



Technical note

Regeneration of alkali leaching solution through precipitation using calcium hydroxide

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ABSTRACT

Sodium hydroxide (NaOH) is known to be capable of selectively leaching phosphorus from oolitic iron ores. The main limitation on implementing this leaching approach is the expense of NaOH. In order to make sodium hydroxide leaching practical, a method is needed for removing the dissolved phosphorus from solution and inexpensively regenerating the sodium hydroxide. In this work, removal of phosphorus from solution was achieved using calcium hydroxide (Ca(OH)₂), which is significantly less expensive than NaOH. The addition of Francolite seed crystals greatly accelerates the precipitation rate. Up to 99.9% of phosphorus in solution was removed within thirty minutes. A leaching comparison between fresh and regenerated caustic solution was performed, with removal rates of 51.9% and 52.2% respectively, demonstrating that the regenerated leaching solution was still effective for removing phosphorus.

1. Introduction

The removal of phosphorus from iron ore is vital for the production of high quality steel. Iron ores containing close to 1% phosphorus are common, which is much too high to be desirable for steelmaking. In ores where the phosphorus is uniformly distributed through the ore, such as many oolitic iron ores, physical separation is impractical because the phosphorus is not occurring as discrete minerals, and so chemical leaching is necessary. A variety of chemical leaching processes have been investigated (Cheng et al., 1999; Fisher-White et al., 2012; Ionkov et al., 2012; Ionkov et al., 2016; Kokal, et al., 2003; Levintov et al., 2007; Yong-shi et al., 2006), with the most positive results of these investigations shown in Table 1. With the exception of the leaching presented by Yong-Shi, all other processes utilized a roasting step prior to leaching. The roasting step converts the goethite into hematite, which then tends to be more amenable to chemical leaching for the removal of phosphorus. The investigation performed by Yong-Shi et al. (2006) focused on processing the iron ore originating in Changde, China, which as described, is composed mainly of hematite. Being that the ore is already hematite, it correlates well with the other roasted goethite ores with removal rates obtaining 90% when sulfuric acid was applied as the leaching reagent. When Yong-Shi et al. (2006) performed alkaline leaching using sodium hydroxide, a maximum removal of 27.96% was obtained when a 1.0 M NaOH solution was used. As stated

above, the ore processed by Yong-Shi et al. (2006) is mainly hematite, which will react differently than goethite when alkaline leaching is applied.

Alkaline leaching is based on the decomposition of FePO₄ into Fe₂O₃ and soluble PO₄^{−3} under alkaline conditions, as can be seen in the Pourbaix diagram (electrochemical potential vs. pH) for the iron-phosphorus system, shown in Fig. 1 (Bale et al., 2009).

In experiments with alkaline leaching of an ore that originates from Kazakhstan (which had been gravimetrically concentrated to an iron concentration of 49.5% and a phosphorus concentration of 0.7–0.8% (Kokal et al., 2003)), it has become clear that the necessary concentration of NaOH to dissolve phosphorus is sufficiently high that regeneration of NaOH solution is critical. The regeneration of alkaline leaching solutions targets removing phosphorus from solution while still maintaining the alkalinity required for leaching. It is important to keep the alkalinity at a high value as to not incur additional chemical costs that are associated with pH adjustments. Thus, all tests were designed to be applied at a pH of 14 or greater to match the pH of the pregnant leaching solution.

Sodium hydroxide has a market value of \$420/short ton (Chang, 2017), compared to only \$131/metric ton (Kowal et al., 2017, Miller, 2011) for Ca(OH)₂. In addition to the lower chemical costs associated with Ca(OH)₂, using it to precipitate phosphates produces a calcium phosphate byproduct that has a market value of \$77/ton for the

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Table 1
Results of the most promising phosphorus leaching investigations.

Ore body location	Roasted	Leaching solution	Temp. (°C)	Duration (hours)	Initial P Conc. (%)	Final P Conc. (%)	% P Removal	Reference
Western Australia Pilbara	1250 °C 24 h	0.1 M H ₂ SO ₄	60	5	0.126	0.044	67.2	Cheng et al. (1999)
	300 °C	0.1 M NaOH	BP	3	0.146	0.04	74.2	Fisher-White et al. (2012)
Kazakhstan Lisakovsk	1 h 10 wt% NaOH	10 wt% H ₂ SO ₄	Amb.	0.25	0.71	0.05	90.4	Ionkov et al. (2016)
	900 °C	10 wt% H ₂ SO ₄	Amb.	16–18	0.65	0.094	86	Kokal, et al. (2003)
China Changde	1 h 10 wt% NaOH	10 wt% NaOH	55	16–18	0.65	0.092	86.2	Yong-Shi et al. (2006)
	NO ROASTING	0.1 M NaOH	Amb.	2	1.12	–	20.18	
		1 M NaOH		2	1.12	–	27.96	
		1 wt% H ₂ SO ₄		0.33	1.12	–	91.61	
		1 wt% H ₂ SO ₄		1	1.12	–	93.31	

