

Received November 21, 2020; reviewed; accepted April 05, 2021

Reverse flotation of iron ore using amphoteric surfactant: 2-((2-(decyloxy)ethyl)amino)lauric acid

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Abstract: 2-((2-(decyloxy)ethyl)amino)lauric acid ($C_{10}H_{21}CH(NH(CH_2)_3OC_{10}H_{21})COOH$, (LDEA), a novel amphoteric surfactant, has been first utilized as the collector for on-site the reverse flotation of Anqian mixed magnetic concentrates. The separation performances were investigated systematically by flotation conditioning tests, open-circuit flowsheet experiment, and locked cycle flowsheet test. The flotation condition test's results showed that the optimal roughing conditions were pulp pH of 10, starch dosage 1000 g/Mg, and LDEA dosage of 500 g/Mg at 25 °C. Under the optimized flotation conditions, through one roughing - three scavengings locked cycle flowsheet, the iron concentrate with a total iron grade of 68.08% and recovery of 88.20% was obtained. In contrast, the iron grade of the tailings was only 12.32%. Compared with the flotation results of anionic commercial surfactant RA-715 used as the collector, the LDEA has a better separation performance. The iron grade and recovery increased by 0.07% and 0.86% in the iron concentrate, and the grade of the tailings decreased 3.72%, respectively. Besides, the LDEA possesses more advantages, such as a simple flotation process, no activator (CaO) addition, lower collector consumption, and lower pulp temperature. The study revealed that the novel amphoteric surfactant LDEA was an effective flotation collector with good collecting and separation ability on natural iron ores at a comparatively lower temperature.

Keywords: 2-((2-(decyloxy)ethyl)amino)lauric acid, iron ores, reverse flotation, amphoteric surfactant, low temperature

1. Introduction

Iron has great importance due to its wide application in industries, ranking first among the metals in worldwide production and consumption (Nakhaei and Irannajad, 2017). Its low cost and high strength properties make it indispensable in engineering applications such as the construction of machinery and machine tools, automobiles, the hulls of large ships, and structural components of buildings (Greenwood and Earnshaw, 2012). In China, after years of mining, the high grade and easily separated iron ores are decreasing year by year (Li, 2018). Therefore, the exploitation and utilization of low-grade fine-grained complicated refractory iron ores are urgent in need to maintain the rapid and healthy development of the Chinese iron and steel industry (Filippov et al., 2010; Liu et al., 2009; Shen and Huang, 2005; Zhang et al., 2017).

Froth flotation technology is the most effective solution for separating gangue minerals from valuable iron-containing minerals technologically and economically (Liu et al., 2009; Wills and Finch, 2015). Even though the cationic reverse flotation remains the most popular separation in the iron ore industry (Araujo et al., 2005; Filippov et al., 2014), in recent years, the anionic reverse flotation has been successfully practised in the separation. In addition, the utilization of fine-grained iron ores in China's major iron ore area has been concentrated on anionic reverse flotation (Luo et al., 2016b; Ma, 2012; Weng et al., 2013). In the anionic reverse flotation, the gangue minerals, mainly silica (in the form of quartz)

are activated by divalent calcium ions and will be floated out through the interactions with anionic collectors. In contrast, the iron-containing minerals are kept in suspension with depressant starch (Cao et al., 2013; Lima et al., 2013; Liu et al., 2016).

During the froth flotation process, collector properties, especially its solubility in the pulp, adsorption degree onto the mineral surface, and hydrophobicity, play essential effects for achieving the iron concentrate with optimal iron grade and recovery indexes. Among these properties, the solubility of surfactants comes in the first place as the most extensively used anionic collectors are long carbon chain fatty acids, alkyl sulfonic acids, alkyl sulfuric acids, alkyl hydroxamic acids, and their sodium or potassium salts (Araujo et al., 2005; Quast, 2006). In order to increase these collectors' effectiveness and sufficient solubility in solution, the pulp water temperature should be kept at a high level (35 - 45 °C), which leads to a great of non-renewable resources consumption and emission of carbon dioxide and other greenhouse gases (Luo et al., 2015, 2018; Zhu et al., 2015). Besides, due to the extensive use of lime (CaO) as the activator in the anionic reverse flotation, pipelines blockage and affect normal industry production operations. For lime, it is prone to precipitate and form limescale. Furthermore, the higher calcium ion concentration in recycling process water could decrease the total iron grade of the concentrates when the common fatty acids were used as the collector, such as RA-715, KS-II, etc (Jiang and Xu, 2017; Mei et al., 2009).

