



## Short communication

## Potassium chloride production by microcline chlorination

Pablo Orosco<sup>a,b,\*</sup>, María del Carmen Ruiz<sup>a</sup><sup>a</sup> Instituto de Investigaciones en Tecnología Química (INTEQUI), Chacabuco y Pedernera, San Luis, Argentina<sup>b</sup> Facultad de Química, Bioquímica y Farmacia, Universidad Nacional de San Luis, Chacabuco y Pedernera, San Luis, Argentina

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## ABSTRACT

The potassium chloride is one of the most important fertilizers used in agriculture. The current demand of this salt makes interesting the study of potassium chloride production from unconventional potassium resources. In this work the potassium chloride production by chlorination of microcline was investigated. The starting reagents were microcline, hydromagnesite and chlorine.

Non-isothermal and isothermal chlorination assays were carried out in a thermogravimetric device adapted to work in corrosive atmospheres. The temperature effect on potassium extraction and the phase transformations produced during chlorination of microcline were studied. The reagents and reaction products were analyzed by X-ray fluorescence (XRF) and X-ray diffraction (XRD). The experimental results indicated that by chlorination of microcline an important extraction of potassium in the temperature range from 800 to 900 °C was produced. Moreover, at 800 °C the forsterite, enstatite and magnesium aluminate spinel phases were generated.

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## 1. Introduction

Nowadays, the fertilizers have an important role in the efficient production of agricultural products. Potassium chloride (KCl) is one of the most important fertilizers as it supplies potassium to plants, which is an essential element for most of them. This salt performs the function of activating numerous enzymes from vegetal cells. Moreover, it is the main osmotic potential contributor due to its abundance in the cells. Worldwide, the potassium chloride is employed mainly in the fertilizer industry and in less extent in chemical industry for manufacturing hydroxides, carbonates and nitrates. In Argentina, the use of KCl is limited to the agricultural sector. This country is a great consumer of potassium chloride; however, it does not have a big production of this salt. At the present, Argentina imports potassium chloride, mainly from Germany and Canada, principal producers of this salt [1,2].

Argentina has deposits of KCl located in the south of Mendoza province and in the north of Neuquén province. In these deposits, the potassium chloride contains impurities such as NaCl, CaSO<sub>4</sub> and clays. The methods of purification and concentration used are

washing and attrition, flocculation with polyacrylamide, and crystallization and flotation [2,3].

Taking into account the small amount of deposits in Argentina and the cost of processing involved in the commercial elaboration of potassium chloride, it is interesting to study new methodologies to process KCl from deposits and the use of other potassium sources.

Several researchers have studied the potential use of rocks rich in K such as K-feldspars, micas and feldspathoids as potassium sources. The extraction of potassium was carried out through processes of crushing, milling and leaching using HNO<sub>3</sub> as leaching agent. However, the results obtained showed a low efficiency in extracting this metal [4–6]. Kleiv and Thornhill [7] have used mechanical activation and leaching with distilled water and HNO<sub>3</sub> to extract potassium from nepheline syenite and microcline, reaching a considerable metal recovery. Despite the efficient extraction that was achieved, this method requires a high quantity of energy.

The pyrometallurgical process of chlorination can be an alternative way for extracting potassium as KCl from microcline. This method has been effectively used in the extraction of various metals of oxides and minerals in the last decades. This is due to the high reactivity of the chlorinating agent, the selectivity of the reaction, simple treatment of effluents, and low cost of the processes [8–10].

The main aim of this work is to study the feasibility of producing potassium chloride by chlorination of microcline.

\* Corresponding author at: Instituto de Investigaciones en Tecnología Química (INTEQUI), Chacabuco y Pedernera, San Luis, Argentina.  
Tel.: +54 2664426711; fax: +54 2664426711.

E-mail addresses: [porosco@unsl.edu.ar](mailto:porosco@unsl.edu.ar), [pablorosco@gmail.com](mailto:pablorosco@gmail.com) (P. Orosco).

## 2. Experimental procedures

### 2.1. Materials and procedure

The solid reagents used in this work were microcline from San Luis, Argentina, supplied by *Minera Piedra Grande S.A.* and hydromagnesite p.a. quality, provided by Sigma–Aldrich.

The gases used in the different thermal treatment analysis were chlorine 99.5%, supplied by Cofil, Argentina and nitrogen 99.99% provided by Air Liquide, Argentina.

The minerals were mixed in a ratio of 70% (w/w) of hydromagnesite and 30% (w/w) of microcline. This ratio was established in order to generate new phases that promote reactivity of the chlorine with the potassium. The preparation of the mixture, denominated M, was made in a disk mill, and the mixing time was 4 min.

The chemical composition of the minerals and the M sample was determined by X-ray fluorescence (XRF). The results obtained are presented in Table 1.

The primary minerals which are present in the mixture under study were identified by X-ray diffraction (XRD). The diffractogram of the M sample (Fig. 1) shows the presence of the crystalline phases microcline and hydromagnesite.

