



Synthesis and characterization of iron oxide pigments through the method of the forced hydrolysis of inorganic salts



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ARTICLE INFO

Article history:

Received 27 December 2014

Received in revised form

14 April 2015

Accepted 20 April 2015

Available online 29 April 2015

Keywords:

Iron oxide nanoparticles

Forced hydrolysis

Synthetic pigments

Characterization

Precursors

Dispersion in white paint

ABSTRACT

Iron oxide hydroxides were synthesized through forced hydrolysis of iron (III) chloride hexahydrate and iron (III) nitrate nonahydrate that as a function of temperature yield different phases characterized by their colors. The tonalities were related to the chemical composition as well as particle size as shown through energy dispersive spectroscopy, thermogravimetric analysis and dynamic light scattering. Iron chloride yielded Akaganèite with different opaque yellow tonalities and a mean particle size of 196 (50 °C), 253 (70 °C), 235 (90 °C) and 170 nm (110 °C). The nitrate precursor yielded Goethite with yellow tonalities and particle size of 360 (60 °C) and 318 nm (70 °C), and Hematite with red tonalities and particle size of 262 (90 °C) and 261 nm (110 °C). The test dispersion and coloring of the white paint (water-based) showed an effective coating capacity, chemical and physical stability of the dispersant and iron pigments.

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1. Introduction

Iron makes up about 6.3% of the Earth's crust; however it is never found in pure form, but instead, combined with other elements, especially oxygen, yielding iron oxides. These iron compounds possess distinct properties such as coloration, resulting from electron transitions between the *d* (t_{2g} and e_g) orbitals. These colors include yellows and reds, which are responsible for soil colors [1,2].

In living organisms, iron oxides are responsible for the activities of many enzymes, and in this way, they can be applied in biomedicine. Synthetic iron oxides are of great importance in many fields. Some oxides are used as catalysts, in redox processes and as pigments [3–8]. They are used to dye paper, rubber, plastics and cement, paint components, varnishes and enamels, because of the range of colors (yellow, brown, red, etc.) that can be obtained [9].

The colors of synthetic iron oxides differ depending on the method and conditions employed [8–16], as for each oxide or oxide hydroxide phase a specific method is necessary. No reporting was

found in the literature regarding the synthesis of different types of iron oxides employing different forced hydrolysis temperatures in order to obtain pigments with different colors.

This article presents the identification of iron oxide hydroxides synthesized at different temperatures of forced hydrolysis (thermo-hydrolysis), beginning with inorganic precursors iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and iron (III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$). These syntheses yielded nanoparticles with a slight change in the composition that is responsible for the colors in these oxide hydroxides. The principal properties studied were thermal behavior, size distribution, morphology and chemical composition. The instrumental techniques used for characterization were thermogravimetric analysis, energy dispersive spectroscopy, Raman spectroscopy and X-ray diffractometry. Particle size was determined through dynamic light scattering and X-ray diffractometry. Colors were attributed through electronic spectroscopy.

2. Materials and methods

In the synthesis of the iron oxide pigments (IOPs), iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, VETEC) and iron (III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, SYNTH) were used. All reactants

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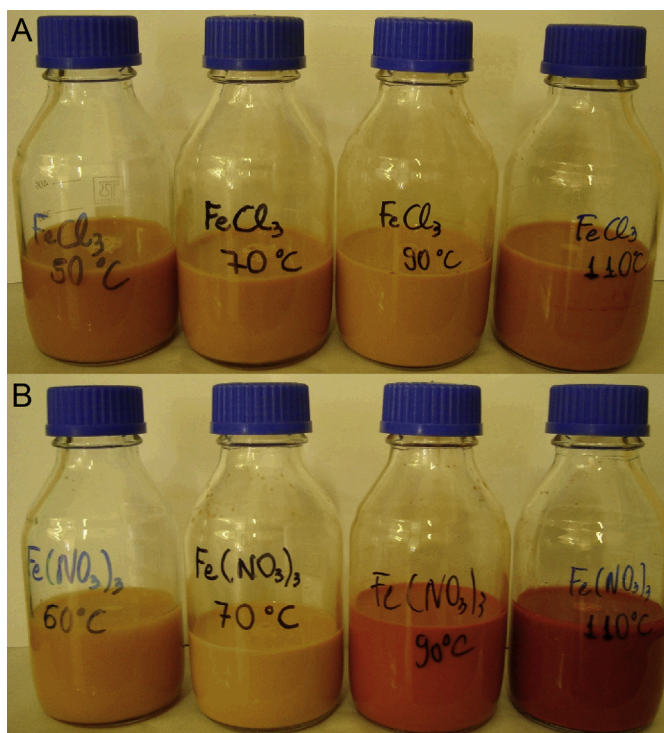


Fig. 1. Photography of the bottles of reactions containing IOPs identified in relation to precursors (FeCl_3 – $\text{Fe}(\text{NO}_3)_3$) and their temperatures the forced hydrolysis.

were of analytical grade, and all solutions were prepared with ultrapure water.

