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Magnetic mapping of hercynite produced by combustion synthesis

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Spinel-type ferrites have attracted great attention due to its structural characteristics and potential applications, specially related to their magnetic properties. Spinel structured materials have FCC structure and general formula AB2O4 [1] and can be synthetized by several methods, including co-precipitation, sol-gel [2] and combustion reaction [3]. The latter is particularly convenient, even for nanoparticles, due the low cost, high reaction speed, that may take only a few minutes, and for being energetically efficient, because the reaction environment releases enough energy for the reaction to occur [4]. In this sense, we have synthetized and characterized hercynite particles (FeAl2O4 – iron-aluminum spinel) with the method described in references [1,3], with citric acid as a combustible. A solution with the iron and aluminum nitrates (in the molar ratio of 1:2) and citric acid, in a stoichiometric proportion, was mixed in a glass evaporation dish. The solution was heated in a hot plate at 500ºC, inside a fume hood until ignition and burning, to produce the solid product in powder. X-ray Diffraction (XRD), Scanning Transmission Electron Microscopy (STEM – JEOL2100F) and Scanning Electron Microscopy (SEM – JEOL7100F) was used to characterize this material.

Figure 1a shows the X-ray diffractogram of this sample, corresponding to a multiphasic material, with the typical structure of FeAl2O4 spinel (JCPDF 01-089-1685) and hematite (JCPDF 00-013-0534). Figures 1b and 1c are secondary electron (SE) and backscattered electron (BSE) images. Those micrographs indicate that this material have a morphology of solid aggregates with two phases, corroborating with figure 1a. Figures 1c and 1d are bright-field (BF) and dark-field (DF) STEM images, confirming the crystalline aggregate nature of this product, with particle size of around 40-50 nm.

Magnetic properties were measured using a scanning magnetic microscope. Through a method of obtaining magnetic maps it's possible to obtain the region of maximum intensity as shown in figure 2a. After this process we use the line method to obtain the magnetic moment of the sample [5]. This method can also be used through magnetic maps in two dimensions on the X and Y axis (See figure 2b).

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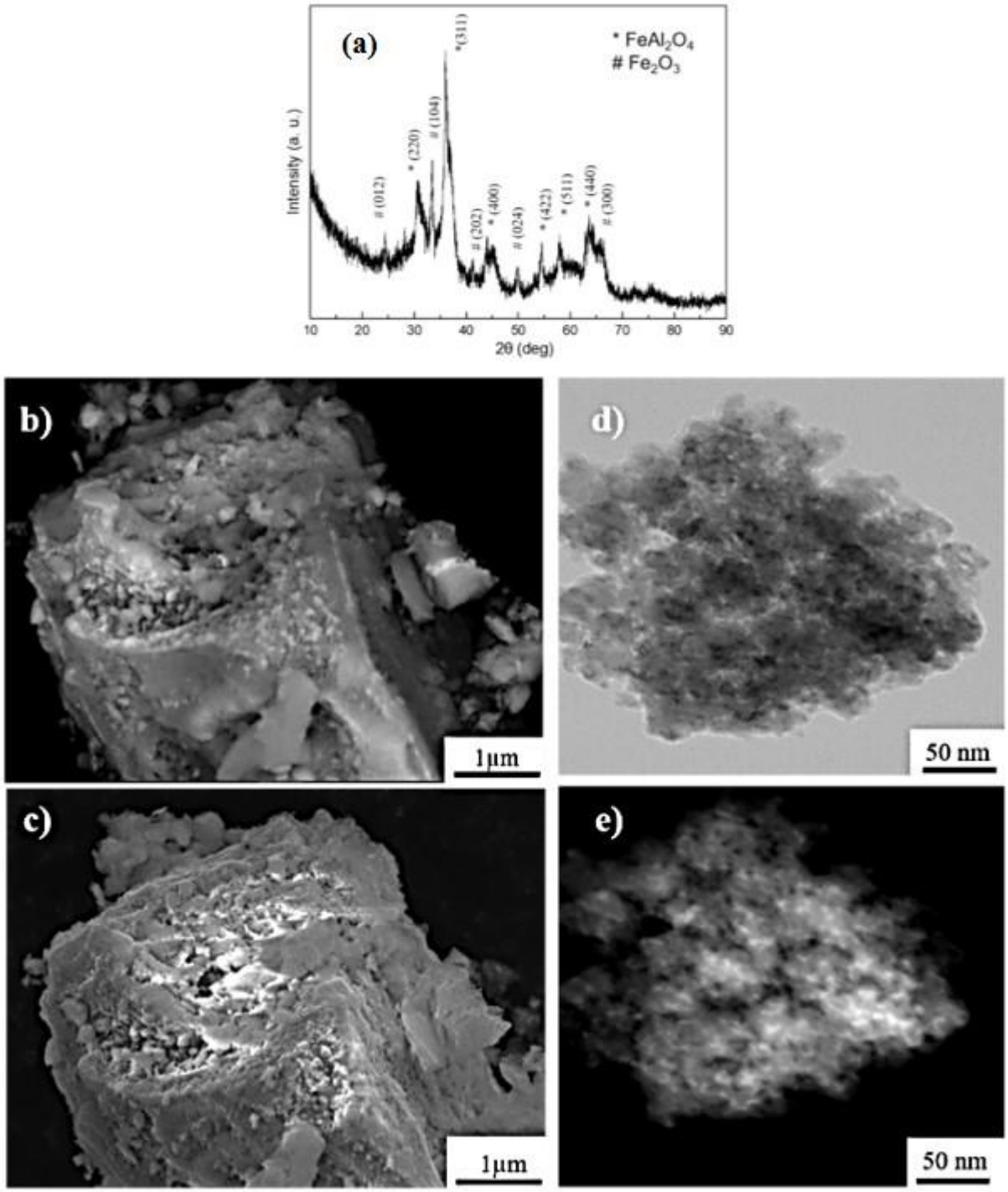

