



A multi-analytical approach to the study of uranium-ore agglomerate structure and porosity during heap leaching



Emerence Hoummady^{a,*}, Fabrice Golfier^a, Michel Cathelineau^a, Laurent Truche^{a,b},
Nicolas Durupt^c, Jean-Jacques Blanvillain^c, Jeremy Neto^c, Eric Lefevre^a

^a Université de Lorraine, CNRS, CREGU, GeoRessources lab., 54518 Vandoeuvre-lès-Nancy Cedex, France

^b ISTerre, UMR 5275 Université Grenoble Alpes, CNRS, 1381 rue de la Piscine, BP 53, F-38041 Grenoble Cedex 09, France

^c AREVA Mines, SEPA, 2 route de Lavaugrasse, 87250 Bessines-sur-Gartempe, France

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ABSTRACT

Agglomeration is commonly used for processing clay-rich ores in order to prevent undesirable effects, especially the risks of plugging or preferential channelling within the heap leaching piles. As the mechanical and chemical stability of the agglomerates is of crucial importance for the behaviour of the pile, a detailed characterization of the structure and porosity of agglomerates seems necessary. In the present study, uranium-bearing clay rich ore was agglomerated and agglomerates were studied before as well as after 10 days of leaching. A multi-analytical approach (by X-ray tomography, SEM, XRD and MIP analyses) was used for the study of the mineralogy, porosity distribution and textures of the agglomerates. X-ray tomography and SEM images showed that agglomerates were the result of coalescence and layering of micro-agglomerates where each one in turn was composed of a nucleus embedded within phyllosilicates and an aluminous silicate matrix. MIP analyses highlighted that unleached agglomerates had a low connected porosity (between 3 and 7%). During the 24 first hours of process, the connected porosity increased by a factor 2 because of the leaching of the aluminous silicate matrix and about 60% of the uranium was recovered. During the next 9 days, the formation of a secondary aluminous silicate matrix derived from the dissolution of illites resulted in the plugging of mesopores and therefore led to a decrease of porosity and uranium extraction extent.

1. Introduction

Heap leaching is a common industrial mining technique consisting of percolating a leaching solution through an ore pile of 4 to 10 m height to extract copper, nickel or uranium. The process relies on fluid-rock interactions and is driven by several physical and chemical factors such as temperature and reagent flow rate (Bartlett, 1997; Ghorbani et al., 2016; Petersen, 2016). Ore mineralogy and particle size distribution especially influence leaching. Fine particles and clays are indeed often the source of plugging within heaps (Ghorbani et al., 2016). This causes permeability issues and channelling, such that some parts of the heap might not be wetted by the leaching solution. This effect compromises the efficiency and the homogeneity of the leaching process.

To enhance the leaching of low grade ores containing clays and fine particles, nickel, copper and uranium ores are agglomerated (Dhawan et al., 2013; Ghorbani et al., 2016). This process occurs after crushing to change the size distribution by bonding fine particles, using capillary

forces, cohesion forces between particles and adhesion forces (e.g. hydrogen bonds, electrostatic and Van der Waals forces). Basically, agglomeration occurs when the sum of the forces tending to disperse the ore are lower than the sum of the attraction forces between fine particles (Pietsch, 2002). This process is claimed to improve the heap permeability and prevent fine particles migration during leaching (Dhawan et al., 2013).

For heap leaching, agglomerates are formed by the adhesion of ore particles mixed water within a rotation drum. Several consecutive steps can be identified during agglomerate formation (Iveson et al., 2001): (i) wetting of particles, (ii) growth of the agglomerates (usually subdivided into the three following steps: nucleation, coalescence of the nuclei and layering of fine particles around the agglomerates), (iii) consolidation and compaction of the agglomerates, and finally, (iv) fragmentation and abrasion. In some cases, a binder can be added to the solid phase prior to wetting or to water to strengthen the agglomerates and promote agglomeration of fines (Pietsch, 2002). The choice of binder depends on the ore mineralogy and leaching solution. Agglom-

* Corresponding author at: Université de Lorraine, CNRS, CREGU, GeoRessources lab., 54518 Vandoeuvre-lès-Nancy, Cedex, France.
E-mail address: emerence.hoummady@univ-lorraine.fr (E. Hoummady).

erates are stacked in the heap for up to 2 weeks before leaching. During that time, bonds between particles are formed (Pietsch, 2002).

However, even with agglomeration, plugging can occur during leaching. This issue, which causes lower leaching efficiency, is related to agglomerate properties. Important efforts have been made to investigate these properties and improve the agglomeration process. As a result, it is now recognized that the operating parameters such as the agglomeration time within the rotation drum, the nature and the rate of the binder and the liquid/solid ratio during agglomeration influence agglomerate morphology and size distribution (Bouffard, 2008; Lewandowski and Kawatra, 2009; Nosrati et al., 2013, 2012a; Velarde, 2005; Vethosodsakda et al., 2013). The agglomeration time must be long enough to produce big agglomerates (Nosrati et al., 2012a; Pietsch, 2002). According to Bouffard (2008), the agglomeration time should exceed 2 min. The choice of the binder is also important: a good binder must have both the adequate physico-chemical properties to improve agglomerate strength and leaching efficiency, and a low cost. In the case of acid leaching, sulfuric acid as a binder is commonly used as it initiates dissolution of copper, nickel or uranium minerals before the start of heap leaching. Other binders have been tested such as stucco or polyacrylamides (Kodali et al., 2011; Lewandowski and Kawatra, 2009) but they are rarely used at the industrial scale due to their cost. Finally, the impact of moisture content within agglomerates has also been investigated. This parameter influences capillary forces within the agglomerates, for optimal performances, agglomerates should contain an optimal water content. An increase of agglomerates water content especially increases agglomerate size (Vethosodsakda et al., 2013).

