

APPLICATION OF CUBIC MAGNETITE TO THE SYNTHESIS OF SUPERPARAMAGNETIC CELLULOSE BEADS FOR ENZYME IMMOBILIZATION. José R. Correa(1), Dora Canetti(1), Eduardo Bordallo(2), Jacques Rieumont(1), Javier Dufour(3). (1) Facultad de Química, Universidad de La Habana, Zapata y G. CP 10400, Havana, Cuba; (2) Unión de Investigación-Producción de la Celulosa del Bagazo Cuba-9. Pablo Noriega, CP: 33500, Havana Cuba. (3) Escuela Superior de Ciencias Experimentales y Tecnología, Universidad Rey Juan Carlos, 28933 Madrid, Spain. Email: correa@fq.uh.cu.

Iron oxides have a great industrial importance because of its vast application: magnetic recording supports, toners, pigments, catalysts, gas sensors, magnetic fluids and health.

Magnetite (Fe_3O_4) historically is one of the iron oxides which have brought about more interest because of its particular characteristics. Several synthesis methods have been explored in order to obtain Fe_3O_4 with particular magnetic properties. In the case of ferrofluids [1], cubic morphology could be used, with the advantage of obtaining magnetite directly from $\text{Fe}(\text{OH})_2$ precipitate.

Likewise, magnetic dispersed systems in polymeric matrices constitute a new generation of materials, which gains access in biochemistry, biotechnology, medicine, etc. [2]. The objective of the present work is the synthesis, characterization and application of magnetite with cubic morphology in the formation of a composite material. The so called material consists in encapsulated magnetite by a cellulose layer which is further modified, in order to use it as a vehicle in enzyme immobilization. The characterization of both materials includes several solid states analytical methods like IR Spectroscopy, X-Ray Diffraction (XRD), Thermogravimetric analysis and Scanning Electron Microscopy (SEM). Two types of magnetite samples were synthesized from $\text{Fe}(\text{OH})_2$ oxidation at pH 8-9 (MpH8-9) and pH > 11 (MpH>11) respectively. Superparamagnetic particles of cellulose-magnetite were synthesized starting from a solution containing cellulose and magnetite (MpH>11), using the method of emulsion water/oil. Composite particle size average is 100 μm with 2.8% of iron. Cellulose hydroxyl groups were activated by monochloroacetic acid in NaOH. The papain was immobilized using EDAC as copulating agent. It was demonstrated by IR Spectroscopy and X-ray Diffraction that both magnetites were lightly oxidized but the one at lower pH shows also the presence of a goethite phase. In both samples, particles with cubic morphology are obtained and the particle size distribution is of Gaussian type, bimodal, with similar shape to that reported for magnetite nanoparticles [3]. The micrograph of the specimen MpH8-9 (Fig. 1a) shows an abundance of small size particles, while in the MpH>11 sample (Fig. 1b) bigger particles are the majority. In MpH8-9 sample, the smallest particles have an average size of 151 nm and the biggest an average of 661 nm. Conversely, for magnetite MpH>11, the smallest particles attain a size of 224 nm and the biggest mean 566 nm. Experimental results indicate that magnetite obtained at high pH presents different crystallinity with respect to that with pH close to neutrality [4]. Because of phase purity and oxidation resistance, magnetite of pH>11 was selected for the composite synthesis. The formation of magnetite-cellulose composite could be verified by XRD (Fig. 2). The regular distribution of magnetite particles within the spheres (Fig. 3) was also corroborated by iron liberation studies, where a kinetic behavior of zero order was obtained. The amount of immobilized enzyme was 60 mg/g of composite, which retains 27% of its proteolytic activity after conjugation. No variation was observed on the optimum temperature. The enzyme retained 75% of its initial activity after 100-days of storage at 4 °C and 33% after 6 cycles of reuse [5].

References

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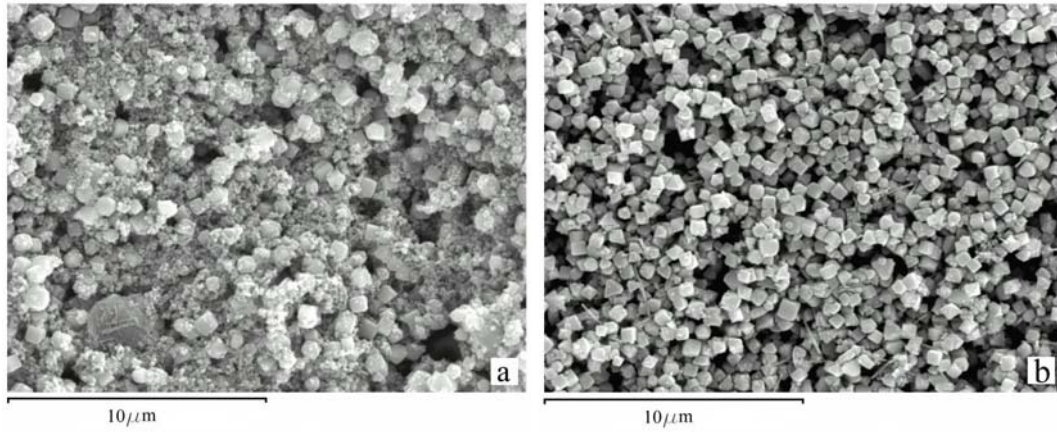


Fig. 1. - Micrographs from SEM of the magnetite obtained at: a) pH 8-9, b) pH > 11.

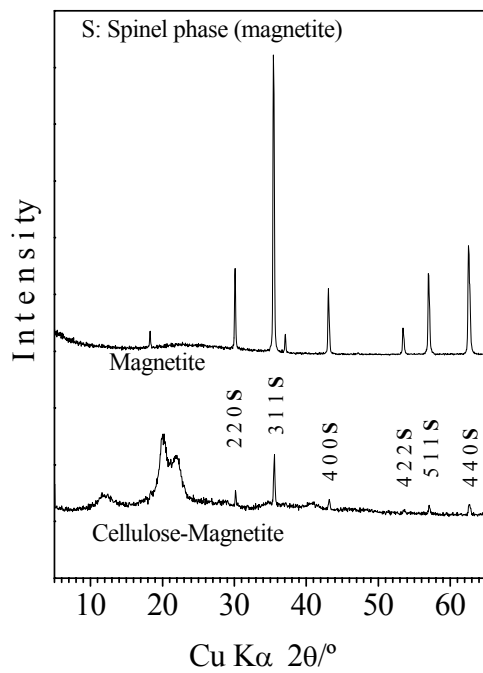


Fig. 2.- X-ray Diffraction patterns for magnetite (MpH>11) and cellulose-magnetite samples.

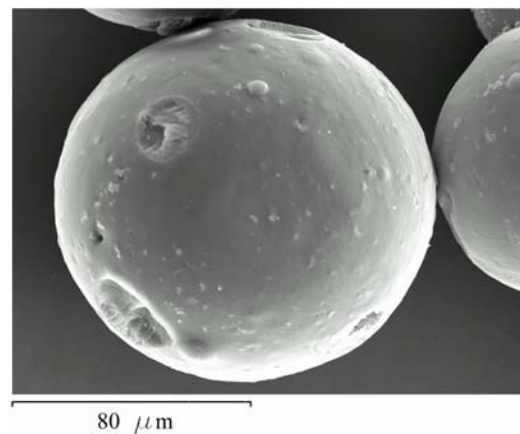


Fig. 3.- SEM micrograph of cellulose-magnetite composite.