



Effects of cooling method on removal of sodium from active roasting red mud based on water leaching



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ABSTRACT

Red mud cannot be directly employed as the raw material of iron-making and construction materials for the existence of sodium element. The effects of cooling methods of furnace, air, water and liquid nitrogen on roasted red mud for recovering Na^+ with water leaching were investigated through the analyses of TG-DTA, QXRD, FTIR, SEM, etc. The faster cooling methods we used, the better leaching performance would be obtained. Liquid nitrogen cooling sample therefore displayed the best leaching result with concentration of $1202 \text{ mg} \cdot \text{L}^{-1} \text{ Na}^+$ at the first leaching stage (~25 wt.% total sodium recovery). Part of cancrinite known as desilication products transformed into NaCaHSiO_4 and nepheline after the roasting process. In the fast cooling red muds, the new generation of $\text{Na}_2\text{Ca}(\text{CO}_3)_2$ could dissolve directly into water; the increase of $\text{Ca}(\text{OH})_2$ concentration to ~4.8 wt.% was beneficial for the dissolution of cancrinite and NaCaHSiO_4 during the leaching process; amorphous phase increasing from ~4.1 to ~13.5 wt.% made sodium be more easily leached out from sodium-containing amorphous phase than the same crystalline phase; fluey flakes or plate-shape particles weakened the aggregation behaviour; the increase of specific surface area from 1.898 to $2.177 \text{ m}^2 \cdot \text{cm}^{-3}$ led to the contact area increasing between particles and leachant, implying that sodium could be more easily leached out from the fast cooling samples.

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1. Introduction

Red mud is generally a rusty solid waste generated from the aluminium industry via the Bayer process, employing this technology in alumina production currently accounts for >95% worldwide (Klauber et al., 2011). It is well known that the aluminium extraction of Bayer process is conducted by the digestion of bauxite with substantial hot sodium hydroxide solution. Given the generation of desilication products (DSP) mainly presented as sodalite or cancrinite with chemical formula of $\text{Na}_6[\text{Al}_6\text{Si}_6\text{O}_{24}] \cdot \text{Na}_2\text{X} \cdot n\text{H}_2\text{O}$ ($\text{X} = \text{CO}_3^{2-}, \text{SO}_4^{2-}, 2\text{Cl}^-, 2\text{OH}^-$ or 2NO_3^-), the losing of sodium hydroxide will accumulate in the red mud during the alumina extraction process (Abdulvaliyev et al., 2015; Zhang et al., 2011). Moreover, this behaviour will limit its application, especially using as iron-making materials, since the low melting point alkali metals circulating in the blast furnace and eroding the lining (Liu and Li, 2015). In the case of large scale producing construction material from red mud, the strong alkalinity characteristic also brings a negative effect on the application amount of red mud, such as scumming, and weakens the intensity of building materials owing to the alkali-aggregate reaction in the concrete (Tsakiridis et al., 2004; Zhu et al., 2015).

Damming and stockpile are both the primary disposal methods for red mud, while the high sodium content and fine structure of this kind of solid waste could easily deteriorate soil structure as well as intensify the risk of environmental problems such as water pollution and haze (air pollution) to surrounding residents (Li et al., 2014; Ruyters et al., 2011). The unappropriated disposal of red mud not only wastes the other resources but also causes the secondary environmental pollution. Accordingly, it is completely necessary for us to do some work to reduce the concentration of sodium in red mud prior to its further comprehensive utilization.

