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INVESTIGATION OF QUARTZ FLOTATION FROM DECARBURIZED VANADIUM BEARING COAL

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Abstract: Ether diamine (Fm 2835-2L) was used as a collector for flotation of quartz. It allows flotation of quartz from mica and calcite. The adsorption mechanism of Fm 2835-2L on quartz was investigated by flotation tests, zeta-potential measurements and infra-red (FTIR) spectra measurements. Results show that Fm 2835-2L adsorbs on the quartz surface in physical adsorption with no new products, changing its zeta potentials, and increasing its hydrophobicity. The effect of calcium ions on flotation of quartz was investigated by flotation tests and zeta-potential measurements. Results show that under neutral or weakly acidic conditions calcium cation can adsorb onto the surfaces of quartz, increase the zeta potential of quartz particles, which in turn causes weaker aggregation of quartz particles and lower flotation recovery of quartz. Under the alkaline conditions the hydrolytic components of calcium are also adsorbed on the quartz surface and increase the zeta potential of quartz particles, which causes stronger aggregation of quartz particles and higher flotation recovery of quartz. However, the hydrolytic components such as $CaOH^+$, $Ca(OH)_{2(aq)}$ and $Ca(OH)_{2(s)}$ were not formed in significant amounts in the best flotation tests.

Keywords: mica, vanadium-bearing coal, flotation, quartz, ion dissolution, calcium ion

Introduction

In recent years, the enrichment of vanadium-bearing coal has becoming an important issue (Ni et al., 2010; Wu et al., 2008). Many studies have been devoted to reduce the cost of vanadium extraction from coal and to facilitate utilization of resources below cut-off grade (Zhao et al., 2013a; Zhao et al., 2013b). However, there a limited number of papers on mechanism of the vanadium materials enrichment process. Therefore, it is necessary to study the effect of various parameters on enrichment process.

In the vanadium-bearing coals, about 90% vanadium occurs in muscovite. Quartz is the main gangue mineral in the muscovite ores, which results in a low purity of muscovite and limits its wide applications. Therefore, there is an urgent need for improvement of beneficiation of low grade muscovite ores. In many studies quartz is removed by screening and gravity separation due to differences in shape, hardness and density of ore components. However, these methods provide poor efficiency in case of particles below 50 μ m in size (Wang et al., 2014).

Ether monoamine and ether diamine are used as the collectors for flotation of silicate gangue minerals in the iron ore industry. It is known that diamines are strong collectors for quartz in comparison to monoamines with comparable chain length (Ma et al., 2009; Smith and Scott, 1990; Scott and Smith, 1991). This is due to smaller pK_a values of diamines in comparison to monoamines and the doubly charged amine species of the diamines promoting stronger flotation of quartz (Scott and Smith, 1991).

In practical mineral separations, unavoidable metal ions such as Fe(III), Mg (II), and Ca (II) can profoundly affect the flotation of minerals (Fan and Rowson, 2000; Zhu et al., 2012). These ions can activate the gangue minerals, thereby increasing their flotation by promoting collector adsorption (Ejtemaeia et al., 2012). Wang and Yu (2007) investigated the effects of metal ions on the flotation of spodumene and beryl sand and found that Fe(III) and Ca(II) ions improved flotation. However, it largely depends on the type of collector used (i.e. anionic, cationic, or amphoteric). In most cases, metal ions are highly detrimental to the process during flotation of the oxide minerals. The activation of the silicates by metallic cations impairs the selectivity of the separation of the oxides and silicates such as beryllium minerals from quartz (Ejtemaeia et al., 2012). Fornasiero and Ralston (2005) studied the Cu(II) and Ni(II) activation in the flotation of quartz, lizardite and chlorite minerals. They found that quartz can be activated by these metal ions to float with xanthate at pH 7nickel hydroxides are Since the copper and stable species. their 10. adsorption/precipitation on the mineral surface improves the flotation of these minerals. All metal ions such as Fe(III), Mg(II), Ca(II), Cu(II), and Ni(II), may have potential as activators in the flotation of albite, spodumene, and quartz minerals.