2.1. Synthesis of iron oxide pigments – IOPs

Iron chloride (6.76 g) and iron nitrate (10.10 g) were dissolved in 250 mL of water and stored in autoclavable flasks (limit of 150 °C).

These solutions were transferred to a thermal reactor (QUIMIS, model Q-819v). Syntheses were carried out at 50 °C, 60 °C, 70 °C, 90 °C and 110 °C for a period of 24 h. This period corresponds to the time necessary for the formation of iron oxide hydroxide crystallites [7]. In the studied condition, iron (III) chloride precipitated at 50 °C, while iron (III) nitrate precipitated at 60 °C. All IOPs were washed with ultrapure water using ultracentrifugation at the speed of 10,000 rpm. The pH of the solutions was monitored and remained constant, with values near 3. Fig. 1 shows photographs of the flasks with their respective suspensions, in which the different colors and their tonalities can be observed. The suspensions were frozen (at –7 °C) and lyophilized, and then the IOPs in powder form were characterized as to their structure, morphology and spectroscopic behavior.

2.2. IOPs dispersion in white paint

In order to verify the compatibility and chemical stability of IOPs, they were dispersed in commercial white water-based paint. The dispersion was carried out in the proportion of 100 mg of IOP in 1 mL of water, and then mixed with 1 mL of white paint.

2.3. Characterization techniques

The thermal behavior of the IOPs was studied with a simultaneous thermal analyzer (TG-DTG-DTA) manufactured by Seiko, model 6300. Samples were heated in alumina pans, and the heating ramp was from 30 to 1200 °C, using a heating ratio of 20 °C/min in a compressed air atmosphere (200 mL/min). Chemical analysis were studied through energy dispersive spectroscopy (EDS), model SwiftED-3000, with a tungsten filament as a source of electrons, operated at 15 kV. X-ray diffractometry (XRD) was performed in a Bruker diffractometer, model D2 Phaser, equipped with a copper cathode ($\lambda = 1.5418 \text{ \AA}$), operated at 30 kV, current of 10 mA, with a working window of 2°–90° (2 θ) and increment of 0.02 °/s. XRD data were treated with the software EVA (Bruker version 1.1) and indexed according to ICDD-PDF2 2009 cards. Raman spectra were

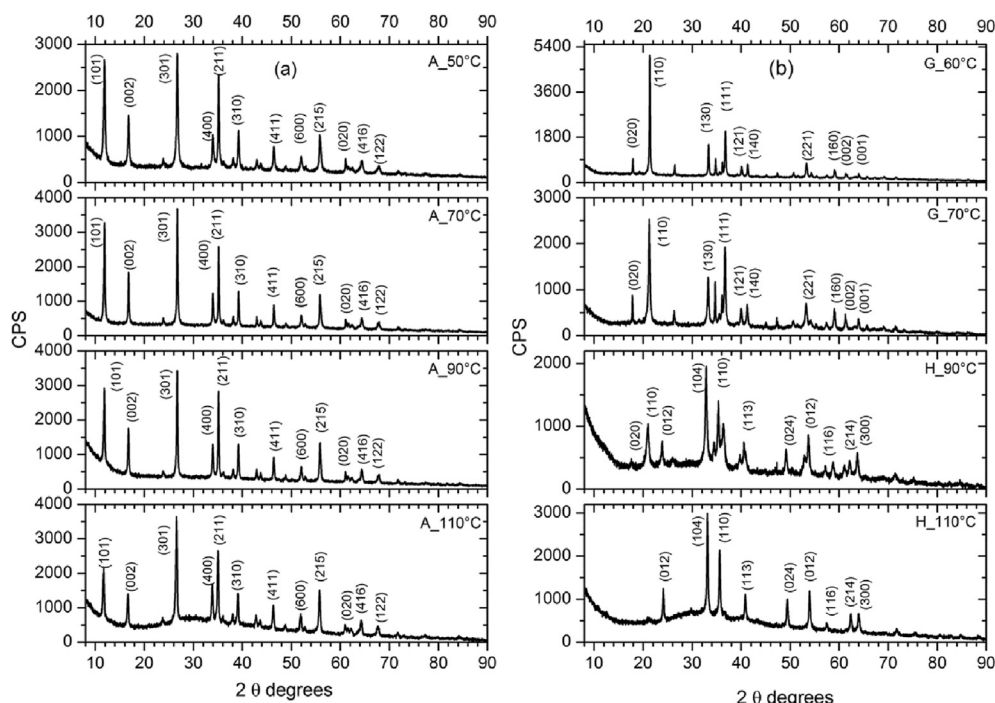


Fig. 2. XRD of the IOPs synthesized according to precursor: A) ferric chloride (FeCl_3) and B) ferric nitrate ($\text{Fe}(\text{NO}_3)_3$).

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