In our previous paper, the novel amphoteric surfactant 2-((2-(decyloxy)ethyl)amino)lauric acid had been synthesized. Later, the collecting performance and adsorption mechanism of the collector on quartz mineral surface had been investigated through zeta potential, FT-IR, XPS measurements, and molecular dynamic simulations, in conjunction with the results of quartz mineral micro-flotation tests (Luo et al., 2018; Luo et al., 2019). The flotation results showed that LDEA exhibited an excellent collecting performance on quartz minerals at a wide pulp pH range (about 6 - 11) with a low collector concentration of 15.0 mg/dm³ at a relatively lower temperature 15 °C. The adsorption mechanisms reveal that the LDEA could be adsorbed onto the surface of pure quartz in the forms of electrostatic and hydrogen-bonding interactions at pH 10. However, the natural iron ores are more complex than the single mineral for their mineral compositions. Moreover, the froth flotation of raw iron ores seems like an excellent example of an engineering "system" in that the various important parameters are highly interrelated (Kawatra, 2011). Therefore, it is meaningful to verify the collecting ability and selectivity of the novel surfactant LDEA when used as the collector for the reverse flotation of natural iron ores.

In this paper, the froth flotation performances of the LDEA were systematically investigated in consideration of pulp pH, starch dosage, collector dosage, CaO dosage as well as pulp temperature firstly. Based on the optimal reagent system obtained from flotation conditioning experiments, the open circuit flowsheet experiment was carried out to determine the optimal locked cycle flowsheet, which would be later used to achieve the iron concentrate products. Compared with the flotation results of on-site surfactant RA-715 used as the collector, the flotation separation performances of the novel LDEA were evaluated for further industrial application, as a substitute for RA-715, in cutting the energy consumption and protecting the environment.

2. Materials and methods

2.1. Materials

The natural iron ores used in the flotation condition tests and open/locked circuit flowsheet tests were mixed magnetic separation concentrates supplied by the Anqian iron mine plant in China. The iron ore samples were firstly characterized using chemical analysis and XRD analysis. The results are shown in Table 1 and Fig. 1, respectively.

The content of valuable total iron (TFe) is 44.83%, and the main chemical compositions of the natural iron ores are FeO (14.20%) and SiO₂ (38.83%), while the other chemical compositions are less than 1% (Table 1). As shown in Fig. 1, the elemental iron mainly occurs in magnetite and hematite forms which are associated with the gangue minerals, including quartz, chlorite, and amphibole.

Meanwhile, the iron in the mixed magnetic concentrates is distributed in magnetite (84.65%), hematite (12.05%), carbonate (1.43%), and iron silicate minerals (1.27%) as well as in iron sulfide minerals (0.60%) (Table 2). The particle size distribution and TFe content in each size fraction of the samples are presented in Table 3. It shows that the -0.038 mm fraction is 63.37 % by mass weight and

has a high iron content of 85.16%, indicating the anionic/amphoteric reverse flotation is suitable for recovery the iron resources from natural iron ores.

Table 1 Chemical element analyses of the natural iron ores (wt. %)

Composition	TFe	FeO	SiO ₂	Al ₂ O ₃	MgO	K ₂ O	CaO	P	S
Content	44.83	14.20	38.83	0.22	0.63	0.03	0.39	0.014	0.007

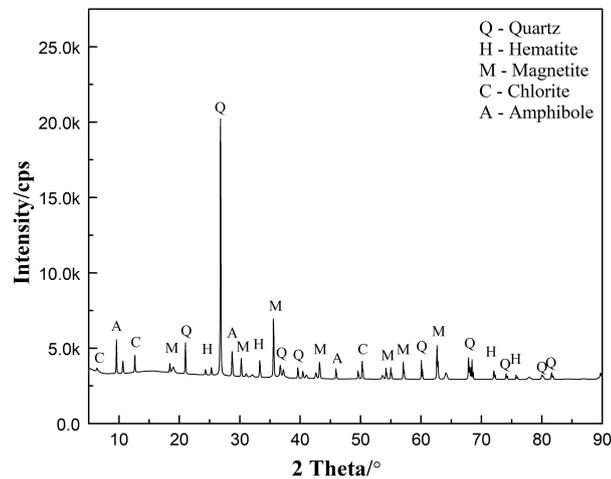


Fig. 1. X-ray diffraction patterns of the natural iron ores

Table 2. Chemical phase analysis of Fe and its distribution in the natural iron ores

