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Effects of water hardness on selective flocculation of diasporic bauxite

LIU Wen-li, SUN Wei, HU Yue-hua

School of Minerals Processing and Bioengineering, Central South University, Changsha 410083, China

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Abstract: The effect of electrolyte on settling behavior of kaolinite was studied. Effects of hard water on selective flocculation of diasporic bauxite was tested and the measures were taken to eliminate the effects of Ca^{2+} and Mg^{2+} in hard water. The results indicated that, not only the concentration of electrolyte ions but also the ionic valence of the electrolyte ions affects the settling behavior of kaolinite; hard water significantly affects its selective flocculation owing to Ca^{2+} and Mg^{2+} ; general dispersants could not eliminate the effects of Ca^{2+} and Mg^{2+} . Self-made softening agent in our lab could weaken or eliminate the effects of hard water on flocculation processes. The results of molecular dynamics simulation show that softening agent molecules could restrict Ca^{2+} and prevent them from playing their roles, so as to eliminate the effects. The continuous pilot experiment results of bauxite flocculation were even better than those obtained in laboratory.

Key words: softening agent; hard water; bauxite; dispersant; flocculation

1 Introduction

Diasporic bauxite in China is characterized by high content of aluminum oxide and silica but low ratio of Al_2O_3 to SiO_2 (average 5–6). Bauxite can be treated by Bayer process to produce aluminum oxide [1], unless the mass ratio of Al_2O_3 to SiO_2 is larger than 10 [2,3], Bayer process could not economically produce aluminum oxide from diasporic bauxite because the high content of SiO_2 caused high NaOH and energy consumption. Therefore, it is necessary to effectively remove aluminosiliate minerals such as kaolinite, pyrophilite and illite from bauxite to increase Al_2O_3/SiO_2 ratio (A/S) of bauxite [4–6].

In diasporic bauxite ore, diaspore, the valuable mineral, accounts for only 70% of total mass, and the gangue minerals, such as kaolinite, pyrophyllite and illite, take up 30% mass yield. To separate these silicate minerals from diaspore, flotation provides a promising way. In previous studies, the direct flotation using oleic acid as collector [2,3] has been employed to de-silicate diasporic bauxite ore and proved to be an efficient method.

However, direct flotation can not solve such problems as dewatering and the effect on aluminum

metallurgy [7,8]. Thereby, the reverse flotation with cationic collector was developed and some achievements were obtained.

Flocculation of fine particles using polymeric materials flocculant(s) and separation of such aggregates from particles of other component(s) in the dispersed phase is known as selective flocculation. Many researches on selective flocculating diaspore from diasporic bauxite have been carried out [9-12]. Some excellent results have been achieved in soft water system [13-16].

However, in Henan and Shanxi Provinces, China, where located the main diasporic bauxite deposits in China, the water is hard water containing a mass of Ca^{2+} and Mg^{2+} which will greatly affect the selective flocculation process of diasporic bauxite. The effects of electrolyte ions on dispersion and settling behaviors of diaspore and kaolinite directly affect the separation process efficiency of them.

This work aims to study the effects of the electrolyte in hard water on selective flocculation of diasporic bauxite, to explore the ways to eliminate or weaken the effects of Ca^{2+} and Mg^{2+} and discuss the elimination mechanism through molecule dynamics simulation method.

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2 Experimental

2.1 Materials and reagents

Lumps of kaolinite were taken from Shanxi Province, China, and then crushed and ground in a laboratory porcelain mill. Particles with an average diameter of $3.85 \,\mu\text{m}$ were collected and used in settling tests. The chemical component of the material was analyzed, and the results are listed in Table 1.

Table 1 Analytical results of chemical component for kaolinite(mass fraction, %)

SiO_2	Al_2O_3	MgO	CaO	TFe	TiO_2	K_2O	Na ₂ O	$\mathrm{H_2O^+}$	Loss
43.67	39.2	0.068	0.010	0.32	1.98	0.094	0.028	13.65	13.98

Bauxite ore used in these experiments were sampled from Henan deposits in China. Based on X-ray powder diffraction (XRD) analysis (Fig. 1), the compositions of the bauxite ore were diaspore (59.8%), illite (14%) and kaolinite (15%) (Table 2). The chemical composition of the bauxite ore is given in Table 3. The contents of Al_2O_3 and SiO_2 in the bauxite ore are 62.18% and 13.28%, respectively, resulting in an A/S of 4.68.



