



## Surface magnetization of siderite mineral

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### ABSTRACT

Surface self-magnetization of siderite is achieved by generating ferromagnetic substance on the surface of siderite by adjusting slurry temperature, pH value, stirring rate and reaction time. No addition of any iron-containing reagent is required. The temperature of 60 °C, NaOH concentration of 0.10 mol/L; stirring rate of 900 r/min and the reaction time of 10 min are the optimal conditions. The results show that the siderite recovery in magnetic separation increased from 26.9% to 88.8% after surface magnetization. Magnetization kinetic equation is expressed as  $1 - [1 - (\varepsilon - 0.269)]^{1/3} = Kt$ . Activation energy for the magnetization reaction is 4.30 kJ/mol. VSM, SEM and XPS were used to characterize the siderite, and results show that the saturated magnetization ( $\sigma_s$ ) of siderite increased from 0.652 to 2.569 A m<sup>2</sup>/kg, the magnetic hysteresis was detected with a coercive force of 0.976 A/m after magnetization; Fe2P3/2 electron binding energy changed which reflects the valence alteration in iron on the surface and the formation of ferromagnetic Fe<sub>3</sub>O<sub>4</sub>.

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### 1. Introduction

Magnetic separation in mineral processing is based on magnetic properties of constituent mineral particles. In some cases, natural magnetism of the minerals is very weak or there is no obvious magnetic difference among different minerals, and consequently it is very difficult to get good enrichment or separation. Fortunately, some pioneering research work showed that the magnetic property of minerals can be altered by surface magnetization. Parsonage pointed that the normal magnetic separating method works well if there was 0.01–0.1% magnetite covering on the surface of the mineral [1].

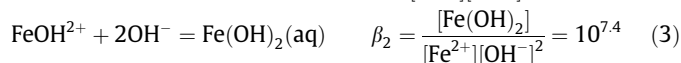
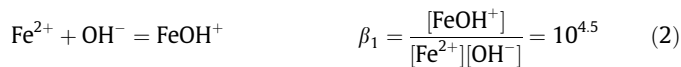
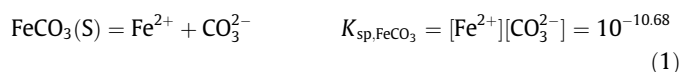
The above-mentioned magnetite, a vital substance in effective surface magnetization, is called “magnetizing seeds”. The surface magnetization is used in separation of weakly-magnetic or non-magnetic minerals or elimination of the impurities [2]. Fine particles of iron mineral siderite were recovered well by selective magnetic coating [3]. Anastassakis conducted the selective magnetization test on magnesite and quartz minerals using finely ground magnetite as a magnetizing seed, and theoretically figured out the interactive energy of different minerals in pulp [4,5]. Hwang reported that magnetite packed by organic surfactants was an effective magnetizing reagent in separating rutile and quartz as well as alumina and calcite [6]. The techniques of surface magnetization are also used in water treatment, recycling heavy metals and phosphate particles [7,8]. In comparison with this normal surface magnetization by using magnetizing seed of finely ground

magnetite powders, the author compounded the magnetizing seed Fe<sub>3</sub>O<sub>4</sub> particles directly in the pulp, and simultaneously magnetized limonite mineral [9]. In this way, the solutions of ferric and ferrous chlorides were added into the pulp containing limonite, and then the pulp pH was adjusted to 10.5 by adding ammonia water. The recovery of limonite in magnetic separation reached 91.6% on the conditions of Fe<sup>2+</sup> to Fe<sup>3+</sup> molar ratio of 3:1 and Fe<sup>3+</sup> concentration of  $7.5 \times 10^{-3}$  mol/L.

Furthermore, based on the slight dissolution of siderite mineral in aqueous solution, surface self-magnetization was achieved only by adjusting conditions of magnetization reaction without any addition of iron-containing reagents.

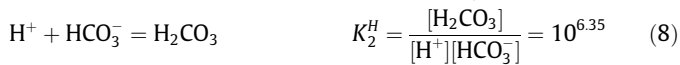
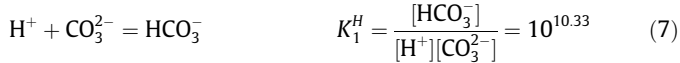
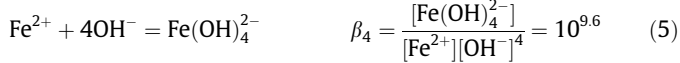
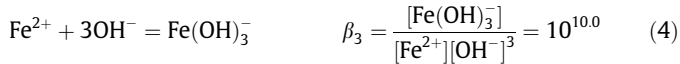
### 2. Dissolution equilibrium theory of siderite solution with surface magnetization