	TFe	Distribution/ %				
		In magnetite	In hematite	In silicate	In carbonate	In sulfide
Sample	44.83	37.95	5.40	0.57	0.64	0.27
	100.00	84.65	12.05	1.27	1.43	0.60

Table 3. Particle size distribution analyses of the natural iron ores and TFe content in each fraction

Size fractions/mm	Yield/ %	Grade/ %	Distribution/ %
+ 0.150	1.65	9.66	0.36
-0.150 + 0.074	15.45	9.52	3.28
-0.074 + 0.038	19.53	25.70	11.20
-0.038	63.37	60.25	85.16
Total	100.00	44.83	100.00

2.2. Reagents

The novel amphoteric surfactant 2-((2-(decyloxy)ethyl)amino)lauric acid was used as the collector and synthesized in the laboratory (Luo et al., 2018). Non-modified corn starch provided by the Anqian plant was used as the depressant, and the starch solution was freshly prepared to avoid degradation. Industrial purity of lime (CaO) was used as the source of Ca(II) supplied by the Anqian plant. Ultra-pure and laboratory tap water was used throughout the actual ores froth flotation tests.

2.3. Bench-scale flotation tests

Flotation condition tests of Anqian mixed magnetic concentrates were conducted with an XFDIV0.5 flotation machine with a 0.5 dm³ cell. 240 g samples were added into the cell (pulp density 40% by weight), in which 0.360 dm³ water was filled later. The slurry was agitated for 3 min at a stirring speed of 1900 rpm before adding 5% HCl or 5% NaOH solution to adjust pulp pH to a specific value. Starch,

lime, and LDEA were added into the cell with a 3 min interval continually. The suspension was agitated for 3 min after adding all the desired amount of reagents, and the flotation separation was conducted for 5 min. The froth products and tailings were weighed and assayed separately after drying, and the recovery was calculated based on the weight and elemental contents of the products obtained. The flowsheet of flotation condition experiments was presented in Fig.2. Considering both the TFe grade and TFe recovery indexes, it could be easy to compare the flotation performance under different conduction conditions. Moreover, the flotation open-circuit flowsheet experiment (Fig. 3) was conducted under the achieved optimal flotation conditions.

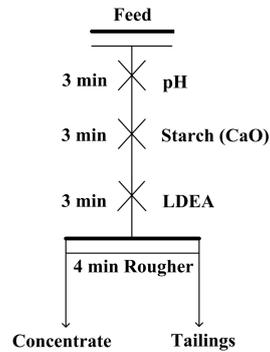


Fig. 2. The flowsheet of flotation condition experiments

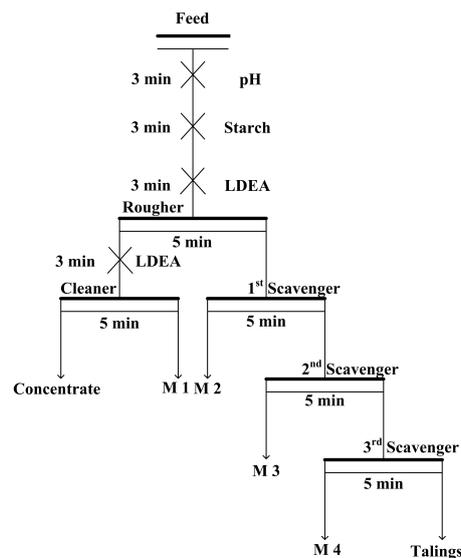


Fig. 3. The flowsheet of flotation open circuit experiment (M - middling product)

3. Results and discussion

3.1. Results of flotation condition experiments

Under the flotation conditions of starch dosage 1200 g/Mg, collector dosage of 800 g/Mg LDEA at room temperature 25 °C, the flotation separation performances of the natural iron ores as a function of slurry pH are presented in Fig. 4.

