

SPECTROSCOPIC CHARACTERIZATION OF IRON-ORE FROM ITAKPE, NIGERIA

Adegoke H. I^{*} and Adekola F. A.

Department of Chemistry, University of Ilorin, P.M.B. 1515, Ilorin, Nigeria

E- mail: ihalimat@yahoo.com; TEL: +2348056455552

+ Correspondence author

ABSTRACT

Physico-chemical and surface characterization of iron ore from Itakpe deposits, Nigeria have been carried out using X-ray fluorescence (XRF) and X-ray diffractometry (XRD) among others. The physicochemical parameters studied were pH, total organic carbon and point of zero charge. The results revealed that the pH, total organic carbon and point of zero charge of the sample are 6.66, 1.68% and 7.26 – 7.70 respectively. The elemental analysis by XRF showed that the iron ore is constituted of Fe (36.04%), O (58.51%) as the major elements, with Cr, Mn, and Zn occurring as impurity elements. Most other impurities found in the ore are silica, alumina and limestone. The XRD confirmed the originality of the iron ore sample and it revealed the presence of hematite (Fe_2O_3) peaks at 33.15, 62.45 and magnetite (Fe_3O_4) peak at 44.70.

Keywords: Iron ore, characterization, Point of zero charge

INTRODUCTION

Iron ores are minerals from which metallic iron can be economically extracted. The ores are usually rich in iron oxides and vary in color from dark grey, bright yellow, deep purple to rusty red. The iron itself is usually found in the form of magnetite, hematite, goethite, limonite or siderite (Min. Inf. Inst., 2006). Iron ore can also be perceived as any naturally occurring iron-bearing formation that contains sufficient quantity and satisfactory form to allow its economic recovery (Cornell and Schwertmann, 2003).

Iron oxides are composed of Fe together with O and/or OH. Some characteristics features of iron oxides include low solubility and thereby high stability, conspicuous colours and high surface area making them effective sorbents for a number of dissolved species (Cornell and Schwertmann, 2003). There are seventeen known iron oxides differing in composition, valence of iron and most importantly in the crystal structure. The degree of crystal order however, could vary and thus may depend on the conditions under which the iron oxides are formed (Cornell and Schwertmann, 2003).

A comprehensive characterization of iron ore is important prior to possible industrial applications. Iron ores from different Brazilian locations (ganga, cryptomelane, hematite and chalcocine ores) have been characterized with the application of Mossbauer spectroscopy. The spectra obtained from the various ores revealed that the ores contain a mixture of natural forms of hematite ($\alpha\text{-Fe}_2\text{O}_3$), goethite ($\alpha\text{-FeOOH}$), cryptomelane ($\text{KMn}_8\text{O}_{16}$), quartz, chlorite and copper ore as the main crystalline phase depending on the type of ore (Costa *et al.*, 1995).

Economic deposits of iron ore in Nigeria have been established for Itakpe with over 182 million metric tonnes. Other locations of economic interest are Ajabonoko Hill and Choko-choko (Ajaka, 2009). The iron ore at Itakpe has been characterized to be of ferruginous quartzite (Ola *et al.*, 2009). It is also considered to be of low grade and thus requires upgrade before it could be fed into a blast furnace (Kollere and Ajiboye, 2009).

The leaching behavior of Itakpe iron ore in mineral acids has also been reported in the literature (Baba *et al.*, 2005; 2007 and Nwoye and Ndilu, 2009). A good number of studies have also been carried out on the engineering properties and beneficiation of Itakpe iron ore (Olubambi and Potgieter, 2005 and Das *et al.*, 2007). Despite the tremendous efforts of these researchers, there exist important information gap on the surface properties such as point of zero charge.

The aim of the present study is to characterize the Itakpe iron ore with special bias to the surface and physico-chemical parameters and with the view of evaluating its potential in the environmental application. This paper therefore discusses the results of XRF, point of zero charge and XRD investigation of Itakpe iron ore.