The objective of the present investigation is to improve understanding of the effect of metal ions on quartz flotation and the underlying adsorption mechanism of ether diamine as collector in flotation of quartz from vanadium-bearing mica present in coal. Flotation performance of Fm 2835-2L as the collector was assessed by flotation tests using single minerals and a decarburized sample of vanadium-bearing coal. Their adsorption on minerals was analyzed by zeta-potential measurement and the Fourier transform infrared (FTIR) spectroscopy. The effect of ion dissolution in flotation system on the quartz flotation during enrichment process of vanadium-bearing coal was studied by different flotation technology and ion dissolution. In this paper, the results of these investigations were discussed and the role of ether diamine collector and calcium ion dissolution in quartz flotation is delineated.

Materials and methods

Materials

The vanadium-bearing stone coal was collected from Teng-da Mining and Metallurgy Co. Ltd., Hubei, PR China. Around 200 kg of representative ore samples were crushed to below 2 mm size with two-stage jaw crusher and one-stage roll crusher. The materials were then well mixed and divided into 2 kg samples for mineralogical and pre-concentration studies. Quartz was obtained from the mineral processing laboratory of Wuhan University of Technology. The chemical analysis showed that it contained more than 99% SiO₂ and was sufficiently pure for research purposes. Ether diamine (Flotigam 2835-2L from Clariant) was used as collector for quartz. The 2# oil, also known as terpenic oil, a mixture of higher alcohol, widely used as foaming agent or collector in the non-ferrous metal flotation was also used. Other chemicals employed in the test were of A.R. grade. Single mineral experiments were carried out with distilled water only.

Methods

The crushed materials were firstly decarburized in a SXZ-10-B muffle furnace at 700 °C for 90 min. The loss on ignition was between 12 and 13%. Then, the decarburized sample was mixed and divided into 200 g samples for quartz flotation. In the flotation NaOH and H_2SO_4 were used as modifiers, sodium silicate as inhibitor, Fm 2835-2L as collector, and oil as foaming agent. The flotation of decarburized sample was conducted in a single XFD II 0.5 flotation cell at the pulp density of 50%.

Single quartz flotation tests were conducted in a flotation cell having 40 cm³ in volume. In each test, the mineral sample of 2 g was placed into flotation cell to disperse for 3 min, followed by adjusting the pH value for 3 min, and then mixed with the collector Fm 2835-2L for 5 min, floated for 5 min. Finally, the product was filtered, dried, weighed and analyzed.

The determination of vanadium, silicon and calcium content was performed in accordance with test Methods of Vanadium in Coal Standard (GB/T19226-2003) and by the potassium dichromate volumetric method. Others analyses were performed with the Xios advanced X-ray fluorescence (XRF) analyzer.

X-ray diffraction (XRD) analysis was conducted with a D/Max-IIIA X-ray diffractometer using Cu-K α radiation, voltage 40 kV, current 30 mA at the scanning rate of 15°/min from 3° to 70°. The phases were identified by comparison of the peak positions and *d* values with the data published by the International Centre for Diffraction Data (ICDD).

Detailed mineralogy on the head ore and decarburization samples were done using a Leica DMLP polarization microscope while the quantitative evaluation of minerals by scanning electronic microscopy (QEMSCAN).

In order to characterize the nature of the interactions between the collector and quartz particles, the infrared spectra of collector as well as the samples with or without

collectors pretreated were measured by the KBr technique. Fourier transform infrared (FTIR) spectra of the samples were recorded with a Nicolet Model Nexus 670 (USA) instrument using KBr pellets at a resolution of 4 cm^{-1} .

Zeta potential of quartz was determined with a Coulter Delsa440sx Instrument (Beckmen-Coulter/USA). The measurements were conducted by inserting 0.5 g of mineral into 100 cm³ of surfactant solution. NaOH (1%, mass fraction) and H₂SO₄ (1%, mass fraction) solutions were used for adjusting pulp pH. An average of six measurements was performed for each mobility point.