The pulp pH not only affects the surface chemistry of most minerals but also influences the state of ionization of the collectors, which eventually could determine the adsorption mechanisms of the collector on the mineral surfaces and separation performance (Fuerstenau and Pradip, 1984; Fuerstenau and Palmer, 1976; Leja, 2012). As shown in Fig. 4, the TFe recoveries in the iron concentrates were lower than 20% in weak acid to the neutral environment (pH 4.0 - 7.0). It may be due to the weak depressing of corn starch for the iron-containing minerals. With increasing pH from 7 to 10, the recovery of TFe sharply risen from 18.39% to 69.33% with TFe grade risen from 55.98% to 63.16%, respectively;

continually increasing the pulp pH to 11.5, the effective concentration of the LDEA species descended and resulted in the TFe grade decreasing gradually to 59.55%. The optimal pulp pH was chosen as 10, at which the better flotation separation performance was achieved. Under the pH 10, starch dosage of 1200 g/Mg at 25.0 °C, the influence of LDEA dosage on the flotation separation performances is shown in Fig. 5. As shown in Fig. 5, increasing the LDEA dosage from 300 g/Mg to 1000 g/Mg, the TFe grade of the iron concentrate risen from 50.85% to 69.64%, in the meanwhile the TFe recovery of the iron concentrate decreased from 89.96% to 47.73%.

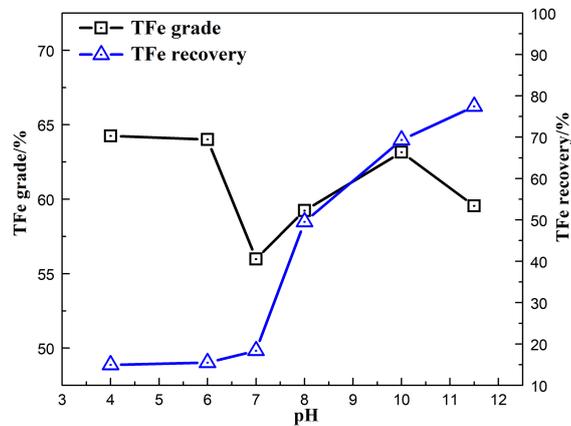


Fig. 4. The separation performances of the natural iron ores as a function of pH

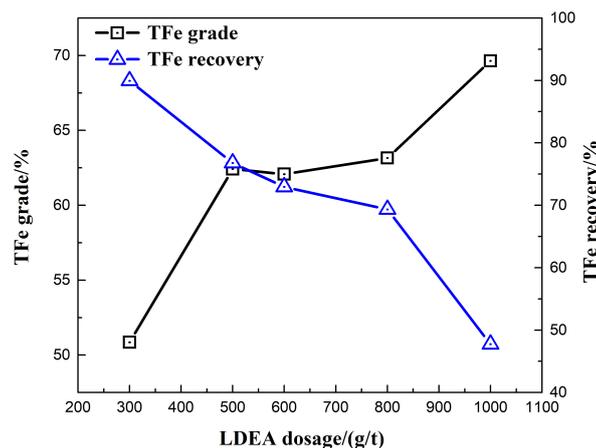


Fig. 5. The separation performances of the natural iron ores as a function of collector LDEA dosage

Fig. 6 shows the flotation separation performances as a function of starch dosage under slurry pH 10, LDEA dosage 500 g/Mg at 25.0 °C. The influence of starch dosage is opposite to that of LDEA dosage on flotation performances with considering both TFe grade and TFe recovery indexes of the iron concentrates. The optimum starch dosage was decreased to 1000 g/Mg, where the TFe grade of the concentrate increased from 62.42% to 67.28% compared with the results when starch dosage 1200 g/Mg was used as the depressant.

