Synthesis and Characterization of Hydrocalumite-type Material Employing Bauxite-washing Residues from Amazon Region (Brazil)

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Abstract

This work deals with the synthesis of anionic clay with hydrocalumite-type material employing Amazonian bauxite tailings. The influence of temperature was investigated in the formation of this lamellar product. The product characterization was performed with X-ray diffraction, X-ray fluorescence, infrared and Raman spectroscopy, scanning electron microscopy and transmission electron microscopy with EDS. The results have revealed that bauxite wastes can convert into hydrocalumite by hydrothermal treatment at 75 °C for 2 days, with a high degree of crystallinity. The XRD pattern, IR and Raman bands of the hydrocalumite-type structure were identified. The results have shown that this type of industrial residue could be a low cost raw material for layered double hydroxide synthesis.

Keywords: synthesis, characterization, hydrocalumite, bauxite residues, Amazon Region.

1. Introduction

Hydrocalumite, $Ca_8Al_4(OH)_{24}(CO_3)Cl_2(H_2O)\cdot 1.6(H_2O)_8$, is a layered double hydroxide which occurs generally in soils and ores. The structure consists of brucite-type sheets linked MO_6 ($M = Ca^{2+}, Al^{3+}$) octahedra with anions and water molecules in the interlayer space. Other cations and anions can be used to obtain hydrocalumite-type materials with unique physical and chemical properties [1 – 3].

Since hydrocalumite possesses excellent ion exchange, surface chemistry, thermal behavior, electrochemical and photocatalytic properties, an extensive research has been carry out to investigate it as selective anionic-exchange, adsorbent, drug deliveries, support catalysts, catalysts, photocatalysts, and for cement industry [3 – 8].

Hydrocalumite-type materials are traditionally synthesized by co-precipitation and hydrothermal synthesis from pure reactants as starting materials and routes from natural sources (mineral, ores, and residues of the mining industry) as low cost starting material are limited [2 - 8]. Studies on the conversion of tailings into layered double hydroxides (and other technological materials) are very important because they can help in reducing the storage of by-products in the environment, as well as the possibility of obtaining products of technological importance [9].

In this work, a facile and low-cost synthesis of Fe-hydrocalumite-type material employing bauxite washing residues from Amazon Region is described.

2. Material and Methods

2.1. Synthetic Route

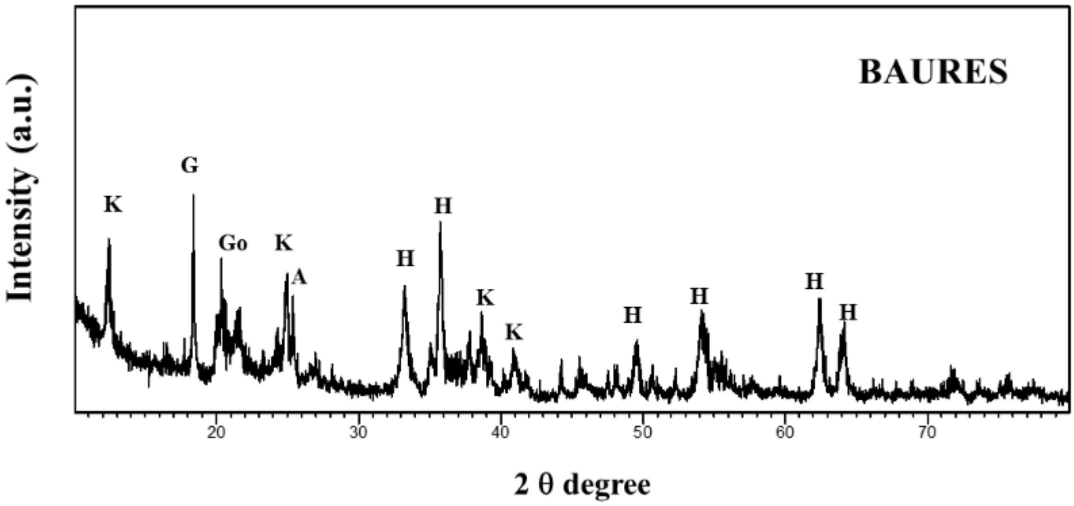
Bauxite residues from Amazon (BAURES) were employed as raw material for synthesis of hydrocalumite. About 1 g of BAURES were added to HCl solution and heated at 90 ºC to obtain a rich solution in Fe^{3+}. Later, the ideal stoichiometric ratio ($Ca^{2+}/ Fe^{3+} = 2:1$) for obtaining hydrocalumite was reached adding a mass of 1.5 g of $CaCl_2 \cdot 2H_2O$ (Sigma Aldrich) to the solution. Finally, NaOH solution was added dropwise slowly under stirring at 25 °C. The influence of temperature was studied at 0, 25, 50 and 75 °C.

2.2. Characterization

The chemical composition of BAURES was obtained by X-ray fluorescence (Axios Minerals, from Panalytical) spectrometer. X-ray diffraction (XRD) was performed in a D2Phaser diffractometer (Bruker), copper tube ($CuK\alpha = 1.5406$ Å) at 30 kV and 10 mA. FT-IR spectra was obtained by using a spectrometer Vertex 70 (Bruker). The Raman spectrum was performed in a Bruker FT 100/S spectrophotometer, with a laser excitation YAG:Nd operating at 1064 nm. The morphology of the synthesized material was obtained by scanning electron microscopy Vega-Tescan, current of 90 mA, voltage of 20 kV and working distance of 15 mm. HR-TEM (Tecnai G2-20 SuperTwin FEI - 200 kV) with EDS measurements were also used to obtain the morphology and microanalysis of the final synthetic product.

3. Results and Discussion

The X-ray diffraction pattern of the bauxite residues (BAURES) which was employed as the starting material is presented in Figure 1. The mineralogical composition showed that kaolinite, hematite, goethite and gibbsite were the main component. The chemical composition showed Fe_2O_3 (~ 50 w.t. %), Al_2O_3 (~ 15 w.t. %) and SiO_2 (~ 10 w.t. %) as main constituents.



Legend: K = kaolinite, G = gibbsite, Go = goethite, A = anatase, H = hematite

Figure 1: XRD pattern of BAURES.

In order to study the influence of crystallization temperature on the formation of hydrocalumite, the crystallization temperature was changed from 0 to 75 ºC for 2 days. Figure 2 shows the

4. Conclusions

The bauxite washing residues composed of kaolinite, goethite, hematite, gibbsite, anatase and quartz were successfully converted into Fe-hydrocalumite-type material. The hydrothermal synthetic route described here showed that temperature influenced the synthesis of well-crystallized lamellar nanomaterial; ideal conditions were around 75 °C for 2 days.

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6. References

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