The sodium removal or recovery methods typically consisting of water/seawater leaching, acid leaching/neutralization, bacteria leaching, CO_2 pressure leaching, lime-hydrothermal treatment and lime-soda sintering coupled with water leaching can be applied to recover sodium from red mud (Chen and Long, 2015; Cresswell et al., 1987; Guoli et al., 2012; Hrishikesan, 1977; Şayan and Bayramoğlu, 2001; Zhang et al., 2012; Zhong et al., 2009; Zhu et al., 2015). In respect of direct water leaching method, which can easily and effectively separate out the dissoluble sodium ions from red mud and yet not take into account most of the chemical bonded ones. However, the active roasting before water leaching process is beneficial to increasing the dissolving capacity of sodium-containing phase because of the phase transformation of DSP $\text{Na}_6\text{CaAl}_6\text{Si}_6(\text{CO}_3)\text{O}_{24} \cdot 2\text{H}_2\text{O}$ into dissoluble mineral phases of $\text{NaOH} \cdot \text{H}_2\text{O}$ and $\text{Na}_2\text{Ca}(\text{CO}_3)_2$ or other crystallized sodium aluminates (Zhu et al., 2015). In comparison to high temperature calcium carbonate

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Table 1

The main chemical composition of red mud measured by X-ray fluorescence (wt.%).

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	Na ₂ O	TiO ₂	K ₂ O	SO ₃	MgO	Others ¹	CaO/SiO ₂ molar ratio	Na ₂ O/Al ₂ O ₃ molar ratio
20.23	19.86	24.22	18.10	6.11	6.69	2.56	0.94	0.61	0.68	~1.0	-0.5

¹ Others include the trace amount of components (<0.5 wt.%), such as ZrO₂, P₂O₅, Cr₂O₃, SrO, MnO, NbO, Y₂O₃ and NiO.

smelting, active roasting seems to be much more energy saving and economically feasible despite ~80% Na recovery lower than that 90% recovery value with smelting at 2.0 CaO:SiO₂ mass ratio, 1400 °C for 3 h and water leaching at 1:5 solid to liquid mass ratio, 60 °C for 8 h (Bruckard et al., 2010; Zhu et al., 2015). 80.7% of similar sodium leaching result can also achieved with roasting condition of 2.0 Ca:Si molar ratio, 1.0 Na:Al molar ratio, 1:200 coal char to red mud mass ratio and subsequently leaching at 60 °C for 15 min, 1:2 solid to liquid mass ratio (Liu et al., 2012). On the one hand, the augmenting CaO contend could reduce the SiO₂ thermodynamic activity in the roasting process; on the other hand, the sodium dissolubility will be improved in the water leaching process (Liu and Li, 2015). Without the addition of lime or soda to red mud before the roasting process, the dealcalization of red mud could also reach 82% with four water leaching stages at 90 °C, 60 min, 7 mL·g⁻¹ liquid to solid ratio (Zhu et al., 2015).

The hydrothermal process demonstrates an excellent performance to recover sodium from red mud and other valuable elements simultaneously. In the early 1990s, Cresswell (Cresswell and Milne, 1982) and Solymar (Solymar et al., 1997) et al. had obtained the favourable sodium recovery result with the hydrothermal treatment. The treatment is generally carried out by cooking the mixture of lime, soda and red mud under the condition of 150–300 °C, 0.1–10 MPa, 3–8 liquid to solid mass ratio, 10–240 min leaching time to transform DSP into andradite-grossularite hydrogarnet Ca₃[Al, Fe]₂(SiO₄)_{3-x}(O₄H₄)_x (0 < x < 3) (Cao et al., 2013; Cresswell and Milne, 1982; Zhang et al., 2011; Zhong et al., 2009). Only one thing to note here is that this method is not quite applicable to Fe-lean red mud as a result of the low isomorphous substitution of Fe to Al in silicate hydrogarnet and the phase NaCaHSiO₄ emerging (Zhang et al., 2011). All the methods mentioned above can be concluded that the sodium-containing complex salts should be subjected to structure changes or phase transitions with the aid of lime/reductive/soda/reducing roasting/smelting first and then the minerals obtained can be available for hydrolysis process to leach out sodium into water at a mild temperature (Bruckard et al., 2010; Chen and Long, 2015; Cresswell and Milne, 1982; Cresswell et al., 1987; Guoli et al., 2012; Hrishikesan, 1977; Zhang et al., 2012; Zhang et al., 2011; Zhong et al., 2009; Zhu et al., 2015). In addition, a specific pressure, higher leaching temperature and longer duration time have a positive effect on the leaching process, but at the same time these conditions will restrict the commercial application by large-scale.