As we all know, collectors' solubility and selectivity affect the flotation separation performance, mostly determined by the pulp temperature. The separation performances of the natural iron ores as a function of pulp temperature are shown in Fig. 7. The TFe grade of 65.32% and TFe recovery of 64.43% in the iron concentrate was obtained at 20 °C under the flotation conditions of pulp pH 10, starch dosage 1000 g/Mg, and LDEA dosage 500 g/Mg. It means the new amphoteric LDEA showed a strong collecting ability and excellent selectivity for the flotation separation of the natural iron ores at a relatively lower temperature. With increasing the pulp temperature from 20 °C to 25 °C, the TFe grade of the concentrate increased to 67.28%, whereas the TFe recovery decreased to 61.80%. Continually raising the pulp temperature to 40 °C, the TFe grade of the concentrate slightly increased about 2.35% as TFe recovery of the iron concentrate decreased about 3.60%, respectively, which illustrates that the

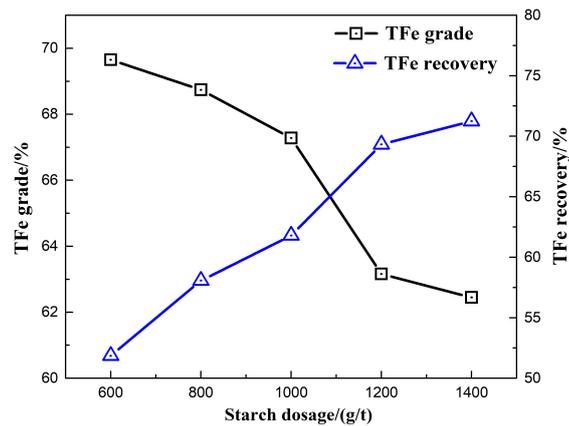


Fig. 6. The separation performances of the natural iron ores as a function of depressant starch dosage

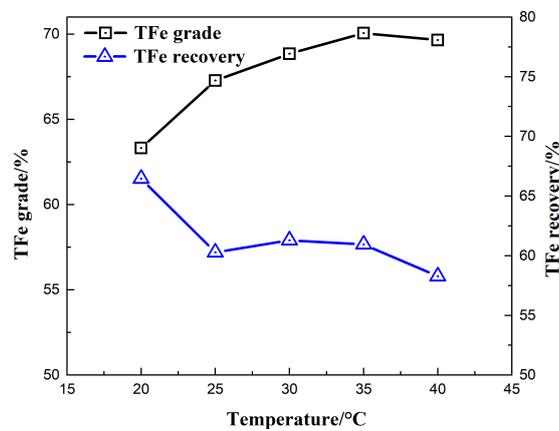


Fig. 7. The separation performances of the natural iron ores as a function of pulp temperature

LDEA exhibited an excellent flotation separation performances in the pulp temperature range from 20°C to 40°C. The appropriate temperature was chosen at 25 °C, which was on-site room temperature with easy flotation operation in the laboratory.

Under the flotation conditions of pulp pH 10, starch dosage 1000 g/Mg, LDEA dosage 500g/Mg at 25 °C, the effect of CaO dosage on the flotation separation performances of the natural iron ores is shown in Fig. 8. It is well-known that the pure quartz mineral could not be floated out with only fatty acid collectors. The DFT calculation results indicate that calcium ions act as a bridge between the α -quartz (101) surface and modified fatty acid collector (Zhu et al., 2016), resulting in the adsorption of fatty acid collectors onto calcium-activated quartz surface. In cationic reverse flotation system, the presence of calcium ions may depress the recovery of quartz mineral due to the following reasons: i) the interactions between the cationic collectors and quartz surfaces are foremost in the form of electrostatic force (Sayilgan and Arol, 2004); ii) the adsorption of calcium ions on quartz surfaces increases the quartz surface potential which further weakens the adsorption force of the collector on the quartz surface.

In Fig. 8, the CaO dosage has a slight effect on the iron ore flotation performance generally may relate to the amphoteric molecular structure of the LDEA, which containing the carboxyl group (-COOH) and amino group (-NH₂) simultaneously. The iron concentrate with TFe grade of 67.05% and recovery of 61.07% was obtained at 0 g/Mg CaO dosage (ultra-pure water used). With continually increasing the CaO dosage to 400 g/Mg, the TFe grade of the iron concentrate increased less than 1.6%, and the respective TFe recovery of the concentrate decreased less than 6%. The results illustrate the following conclusions: i) LDEA could float out the gangue and unliberated particles without CaO addition; ii) the increasing of CaO dosage could enhance the collectivity of LDEA for the unliberated particles and larger size gangue minerals, which resulted in the increasing of the TFe grade of the concentrate; iii) the novel collector LDAE has a better tolerance to calcium ions in water compared with the collector RA-715 (Jiang and Xu, 2017).

