PHYSICS

MORPHOLOGICAL, STRUCTURAL AND MAGNETIC CHARACTERIZATION OF BLACK SANDS OF THE GUAJIRA – COLOMBIA

CARACTERIZACIÓN MORFOLÓGICA, ESTRUCTURAL Y MAGNÉTICA DE ARENAS NEGRAS DE LA GUAJIRA – COLOMBIA

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ABSTRACT

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This article shows the morphological, structural and magnetic properties of the black sand on the surface soil obtained in the Guajira-Colombia. Morphological characterization of samples is performed by SEM. The compositional analysis of the compounds and elements was carried out through the XRF technique and by applying the Rietveld method to the X-ray diffraction data by quantifying the polycrystalline phases present in the simple such as ilmenite and magnetite. Magnetic characterization was performed by obtaining the hysteresis curve of magnetization as a function of applied fields at 300K temperature. Results evidence a ferromagnetic behavior of sample with magnetic parameters that permit to discuss the origin of magnetic character in the black sands. From heat treatments on air at different temperatures, the present magnetic phases are studied in order to determine the possibility obtaining of a single phase.

Keywords: Black sand, ferromagnetic, ilmenite, magnetite

RESUMEN

En este artículo se muestran las propiedades morfológicas, estructurales y magnéticas de la arena negra obtenidas sobre la superficie del suelo en la Guajira - Colombia. La caracterización morfológica de las muestras se lleva a cabo por SEM. El análisis de la composición de los compuestos y elementos se llevó a cabo a través de la técnica de XRF y mediante la aplicación del método de Rietveld a los datos de difracción de rayos X mediante la cuantificación de las fases policristalinas presentes en la muestra tales como ilmenita y magnetita. La caracterización magnética se realizó mediante la obtención de la curva de histéresis de la magnetización en función de los campos aplicados a 300K de temperatura. Resultados evidencian un comportamiento ferromagnético

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de la muestra con parámetros magnéticos que permiten analizar el origen de carácter magnético en las arenas negras. Los tratamientos térmicos se realizaron en aire a diferentes temperaturas, donde se estudiaron las fases presentes con el fin de determinar la posibilidad de la obtención de una sola fase.

Palabras claves: Arena negra, ferromagnéticos, ilmenita, magnetita

1. Introduction

Many materials that are currently used are mined [1] and it is known that the chemical composition of these minerals depends on its location. Minerals such as magnetite and ilmenite are of great interest due to their crystal structures because they show the presence of iron and titanium, which are materials with a wide application in fields such as aeronautics, military industry and some applications in biomedicine. The hematite phase of iron oxide is used as a pigment in the manufacture of paints. Studies in sands such as the one conducted by Muta'âlim and others which are magnetic sands found on beaches in Indonesia, show the presence of iron-based compounds and titanium as shown in this paper [2]. In this paper the chemical and morphological description of the black sands of La Guajira Colombia and results of the magnetic response are presented. Experimental data of x-ray diffraction are compared with the results obtained by fluorescence spectroscopy X-ray, which shows the presence of compounds based on iron, titanium, quartz, zircons and others. Images found by scanning electron microscopy show the morphology of minerals which are consistent with results of the fluorescence and diffraction characterization. Finally, it shows the ferromagnetic behavior of the sample at room temperature, given that the composition of the sample is mostly magnetite.

2. Experimental

Obtaining black sands samples used in this study were collected from the Mingueo beach in the municipality of Dibulla in the department of La Guajira in Colombia, 90km east of the Sierra Nevada de Santa Marta, in a territory which is part national park of Sierra Nevada de Santa Marta.

The measurements of X-ray diffraction (XRD) was performed on a sample diffractometer with rotating Bragg-Brentano (Philips X'pert-Pro), the XRD data are taken at 2θ between 10° to 90° using radiation CuKα with a wavelength of 1.54 Å and a detector RTMS (remote traffic microwave sensor). Refinements of the experimental data were performed through the GSAS and PowderCell 2.4 codes. From the X'Pert Highscore 2.0 semiquantitative concentrations of samples were determined.

Mineral samples were reduced in grain size with a fritsch ball mill at *400 rpm* for *3 min* obtaining a grain size of *300 μm*.

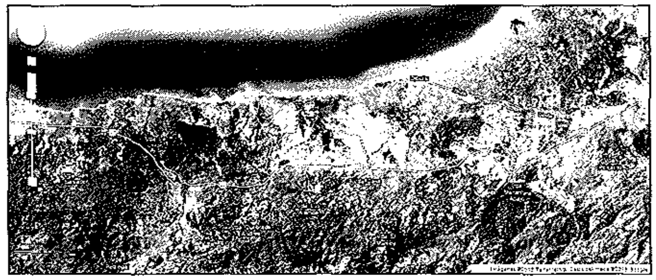

Figure 1. Map of Mingueo beach in municipality of Dibulla.

