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Synthesis and characterization of copper and cobalt containing kanemite

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Abstract Cu- and Co-kanemite, with Si/Cu and Si/Co ratio between 200 and 100, were synthesized and parameters such as copper, cobalt source, gel composition and crystallization time have been explored. The results, such as the expansion of the unit cell or the difference of the tetrahedral of silicon, demonstrated that almost all the copper and cobalt has been incorporated into the kanemite framework, even for the gel composition with the lowest Si/M ratio, rendering crystalline kanemite-type particles. Therefore, it is an effective method for synthesizing kanemite with active centres, to be used as an acidic catalyst and as a possible precursor in adsorption of metals.

Many efforts have recently been devoted to the design and construction of inorganic-organic nanomaterials due to potential applications as novel functional materials. Wide varieties of material combinations and synthetic strategies have been employed. Intercalation of organic species into the interlayer spaces of inorganic layered crystals provides unique nanostructures consisting of alternating layers of inorganic and organic moieties[1].

Na-Kanemite, of ideal formulation $NaHSi_2O_5.3H_2O$ is a layered material consisting of single layers of SiO_4 tetrahedra. This structure can support a larger specific surface area then it is possible with other hydrous sodium silicates, such as magadiite, kenyaite etc. The interest in kanemite stems from its high charge density (higher than clays), high reactivity between the silicate sheets and potential uses as a catalyst support and novel adsorbent. In addition, kanemite is used as the starting compound for the synthesis of the microporous and mesoporous materials, which have novel catalytic adsorption properties[2] and controlled release of drug.

The structural characterization of the samples was carried out by X-ray diffraction, infrared spectroscopy, scanning eletronic microscopy and ^{29}Si CP/MAS NMR spectroscopy. The x-ray difratogram indicated a good crystallinity, with characteristics peaks of a lamellar material. The FTIR spectrum of Na-kanemite presents bands between 1300 to 950 cm^{-1} that are associated with asymmetric stretching of Si-O-Si ligation as shown in Fig. 1. The stretching around 460 cm^{-1} refers to Na-O bond. For the modified kanemite it is observed the same bands from Na-Kanemite with few changes in their intensities. Figure 2 presents XRD of Na-kanemite and modified with copper and cobalt with maintenance of crystallinity Further it was observed a discrete increase in the interlamellar distance (from 1.016nm for Na-Kanemite to 1.025 and 1.019 nm to Cu and Co-Kanemite respectively. The SEM of kanemite (Fig. 3) presents simetric morfology with paralell plates, suggesting a lamellar material, as observed for XRD. Co-Kanemite presented two phases and Cu-Kanemite was exfoliated.

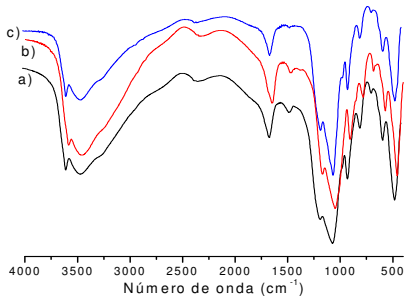

Figure 1: FTIR Spectroscopy **a)** Na-kanemite **b)** Co-kanemite and **c)** Cu-kanemite

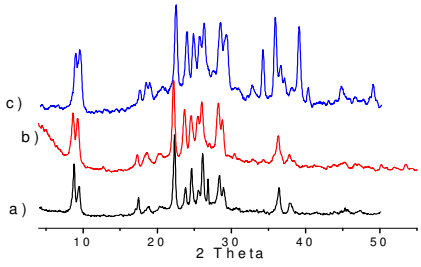

Figure 2: X-ray diffraction: **a)** Na-kanemite **b)** Co-kanemite and **c)** Cu-kanemite.

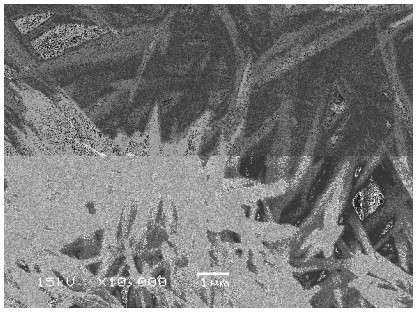

Figure 3: SEM of Na-kanemite

[1] N. Takahashi and K. Kuroda, *Langmuir* **2007,** *23,* 10765-10771

[2] M. D'Amore and L. Marchese, *Chem. Mater.* **2008,** *20,* 4980–4985