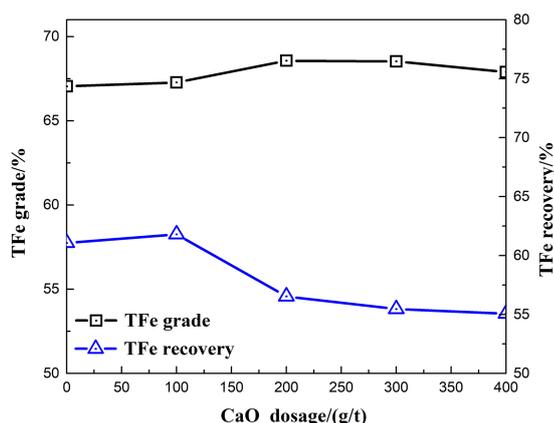


Fig. 8. The separation performances of the natural iron ores as a function of CaO dosage

3.2. Results of flotation open-circuit and locked cycle experiments

Under the conditions of pulp pH 10, starch dosage 1000 g/Mg, and LDEA dosage 500g/Mg at 25 °C, the flotation open-circuit experiment following one roughing - one cleaning - three scavengings flowsheet (Fig. 3) (half collector dosage added in cleaning operation and no collector added in scavenging operation) was conducted to investigate the locked cycle flowsheet. The result of the flotation open-circuit experiment is shown in Table 4.

Table 4. Results of the flotation open-circuit flowsheet experiment (M - middling product)

Product	Yield/%	Grade/%	Recovery/%
Concentrate	18.85	70.34	29.30
M 1	20.13	69.60	30.96
M 2	13.45	63.91	19.00
M 3	7.08	58.86	9.20
M 4	3.32	53.34	3.90
Tailings	37.16	9.30	7.64
Total	100.00	44.37	100.00

It was found that the TFe grade of 70.34% and TFe recovery of 29.30% in the iron concentrate was obtained through one roughing - one cleaning - three scavengings reverse flotation open-circuit flowsheet. Considering the TFe grade of 69.60% in middling 1 product close to that of the iron concentrate, it was reasonable to reduce the cleaning operation. Meanwhile, the TFe grade of the tailings was only 9.30%, demonstrating that the thrice scavenging process is enough to recover the iron resource. Followed the one roughing with three-scavengings flowsheet was applied to conduct the locked cycle flowsheet flotation separation experiment.

The optimized flotation conditions and the flotation flowsheet of the locked cycle experiments when using LDEA and RA-715 (Guo et al., 2016) as the collectors are listed in Fig. 9 and Fig. 10. The results of bench-scale locked cycle experiments are shown in Table 5.

As shown in Table 5 and Fig. 9, the iron concentrate obtained the indexes of TFe grade of 68.08% and TFe recovery of 88.20%, and the TFe grade of the tailings was only 12.32% at 25 °C through one roughing and three scavengings flotation operation. For RA-715, the optimal flotation separation of the natural iron ores could be achieved under the conditions of pulp pH 11.5, depressant starch dosage 1100 g/Mg, roughing collector RA-715 dosage 530 g/Mg, cleaning collector RA-715 270 g/Mg at 40 °C.

It is obvious to be seen that the novel collector possesses a better flotation index where the TFe grade and TFe recovery increased 0.07% and 0.86 % in concentrate and a TFe grade of 3.72% decreased in tailings. Besides, the advantages such as a simple flotation process, no calcium ions addition, lower reagent consumption of depressant starch dosage and collector dosage, and lower temperature were found when LDEA was used as the collector. The results revealed that the novel amphoteric surfactant LDEA was an effective flotation separation collector with good collecting and separation ability on

natural iron ores at a comparatively lower temperature and could be considered as a substitute for the collector RA-715.

