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PROPERTIES OF FATTY ACID/DODECYLAMINE MIXTURES AND THEIR APPLICATION IN STEAM COAL REVERSE FLOTATION

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Abstract: A series of surfactant mixtures which consist of DAH (dodecylamine hydrochloride) and fatty acids (FAD) were made in this study. The characteristics of these fatty acids/DAH mixtures, including surface tension, critical micelle concentration (CMC) and adsorption onto coal were investigated. The reverse flotation results with these fatty acids/DAH mixtures (FAD) and DAH were compared with each other. Experimental results showed that the fatty acids/DAH mixtures have lower CMC and γ_{cmc} than DAH. The adsorption test indicated that more amine molecules would adsorb preferentially onto the bubbles surface in FAD solutions than that in DAH solutions. Reverse flotation results showed that about 50% surfactant dosage was saved under the same froth product yield. Only 28% froth product yield was obtained in the presence of 1.66 kg/Mg DAH. However, when 0.83 kg/Mg hexadecanoic acid/DAH mixture (C16D) surfactant was used, the froth product yield reached 29%. Dodecanoic acid/DAH mixture (C12D) surfactant showed better performance with a high mineral matter recovery similar to that with tetradecanoic acid/DAH mixture (C14D) and higher combustible recovery than with C14D and C16D.

Keywords: steam coal, surfactant mixture, reverse flotation, fatty acids, surface activity

Introduction

Even though the coal reverse flotation process has some obvious advantages over forward flotation and a development period of around 50 years starting from 1960s, it is still an established route neither for ash reduction nor for pyrite removal. This indicates that there is scope of improvement and more work needs to be done in order to bring the process to the commercialization stage. The idea of reverse flotation was first patented by Eveson in 1961. The concept was further developed for the selective removal of pyritic sulphur from US coals by several investigators (Baker et al., 1973; Miller, 1973, 1975, 1978; Miller and Deurbrouck, 1982; Lin, 1982; Miller et al.,

1984). Consequently, the study of reverse flotation of coal with a motive to lower the ash content of coal was carried out by many scientists and researchers (Stonestreet and Franzidis, 1988, 1989, 1992; Pawlik and Laskowski, 2003a, 2003b; Ding and Laskowski, 2006a, b; Patil and Laskowski, 2008, Zhang et al., 2013; Ozturk and Temel, 2013; Xia and Yang, 2013a; Zhang and Liu, 2015).

The driving force behind the works done so far to develop reverse flotation as a viable processing tool to produce clean coal is the advantages associated with it. Coal makes up around 60% to 80% of the flotation feed and as the coal rises, with it gangue particles also rise which cannot be avoided. This problem is known as ash entrainment which is inevitable during direct flotation and adversely affects the quality of product (Angadi and Suresh, 2005; Angadi et al., 2012). Here coal reverse flotation can provide a good substitute as the gangue particles are floated with some coal entrainment. But in comparison to ash entrainment, coal entrainment will be less significant due to the coarser size of coal particles and smaller physical mass transfer of coal into the froth. Another benefit is the reduction of mass transfer as the gangue material makes up 15% to 35% of the feed. So the concentrate mass is lowered and the overall carrying capacity of the froth is reduced which implies that reverse flotation can be carried out at higher pulp densities and using small sized equipment. The gangue minerals in comparison to coal have a consistent chemical composition and well defined structures which impart them uniform floatability. Above all, these coal reverse flotation may prove to be a better option for coals with poor floatability characteristics which do not give sufficient recovery with forward flotation.

With the increasingly serious air pollution in China, coal slime in steam coal preparation plants need to be upgraded with flotation technology. However, it is difficult to float these coal slimes in conventional coal flotation processes due to its low hydrophobicity. Compared with flotation of coal slimes in coking coal preparation plants, a certain amount of ash reduction can be acceptable for coal slimes in steam coal preparation plant. Therefore, reverse flotation of steam coal slimes should be more suitable.