Results and discussions

Mineralogy of decarburization

Roasting decarburization was employed to eliminate the effect of carbon on mineral separation. The roasting temperature was 700 °C due to beneficial effect of this temperature on the subsequent leaching process and mineral surface property. The decarburized sample was analyzed using the quantitative evaluation of minerals approach by scanning electron microscopy (see Fig. 1). The chemical and mineral compositions of the decarburized samples are shown in Tables 1 and 2, respectively. The grade of V_2O_5 after roasting increased from 0.71% to 0.82%, pyrite was converted to hematite, and calcite was decomposed into CaO and CO₂ because of the local overheating of muffle. Silicate minerals were mainly in the form of amphibole and olivine, whereas sulfate minerals were primarily composed of calcium, sulfur, and other elements. The calorific value produced in the roasting process can be used for a subsequent leaching.



Fig. 1. Mineralogical phase of decarburization sample (-150 +75 µm)

Table 1. Chemical composition of decarburized sample (%)

Element	V_2O_5	SiO ₂	Al_2O_3	Fe ₂ O ₃	K ₂ O	Na ₂ O	CaO	MgO	S	С
Content	0.82	54.31	10.59	5.67	4.90	0.35	7.35	3.13	1.90	2.72

Mineral	Quartz	Calcite	Muscovite	Feldspar	Iron mineral	Kaolinite	Silicate	Sulfate	Other
Content	34.21	5.97	12.81	12.72	12.88	5.00	3.52	3.60	9.29

Table 2. Mineral composition of decarburized sample (%)

Single mineral flotation

To investigate the flotation of quartz alone, the Fm 2835-2L collector was used. The results are summarized in Fig. 2. The recovery of quartz increases with the concentration of Fm 2835-2L. When the concentration is greater than 50 mg/dm³, the recovery reaches a plateau. The effect of the pH on the flotation of quartz in the presence and absence of Fm 2835-2L is shown in Fig. 2 b. The results indicate that the recovery changes a little with the pH value from 5.0 to 9.0 (\pm 0.2) in the presence of Fm 2835-2L. The recovery decreases sharply to 84.8% at about pH 10.0. The highest recovery at pH 7.1 is below 30% in the absence of collector. This result indicates that quartz does not have natural floatability. It also corresponds with the results of other invetsigations.



Fig. 2. Effect of pH and collector concentration on quartz flotation, a) relation between recovery and collector concentration at pH 7.1, b) relation between recovery and pH with and without collector (2# oil content was 30 mg/dm³ while Fm 2835-2L was 50 mg/dm³

The adsorption mechanism of collector on quartz was investigated using the infrared analysis and zeta potential, as shown in Figs 3 and 4. The IR spectra of quartz, Fm 2835-2L, and the interaction product are given in Fig. 3. The IR spectrum of quartz (see Fig. 3 curve 1) agrees well with the standard spectrum of SiO2. It exhibits two strong Si—O absorption band at about 1100 cm^{-1} and 500 cm^{-1} . The Si—O and Si—O—Si stretching band can be seen in the wavenumber range of 1081.87-1170.58 and $779.10-796.46 \text{ cm}^{-1}$ respectively. After the interaction with Fm 2835-2L, the IR spectrum of quartz (see Fig. 3 curve 2) gives no other characteristic absorption peaks showing that the adsorption on the surface of the quartz is physical.



Fig. 3. Infrared spectra of quartz, quartz after action of Fm 2835-2L and pure Fm 2835-2L



Fig. 4. Relationship between zeta potential of quartz and pulp pH with or without collector and regulator

Zeta-potential of quartz in the presence of no reagent, Fm 2835-2L, CaCl₂ and mixed Fm 2835-2L and CaCl₂ as a function of pH are illustrated in Fig. 4. The quartz iso-electric point (iep) is about 2.0. The results indicates that the ether diamine (Fm 2835-2L) has a strong influence on the quartz zeta-potential and increases the charge on quartz at all pH values. Quartz is negatively charged when the pH exceeds 2.0. The positively charged Fm 2835-2L absorbs on the quartz surface through electrostatic interactions. However, when the pH of the solution is less than 2.0, quartz is positively charged.