Solid siderite mineral slightly dissolves in an aqueous solution, so it is possible to supply the substances related to Fe<sup>2+</sup> which are needed in compounding of Fe<sub>3</sub>O<sub>4</sub>. The solubility equilibrium equations of siderite–water system were built as below. In the case of no oxidation during the hydrolysis of siderite, the reactions were as follows [10]:



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where  $\beta_1, \beta_2, \beta_3, \beta_4$  are formation constants; and  $K_1^H, K_2^H$  the protonation constants. Given that [Fe] and [C] are the sums of the various chemical components of iron and carbon in the solution, respectively, therefore,

$$[\text{Fe}] = [\text{Fe}^{2+}] + [\text{Fe}(\text{OH})^+] + [\text{Fe}(\text{OH})_2(\text{aq})] + [\text{Fe}(\text{OH})_3^-] + [\text{Fe}(\text{OH})_4^{2-}] \\ = [\text{Fe}^{2+}](1 + \beta_1[\text{OH}^-] + \beta_2[\text{OH}^-]^2 + \beta_3[\text{OH}^-]^3 + \beta_4[\text{OH}^-]^4) \quad (9)$$

$$[\text{C}] = [\text{CO}_3^{2-}] + [\text{HCO}_3^-] + [\text{H}_2\text{CO}_3] \\ = [\text{CO}_3^{2-}](1 + K_1^H[\text{H}^+] + K_1^H K_2^H[\text{H}^+]^2) \quad (10)$$

When  $\text{Fe}(\text{OH})_2$  is not present in the solution,  $[\text{Fe}] = [\text{C}]$  in a closed siderite–water system, namely,

$$[\text{CO}_3^{2-}] = \frac{[\text{C}]}{1 + K_1^H[\text{H}^+] + K_1^H K_2^H[\text{H}^+]^2} = \frac{[\text{Fe}]}{1 + K_1^H[\text{H}^+] + K_1^H K_2^H[\text{H}^+]^2} \quad (11)$$

Considering Eqs. (1) and (11), the relation between  $[\text{Fe}^{2+}]$  and pH values can be established:

$$[\text{Fe}^{2+}] = \sqrt{\frac{K_{\text{sp,FeCO}_3}(1 + K_1^H[\text{H}^+] + K_1^H K_2^H[\text{H}^+]^2)}{1 + \beta_1[\text{OH}^-] + \beta_2[\text{OH}^-]^2 + \beta_3[\text{OH}^-]^3 + \beta_4[\text{OH}^-]^4}} \\ = \sqrt{\frac{10^{-10.68} + 10^{-0.35-\text{pH}} + 10^{6-2\text{pH}}}{1 + 10^{\text{pH}-9.5} + 10^{2\text{pH}-20.6} + 10^{3\text{pH}-32} + 10^{4\text{pH}-46.4}}} \quad (12)$$

When  $\text{Fe}(\text{OH})_2$  is generated in the solution, according to Eq. (6), the relation between  $[\text{Fe}^{2+}]$  and pH values can also be established:

$$[\text{Fe}^{2+}] = \frac{K'_s}{[\text{OH}^-]^2} = 12.9 - 2\text{pH} \quad (13)$$

According to Eqs. (12) and (13), two relation curves for  $\text{Fe}(\text{CO})_3$  and  $\text{Fe}(\text{OH})_2$  in solubility equilibrium are plotted  $\lg[\text{Fe}^{2+}]$  versus pH value, respectively as shown in Fig. 1. When pH value is 8.73, the two lines intersect, when  $\text{pH} > 8.73$ ,  $[\text{Fe}^{2+}][\text{OH}^-]^2 > K'_s$ , the balance of chemical reaction (6) shifts to the left, so  $\text{Fe}(\text{OH})_2$  is generated

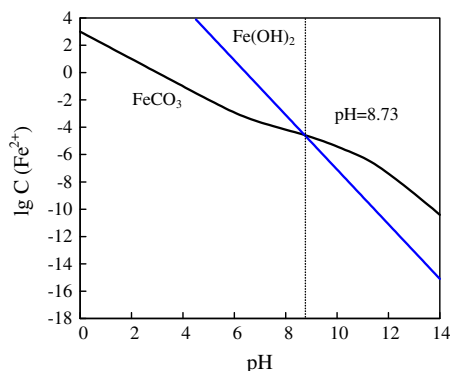


Fig. 1. Effect of pH on distribution of dissolved  $\text{Fe}^{2+}$  species in systems of  $\text{Fe}(\text{CO})_3(\text{s})/\text{water}$  and  $\text{Fe}(\text{OH})_2(\text{s})/\text{water}$ .

