

Iron oxihydroxy nanostructured in the clays montmorillonite

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Abstract – The synthesis of akaganeite by hydrolysis gives only approximately 30% yield, but is the most common method found in the literature [3]. Another way to synthesize akaganeite is in basic solutions, but this route requires much time [3]. In this work, emphasis is given to the synthesis of the iron oxyhydroxide known as akaganeite through a new method of basic precipitation of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The resulting brown-colored material, apparently homogeneous, was washed by decantation and maintained in aqueous medium for purposes of manipulation. For structural and spectroscopic characterization, the material was dried at room temperature, in a vacuum, for 24 hours.

Hybrid and mixed sol-gel materials have aroused great interest due to their versatility, ease of preparation, and wide range of applications [1]. They can be obtained from combinations of organic and inorganic compounds using various methods [1]. Among these components are clays, which, combined with oxides, oxyhydroxides and humic substances, are responsible for many important natural processes [2]. One of these processes is the stabilization of the reactivity and structure of iron oxyhydroxides [2].

The synthesis of akaganeite by hydrolysis gives only approximately 30% yield, but is the most common method found in the literature [3]. Another way to synthesize akaganeite is in basic solutions, but this route requires much time [3]. In this work, emphasis is given to the synthesis of the iron oxyhydroxide known as akaganeite ($\beta\text{-FeOOH}$) through a new method of basic precipitation of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The iron salt (23.15 g) was dissolved in 300mL of water and 10 mL of concentrated HCl was added. This solution was heated to 80 °C, after which 42 mL of NH_4OH was added. The final pH of the supernatant solution was approximately 11. The resulting precipitate iron oxyhydroxide was washed to remove the excess OH^- . This precipitate was added to a clay suspension (2.0 %) and stirred for a few minutes and allowed to rest. The resulting suspension was washed to remove the excess of iron oxyhydroxide.

The resulting brown-colored material, apparently homogeneous, was washed by decantation and maintained in aqueous medium for purposes of manipulation. For structural and spectroscopic characterization, the material was dried at room temperature, in a vacuum, for 24 hours. XRD analyses showed that the major phase is akaganeite ($2\theta = 26.85^\circ$), (Fig. 1). Intercalation of the material between the layers occurred, evidenced by a variation in $d(001)$ [2]. FTIR spectra showed (Fig. 2) peaks typical of the formation of the akaganeite phase (1630cm^{-1} , 626cm^{-1} and 490cm^{-1}). Electronic spectra show how the clays promote a reorganization of the material, permitting and intensifying electron-transfer processes. EDX analysis suggests that at least part of the iron oxyhydroxide is distributed on the clay surface. These data also show the synergism between the clay and the iron oxyhydroxide.

These materials were tested and show promise in the breakdown of dyes by a Fenton-like process, and as glucose sensors [2].

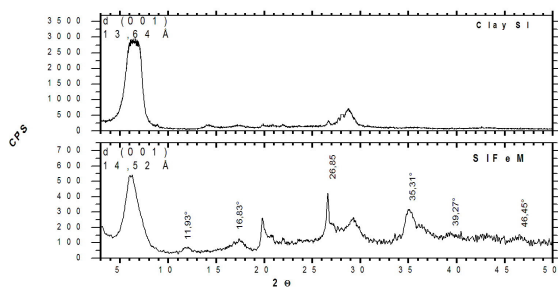


Figure 1: XRD for the clay and mixed material

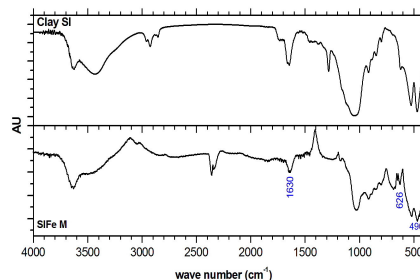


Figure 2: FTIR spectra for the clay and mixed material.

References

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