SYNTHESIS AND CHARACTERISATION OF γ-Fe_2O_3 PARTICLES

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INTRODUCTION: Nanoscaled particles showing a superparamagnetic behaviour have been intensively studied these past years for biomedical applications. Nevertheless, in vivo as well as in vitro applications still suffer from the lack of well-defined particles. One of the major challenges remains the synthesis of particles with a narrow size distribution.

METHODS: The aim of this work is to synthesize and characterise improved ferrofluids for cell separation and drug delivery. The size-controlled precipitation of iron oxide particles and the coating step are the two main parts of the project. The nanoparticles are synthesized by co-precipitation of iron-based salts in different media. The use of various compounds such as dextran, starch, polyvinyl alcohol (PVA), sodium-dodecylsulphate (SDS) and silica allows to obtain stable colloids. The particles composition and morphology are characterized using TEM, XRD and FTIR. SQUID magnetometery is used to investigate the magnetic characteristics of the particles but this technique is discussed more in detail in the presentation D5.

RESULTS: TEM pictures show ellipsoidal particles. A statistical analysis based on hundred particles per sample lead to an average size of less than 10nm with an ellipse aspect ratio of about 1.2. XRD patterns show a wide amorphous background due to the presence of polymer, nevertheless typical peaks, which can be attributed to nanocrystalline magnetite (Fe_3O_4) or maghemite (γ-Fe_2O_3) are also present. The size calculated from these data using the Scherrer formula confirms the TEM results. FTIR spectrometry led to the conclusion of a defect magnetite structure with a lattice parameter in between the one of bulk maghemite and magnetite.

DISCUSSION & CONCLUSIONS: Up to date, the studies revealed that stable ferrofluids at ambient temperature and neutral pH could easily be synthesised using "bio-compatible" compounds as stabilisers. The next step is to try and get a firmly attached coating around each particle. The study of the coating step is in progress and the conformation investigation of some polymers at the particles surface is planned. The size distribution of iron oxide particles is still too broad but should be improved by using a Segmented Flow Tubular Reactor (SFTR) for the synthesis.

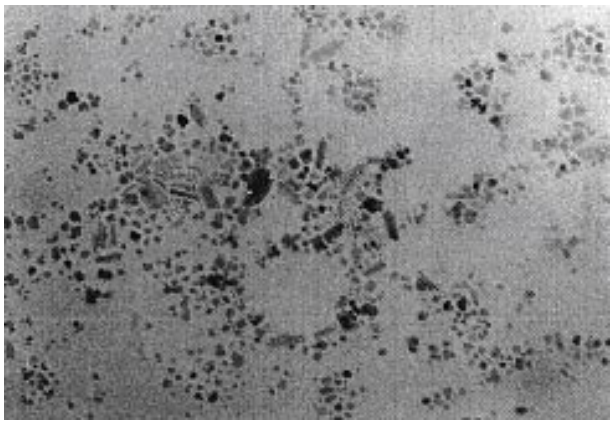


Fig. 1: Bright field TEM image of iron oxide particles stabilised with SDS. The sample consists of stable colloids at physiological pH, diluted and dried on a copper grid. The agglomerated structure is thought to be mainly due to the drying step

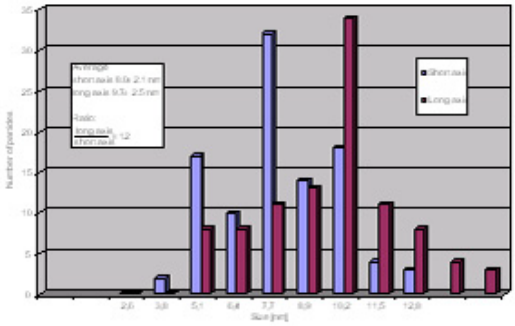
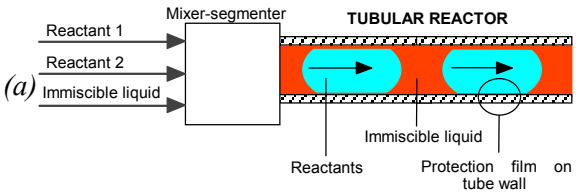


Fig. 2: Length distribution of the two axes of the particles (considered as ellipsis) from the TEM picture..



(b)

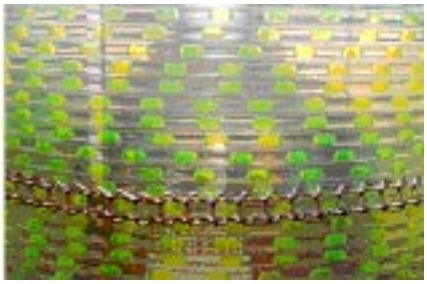


Fig. 3: Principle of a Segmented Flow Tubular Reactor (a) and setting in use (b). Such a setting allows a continuous synthesis with the advantage of producing identical particles over time.