Fig. 1 XRD pattern of bauxite ore sample

 Table 2 Minerals composition of bauxite ore (mass fraction, %)

Diaspore	Kaolinite	Illite	Calcite	Hematite	Ananaes+rutile
59.38	15	14	1.5	5.1	3

Table 3 Chemical composition of bauxite ore (mass fraction, %)

Al_2O_3	SiO_2	$\mathrm{Fe_2O_3}$	TiO ₂	CaO	K_2O	Na ₂ O	MgO	Lose
62.18	13.28	5.13	3.01	0.84	1.48	0.025	0.30	13.16

As the ion additive, ammonium chloride, calcium chloride and potassium chloride were analytical grade hydrochloric. Sodium carbonate, sodium hexametaphosphate, sodium silicate, and sodium hydroxide were of analytical grade and used as dispersant. Softening agent was provided by our lab, whose molecular structure is shown in Fig. 2. Distilled water was used in settling tests of kaolinite.

$$- CH_2 - CH_1 - CH_1 - COOH$$

Fig. 2 Molecular formula of softening agent

2.2 Settling tests

In the settling tests, the kaolinite suspension was prepared by putting 3 g mineral sample into a glass beaker with 40 mL distilled water. The resultant suspension was stirred with an electromagnetic stirrer for 8 min at a speed of 1300 r/min for fully dispersion and 3 min at a speed of 300 r/min after adding flocculant or pH regulator. The suspension was poured gently into a 100 mL measuring cylinder, which was inverted 10 times. The cylinder was then kept immobilization on the level bed for 15 min, and the top 50 mL suspension was extracted by a latex tubing. The dried mass of solids was obtained after the bottom 50 mL suspension was leached. Results of the settling tests were reported in terms of aggregation efficiency (E_a), which was defined as:

$$E_a = m_0 / m \times 100\%$$
 (1)

where M_0 represents the dried mass of solids in the bottom 50 mL suspension; *m* represents the total mass of sample.

2.3 Flocculation experiments

The flocculation experiments of bauxite were carried out with soft water and hard water respectively to study the effects of hard water on selective flocculation process of bauxite.

The selective flocculation experiments of bauxite were carried out in a Lucite settling bucket of 20 cm in diameter with a capacity of 6000 mL according to the flow sheet shown in Fig. 3. Additional parameters on desilicating tests are also given in Fig. 3. For the desilicating balance calculations, desilicating products such as feed, tailings and concentrate were subjected to chemical analyses for determination of Al_2O_3 and SiO_2 .

2.4 Molecule dynamics simulation

The software package, Discover in Material Studio (Accelrys Software, Inc) with COMPASS force field, was used to study the process of softening agent restricting Ca^{2+} and Mg^{2+} to eliminate the negative effects on selective flocculation. Discover is a molecular simulation program in Material Studio, which provides a broad range of simulation methods to study molecular systems. The force fields in Discover provide the function of predicting the structure, energy, and



Fig. 3 Flow sheet of selective flocculation experiments

properties of organic, inorganic, and biological systems as well as gas, liquid and solid. Molecular dynamics simulations generate information at microscopic level, including atomic positions and velocities.

The isothermal-isometric molecular dynamics simulations (NVT-MD) [17,18] method was employed. In NVT ensemble, temperature change was controlled using Andersen method. Potential energy of each atom was derived from the COMPASS force field. The cutoff distance for van der Waals and electrostatic forces was 9.5 Å.

For simplification, molecular dynamics simulation was carried out in vacuum. The simulation box has dimensions of 100 Å \times 100 Å \times 100 Å, containing 5 softening agents, 5 carbonate ions, 5 calcium ions and 5 hydrogen ions. The number of these molecules is determined by the dosage of softening agent in practice. Total simulation time is 1000 ps, the time step is set to 1 fs, and the neighbor list updated every 5000 steps. The last 50000 steps were used for the analysis of softening mechanisms of softening agent.

3 Results and discussion

3.1 Effects of electrolyte on dispersion and settling behavior of kaolinite

The effects of electrolyte on the settling behavior of kaolinite are presented in Fig. 4. Under the given conditions the effects of cations on colloidal stability are significant. Figure 4 indicates a strong increase of sediment yield of particles with increasing ionic concentration of the solution. When the concentration of cations reaches a certain value, the sediment yield begins to decrease. What is more, this significant increase depends not only on the ionic concentration but also on the valence of the cations. The trivalent Al^{3+} is most effective in aggregating the kaolinite particles, and the divalent Ca^{2+} ions are more effective than the

monovalent Na⁺. These results are consistent with the Hardy–Schultz theory. HUANG [14] and CHEN [15] obtained the similar results.