EXPERIMENTAL

Sample Collection and Preparation

The iron-ore sample was collected from the National Iron Ore Mining Company (NIOMCO) Itakpe, Kogi State, Nigeria. 100g of the sample was collected using a clean stainless trowel and placed in a clean polythene bag. The sample was triply bagged to prevent transit damage and transported to the laboratory. The sample was purified by washing with deionized water, filtered and the residue was air-dried at room temperature for 48 hours.

Sample Characterization

The sample was characterized based on some physical and chemical properties; colour, texture, pH, moisture content, bulk density, organic carbon and organic matter.

(i) The **moisture content** of the sample was determined by heating to constant weight at 105°C. The bulk density was determined by using Archimedes principle and was calculated by taking the weight and volume difference of the measuring cylinder and calculating using mass/volume (Adekola and Adegoke, 2005).

(ii) The measurement of the **pH** of the sample in water and CaCl_2 solution was carried out with a pH meter (model PHS -25) in a 1:10(w/v) suspension of sample in water and 1: 2 (w/v) suspension of sample in 0.01mol/dm³ calcium chloride solution (Black 1979). Aliquots of 5g of sample shaken with 50cm³ distilled water and with 10cm³ of 0.01mol/dm³ CaCl_2 and left for 30 minutes, the pH of the suspension was taken (Patkowaka *et al.*, 2005).

(iii) The **total organic carbon** was determined by adding 10cm³ of 1.0mol/dm³ $\text{K}_2\text{Cr}_2\text{O}_7$ to 2g of the sample in a 250cm³ conical flask. The mixture was swirled until the sample dispersed. 10ml of concentrated tetraoxosulphate (VI) acid was added to the mixture and swirled until the reagent became mixed. The flask was then allowed to stand for 30 minutes. Further 10ml of distilled water was added and 3 drops of ferroin indicator was added and titrated with iron (II) sulphate solution (Black, 1979). The experiment was carried out in triplicate and a blank titration was carried out in the same manner.

Point of Zero Charge (pzc) determination

The point of zero charge using mass titration method (Balderaz *et al.*, 2006; Preocanin and Kallay 1998) was performed by weighing 0 to 2.0g of the sample in seven different conical flasks. 20cm³ of 0.01 mol/dm³ KNO_3 was added to the sample in the flasks and the mixture was shaken on a mechanical shaker (HY-2 Orbital shaker model) for 24 hours. The pH of the mixture was measured after shaking and was adjusted by adding HNO_3 or KOH depending on the pH (Preocanin and Kallay, 1998). The experiment was repeated and recorded for 0.001 mol/dm³, 0.01 mol/dm³ and 0.1 mol/dm³ KNO_3 respectively. The plot of the pH against the mass of sample for the three different ionic strength and point of intersection was noted as the point of zero charge (pzc).

Spectroscopic Characterization

The original sample was subjected to spectroscopic (XRF and XRD) analyses. The elemental analysis of the sample was carried out using X-ray fluorescence (Canberra model). The phase study of the sample was determined using X-ray diffractometer (Phillips Scientific model). This was carried out by pulverization and screening of the sample to the required mesh size. A 0.5g of sample was weighed and placed in the sample holder of about 10mm diameter and smoothened to obtain a polished sample phase. The sample holder was then introduced into the sample cavity and scanned continuously at 0.020° angle internal from 20° (2) to 70° (2) at 0.4000 sec per step (Hutchison, 1974). Both XRF and XRD were carried out at the Centre for Energy Research of Obafemi Awolowo University Ile-Ife Nigeria.

RESULTS

Sample Properties:

The properties of iron-ore sample evaluated are summarized in Table 1.

Table 1: Physical properties of the sample

Properties	Description
Colour	Black/grey
Texture	Brittle
Moisture content (%)	8.68
pH in H ₂ O	6.66
pH in CaCl ₂ solution	7.38
% organic carbon	1.73
% organic matter	2.99
Bulk density(g/cm ³)	2.56
Pzc	7.26 – 7.70

The results of the XRF analysis are shown in Table 2.