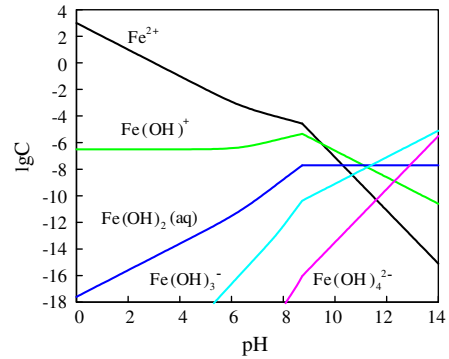


Fig. 2. Distribution of dissolved iron species versus pH in the system of siderite/water.

and  $[\text{Fe}^{2+}]$  obeys the Eq. (13), when  $\text{pH} < 8.73$ , there is no  $\text{Fe}(\text{OH})_2$  generated, and  $[\text{Fe}^{2+}]$  obeys the Eq. (12).

According to Eqs. (2)–(5), concentration relations of other complexes of iron and  $[\text{Fe}^{2+}]$  in the siderite solution are described as follows:

$$\lg[\text{Fe}(\text{OH})^+] = \lg \beta_1 + \lg[\text{Fe}^{2+}] + \text{pH} - 14 \quad (14)$$

$$\lg[\text{Fe}(\text{OH})_2(\text{aq})] = \lg \beta_2 + \lg[\text{Fe}^{2+}] + 2(\text{pH} - 14) \quad (15)$$

$$\lg[\text{Fe}(\text{OH})_3^-] = \lg \beta_3 + \lg[\text{Fe}^{2+}] + 3(\text{pH} - 14) \quad (16)$$

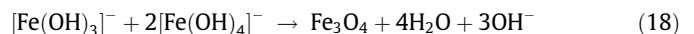
$$\lg[\text{Fe}(\text{OH})_4^{2-}] = \lg \beta_4 + \lg[\text{Fe}^{2+}] + 4(\text{pH} - 14) \quad (17)$$

Based on the Eqs. (14)–(17), a graph is drawn to show the concentrations of various dissolved components in a saturated siderite solution, as shown in Fig. 2.

So, when  $\text{pH} > 8.73$ ,  $\text{Fe}(\text{OH})_2$  is generated in the siderite solution; with the increasing value of pH, the concentration of  $[\text{Fe}^{2+}]$  decreased while the concentrations of  $\text{Fe}(\text{OH})_2(\text{aq})$ ,  $\text{Fe}(\text{OH})_3^-$  and  $\text{Fe}(\text{OH})_4^{2-}$  increased gradually.

There are many different mechanisms to explain the formation of strong magnetic matter  $\text{Fe}_3\text{O}_4$ . The more acceptable mechanisms are as follows [11]:

Firstly,  $\text{Fe}^{2+}$  and  $\text{OH}^-$  are converted to  $\text{Fe}(\text{OH})_2$ ; secondly,  $\text{Fe}(\text{OH})_2$  is oxidized to  $\text{Fe}(\text{OH})_3^-$  and  $\text{Fe}(\text{OH})_4^{2-}$  hydroxy complexes; finally,  $\text{Fe}(\text{OH})_3^-$  and  $\text{Fe}(\text{OH})_4^{2-}$  are transformed to  $\text{Fe}_3\text{O}_4$  through the dehydration reaction.



Hence, the siderite solution, especially in alkaline condition, supplies  $\text{Fe}(\text{OH})_2(\text{aq})$ ,  $\text{Fe}(\text{OH})_3^-$  and  $\text{Fe}(\text{OH})_4^{2-}$  which are the species used for the synthesis of  $\text{Fe}_3\text{O}_4$ . In the other words, in siderite slurry, it is possible to form strongly-magnetic  $\text{Fe}_3\text{O}_4$  substance on the surface of siderite by adjusting slurry pH value.

### 3. Experimental

Siderite used in experiments was prepared from magnetic separation in the lab. It contained 80.0% of  $\text{FeCO}_3$  and 85% of the particles were of size  $-0.038$  mm.

Agents are sodium hydroxide (AR), hydrogen peroxide (AR), potassium permanganate (AR).

Ten gram of the sample and 40 mL of distilled water were used in the single mineral tests. After proper preheating and mixing, NaOH was added into the solution. Then the stirring speed was adjusted to 900 r/min, and it lasted for 10 min (except special instruction). Subsequently, the magnetic separation was carried out using a wet-intermittent high gradient magnetic separator (magnetic density was 2100 Gs). The recovery rate of siderite was used to evaluate the phenomena of surface magnetization.

HH-15 vibrating sample magnetometer (VSM) was used to measure the relationship between magnetic moment and magnetic

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