So far these parameters—roasting condition of lime & soda concentration, roasting atmosphere, temperature, time and leaching condition of temperature, pressure, duration time, pH for recovering sodium and other valuable elements from red mud—have been widely researched, whereas there was little attention that is paid to the influence of cooling methods on the dealcalization of roasted red mud with water leaching method. Accordingly, the objective of this work is to monitor effects of the various cooling methods, namely furnace cooling, air cooling, water cooling and liquid nitrogen cooling, on the leaching of sodium from the roasted red mud with five water leaching stage at a specific temperature and residence time.

2. Experimental

2.1. Materials

Bayer red mud used in the present study was sampled from a subsidiary company of Chalco in Guizhou province of China. The chemical

composition of sample was determined by X-ray fluorescence (XRF-1800, RIGAKU Co., Japan) analysis, as listed in Table 1.

2.2. Methods

Thermal behaviour was conducted by Thermogravimetric-differential thermal analysis (TG-DTA, HCT-4, Henven, China) with powder red mud (<74 μm) at a heating rate of 10 °C·min⁻¹ and a range of 25–1200 °C under normal atmosphere. Mineral phases were measured by X-ray powder diffraction (XRD, TTRIII, RIGAKU Co., Japan) with a 2θ scan range of 10–100°, conducting at 40 kV and 40 mA with Cu Kα radiation (λ = 1.5418 Å) at 25 °C, a step size of 0.02° and a counting time of 1 s. Phase identifications were carried out by using Jade software of Materials Data, Inc. (MDI), supported by the Powder diffraction file database (PDF22004) of the International Centre for Diffraction Data (ICDD) and the Inorganic crystal structure database (ICSD). The room temperature Fourier transform infrared spectra (FTIR, Nicolet iS5, American) of samples were carried out by embedding the powder specimen in the fine KBr (analytical reagent) pellet to support the XRD results. The quantitative X-ray diffraction (QXRD) techniques were performed with General Structure Analysis System (GSAS) software based on Rietveld refinement method (Toby, 2001). The reliability of QXRD result was characterized by CHI², -Bknd wRp, -Bknd Rp and R(F²), which can be computed from (Liao et al., 2015; Toby, 2006)

$$CHI^2 = \frac{\sum w(I_o - I_c)^2}{N - P} \quad (1)$$

$$wRp = \sqrt{\frac{\sum w(I_o - I_c)^2}{\sum wI_o^2}} \quad (2)$$

$$Rp = \frac{\sum |I_o - I_c|}{\sum I_o} \quad (3)$$

$$R(F^2) = \frac{\sum_{hkl} F_{o,hkl}^2 - F_{c,hkl}^2}{\sum_{hkl} F_{o,hkl}^2} \quad (4)$$

Wherein, *I* is intensity; *w* is weight assigned to each step intensity; *F* is structure factor; *N* is the total number of the observations in all histograms; *P* is the number of parameters in the least squares refinement. The subscript O and C identify the observed and calculated values.

In order to reduce the errors and quantify the amorphous phase, ~20 wt.% ($\frac{m_{CaF_2}}{m_{redmud}}$) CaF₂ (analytical reagent) was added to the samples as the internal standard material (Liao et al., 2015). Thus, these concentrations of reported phase can be corrected proportionately via the following relationship (Kolb et al., 2012).

$$Corr(W_a) = W_a \frac{STD_{known}}{STD_{measured}} \quad (5)$$

The amorphous phase *W_{amorphous}* value can then be derived from:

$$W_{amorphous} = 1 - \sum_{j=1}^n Corr(W_j) \quad (6)$$

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