Although coal reverse flotation displays these advantages, it is still not brought to the commercialization stage. The main obstacle is the huge dosage and the expensive price of the collector. Thus, many scientists and researchers have studied on many ways to decrease the dosage of the collector. According to the papers by Pawlik and Laskowski (2003b) and Ding and Laskowski (2006a), dodecyltrimethyl ammonium chloride (DTAC) was used as collector which showed good separation but not at dosage lower than 6 kg/Mg. They found that for hydrophilic oxidized and sub bituminous coals high dosages of amine (around 4 ~ 6 kg/Mg), were needed to initiate the reaction. In addition, they found that the use of PAM (polyacrylamide) along with the zero-conditioning method brought down the amine dosage from 6 kg/Mg to 1.375 kg/Mg at rejection yield more than 80% (Ding and Laskowski, 2006b). In this paper, mixtures of DAH (dodecylamine hydrochloride) and fatty acids were made and used as mineral collectors. Generally, surfactant mixtures can have a number of synergistic

advantages over the use of a single surfactant type. Many studies have been conducted on mixed micellar systems that consist of two surfactants (Fang et al., 2003; Penfold et al., 1996; Xiao et al., 2000; Rubingh and Jones, 1982; Xue et al., 2009). Among various surfactant combinations, mixed cationic–nonionic surfactants are of great interest because of their strong synergistic effects on the interfacial properties and spontaneous vesicle formation behavior in aqueous phases (Desai and Dixit, 1996; Penfold et al., 1997; Vora et al., 1999). A system that contains two surfactants of different charge is called a catanionic system. The attraction between the different charged head groups will lead to a decrease in the area per head group (Herrington et al., 1993). However, there are limited studies on the application of surfactant mixtures in coal reverse flotation.

In this paper, five different fatty acids were mixed with DAH respectively. The characteristics of these fatty acids/DAH mixtures, including surface tension, critical micelle concentration (CMC) and adsorption onto coal were investigated. And the reverse flotation results with these fatty acids/DAH mixtures and DAH were compared with each other. In this paper, gangue minerals are floated into froth as froth products, clean coals are left in flotation pulp as concentrate.

Experiment method and procedure

Samples and reagents

Coal samples were obtained from the Steam Coal Preparation Plant in the Shanxi Province. The over 0.5mm size materials were removed. The proximate analysis of coal samples (air-dried) is given in Table 1, where M_{ad} is the moisture content, V_{ad} the volatile content, FC_{ad} the fixed carbon content, A_{ad} the ash content, and S_t is the total sulfur content.

Table 1. Proximate analysis of oxidized coal sample (air dried)

M_{ad} (%)	V_{ad} (%)	FC_{ad} (%)	A_{ad} (%)	S_t (%)
6.02	30.47	38.01	25.5	0.8

The size composition of coal samples is given in Table 2. The weight of -0.04mm size fraction is the highest which is 68.34%. Meanwhile, the ash content of -0.04mm size fraction is the highest which is 33.01%.The size composition of coal samples indicates that the coal is primarily made up of fine coals with high ash content.

X-ray diffraction is used to study the composition of coal samples. The XRD test was performed at room temperature. The samples were ground to $-50\mu\text{m}$. The step size of the test is 0.01° and the speed is $8^\circ/\text{min}$. The scanned area is $5^\circ\sim 80^\circ$.

Table 2. Size composition of coal samples

Size fraction (%)	Weight (%)	Ash content (%)
-0.5 + 0.25	3.25	8.66
-0.25 + 0.125	9.38	9.52
-0.125 + 0.074	9.45	9.88
-0.074+0.045	9.58	10.21
-0.045	68.34	33.01
Total	100.00	25.65

Figure 1 showed that the main gangue mineral in the coal sample is kaolinite and quartz. The IEP of kaolinite was at pH 1.8. And the IEP of quartz was 2.7. Thus, in these experiments they could be floated by cationic collector.