CaCl₂ also has a strong influence on the quartz zeta-potential and increases the charge of quartz at all pH values but surface is negatively charged up to pH 10. Figure 4 also indicated that when both Fm 2835-2L and CaCl₂ interact with quartz, the zeta-potential of quartz increases only in the alkaline solutions. The reason may be that when calcium cations are present in the flotation pulp, under neutral or weak acidic conditions, these cations decrease aggregation of quartz particles by increasing the zeta potential of the quartz particles. Therefore, the flotation recovery of quartz declines in the presence of calcium ions, while under the alkaline conditions, the adsorption of other components of calcium increases the zeta potential of quartz particles and improve its recovery.

Quartz flotation from decarburized vanadium-bearing coal

According to the flowsheet of the process shown in Fig. 5, the flotation of quartz from the decarburized sample of vanadium-bearing coal was studied using Fm 2835-2L as collectors in the presence of Ca (II). Based on the results of single quartz flotation, 50g/Mg of Fm 2835-2L was used at pH 7.0 in the absence of additives and in the presence of Ca (II) which was leached out from the decarburization sample. The results in Table 3 indicate that concentrate grade was 51.13% SiO₂ and it was lower than that in the feed (54.31%). Thus, quartz was not enriched by flotation with Fm 2835-2L. However, the minerals containing vanadium were slightly better recovered than quartz because V₂O₅ content increased to 1.04% from 0.82%. It is known that all the amines are relatively strong collectors for quartz (Wang et al., 2014; Liu et al., 2011; Vidyadhar and Hanumantha, 2007) and it has been confirmed in the single quartz flotation. However, the obtained results (Table 3) contradict this fact when flotation is performed for the decarburized sample of vanadium-bearing coal. Probable surface chemical phenomena giving rise to the observed experimental results will be discussed in the following section.

Production	Viald	V ₂ O ₅			CaO	SiO ₂		
	rield	Grade	Recovery	Grade	Recovery	Grade	Recovery	
Concentrate	34.67	1.04	43.97	2.01	9.48	51.13	32.64	
Tailing	65.33	0.70	55.77	10.18	90.52	56.00	67.36	
Feeding	100.00	0.82	100.00	7.35	100.00	54.31	100.00	

Table 3. Results of removing quartz from decarburized coal without pre-desliming. Numbers in %

It was known that pre-desliming improves vanadium enrichment (Bian, 2014). In this part of investigations, classification by sedimentation was used to remove slimes below 0.04 mm in size and the coarse grains above 0.04 mm. Next, to further remove the slimes, flotation at pH 9.4 with 2# oil was performed, followed by a second flotation to remove quartz at about pH 7.0 with Fm 2835-2L as the collector. The results of the process are shown in Fig. 6 and Table 4.



Fig. 5. Flowsheet for removing quartz from coal

Production	Yield —	V	⁷ ₂ O ₅	C	CaO	SiO ₂	
		Grade	Recovery	Grade	Recovery	Grade	Recovery
Slime	22.22	0.84	22.76	7.01	21.19	62.62	25.62
Slime	12.69	0.92	14.24	5.68	9.81	36.62	8.56
Concentrate	33.02	0.67	26.98	1.52	6.83	75.00	45.60
Tailing	32.07	0.93	36.37	14.25	62.17	34.25	20.22
Feeding	100.00	0.82	100.00	7.35	100.00	54.31	100.00

As Table 4 demonstrates, quartz was enriched in concentrate to 75% of SiO₂ through classification and flotation desliming. 2# flotation product (slime) contained fine grains also noticed by Bian (2014). One reason of better separation results may be that the pre-desliming process removed the detrimental ions from the flotation system. The addition of Fe^{3+} may be another reason of improvement of quartz recovery. Zhang et al. (2014) proved that Fe(III) was an activator for the flotation of quartz. To discuss this issue, ion dissolution would be studied in the following part of paper.