Morphologic analysis was carried out with a scanning electron microscope FEI Quanta *200 in* a vacuum environment *10* to *130 Pa*, due to variations in intensity it can be observed the analyzed surface topography, where the lighter areas correspond to the phases of higher atomic weight, useful a solid state detector. The fluorescence analysis was performed on a petro AXIOS PANalalytical brand equipment with a reduced grain size produced by a ball mill described above, it was taken *8 g* of the obtained powder, where this powder was combined with *0.8 g* of wax, after that it was placed in a *40 mm* diameter mold, and then it was press at *100 kN* for *20 s*, resulting in a pellet. The scanning was performed at *2θ* in a range *10°* to *150°* using Super Q Manager software. This technique allows us to find the elements present in the sample, and it should be noted that there may be elements of concentrations below *100 ppm* which the equipment cannot detect. For measuring the magnetic properties it was used the magnetic power measurement system MPMS quantum design. The magnetic moment was measured at room temperature, by using the hysteresis measurement through the application of magnetic fields from *-5000 Oe* up to *5000 Oe*; the frequency used was *40 Hz*, and the trial was conducted between *250 mV – 25 mV*.

3. Results and Discussion

X-ray fluorescence Analysis shows the existence of different phases present in the sample which can be seen in Table1.

The spectroscopic analysis of X-ray fluorescence indicates that the iron is the element with the highest proportion in the count made, following by the titanium and silicon. These results can be verified in Table 1 which shows that *Fe*, *Ti* and *Si* are elements found in higher proportion in the sample. These results are compared with the analysis of X-ray diffraction in

Table 1: Results of X-ray fluorescence in the sample of black sands from la Guajira

Name*	Symbol	Composition (%)
Sodium	Na	1.6
Magnesium	Mg	0.7
Aluminium	Al	1.9
Silica	Si	10.8
Phosphorus	P	0.2
Sulphur	S	0.3
Potasium	K	0.2
Calcium	Ca	1.2
Titanium	Ti	21
Chromium	Cr	<0.1
Manganese	Mn	1.6
Iron	Fe	56.7
Zinc	Zn	<0.1
Zirconium	Zr	2.5
Yttrium	Y	<0.1
Niobium	Nb	0.1
Cerium	Ce	0.1
Hafnium	Nf	0.2
Chlorine	Cl	0.9

* The "elements" present make oxides.

which verifies that the phases present and the percentages of each of them coincide in the two methods of analysis. Consequently, it can be stated that present content of the sample phases are magnetite, ilmenite, quartz and zircon. Figure 2, show the difractogram performed on the sample of black sands from La Guajira where the characteristic peaks of the phases present in the sample can be appreciated, which are

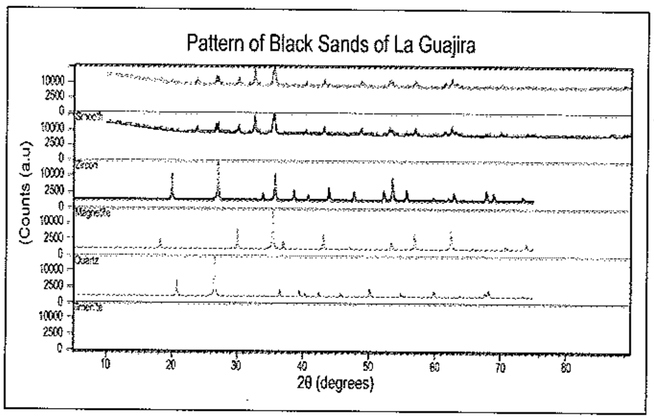

Figure 2. XRD pattern of black sands from Guajira.

identified as magnetite, ilmenite, quartz, zircon and others with lesser degree.

The lattice parameters and crystal structures found for the compounds in the sample of black sand are compared with those reported by other authors and are consistent with the obtained by this work. Using the Rietveld refinement method the quantitatively phases of black sands samples were found. Results are consistent with the values in Table 1 and stoichiometry of the different phases which are around 37% magnetite, 42% ilmenite, 14% quartz and 7% zircon. After the sample was separated magnetically, it was heated at *650 °C*; it can be observed in the figure 3 the presence of hematite and titanium oxide phases.

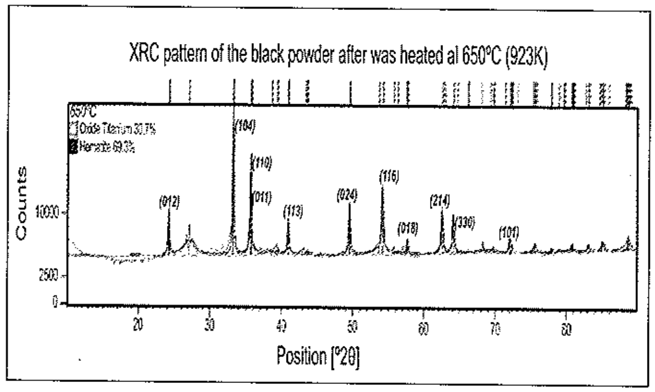

Figure 3. XRD pattern of the black powder after it was heated at 650 °C.

At the end of Table 2 it shows the found parameters of the new lattice of the crystalline phases where a comparison is made with results reported by other authors, and it is found a similarity in their parameters. The quantification of the sample indicates that 80% belongs to the hematite phase and 20% to the titanium oxide phase.