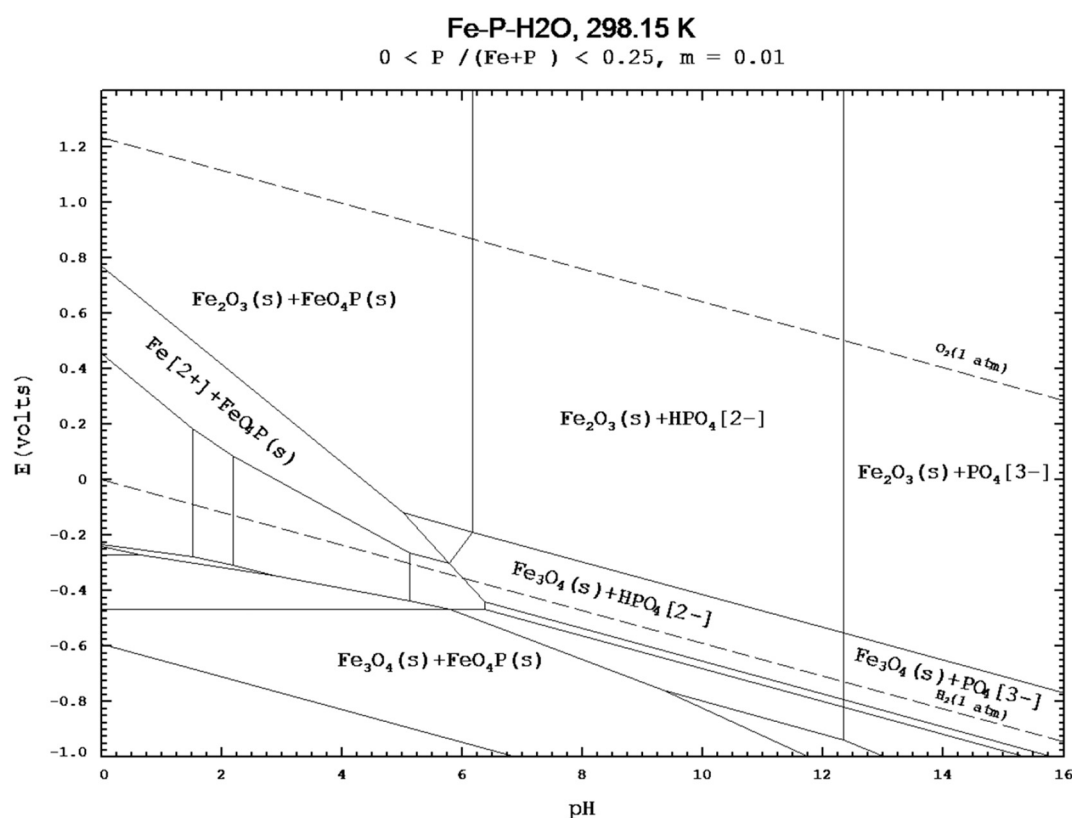


Fig. 1. Pourbaix diagram for an iron-phosphorus system (Bale et al., 2009). Targeted range for alkali leaching with be above pH 12.5.

fertilizer market (Jasinski, 2017). As a result, regeneration with lower cost Ca(OH)₂ will make the overall process much more economical than attempting to carry out the leaching with NaOH alone. A key benefit when applying the economic analysis for the chemical costs is that the amount of sodium hydroxide required is dependent on the quantity of ore, while the quantity of calcium hydroxide is dependent on the quantity of phosphorus in the ore. Applying the aforementioned costs to a theoretical operating plant suggested by Kokal et al. (2003) processing 900,000 tons of ore per year, a baseline chemical cost of using only a 5 wt% sodium hydroxide solution for leaching was determined. The associated cost for the application of sodium hydroxide (dry) to ore at a weight ratio of 0.5, results in chemical costs of \$208.34 MM per year or \$231 per ton of ore. The cost of heating and any additional processing was neglected at this point. After determining the optimal conditions for precipitation, these operating costs will be reevaluated to include the regeneration of the alkaline solution.

As precipitation is the focus of this work, the precipitation that is

being investigated will be a part of an overall process that aims to reduce phosphorus levels within an oolitic iron ore. Precipitation aims to regenerate an alkaline leaching solution to aid in phosphorus leaching.

1.1. Theory of regeneration by precipitation

In order to remove the phosphorus from the NaOH solution, precipitation was utilized. The solution that has been found to be most effective for leaching iron ore has a pH of 14, and after leaching the ore it contains approximately 150 mg/L PO₄³⁻. The desired regeneration shall be able to precipitate PO₄³⁻ without needing to change the pH.

A review of the literature found that the major current application for precipitation of soluble phosphorus is as part of wastewater treatment. Wastewater treatment targets phosphorus removal to produce clarified waters that can be reintroduced into local watersheds, which is carried out at a pH near 7. There are three main precipitation techniques followed in wastewater treatment, with many other hybrid

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