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Figure 1. (a) X-ray diffractogram of the synthesis final product; (b) SE-SEM (c) BSE-SEM and (d) (e) STEM images, respectively

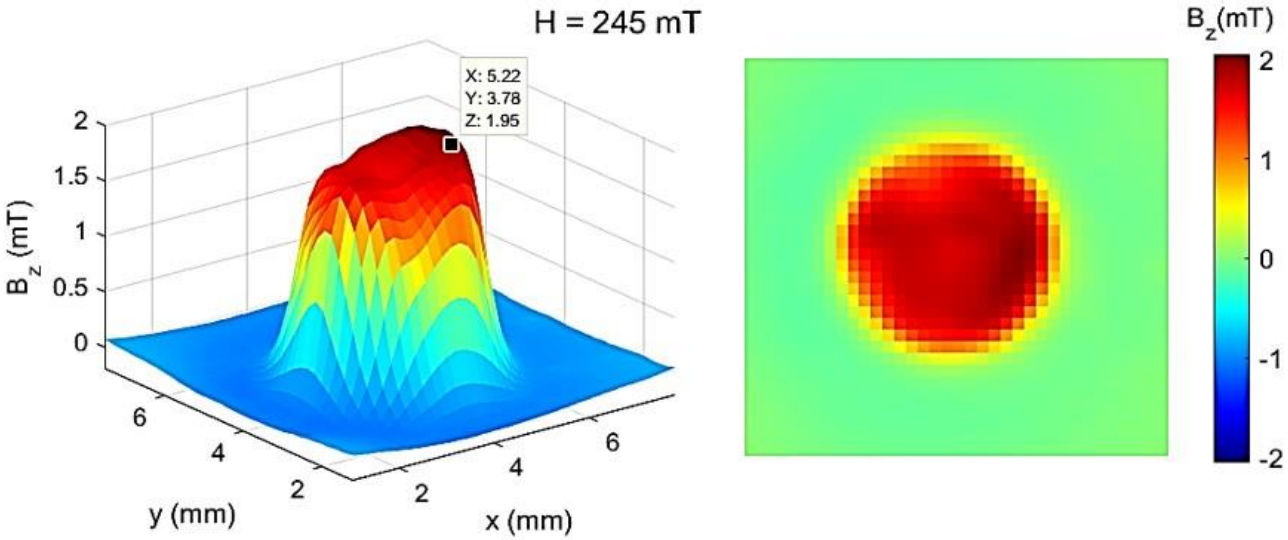

Figure 2. (a) Magnetic mapping of hercynite particles in 3 dimensions; (b) Magnetic mapping of hercynite particles in 2 dimensions.

References

[1] Silva, J. M., Araujo, J. F. D. F., Brocchi, E., &Solorzano, I. G. (2020). Ceramics International, 46(11), 19052-19061.
[2] Millican, S. L., Androshchuk, I., Tran, J. T., Trottier, R. M., Bayon, A., Al Salik, Y., & Weimer, A. W. (2020). Chemical Engineering Journal, 401, 126015.
[3] Loreto, R., Silva, J., Araujo, J., & Solórzano, G. (2020). Microscopy and Microanalysis, 26(S2), 2154-2155.
[4] M. Chai et al. Chemistry Letters 43 (2014), 1743-1745.
[5] J. F. D. F. Araujo et al., J. Magn. Magn. Mater. 499 (2020), 166300

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