Agglomerate morphology may influence heap leaching efficiency. Numerous tests, such as soaking, permeability, size distribution, conductivity, and strength, have been performed to control agglomerate morphology (Bouffard, 2005; Dhawan et al., 2013; McFarlane et al., 2011; Pietsch, 2002; Lewandowski and Kawatra, 2009). According to these tests, ideal agglomerates should be sufficiently strong to remain porous under the weight of the heap (Liu et al., 2012). McClelland (1988) concluded that if agglomerates don't disintegrate after 24 h of soaking in water or acid, they should be strong enough to remain intact during clay swelling. Similar soaking tests were also used to quantify fine particles migration (Lewandowski and Kawatra, 2009).

Apart from Kodali et al. (2011) studies, few have focused on the evolution of the agglomerate structure in batch leaching. In addition, petrophysical properties of agglomerates, such as porosity, were insufficiently investigated and the link between microstructure and the properties of an agglomerate is not clearly identified at this point, with the exception of the recent studies by Quaicoe et al. (2013) and Nosrati et al. (2013, 2012b). These authors have analysed internal microstructure and porosity of dried nickel lateritic ore agglomerates with X-ray tomography and SEM analyses. They highlighted that agglomerates appeared to have heterogeneous structures and to be composed of sub agglomerates, due to their mechanisms of formation. They also showed that the majority of the agglomerate porosity was located between these sub agglomerates. In addition, when agglomerates dried, their porosity, permeability and compressive strength increased, due to the evaporation of the binder within the agglomerates and crystallization of leached species.

When reviewing the work produced so far, however, all the previous studies on microstructure focused only on nickel laterite or copper-ore agglomerates. To the best of our knowledge, no publication has previously examined the specific behaviour of uranium-ore agglomerates in spite of their mineralogical and chemical differences with nickel laterite or copper ores. Few studies have investigated the evolution of agglomerate structure during leaching. Thus, our work aims at analysing uranium-ore agglomerates and beginning to shed light on the agglomerate microstructure and its related properties. For this purpose, a multi-analytical approach (e.g. X-ray Computed Tomography (CT), SEM analyses, mercury intrusion porosimetry (MIP) and X-ray diffraction

(XRD)) has been adopted. Since MIP and X-ray CT reveal information about the pore distribution and connectivity and microscopy analysis gives an insight on pore geometry and mineralogy, a combination of these techniques was ideal for obtaining a complete picture of agglomerate structure. The influence of sulfuric acid as a binder on agglomerate architecture and porosity has also been studied.

2. Materials and methods

2.1. Ore, agglomeration and leaching processes

Agglomerates were produced from clay sandstone containing about 900 ppm of uranium and > 10% clays, mainly kaolinites, illites, mixed layered illite-smectite phases and chlorites, provided by AREVA from Somaïr, Niger. It also contains a few carbonates (mainly calcite) and no gypsum. Most of the uranium was hosted by clay minerals, especially chlorites. The high clay content of this ore rendered it a good material for agglomeration.

The ore was agglomerated with water and sulfuric acid, at a ratio of 25 kg of acid per 1000 kg of ore, at a liquid/solid ratio of 0.08 kg/kg, within a cement mixer at a speed of 32 rpm. Half of the water was mixed with dry ore. Sulfuric acid was mixed with the rest of the water was added into the mixer and left to agglomerate for 3 min. These conditions are considered as standard agglomeration conditions and the resulting material is hereafter referred to as acid-bound agglomerates. A second batch of agglomerates (water-bound agglomerates) was made without sulfuric acid.

A particle size analysis of the raw ore was conducted, highlighting particles ranging from < 80 µm to 10 mm. In addition, image analysis using ImageJ software was conducted on agglomerates ranging from 1 to 40 mm to determine diameters of the agglomerates. The majority of agglomerates had a diameter larger than 3 mm, which confirmed that fine particles have been gathered into bigger ones.

A representative batch of 10-mm-diameter agglomerates was leached in a column over 10 days with 10 g/L sulfuric acid solution and a flowrate of 7.2 mL/h (i.e. 5.7 L/m²/h, which is a typical irrigation rate for heap leaching according to Petersen, 2016). The leached solution was sampled daily and analysed by ICP OES. The extent of extraction of each element was calculated as moles contained in the leach solution to the total moles within the ore. For purposes of comparison, the analysed elements were classed into three groups, according to their leaching behaviour.

At the end of leaching, agglomerates rested in the open air for 1 h. Three other columns were leached in parallel, for 2, 5 and 7 days respectively.

2.2. Agglomerate analysis

A multi-analysis approach was used to study agglomerate structure and porosity before and after leaching. These analyses were done on both unleached and 10 days leached agglomerates.

Wet agglomerates of each batch of agglomerates were firstly analysed by X-ray tomography with a *Nanotom Phoenix* tomograph. This technique enabled imaging of the internal structure of the agglomerates in a non-destructive manner. Results were then processed using Avizo and VGStudio Max software to investigate changes in pore connectivity. To visualise the agglomerate structure, scanning electron microscope (*Hitachi S-4800*) (SEM) and micro energy-dispersive X-ray spectroscopy (EDS) analysis were also performed on 5 freeze-dried agglomerates of each batch. Freeze-drying removed water without disrupting agglomerate structure, as shown by Pret (2003). Agglomerates used for SEM polished sections were impregnated with poly methyl methacrylate (PMMA) resin, following the protocol of Sammaljärvi et al. (2012). XRD analysis of fines inferior to 80 µm after agglomeration and crushing was performed to better understand mineralogical changes induced by the agglomeration solution.

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