



Amenability for processing of oolitic iron ore concentrate for phosphorus removal

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ABSTRACT

Beneficiation routes aimed at dephosphorisation of oolitic gravity magnetic concentrate and involving a combination of roasting, re-grinding, magnetic separation and water and acid leaching are investigated. Roasting was carried out at 900 °C for 1 h without or with lime or sodium hydroxide as roasting additives. When additives were used, cement phases of Si–Al–Na–Ca–O type were detected as well as the mineral giuseppettite. During the thermal process sodium silicate is liquefied and the newly formed phases coat the oolites and penetrate inside the cracks. Energy Dispersive Spectroscopy analysis has indicated that the zone surrounding the oolites consists of Na, Al and Si phases with part of phosphorus being captured there. As a result of the alkaline roasting, goethite is partly transformed to magnetite and this reduction is reinforced with an increase in sodium hydroxide dosage. Investigation of redistribution of phosphorus shows that it could be only partly separated if leaching is not accompanied by re-grinding and physical separation. The recommended dosage of the reductive agent for the final flowsheet is 8 mass% ratio to concentrate. Grinding to a mean size of 0.040 mm, with water and acid leaching and double magnetic separation creates conditions to obtain a high-quality iron concentrate with 65.97% Fe and recovery of 92.43%, with simultaneous decrease in the phosphorus content from 0.71% to 0.05%.

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1. Introduction

Compared to magnetite and hematite, goethite iron ores have a quite limited share as raw resources in steel making. Their use is restricted mainly due to the observed spalling during firing and because of reduced capacity of blast furnaces. Owing to the forecasted depletion of the available direct shipping high grade iron ores, a constant growth of the goethite share could be expected. The “Lester Plan B 2.0” suggests that iron ore could run out within about 64 years based on an extremely conservative extrapolation of 2% consumption growth per year (Brown, 2006). In parallel with this trend, the utilization of high phosphorus iron ores will increase (Bristow, 2011), and consequently enabling dephosphorisation technologies should be developed. Finding a solution to this problem will have significant economic importance, since it will enable huge new iron ore reserves to be included in the resource base. Very often iron minerals found in oolitic limonite ores are cemented by the Al-silicates or even worse, phosphorus is seen as phosphatic groups embedded in the goethite structure which renders these ores very difficult to beneficiate by physical dressing, (Graham, 1973; Peixoto, 1991; Zhu et al., 2010). The beneficial effect of roasting on phosphorus leaching is already being investigated for Lisakovskiy oolitic iron ore in Kazakhstan, and as a consequence roasting is included in

the flowsheet of the Experimental Dephosphorisation Plant in Lisakovsk, (Akotov et al., 1969; Kotov et al., 2003; Volovyuk et al., 2010). During the thermal process, phosphorus is driven out from the crystal lattice of hematite forming blebs along the cracks and particle surfaces (Gooden et al., 1974). The influence of various additives in roasting on the dephosphorisation efficiency of iron ores has been studied by adding metals and ammonium halides, namely CaCl₂ or NH₄Cl, MnCl₂, ZrCl₂, CuCl₂, and LiCl (Feld et al., 1966). The ore has been roasted for 2 h at a temperature range between 500 °C and 1200 °C, the optimum one being determined as 900 °C. The most economical salt has been found to be the CaCl₂, and within a 30 min leaching with mineral acid at 30 °C, 23% of the phosphorus has been removed. The high phosphorus oolitic hematite ores are more amenable to dephosphorisation by physical separation than the limonitic ones (Jiang et al., 2008). Following beneficiation of such ore graded at 37.51% Fe and 0.88% P by spiral chute and shaking table, an iron concentrate with 58.67% Fe and 0.52% P at 51.93% recovery has been obtained. The concentrate has been leached with sulphuric acid (1%) at 10% solids which brought the final product to 59.26% Fe and 0.056% P. The removal of phosphorus from iron ore using a low temperature heat treatment with additives, followed by alkaline or water leaching, has been tested by Fisher-White et al., (2009), with phosphorus being lowered from 0.145–0.075%. One of the conclusions drawn from this study is that lower level of phosphorus could be achieved if the heating temperature is kept above 800 °C.

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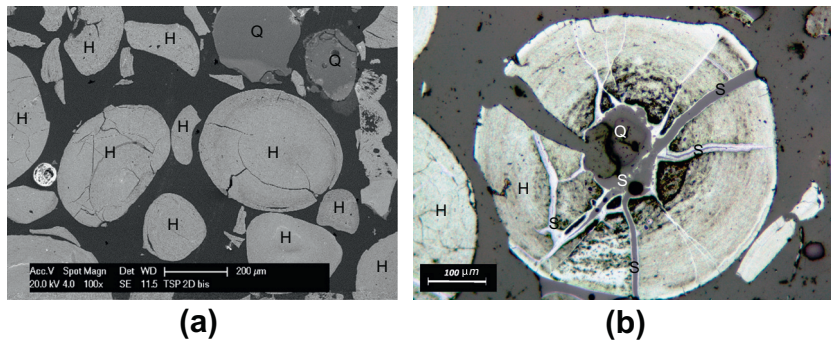


Fig. 1. Section views of roasted samples; (a) SEM of roasted without additive oolites, and (b) optical image of roasted with NaOH (H – hematite, Q – quartz and S – sodium silicate).

