (1999).
[7] Al. Manoliu et al., 8th Int. Conf. Magn, Liquid, Bremen, 2001.
[8] C. Cotae, Z. Kelemen, N. Olaru, M. Cotae, Patent Ro, 93111, 1986.
[9] L. M. Lacava, B. M. Lacava, R. B. Azevedo, Z. G. M. Lacava, N. Buske, A. L. Tronconi, P. C. Morais, J. Magn. Magn. Mater. **225**, 79 (2001).
[10] M. Rasa, B. W. M. Kuipers, A. P. Philipse, J. Col. Interface Sci. **250**, 303 (2002).
[11] P. Licinio, A. V. Teixeira, G. A. M. Safar, M. S. Andrade, L. C. Meira-Belo, U. A. Leita, J. Magn. Magn. Mater. **226-230**, 1945 (2001).