Fig. 6. Flowsheet for removing quartz from coal after pre-desliming

Influence of roasting on ion dissolution in flotation process

In flotation process, separation can be affected by polyvalent metal cation present in the slurry. The reason may be that the change of mineral composition and crystal structure after roasting lead to dissolution of ion. Therefore, polyvalent metal cations concentration in three kinds of slurry was determined by ICP and the results were shown in Table 7. Coal and decarburized coal were milled to -0.074 mm 63% and then mixed with aqueous solution and stirred for 10 min. The supernatant of the mixture

was used for ion concentration determination by means of ICP. The ion concentration of the flotation slurry of decarburized and deslimed stone coal was also tested by the same method. Otherwise, the ion concentrations of the three kinds of slurry were tested at the same slurry concentration, stirred for the same time (10 min), rest for 5 min to produce the supernatant.

Sample	Si^{4+}	Al^{3+}	Fe ³⁺	Mg^{2+}	Ca ²⁺	Ba ²⁺	
The head ore of coal	2.153	1.169	3.286	24.41	418.900	0.127	
Decarburized coal	5.040	0.304	0.234	30.56	1267.000	0.874	
Decarburized and deslimed coal	1.317	0.164	0.004	1.278	425.500	0.235	

Table 5. Polyvalent metal cation ion concentration in slurry (mg/dm³)

As shown in Table 5, the iron concentration in slurry changes sharply after the roasting decarburization of the stone coal. The concentration of Al^{3+} and Fe^{3+} ion decreased, while the concentration of Si^{4+} , Mg^{2+} , Ca^{2+} and Ba^{2+} increased. However, the ion concentration in solution after desliming decreased sharply. Studies of Ejtemaeia et al., 2012; Fornasiero and Ralston, 2005; Lev et al., 2012; Xu et al. 2013 have shown that in the sodium oleate flotation system and pH 8~10 the ions of Al^{3+} , Fe^{3+} and Ca^{2+} strongly activate the phyllosilicates minerals, while in dodecylamine flotation system, those ions have a strong suppression of the phyllosilicate minerals, while higher valence ions have a stronger suppression. Hence, most of polyvalent metal ions in the slurry can decrease the separation of calcite and mica. Roasting decreased Al and Fe content in the trivalent state, increased significantly Mg, Ca and Ba content in the divalent state. Roasting process resulted in the change of ion concentration in slurry, which is unfavorable for flotation.

Solution chemistry of Ca²⁺ in quartz flotation

The adsorption of calcium ions on quartz was influenced by the pulp pH significantly. The results of previous research on the relationship between pH and calcium ions adsorption on the quartz indicated that calcium ions are adsorbed on the surface of the quartz when the pH value was above 10.7 and the adsorption reached the peak when pH was 12.3 (Wang and Hu, 1987). It was known that metal ions activation of mineral was mainly due to the formation of hydroxy complexes, which required strict pH conditions. Therefore, the dissolved calcium species were studied. The following equilibrium equations can be considered:

$$Ca^{2+} + OH^{-} \square CaOH^{+}, \quad K_{1} = \frac{C_{CaOH^{+}}}{C_{Ca^{2+}} \times C_{OH^{-}}},$$
 (1)

$$Ca^{2+} + 2OH^{-} \Box \quad CaOH^{+}, \quad K_{2} = \frac{C_{Ca(OH)_{2(aq)}}}{C_{Ca^{2+}} \times C_{OH^{-}}^{2}},$$
 (2)

$$Ca(OH)_{2(s)} \square Ca^{2+} + 2OH^{-}, \quad K_{zp} = C_{Ca^{2+}} \times C_{OH^{-}}^{2}$$
 (3)

The balance for Ca is:

$$C_{Ca} = C_{Ca^{2+}} + C_{CaOH^{+}} + C_{Ca(OH)_{2(aq)}}$$
(4)