Fig. 4 Effects of electrolyte on settling behavior of kaolinite (Suspension concentration: 3%; Settling time: 15 min; Natural pH)

3.2 Effects of water hardness on selective flocculation

Different water sources were used in the experiments to study the effects of water hardness on selective flocculation processes of diasporic bauxite. The water samples were collected respectively from Changsha of Hunan Province and Zhengzhou of Henan Province, China. The analysis results from the Water Quality Monitoring Center of Changsha are shown in Table 4.

Table 4 Water quality monitoring results

Sample	Monitoring number Source		Hardness/ $(mg \cdot L^{-1})$
1	W2009SZ0004	Running water of Changsha	27
2	W2009SZ0005	Factory water of Zhengzhou	356
3	W2009SZ0006	Running water of Zhengzhou	267
4	W2009SZ0007	Yellow River in Zhengzhou	251

Table 5 presents the results of flocculation with different water sources, in which water hardness plays a negative role in selective flocculation process. In hard water samples the recovery and A/S of concentrate are especially lower than those in soft water samples.

Incomplete dispersion of the bauxite particles is regarded as an important reason for the failure of the selective flocculation processes. As we all know, complete dispersion is the premise of selective flocculation process. Several dispersants were adopted in the flocculation experiments in hard water for complete dispersion of the particles. The results are listed in Table 6.

Plenty of Ca^{2+} and Mg^{2+} in water would occupy the active site on flocculant molecular and reduce their efficiency [19,20], leading to the competitive adsorption between ions and mineral particles.

From the results, we can see that the dispersants have positive effects on dispersion of particles in hard water since the recovery of concentrate is higher. The dispersion efficiency of the combination of sodium carbonate and sodium silicate is the best. However, it also should be noticed that the A/S of concentrate is still low due to the effects of Ca^{2+} and Mg^{2+} . Dispersants could make the mineral particles completely disperse but could not eliminate the effects of Ca^{2+} and Mg^{2+} in hard water. When the dispersants are added in hard water, Na_2CO_3 , Na_2SiO_3 and NaOH would react with Ca^{2+} and Mg^{2+} and generate precipitates such as $CaCO_3$, $MgCO_3$, $Ca(OH)_2$. The chemical reaction equations are shown as follows:

$$Na_{2}CO_{3} + Ca^{2+}(Mg^{2+}) \rightarrow CaCO_{3}(MgCO_{3}) \downarrow +2Na^{+}$$

$$(2)$$

$$Na_{2}SiO_{3} + Ca^{2+}(Mg^{2+}) \rightarrow CaSiO_{3}(MgSiO_{3}) \downarrow +2Na^{+}$$

$$(3)$$

$$2NaOH + Ca^{2+}(Mg^{2+}) \rightarrow Ca(OH)_{2}(Mg(OH)_{2}) \downarrow +2Na^{+}$$

$$(4)$$

These precipitate particles in water tend to be adsorbed on flocculant molecule and prevent the mineral particles from being adsorbed. Since the precipitate

Water hardness/(mg.I ⁻¹)	Droduat	Viold/0/	Grad	le/%	Recovery/%		A /S
water naruness/(ing.L)	FIOduct	1 leiu/ 70	Al_2O_3	SiO ₂	Al ₂ O ₃	SiO_2	A/3
	Tailing	19.86	46.20	26.28	15.14	40.03	1.76
27	Concentrate	75.71	64.20	9.76	84.86	59.97	6.58
	Feed	100.00	60.62	13.04	100.00	100.00	4.65
	Tailing	65.05	60.8	14.95	64.16	69.24	4.21
267	Concentrate	34.95	63.2	11.95	35.84	30.76	5.29
	Feed	100.00	61.64	13.58	100.00	100.00	4.54
	Tailing	69.77	61.05	14.56	69.24	75.37	4.19
356	Concentrate	30.23	62.59	10.98	30.76	24.63	5.70
	Feed	100.00	61.51	13.48	100.00	100.00	4.56
	Tailing	63.27	60.85	13.85	62.44	67.30	4.39
251	Concentrate	36.73	63.05	11.59	37.56	32.70	5.44
	Feed	100.00	61.66	13.02	100.00	100.00	4.74

Table 5 Results of flocculation with different water source

 Table 6 Selective flocculation experiments using different dispersants in hard water