### 2.2. Equipment

The experimental chlorination assays were performed in a thermogravimetric system designed in our laboratory [11]. The device is provided of a quartz reactor placed inside an electric furnace equipped with a temperature controller.

The measurement system of mass is an analytical scale (Mettler Toledo AB204-S/FACT, maximum sensitivity of 0.0001 g) connected to an automatic data logger.

The X-ray diffraction analysis of reactants and reaction products was performed on a Rigaku D-Max-IIc equipment with Cu K $\alpha$ , operated at 35 kV and 30 mA. The composition of microcline and hydromagnesite mixture and the chlorination residues were determined by X-ray fluorescence using a Philips PW 1400 equipment.

### 2.3. Procedure

Isothermal and non-isothermal chlorination assays were carried out using masses of approximately 1 g of powder sample. For the different thermal treatments, flows of 50 ml/min of N<sub>2</sub> and 100 ml/min of Cl<sub>2</sub>/N<sub>2</sub> (50%, v/v) were used.

In each non-isothermal experiment, the samples were calcined in the gaseous mixture Cl<sub>2</sub>/N<sub>2</sub> (50%, v/v) at a heating rate of 5 °C/min until a temperature of 900 °C was reached. The mass change was recorded as a function of temperature.

In each isothermal assay, the reaction temperature was set and N<sub>2</sub> fed into the reactor. Once this temperature was reached, the sample was placed inside of the reactor and the Cl<sub>2</sub>/N<sub>2</sub> (50%, v/v)

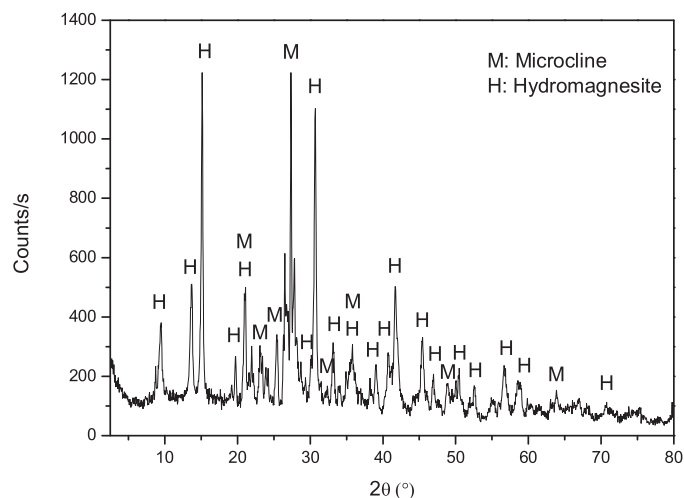


Fig. 1. M sample diffractogram.

was allowed to flow during a reaction time of 15 min. When this period of time was over, the flow of Cl<sub>2</sub> was interrupted, and the sample was purged with N<sub>2</sub> while the reactor was cooled down.

### 2.4. Additional trials

Isothermal chlorination assays were carried out between 650 and 900 °C in Cl<sub>2</sub>/N<sub>2</sub> (50%, v/v) atmosphere during 120 min to determine the optimum temperature for potassium chloride production.

### 2.5. Washing and filtration of the chlorination residues

All the residues from the isothermal chlorination assays performed to reaction times of 15 min and 120 min were subjected to processes of washing and filtration with water at room temperature in order to remove the chlorides generated during the reaction. Then, the residues obtained were dried in an oven at 100 °C. In some cases, the filtering liquid was evaporated in an oven at 100 °C.

The washed and dried residues, corresponding to a chlorination period of 15 min were analyzed by XRD. Also, the crystallized solid from evaporation of filtering liquid obtained by washing of the chlorination residue of the M sample at 700 °C was analyzed by XRD.

The washed and dried residues from the chlorination assays performed during 120 min were analyzed by XRD and XRF.

## 3. Results and discussion

The results of non-isothermal calcination assays performed in Cl<sub>2</sub>/N<sub>2</sub> mixture are shown in Fig. 2.

The thermogravimetric curve of M sample presents three zones of mass change. The first and second zones correspond to the phenomena which produce mass losses, whereas a mass gain occurs in the third zone. The first region can be observed in the range between 90 and 360 °C. The second zone takes place in the range between 360 and 480 °C. The last region can be noted between 700 and 900 °C.

With the purpose of clarifying the phenomena that may occur during thermal treatments in Cl<sub>2</sub>, additional isothermal assays in Cl<sub>2</sub>/N<sub>2</sub> mixture were performed in the temperature interval ranging from 360 to 900 °C. The residues of these assays were analyzed by XRD. Fig. 3 shows the diffractograms of the

Table 1  
Chemical composition (% w/w) of the starting minerals and M sample.

	Mineral		Sample M
	Microcline	Hydromagnesite	
Al <sub>2</sub> O <sub>3</sub>	20.87	–	6.26
SiO <sub>2</sub>	67.70	–	20.31
K <sub>2</sub> O	10.45	–	3.14
CaO	0.19	–	0.06
MgO	–	43.10	28.01
Fe <sub>2</sub> O <sub>3</sub>	0.70	–	0.02
LOI	–	56.90	39.94

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