In Figure 4 it can be seen some maximized phases present in the sample such as quartz, magnetite and ilmenite. The grains have no crystalline forms due to erosion process.

Hysteresis curve shows a ferromagnetic behavior of the samples; this is because as noted above in the analysis of the x-ray diffraction phases is present a good amount of iron in their crystal structures as magnetite [5]. The hysteresis curve shows that Crystallites found in the black sands samples possess ferromagnetic behavior which can be attributed to the high concentration of magnetite in the material.

The magnetic parameters obtained were: *32.7 emu/g* saturation magnetization on *4.9 kOe*, *2.5 Oe* coercive field and *0.07458 emu/g* remnant magnetization.

Table 2: Lattice parameters of the phases present in the black sand of la Guajira.

Phase	Lattice Parameter (Å)	Ref.	Crystalline Structure
Magnetite (Fe_3O_4)	*a=8.3940*		Cubic
Ilmenite ($FeTiO_3$)	*a=5.09* *c=14.093*	*a=5.088* *c=14.093 [6]*	Hexagonal
Quartz (SiO_2)	*a=4.9148* *c=5.4062*		Hexagonal
Zircon ($ZrSiO_4$)	*a=6.6042* *c=5.9796*		Tetragonal
Hematite (Fe_2O_3)	*a=5.0355* *c=13.7471*	*a=5.0355* *c=13.7471 [7,8]*	Rhombic
Titanium Oxide (TiO_2)	*a=4.6530* *c=2.9650*		Tetragonal

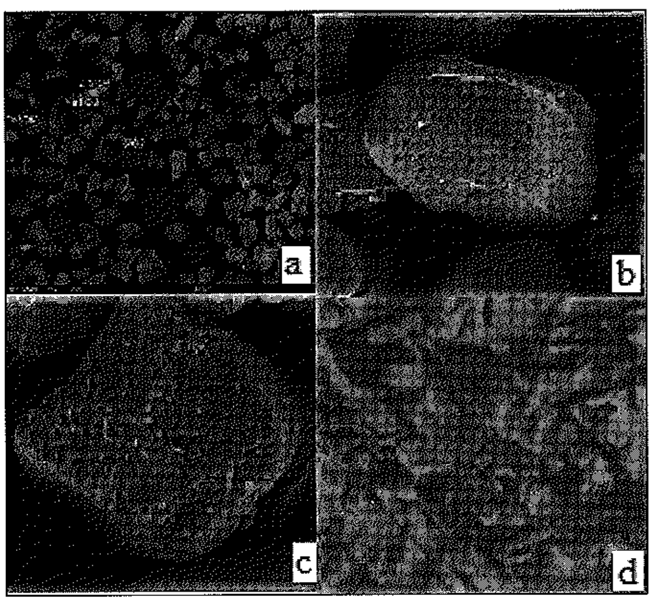

Figure 4. SEM image magnetic sands a) identification of the crystallites, b) and c) ilmenite grains d) magnetite grains

4. Conclusions

X-ray diffraction and X-ray fluorescence showed that the black sands from La Guajira have a high content of magnetite, ilmenite, quartz, and the presence of other elements

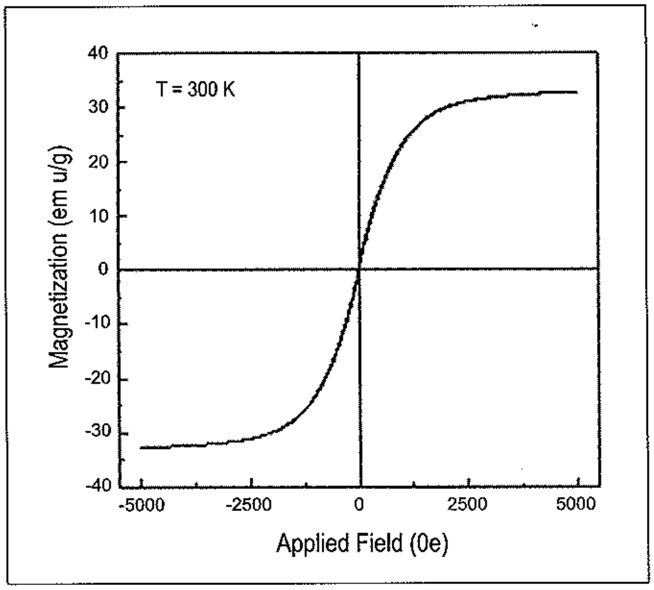

Figure 5. Magnetization as a function of applied field. Hysteresis curve shows the ferromagnetic behavior of the sample.

in low proportion such as zircons. The present phases were 42% of magnetite, 37% of ilmenite, about 10% of quartz, 3% of zircon, and the remaining 8% of the sample represent the presence of other components in lesser amount. Furthermore it can be seen that the heat treatment conducted in the magnetic phases evidenced a change of crystalline structure from magnetite and ilmenite to hematite and titanium oxide. The magnetization curve shows a ferromagnetic behavior due to the presence of minerals such as magnetite and ilmenite where magnetite phase being the largest contributor to this magnetic behavior.

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