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Structural and thermal study of calcium undecanoate

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Abstract

In the present work, an X-ray diffraction (XRD) and thermal study of calcium undecanoate is presented. The measured high-resolution XRD powder pattern of the synthesized salt at room temperature, using synchrotron radiation, showed that the salt is a mixture of monohydrated and anhydrous calcium undecanoate. Calcium undecanoate monohydrate proved to have a monoclinic cell with a symmetry described by the $P2_1/a$ space group. The structure dehydrates at about 100°C. After dehydration, the salt undergoes a phase transformation which results in a thermotropic mesophase. Further heating of the salt leads to decomposition and melting. Ketones are the probable products of decomposition at 400°C.

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1. Introduction

Calcium soaps are materials that serve a wide range of industrial applications as detergents, softeners, plasticizers, greases, lubricants, cosmetics and medicines. Their selection for specific applications is governed by their fundamental properties [1].

Calcium salts of unsaturated carboxylic acids are also of interest because of their presence in the staple food of Mexican and other Central American peoples: the corn tortilla [2].

However, many metal soaps have been poorly characterized and most studies have been carried out several decades ago using XRD and thermal methods available at that time.

Because of their wide use in industry, the knowledge of the structural and thermal behavior of the alkaline metal soaps is of interest.

The present work is part of a more ambitious systematic study of the calcium salts of aliphatic carboxylic acids with different chain lengths [3–5]. It deals with studies of infrared, X-ray, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) of calcium undecanoate carried out in order to investigate the structure and thermal behavior of this calcium salt in the solid state.

2. Experimental

The calcium salt of undecanoic acid was prepared from SIGMA grade $\text{Ca}(\text{OH})_2$ and undecanoic acid ($\text{CH}_3-(\text{CH}_2)_9-\text{CO}_2\text{H}$), using the same procedure previously described [5]. We will call the obtained sample C_{11} .

The XRD powder patterns were measured in a Siemens D5000 diffractometer using $\text{CuK}\alpha$ radiation at 40 kV and 30 mA, with a diffracted beam graphite monochromator. The measurements were performed at room temperature in the $2-50-2\theta$ range, measuring 20 s

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