Condition	Draduat	Viold/0/	Grade/%		Recovery/%		A /S	
Condition	FIOduct	11010/70	Al_2O_3	SiO ₂	Al_2O_3	SiO ₂	A/S	
	Tailing	65.05	60.8	14.95	64.16	69.24	4.21	
$Na_2CO_3 2500 \text{ g/t},$	Concentrate	34.95	63.2	11.95	35.84	30.76	5.29	
nocediant 4 g/t	Feed	100.00	61.64	13.58	100.00	100.00	4.54	
Na ₂ CO ₃ 3000 g/t,	Tailing	7.71	48.2	26.49	5.95	15.46	1.82	
$Na_2SiO_3 1000 g/t$,	Concentrate	92.29	63.6	12.1	94.04	84.54	5.26	
flocculant 15 g/t	Feed	100.00	62.41	13.21	100.00	100.00	4.73	
	Tailing	5.15	44.8	30.71	3.70	11.90	1.46	
Na_2CO_3 4500 g/t, flocculant 15 g/t	Concentrate	94.85	63.4	12.36	96.30	88.10	5.13	
nocculant 15 g/t	Feed	100.00	62.44	13.31	100.00	100.00	4.69	
	Tailing	3.62	49.2	23.71	2.89	6.62	2.07	
Na ₂ SiO ₃ 6000 g/t, Elocculant 8 g/t	Concentrate	96.38	62	12.57	97.10	93.38	4.93	
Tiocculant o g/t	Feed	100.00	61.54	12.97	100.00	100.00	4.74	
NaOH 2500 g/t,	Tailing	10.35	53.0	21.52	8.81	16.63	2.46	
Na ₂ CO ₃ 2000 g/t,	Concentrate	89.65	63.4	12.46	91.19	83.40	5.09	
flocculant 5 g/t	Feed	100.00	62.32	13.40	100.00	100.00	4.65	

The water sample is running water of Zhengzhou

particles have tiny size, they could be adsorbed on flocculant molecule prior to mineral particles.

3.3 Effects of water softening agent on selective flocculation

In order to prevent Ca^{2+} and Mg^{2+} from reacting with dispersants and generating precipitate, before the addition of dispersants, some actions should be taken to eliminate the activity of Ca^{2+} and Mg^{2+} .

Softening agent is a kind of water treatment agent for hard water. The mechanism of softening water is reaction with Ca^{2+} and Mg^{2+} through complexation or chelation. These reactions could restrict Ca^{2+} and Mg^{2+} , but could not generate precipitate. Polyacrylate is a kind of softening agent made in our lab, which was used for hard water treatment. The results are shown in Table 7.

The addition of polyacrylate made a higher A/S and recovery of concentrate in hard water. It also indicates that inadequate or superfluous dosage of softening agent wouldn't produce the desired results in these tests, and an appreciate A/S and recovery of concentrate could be obtained only at a certain dosage.

At an inadequate dosage, the effects of Ca^{2+} and Mg^{2+} in hard water could not be eliminated. When softening agent is excessively added, the redundant molecules are adsorbed on the diaspore particles. The process could obstruct the flocculant molecule to adsorb on diaspore particles, and affect the recovery and A/S of concentrate.

The mechanism of the softening agent is considered that the oxygen atoms of softening agent molecule react with Ca^{2+} and Mg^{2+} prior to the breaking bond on

 Table 7 Selective flocculation experiments using softening agent

mineral particles surface. So appropriate quantity could completely restrict the freedom of Ca^{2+} and Mg^{2+} and no redundant molecule would affect the mineral particles. To verify the inference, the molecule dynamics simulation was carried out, and the results will be exhibited in the following section. The reaction formula is shown below:

$$\begin{array}{c} \left\{ CH_2 - CH \right\}_n^{-} + n Ca^{2+} \longrightarrow \left\{ CH_2 - CH \right\}_n^{-} \\ OH - C & HO - C \\ \parallel & & | \\ O & Ca^{2+} O \end{array}$$
(5)

3.4 Continuous pilot experiment

Continuous pilot experiment was carried out according to the flow sheet shown in Fig. 5. Water used in this experiment was factory water from Zhengzhou shown in Table 3. Pretreatment of the water was carried out before it flew into the flocculation process. After 13 shifts, the average results obtained for each shift are shown in Table 8. The A/S of the concentrate increased from 4.71 to 6.69, and the recovery of Al₂O₃ was 81.25%. These results are even better than those obtained in laboratory.

3.5 Molecule dynamics simulation

Molecule dynamics simulation can reveal the effects of Ca^{2+} and Mg^{2+} on selective flocculation process of softening agent.

Figures 6 and 7 show the beginning state and final state of dynamics simulation of Ca^{2+} adsorbing on polyacrylate.

