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Research paper

Characterization of porous clay ceramics used to remove salt from the saline soils

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ABSTRACT

This paper presents the characterization of porous argil ceramic columns used as a tool to remove salt from the saline soils. The characterization of the samples was performed before and after application for the salt extraction from the soil. Phase, chemical and microstructural characterizations of the whole series of samples were carried out by using X-ray diffraction technique (XRD), ICP-AES measurements and scanning electron microscope (SEM), respectively. Therefore, XRD revealed that quartz is the most important phase. Besides, sodium chloride (halite, NaCl) is the predominant compound formed during the desalination time. The presence of high-temperature phases in the ceramic columns such as diopside and anorthite showed the use of high firing temperatures, in the range 950–1050 °C. In addition, SEM observation on cross-sections clearly showed a network of small dense zones, including quartz grains, interconnected by recrystallized porous phases. These images showed the importance of the pores dimensions and structures on the solution movement and salt deposal. Therefore, it revealed that the porosity heterogeneities could have a great impact on the location of efflorescence at the ceramic column surface. As a consequence, the obtained results allowed us to choose the suitable clay ceramic for the salt extraction from the soil.

1. Introduction

Clay ceramics are materials composed of clays materials, quartz, iron oxides and carbonates (Sánchez-Muñoz et al., 2002; Monteiro and Vieira, 2004). Clay raw materials for ceramic uses have been extensively studied, by e.g. Alliprandi (1979). As the importance of the ceramic material particularly in the developing country, the Tunisian clay formations have been the subject of several studies and researches (Mahmoudi et al., 2008; Hajjaji et al., 2010; Mahmoudi et al., 2010). Furthermore, Tunisia is considered as one of the new important countries for the ceramic industry. There are approximately 70 Tunisian factories specialized in ceramic manufacturing (Medhioub et al., 2012) and the exploration of new clay deposits is important to satisfy demand of clay consuming industries.

In the fabrication process, the raw materials are mixed in proportions taking into account the influence of each component in the properties of the final materials, followed by general processing steps such as pressing, moulding and firing (Sousa and Holanda, 2005). It is well known that the maximum heating temperature, the duration of firing and kiln redox atmosphere are important factors that help to understand the transformations. Furthermore, the relationship between the mineralogical composition of the raw materials and phase changes taking place during their sintering under different conditions was examined by Daskshama et al. (1992) and Jordan et al. (2009). A sintering process takes place between 900 and 1000 °C that consists in the compaction of aggregated particles. Nonetheless, Rice (1987) and El-Didamony et al. (1998) showed that the lattice structures of most clay minerals are collapsed above 950 °C, for example, kaolinite at 950 °C, montmorillonite at 800–850 °C and illite at >850 °C. Additionally, they exhibited that during the firing of a calcareous clay, with a similar composition to the pottery investigated here (with the exception of a lower MgO content), the content of the mineral diopside, which first appeared at 850 °C, gradually increased up to 1050 °C.

Thereby, in order to model ceramic material behavior, Sadowski and Samborski (2003)-Sadowski et al., 2008) showed that the material porosity plays an important role in the material structure and its mechanical behavior. Thus, pores of elliptical or spherical shape arise in the material during the technological process (isotropic pressing and sintering at high temperature (Sadowski and Ro, 1999; Jayaseelan et al., 2002). A promising approach to the analysis of material microstructure has recently been proposed (Sadowski et al., 2005) by the introduction of a so-called grain level micromechanical model.

Moreover, porous ceramics are a class of materials that cover a wide range of structures. This has ultimately led to porous ceramics having a wide range of applications, especially in the field of ceramic industry. They are also used in environmental technologies (e.g. the catalytic converter). These applications strongly depend on the structure, nature,

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pore size, permeability, and specific surface area (Yakub et al., 2012). A promising application of porous clay-based ceramic is their use in desalination. At this juncture, porous ceramic columns formed by argil of traditional pottery were used to remove salt from the saline soils. The chemical and physical characterization of pottery samples in terms of its mineralogical composition provides significant technological information regarding its methods of manufacture, specifically the firing techniques adopted. For this reason, SEM examination of the pottery provides information about the internal morphology developed during firing and the extent of phases of vitrification. Thus, the salt crystallization inside the pores of ceramic or in form of hard crusts had also been confirmed from the analysis of SEM. Additionally, semi quantitative analyses can be gained for some major and minor chemical elements by using the EDX spectrometer attached to SEM equipment. Chemical composition of the whole series of fragments was also determined by using atomic emission spectroscopy performed with an inductively coupled plasma source (ICP-AES). Besides, this technique allowed differentiating the samples through their calcareous nature. In addition, the mineralogical analyses of the clay ceramics were performed using X-ray diffraction method (RXD).

Therefore, the main objective of this paper was to study the chemical, mineralogical and the microstructural characterization that allows the evaluation of the efficiency of the traditional porous ceramic in removing salt from soil. Hence, the obtained results will help understanding the mechanisms responsible for the movement of saline solution and consequently choose the suitable clay ceramic for the salt extraction.

