# The effect of temperature on the preparation of hydrocalumite under microwave irradiation from aluminum saline slag

**Alejandro Jiménez (1*), Alexander Misol (1), Miguel Ángel Vicente (1), Vicente Rives (1), Antonio Gil (2)**

(1) GIR-QUESCAT, Departamento de Química Inorgánica. Universidad de Salamanca, 37008, Salamanca (España)
(2) INAMAT2, Departamento de Química Aplicada. Universidad Pública de Navarra, 31006, Pamplona (España)
* corresponding author: alejm@usal.es

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## INTRODUCTION

Secondary Aluminum Production (SAP) is based on one of the most interesting properties of aluminum: it can be recycled an infinite number of times without losing its properties and quality compared to aluminum produced from bauxite by combination of Bayer and Hall–Héroult processes (Primary Aluminum Production) (Gil, 2005). Different types of wastes are generated during primary and secondary aluminum production (Jiménez et al., 2022). One of the most hazardous is salt cake, which contains a large amount of aluminum, that can be recovered by acid or alkali extraction processes (Jiménez et al., 2022) and the extraction solutions can then be used as sources in the preparation of compounds based on this metal, such as zeolites and Layered Double Hydroxides (LDHs) (Jiménez et al., 2021a; 2021b).

Layered Double Hydroxides (LDH) are anionic clays with a general formula $[M(II)_{1-x}M(III)_x(OH)_2]^{x+}[A^{n-}]_{x/n}\cdot mH_2O$, where M(II) and M(III) are divalent and trivalent cations and A is the interlayer anion (Rives, 2001). When the divalent cation is $Ca^{2+}$, the trivalent cation is $Al^{3+}$, and the anion is chloride, the LDH is called hydrocalumite ($Ca_2Al(OH)_6Cl\cdot 2H_2O$). The effect of the reaction temperature on the properties of hydrocalumite prepared under microwave (MW) irradiation was studied.

## EXPERIMENTAL PROCEDURE

The solids were prepared by the coprecipitation (Rives, 2001) method from an $Al^{3+}$ solution obtained after treating an aluminum slag with aqueous NaOH under reflux conditions and the subsequent removal of silicon species by precipitation upon addition of HCl up to pH = 1 (Jiménez et al., 2021b), the resulting supernatant was submitted to a microwave (MW) treatment. Characterization of the obtained solid was carried out by powder X-ray diffraction, thermal analysis, infrared spectroscopy, element chemical analysis, electron microscopy and $N_2$ adsorption–desorption at –196 ºC. The sample obtained without MW treatment was denoted as ST, and those obtained after MW treatment were denoted as MW – T, T being the temperature treatment in Celsius.

## RESULTS AND DISCUSSION

Following the methodology reported by Jiménez et al. (2022), the aluminum content in the final acid aluminum–containing solution was 7260 mg/L. The PXRD patterns of the solids are shown in Figure 1. Calcite ($CaCO_3$), katoite ($Ca_3Al_2(OH)_{12}$) and hydrocalumite were detected in the solids. Hydrocalumite was detected in all samples, while calcite was only detected in samples ST and MW–90, and its formation may be due to exposure to atmospheric $CO_2$ during sample handling. Although the synthesis was carried out under $N_2$, the reaction mixture was exposed to the atmosphere during handling mainly in the last centrifugation step, and the most alkaline solids may fix enough $CO_2$ to form detectable amounts of calcite. Katoite was clearly detected in samples MW–100 and MW–110, and with very low intensity in MW–120. Hydrocalumite was the only phase detected in samples MW–125 and MW–130.

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The main difference between both samples was the crystallinity, this being higher for sample MW–125. Hydrocalumite and calcite were formed in the sample without MW treatment.

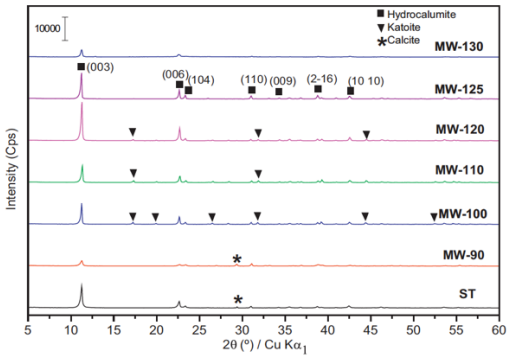


***Fig 1****. X-ray patterns of the prepared solids.*

The theoretical a and c crystal parameter values for hydrocalumite were 5.75 Å and 23.49 Å, respectively. These cell parameters were calculated for the synthesized solids from the interplanar distances of planes (110), and (003) and (006) (a = 2d(110) and c = 3/2[d(003) + 2d(006)]) respectively (Table 1). Cell parameter a varied between 5.75 and 5.77 Å, very close to the theoretical one. Similarly, the c value varied between 23.51 and 23.63 Å, being very similar, although very slightly higher than the theoretical value. Table 1 also shows the crystallite size (D) of the samples (calculated from Scherrer's Equation) along the stacking direction (d(003)) and direction (110) (d(110)).

| Sample | a(Å) | c(Å) | $D_{(003)}$(nm) | $D_{(110)}$(nm) |
|---|---|---|---|---|
| ST | 5.77 | 23.63 | 45 | 85 |
| MW -90 | 5.75 | 23.59 | 29 | 65 |
| MW-100 | 5.76 | 23.61 | 74 | 100 |
| MW-110 | 5.75 | 23.51 | 60 | 102 |
| MW-120 | 5.77 | 23.57 | 62 | 241 |
| MW-125 | 5.76 | 23.63 | 87 | 189 |
| MW-130 | 5.75 | 23.60 | 49 | 110 |

***Table 1****. Parameters determined for the synthesized solids.*

The results showed that the use of the extracted aluminum solution allowed the obtention of hydrocalumite by the coprecipitation method and that the temperature of the MW ageing treatment had a large effect on the formation of side phases, in addition to hydrocalumite.

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