Condition	Draduat	Viald/0/	Grade/%		Recovery/%		A /S
Condition	Product	r leid/ 70	Al_2O_3	SiO ₂	Al_2O_3	SiO ₂	A/3
	Tailing	15.65	50.4	23.15	13.11	29.11	2.18
Softening agent 300 g/t,	Concentrate	84.35	62	10.46	86.89	70.89	5.93
nocculant 4 g/t	Feed	100.00	60.18	12.45	100.00	100.00	4.83
	Tailing	11.77	45.4	25.03	8.91	24.17	1.81
Softening agent 400 g/t, flocculant 4 g/t	Concentrate	88.23	61.9	10.48	91.09	75.83	5.91
	Feed	100.00	59.96	12.19	100.00	100.00	4.92
	Tailing	15.63	48	23.65	12.37	30.33	2.03
Softening agent 400 g/t,	Concentrate	84.37	63	10.06	87.63	69.67	6.26
nocculant 5 g/t	Feed	100.00	60.66	12.18	100.00	100.00	4.98
	Tailing	16.34	49	22.91	13.19	30.40	2.14
Softening agent 500 g/t, flocculant 3 g/t	Concentrate	83.66	63	10.25	86.81	69.60	6.15
	Feed	100.00	60.71	12.32	100.00	100.00	4.93
	Tailing	25.23	10.95	3.54	22.29	36.13	3.09
Softening agent 800 g/t, flocculant 4 g/t	Concentrate	74.77	63.4	10.39	77.71	63.87	6.10
nocculant 4 g/t	Feed	100.00	61.00	12.16	100.00	100.00	5.02

The water sample is running water of Zhengzhou



Fig. 5 Flow sheet of continuous pilot experiment

 Table 8 Average results of shift work in continuous pilot

 experiment

Droduct	Grade/%		A /S	Product	yield/%	Recovery/%	
Floduct	Al_2O_3	SiO_2	A/3	Al_2O_3	SiO ₂	Al_2O_3	SiO_2
Feed	60.97	12.94	4.71	100.00	100.00	100.00	100.00
Concentrate	64.43	9.63	6.69	76.96	75.63	81.25	56.47
Tailing	49.37	23.31	2.12	23.04	24.37	20.28	43.53



Fig. 6 Distance between softening agent molecule and Ca²⁺ at beginning state



Fig. 7 Distance between softening agent molecule and Ca^{2+} at final state

At the beginning, Ca^{2+} , CO_3^{2-} and polyacrylate molecules are dispersant. After dynamics simulation, per Ca^{2+} is restricted by one polyacrylate molecule, which explains how polyacrylate eliminates the hard water effects.

From Fig. 6 and Fig. 7, we could see that at the beginning the distance between the two oxygen atoms (belong to the carboxyl of softening agent molecule) and Ca^{2+} are respectively 29.535 Å and 31.450 Å and at the final state (the stable state), these distance are 1.9 Å and 1.934 Å, which shows that Ca^{2+} and softening agent molecules can make a stable chemical bond, so Ca^{2+} is restricted by softening agent molecules.

Figure 8 shows energy change in the simulation process. The energy increasingly blows down till to a steady value. Therefore, at the final state the system potential is the lowest, and the system is the steadiest.



Fig. 8 Energy change in dynamics simulation

4 Conclusions

1) Electrolyte in solution severely impacts the settling behavior of kaolinite. The effects of electrolyte depend not only on the concentration but also on the ionic valence of the electrolyte ion. The order of ions effect on the settling behavior of kaolinite is $Al^{3+}>Ca^{2+}>K^+$. Settling velocity of particles is faster with the increase of ionic concentration.

2) Hard water greatly affects the flocculating process due to Ca^{2+} and Mg^{2+} . The same dosage of dispersant addition as that in soft water system could not fully make the particles disperse. Although particles fully dispersed with heavy addition of dispersants, the flocculating processes performed unselectively.

3) Certain softening agent could eliminate or weaken the effects of hard water on flocculating processes. Self-made softening agent performed well in these tests, since the A/S and recovery of concentrate were appreciating.

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水的硬度对铝土矿选择性絮凝的影响

刘文莉,孙伟,胡岳华

中南大学 资源加工与生物工程学院,长沙 410083

摘 要:通过絮凝实验和分子动力学模拟的方法,研究硬水中 Ca²⁺、Mg²⁺离子对铝土矿选择性絮凝的影响。结 果表明:除了电解质浓度,电解质离子的价态也对选择性絮凝产生影响;硬水中的 Ca²⁺和 Mg²⁺离子显著地选择 絮凝,且普通的分散剂不能减弱这些影响。实验室自制的软水剂可以削弱甚至消除这些影响。分子动力学模拟结 果显示,软水剂可以限制 Ca²⁺,防止他们发生作用,因此可以消除影响。铝土矿连续絮凝实验结果好于实验室实 验结果。

关键词: 软水剂; 硬水; 铝土矿; 分散剂; 絮凝剂

(Edited by YUAN Sai-qian)