Table 5. Results of bench-scale locked cycle experiments using LDEA and RA-715 as collectors

Collector	Product	Yield/%	Grade/%	Recovery/%
LDEA 25 °C	Concentrate	57.48	68.08	88.20
	Tailings	42.52	12.32	11.80
	Total	100.00	44.37	100.00
RA-715 40 °C	Concentrate	61.30	68.01	87.34
	Tailings	37.70	16.04	12.66
	Total	100.00	47.75	100.00

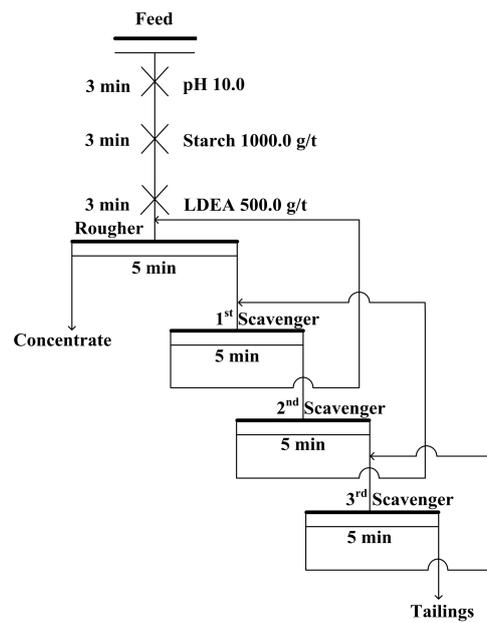


Fig. 9. The optimized flotation conditions and the flotation locked cycle experiment's flowsheet when LDEA was used as the collector

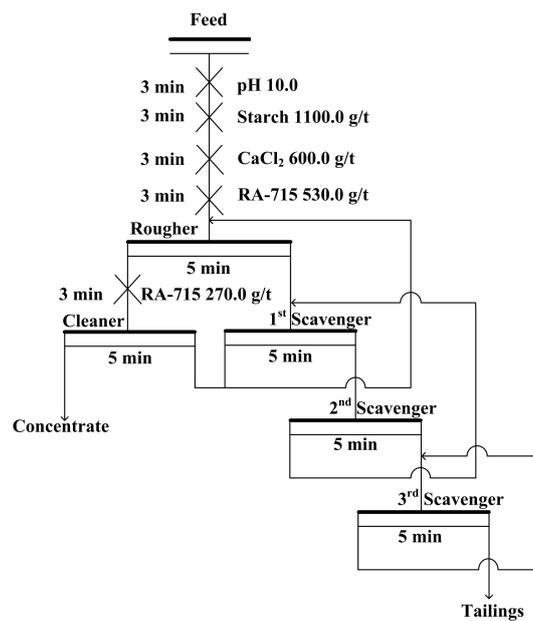


Fig. 10. The optimized flotation conditions and the flowsheet of the flotation locked cycle experiment when RA-715 was used as the collector

3.3. XRD and EDS analyses

The results of mineral composition in the concentrate and tailings obtained through reverse flotation locked cycle flowsheet using LDEA as the collector are shown in Fig. 11.

Compared with the XRD pattern of raw nature iron ores (Fig. 11a), the most of the gangue minerals in the iron ores have been collected to the tailings with the help of LDEA, so that the iron-containing minerals of hematite and magnetite are found in the concentrate (Fig. 11). The results indicate that the novel surfactant LDEA can be considered as a valid collector to separate quartz from hematite and magnetite, which is in agreement with the results of flotation experiments. However, a few characteristic peaks of hematite and magnetite are still shown in Fig. 11c, which might be due to the entrainment of some fine hematite or magnetite particles in the froth bubble.

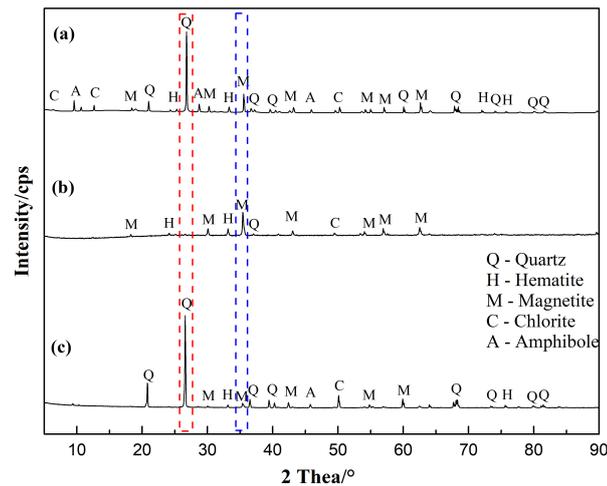


Fig. 11. XRD patterns of raw natural iron ores (a), reverse flotation concentrate (b), and tailings (c)

