## Synthesis and characterization of kanemite from fluoride-containing media: Influence of the alkali cation

## JUAN I. CORREDOR,<sup>1</sup> AGUSTÍN COTA,<sup>2,3</sup> ESPERANZA PAVÓN,<sup>4</sup> AND MARIA D. ALBA<sup>3,\*</sup>

 <sup>1</sup>Unidad de RMN-SCAI, Campus de Rabanales, Edificio Ramón y Cajal, Universidad de Córdoba, 14071-Córdoba, Spain
<sup>2</sup>Laboratorio de Rayos-X, CITIUS, Universidad de Sevilla, Avenida Reina Mercedes, 4b, 41012-Sevilla, Spain
<sup>3</sup>Instituto Ciencia de los Materiales de Sevilla, Consejo Superior de Investigaciones Científicas-Universidad de Sevilla, Avenida Americo Vespucio, 49, 41092 Seville, Spain

<sup>4</sup>Unité de Catalyse et de Chimie du Solide, UCCS, CNRS, UMR8181, Université Lille Nord de France, 59655 Villeneuve d'Ascq, France

## ABSTRACT

Kanemite belongs to the group of naturally occurring sodium silicate minerals that was first found in Kanem, at the edge of the Lake Chad, and has been synthesized in different ways from NaOH-SiO<sub>2</sub> mixtures and used as precursor for the design of microporous and mesoporous materials. The fluoride route to the synthesis of microporous materials is based on the substitution of OH<sup>-</sup> anions by fluoride anions, which may subsequently also play a mineralizing role, and gives rise to materials with higher hydrophobicity and thermal and hydrothermal stability. Moreover, F<sup>-</sup> plays an important role in the incorporation of framework heteroatoms, thereby affecting the activity of the final material. The aim of this study was to synthesize fluorokanemite using different synthetic routes and different F<sup>-</sup> source. The final product was characterized by a combination of methods that provided information regarding the incorporation of fluorine into the framework and the short- and long-range structural order of the fluorosilicate. Kanemite with water content close to ideal was obtained in all cases. The washing process was found to have no effect in the long- or short-range structural order of the layer framework, although it did affect the structure of the cation in the interlayer space of kanemite. The mineralizing agent therefore appears to be the key to the synthesis. Furthermore, it governs the resulting kanemite structure by controlling the formation of hydrogen bonds in the framework, and therefore the degree of lamellar structure condensation.

Keywords: Kanemite, mesoporous, microporous, fluorosilicates, MAS NMR, DRX, mineralizing agent, FSM-16