Table 2: Results of elemental analysis by XRF

Elements	Fe	Cd	Mn	Zn	I	Ca	Ti	S	Cr	K	Ni	Cu	O
Concentration (%)	36.04	3.59	0.53	0.41	0.38	0.17	0.016	0.14	0.011	0.097	0.089	0.029	58.51

O (58.51%) was obtained by difference

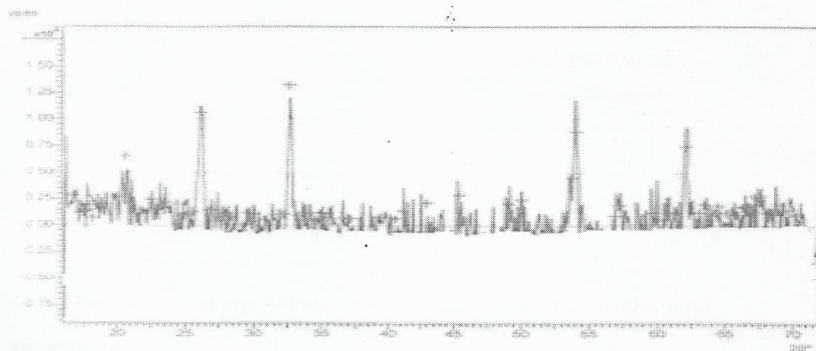


Fig 1: X- ray Diffraction spectra of Itakpe Iron – ore sample

Results of Point of Zero Charge

The experiments were carried out using the mass titration method by weighing 0 to 2.0g of the iron ore sample. A pzc of 7.26 and 7.70 were obtained for the sample after equilibration and acid adjustment respectively. The graphical illustrations are presented in Figs 2 and 3 respectively.