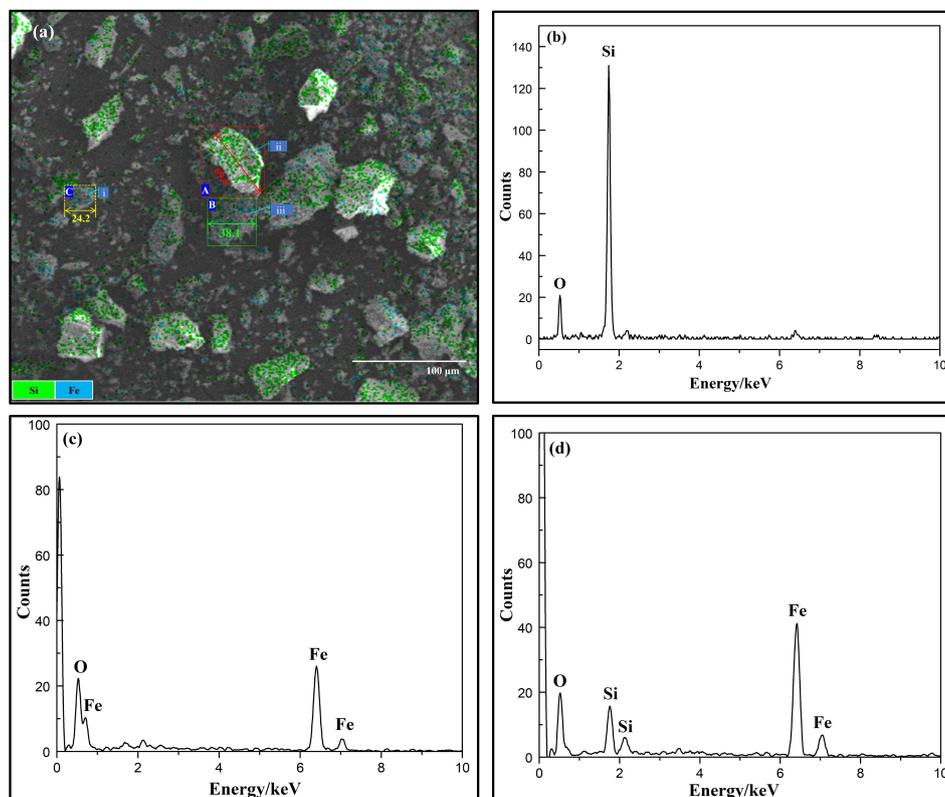


Fig. 12. SEM image and EDS analyses of the reverse flotation tailings when LDEA used as the collector (a - SEM image; b - ii point in particle A; c - i point in particle C; d - iii point in particle B)

SEM image and EDS analyses of the reverse flotation tailings obtained using LDEA as the collector were shown in Fig. 12. As shown in Fig. 12, the gangue mineral quartz particles (Fig. 12a) are evenly distributed in the reverse flotation tailings with larger particle size (A) compared with the hematite or magnetite mineral particles (C). It is noteworthy that the intergrowths were founded with a small particle size (B). The liberated quartz and unliberated particles have been floated out of the raw nature iron ores using LDEA as the collector, while some fine hematite and magnetite could not be depressed by starch (Luo et al., 2016a; Luo et al., 2016b).

4. Conclusion

The novel amphoteric surfactant LDEA has been proven to be an efficient collector for reverse flotation separation of natural iron ores. Under the reverse flotation conditions of pulp pH 10, depressant starch dosage 1000 g/Mg, and LDEA dosage 500g/Mg at 25 °C, through one roughing and three scavengings locked cycle flowsheet, the iron concentrate containing TFe grade of 68.08% with a TFe recovery of 88.20% was obtained. The TFe grade of the tailings was only 12.32%. XRD, SEM with EDS results show that the liberated quartz and unliberated particles could be floated out to the tailings, while some fine hematite and magnetite cannot be depressed by starch. The study demonstrated that the application of amphoteric LDEA in iron ore flotation was prospective to the efficient exploitation and utilization of refractory iron-containing resources.

Acknowledgments

The authors gratefully acknowledge and appreciate the financial support provided by the National Natural Science Foundation of China (Grant No.51274056 and 51474055); and the Open Foundation of State Key Laboratory of Mineral Processing (BGRIMM-KJSKL-2021-07).

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