2. Materials and methods

2.1. Salt extraction method from the soil

This treatment consisted in the application of the porous ceramic column to a soil contaminated by salts. The lower section of the column was vertically inserted into the soil, and the upper section remained above the soil surface as demonstrated in Fig. 1. Indoor and outdoor experiences were conducted in a laboratory prototype to determine the salinity variation in different soil depths and locations as well as the salt germination and deposal on the porous ceramic. In addition, soil samples were analyzed to determine the particle size distribution of the soil used in the experiences. The distribution of the soil grains according to their sizes shows that the soil could be classified as fine sand.

During desalination, the salts are transported from the soil to the ceramic through the porous network. In order to improve the solute transport, a few amount of pure water was added in the soil to obtain an unsaturated moistened soil. Therefore, the efficiency of the treatment was evaluated by quantifying the salts contained in the soil by the measurement of electrical conductivity (EC). Material characteristics, especially mineralogical characterizations and structural can also influence the kinetics and efficiency of the treatment.

2.2. Sample selection

Before the use of ceramic for the desalination, ceramic pieces were taken from the column as presented in Fig. 2. Where, C1 is the ceramic cross section before treatment; C2 is the ceramic cross section after treatment and C3 is external surface ceramic after treatment. The segments of ceramics (C1) were first characterized independently of any specific soil or salt to investigate the relationship between their formulation and their properties. Pottery samples were ground into a fine powder between 10 and 15 min, using an agate motor. All powder samples were dried at 105 °C for 24 h and stored in desiccators until they were analyzed.

2.3. Analytical techniques

2.3.1. Inductively coupled plasma analysis (ICP-AES)

Chemical composition of the whole series of fragments was determined by using atomic emission spectroscopy performed with an inductively coupled plasma source (ICP-AES). After grinding of all the shards into a fine powder, about 0.25 g of each powdered sample was dissolved according to the following mineralisation procedure. An acid mixture formed by 20 ml of hydrofluoric acid (40%), 5 ml of Perchloric acid (70%) and 2 ml of nitric acid (65%), was used to dissolve powdered samples in a Teflon beaker, on a hot plate at 125 °C; then they were evaporated twice to dryness. The residuals were dissolved in 10 ml of hydrochloric acid and 5 ml of demineralised water (MILLIPORE); then they were diluted to 50 ml by using HCl. Thus, the aqueous solutions were filtered prior to analysis by polycarbonate/GMF 0.4 mm syringe filters previously washed with a diluting solution. The filter was tested for a possible release of cations during the filtering. Pure diluting solution was eluted through the filter after washing.

When dissolved by acid attack samples from these shards gave an insoluble black residual, due to carbon as ascertained by means elemental micro-analysis. The presence of carbon induces to hypothesise the use of ash together with clay as raw materials for those handicraft objects. Fifteen elements (Si, Na, Ca, K, Mg, Fe, Al, Ti, Mn, Pb, Cu, Ni, S, Co, Zn) were determined in each sample by ICP-AES.

2.3.2. Microstructural studies

The morphological features were determined by a FEI Quanta 200 scanning electron microscope, equipped with an energy dispersive X-ray analyzer (EDX). Analyses were performed in up to five different areas of the ceramic matrix of each sample by continuous raster scanning at 25 kV. Quantitative analyses were corrected using an automated "ZAF" routine.

2.3.3. X-ray powder diffraction analysis (XRPD)

In order to determine the mineralogical composition of the specimens, the samples were characterized by powder X-ray diffraction analysis (XRD) performed with a D8 Bruker AXS powder diffractometer. The studies were carried out using Cu Kα radiation in a 2θ range of

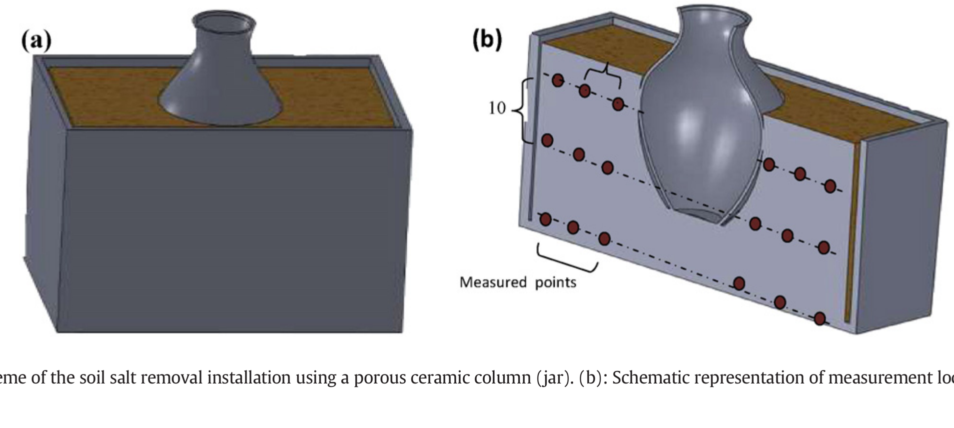

Fig. 1. (a): Scheme of the soil salt removal installation using a porous ceramic column (jar). (b): Schematic representation of measurement locations in the soil.