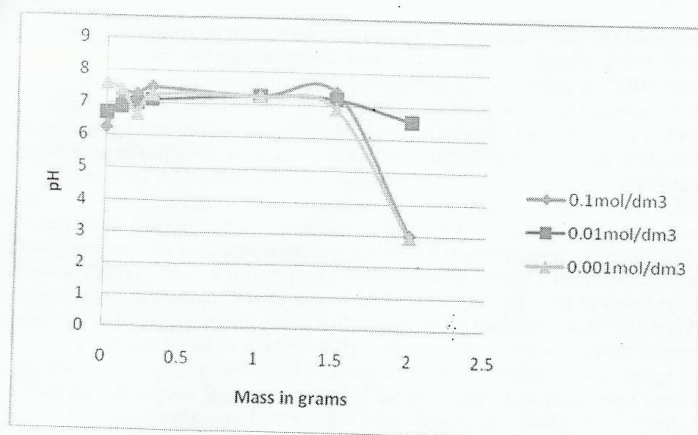


Fig. 2: Point of Zero charge of iron ore after equilibration

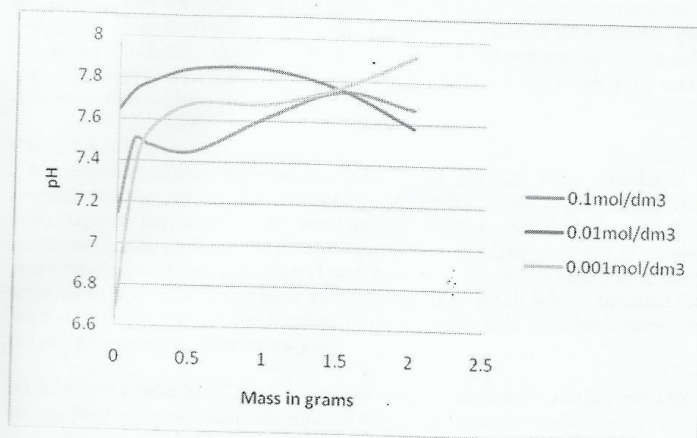


Fig. 3: Point of Zero charge of iron ore after equilibration for acid adjustment

DISCUSSION

From Table 1 the pH of the iron ore was in the range 6.66 for sample's suspension in water and 7.38 for sample's suspension in 0.01 mol/dm³ CaCl₂ solution. These results showed that the sample's solution is near neutrality of about 7.0 ± 0.2. The total organic matter of the sample is 2.99% and this confirms the mineral nature of the sample. The point of zero charge value is in the range of 7.26 - 7.70. This value is in the same range with the values recorded for iron oxides published by Cornell and Schwertmann 2003. A critical examination of the results of pH of the suspension in both water and calcium chloride solution versus the pzc showed that the measurement of pH of sample suspension carried out in CaCl₂ is generally more acceptable and realistic than in water. The measurement in CaCl₂ is realistic as a result of its closeness to the near equilibrium condition unlike in water where there is continuous evolution of the value. This is evidenced from the closeness of pzc to the pH value obtained in CaCl₂ solution.

Table 2 reveals that iron (36.04%) is the dominant element and cadmium (3.59%) is a minor element. The following elements: Mn, Zn, I, Ca, S, Cr, K, Ni, and Cu occurred as trace elements. The

presence of cadmium, an ultra-toxic metal at 3.59% necessitates careful handling of the iron-ore. It is worthy of note that the trace elements present are transition metals. It was observed from the result that the ore is essentially an iron oxide and this is complimented by the XRD's spectra (Fig.1). The result revealed hematite and magnetite peaks. The presence of cadmium in the sample as a minor element shows that cadmium oxide is likely to be present. The level of S (0.14%) is an indication of the presence of sulphidic minerals such as ZnS and CdS in the ore (Das *et al.*, 2007).

From the diffraction spectra patterns of the sample it was observed that the 2 values at 33.000 and 62.500° correspond to that of hematite peaks at 2.668571 and 1.42491 d-spacings respectively while 45.000° corresponds to magnetite peak at 1.99717 d-spacing (Uribe *et al.*, 2006). 2 values at 25.500 corresponds to quartz peak. An earlier XRD examination of Itakpe iron ore by Ola and co-workers (2009) showed major peaks corresponding only to hematite and quartz. The peak for magnetite was not found. The results of XRD in the present work however revealed the presence of magnetite and this was in agreement with the results of Olubambi and Potgieter, 2005.

The result of pzc is in conformity with the value stated by Schwertmann and Cornell for soils rich in oxides of iron (Cornell and Schwertmann, 2003). Point of zero charge (pzc) is the pH where the net particle charge is zero; it is one of the most important parameters used to describe variable-charge surfaces (Parks and Bruyn, 1961; Bourikas *et al.*, 2005). It is a very important parameter because it characterizes the protonation-deprotonation behavior of various types of solid, mainly that of (hydr) oxides in aqueous suspensions (Morais *et al.*, 1976). It is well known that positive or negative surfaces are developed on the surface of solids in aqueous suspensions. The knowledge of the pzc of the materials facilitates the timely choice of surfactant for a specific purpose e.g. in mineral processing (floatation) or the choice of an adsorbent for removal of certain solutes, e.g. from waste water. It makes it possible to predict the pH effect on the phenomena and processes involving adsorption (Kosmulski, 2002).

CONCLUSION

The characterization study of the Itakpe iron ore has been undertaken. The results of this investigation showed that the iron ore contain oxides of iron with hematite constituting the highest percentage in the sample. The point of zero charge values of the sample showed the surface characteristics to be closer to that of hematite sample. The presence of manganese in the ore will make the production of steel easy. There is need to remove cadmium as a heavy metal which could serve as an impediment to obtain pure iron ore. This result further revealed that the sample can be used as an adsorbent for the sorption of heavy metal ions from aqueous solution. Further study is presently on-going on to ascertain this.

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