Taking the logarithm of Eqs. (1), (2) and (3) yields:

$$lg K_{1} = lg C_{CaOH^{+}} - lg C_{Ca^{2+}} - lg C_{OH^{-}}, \qquad (5)$$

$$lg K_{2} = lg C_{Ca(OH)_{2(aq)}} - lg C_{Ca^{2+}} - lg C_{OH^{-}},$$
(6)

$$\lg Ca(OH)_{2(s)} = \lg C_{Ca^{2+}} + \lg C_{OH^{-}}$$
(7)

For lg K₁ = 1.4, lg K₂ = 2.77, pK_{sp} = 5.22 the calcium ion concentration in the decarburized coal flotation system is 1267.000 mg/dm³, thus C_{Ca} = 3.168×10^{-2} mol/dm³. Solving Eqs. (4) to (7), the relation between C_{Ca²⁺}, C_{CaOH⁺}, C_{Ca(OH)_{2(aq)} and pH can be represented by Eqs. (8) to (11):}

$$\lg C_{Ca^{2+}} = -1.5 - \lg(1 + 25.12 \times 10^{pH-14} + 588.84 \times 10^{pH-14}), \qquad (8)$$

$$\lg C_{CaOH^+} = pH - 14.1 - \lg(1 + 25.12 \times 10^{pH - 14} + 588.84 \times 10^{pH - 14}), \qquad (9)$$

$$lg Ca(OH)_{2(aq)} = 2pH - 26.73 - lg(1 + 25.12 \times 10^{pH - 14} + 588.84 \times 10^{pH - 14}), \quad (10)$$

$$lgCa(OH)_{2(s)} = 2pH - 29.75 - lg(1 + 25.12 \times 10^{pH - 14} + 588.84 \times 10^{pH - 14}).$$
(11)

The log c-pH diagram is illustrated in Fig. 7.

The results of Fig. 7 indicated that hydroxy complex of Ca are formed when pH value is above 9.1. It can activate quartz and improve its recovery (Xie, 2011). However, the best pH condition for Fm 2835-2L flotation of quartz is about 7.0. The hydrolytic components (CaOH⁺, Ca(OH)_{2(aq)}, Ca(OH)_{2(s)}) are not formed in insignificant amounts, and only Ca²⁺ can be adsorbed on the surface of quartz and influence its recovery. Based on the available data (Xie, 2011; Fuerstenau and Han, 2002), Ca(OH)_{2(aq)} is not formed at pH values lower than 12.0, until the calcium ion concentration exceeds 5×10^{-2} mol/dm³, higher than 3.168×10^{-2} mol/dm³ in this study. Thus, Ca(OH)_{2(aq)} was not considered in this analysis. Also, at this calcium

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concentration, $Ca(OH)_{2(s)}$ forms when pH exceeds 12.3. Therefore, $Ca(OH)_{2(s)}$ precipitate was not considered here. Solution chemistry analysis of Ca^{2+} agree with the data of Wang and Hu (1987), Xie (2011) and Fuerstenau and Han (2002) and the zeta potential measurement already presented in this study. Thus Ca must be eliminated to improve the quartz floatability when Fm 2835-2L is used as the collector.



Fig. 7. Relation between log c and pH for Ca^{2+} , $CaOH^+$, $Ca(OH)_{2(aq)}$, Ca(OH)_{2(s)} in decarburized coal pulp

Conclusions

Ether diamine collector (Fm 2835-2L) adsorbs on the quartz surface by physical adsorption changing its zeta potentials significantly increasing its flotation. Under neutral or weakly acidic conditions calcium cation can adsorb onto the surfaces of quartz, increase the zeta potential of quartz particles, which in turn causes weaker aggregation of quartz particles and lower flotation recovery of quartz, and the hydrolytic components such as $CaOH^+$, $Ca(OH)_{2(aq)}$ and $Ca(OH)_{2(s)}$ are not formed in significant amounts in the best flotation tests. Ca must be eliminated in quartz flotation from decarburized vanadium bearing coal when Fm 2835-2L is used as the collector.

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