2. Experimental

2.1. Materials and methods

The oolitic gravity magnetic (GM) iron concentrate was supplied by the Lisakovsky iron ore beneficiation plant in Kazakhstan. The plant flowsheet involves ore sieving at 1.6 mm and desliming with further processing through gravity (jigging) and magnetic (low and high intensity) separation stages. The obtained GM concentrate therefore has a relatively narrow particle size distribution with a mean particle size of 0.425 mm. Its principal composition is given as 48.76% Fe, 0.71% P, 12.41% SiO₂ and 5.93% Al₂O₃. The main gangue mineral is quartz observed inside the oolites, cemented with goethite by the clay minerals, and as liberated particles.

Sulphuric acid is employed as the leaching agent, while NaOH, Ca(OH)₂ are used as roasting additives. H₂SO₄, NaOH and Ca(OH)₂ were all analytical grade reagents. De-ionized water is used for the preparation of the acid solutions and leaching suspensions as well as for washing out the salts from the magnetic fractions.

Dry grinding of the concentrate is realized in a laboratory hammer mill. Particle size distribution of ground samples and beneficiation products were determined by a ZETASIZER laser diffraction unit, Malvern Instruments, UK. Chemical composition of the as-received and leached concentrate was determined by sequential XRF XP X-ray spectrometer ARL 9400. Microstructures inspection of raw oolites and roasted products was done by optical microscope OLYMPUS BX60, Scanning Electron Microscope PHILIPS XL 30 and SEM/EDS JEOL system coupled with an OXFORD X-ray detector. The mineralogical analysis was carried out using Philips XRD system with data being processed by X'Pert software.

2.1.1. Roasting

The combination between alkaline roasting and acidic leaching has been successfully applied for nickel recovery from refractory lateritic ores, (Guo et al., 2011; Dong et al., 2009). The same study reports that high leaching efficiency was achieved at mild leaching conditions with optimal results obtained at a mass ratio of sodium hydroxide to ore of 0.6. However, such high dosage does not seem viable for iron ores; therefore in our investigation the additive/concentrate ratio in roasting is fixed at 0.05, i.e. 5% on a dry weight basis. When the samples were intended for observation, roasting additives were supplied at 10 mass%, due to the detection limit of the XRD instrument. Ca(OH)₂ and NaOH were chosen as additives due to their highly alkaline character and affordable cost, making them feasible from an industrial viewpoint. The additive was carefully mixed with 2 kg sample of concentrate to cover oolite particles. Sodium hydroxide was used as a 32% solution, while lime was added in dry form, mixed before water was added and afterwards mixed again. Thus prepared samples were then dried

in an oven at 105 °C. The dried samples were disintegrated using a laboratory hammer mill and further blended with 5 wt.% coke in powder form – used as a reductive agent. The mixture was placed inside covered fireclay crucibles to limit access of air to the sample with care being taken to keep the cover loose for allowing gases (steam and carbon dioxide) out. Samples were roasted at 900 °C for 1 h inside a muffle furnace equipped with a thermal controller (OMRON E5AX) and then cooled down to ambient temperature. Irrespective of the measures undertaken to prevent sample re-oxidation, it has been observed that the material has been predominantly reduced to maghemite (γ-Fe₂O₃) instead of magnetite. In order to assure reduction to magnetite, coke dosage was increased in the last series of tests.

2.1.2. Leaching and magnetic separation

The leaching suspension was prepared by placing 40 g of sample inside a 200 mL beaker. The process starts once 60 mL of sulphuric acid (3, 5 or 10 v/v%) solution is added to the sample. Leaching was carried out at ambient temperature using a teflon stirrer with the agitation velocity kept at 400 rpm.

Magnetic separation was performed inside a laboratory device consisting of a 800 mL beaker with three Nd₂Fe₁₄B permanent magnets with altering polarity, placed around the vessel. The induction measured in the immediate vicinity of the magnets was 0.34 T. Magnetic separation involved a rougher and two cleaning operations with intermediate demagnetization in an alternative magnetic field with decrementing amplitude and a frequency of 400 Hz. The demagnetization step was needed to prevent strong flocculating effects during separation, which are likely to occur due to the higher coercive energy of the artificially obtained magnetite compared to that of natural magnetite. At each operation, the suspension was homogenized by a glass rod for one minute and left for one more minute for non-magnetic particles to settle. The non-magnetic fractions collected from the three operations were joined together. At the end of the test, the suspension was poured out inside a tray and the magnetic fraction, attracted around the magnets was washed with 150 mL de-ionized water.

2.2. Reactions during roasting

Preliminary tests on acid leaching have shown that the phosphorus content could be reduced down to 0.5% through leaching with 1 M sulphuric acid at room temperature for one hour and further down to 0.34% if leaching was done at boiling temperature. The reaction between the solid particles and liquid acid depends on process conditions, porosity of the particles and number of phenomena such as mass transfer, diffusion of reactants and products, heat transfer, provided the reaction is endo- or exothermic and the

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