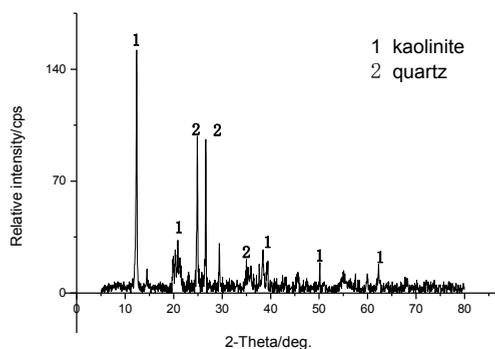


Fig. 1. X-ray diffraction patterns of the sample

The analytically grade DDA (dodecylamine) used in this study was obtained from the Tianjin Guangfu Chemical Research Institute. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used as pH regulators. HCl and NaOH used in this study were of analytical grade obtained from Aladdin Reagent (China). DDA neutralized with HCl regarded as DAH, the solution concentration was 0.135 mol/dm^3 . Reagent grade fatty acids and dextrin were supplied by Aladdin Reagent. The following fatty acids were used: n-octanoic acid, C8 (purity $\geq 98\%$), n-capric acid, C10 (purity $\geq 98\%$), dodecanoic acid, C12 (purity $\geq 98\%$), tetradecanoic acid, C14 (purity $\geq 98\%$) and hexadecanoic acid, C16 (purity $\geq 98\%$). All surfactants and fatty acids were used as received. The full names and abbreviations of all reagents were listed in Table.3. Each fatty acid was mixed with 50 cm^3 DAH solution and stirred with a magnetic stirrer at $50 \text{ }^\circ\text{C}$ until a homogeneous mixture was obtained. The pHs of the mixtures were 3.8–4.1 and the molar ratio of DAH and fatty acid was 1:1.

Table 3. Reagents

Full name	Abbreviation
Dodecylamine	DDA
Hydrochloric acid	HCl
Sodium hydroxide	NaOH
Dodecylamine Hydrochloride	DAH
Fatty acid/DAH mixture	FAD
n-octanoic acid/DAH mixture	C8D
n-capric acid/DAH mixture	C10D
Dodecanoic acid/DAH mixture	C12D
Tetradecanoic acid/DAH mixture	C14D
Hexadecanoic acid/DAH mixture	C16D

Methods

Surface tension measurement

Surface tension measurements were determined at pH=5 by the du Nouy ring method using a LAUDA (TEIC) Tensiometer (Germany). The instrument was calibrated against double distilled water. Measurements were made at intervals of a few minutes until successive values agreed within a standard deviation of 0.1mN/m or less. The CMC values were determined graphically as the intersection point of the two linear portions of the plot (Li et al., 2000).

Adsorption experiments

In these tests, 1 g of the clean coals were preconditioned for 20 min with 25 cm³ of tap water in 100 cm³ glass bottles, placed in a magnetic stirrer at 800 rpm, to ensure complete wetting of the solids. Then 1 cm³ of solutions of different concentrations were added to each sample and the mixtures were conditioned for 1 h at pH=5 which was sufficient to achieve adsorption equilibrium. Then the centrifugate of the solution was used for cationic collector concentration test. Cationic collector concentration was measured using the Eosin Y method (limit of detection less than 10.0 mg/dm³) (Qin and Tan, 2006). The absorbance of DAH–Eosin Y complex was measured with a UV/Vis spectrometer at 516.5 ± 0.5 nm in a 1.0 cm long quartz cuvette respectively. The amount of collector adsorbed was calculated using the expression:

$$n_{ads} = \frac{(C_0 - C_{eq})V}{m} \quad (1)$$

where n_{ads} is the adsorption quantity of collector per gram of coal, C_0 is the initial concentration of collector, C_{eq} is the concentration of collector at a given time after

adsorption, V is the volume of the solution and m is the weight of coal sample. The standard curve of DAH solution was showed in Fig. 2.

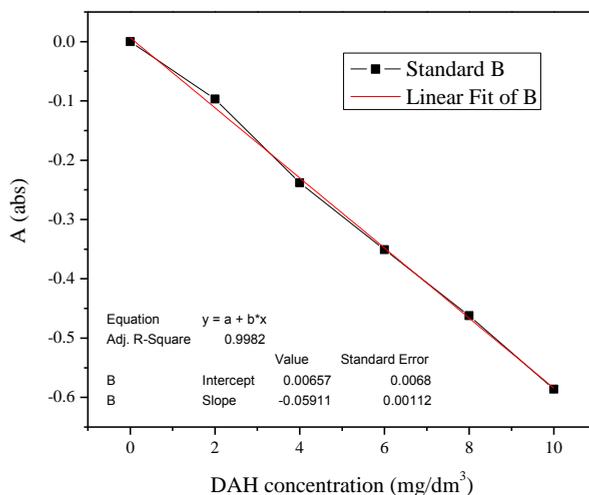


Fig. 2. Standard curve of DAH solution (pH = 5)

Adsorption of DAH and FADs on bubbles

In these tests, different dosages of DAH and FADs were added to a 1 dm³ XFD laboratory flotation cell, respectively. The concentrations of the collectors were 0.27 mmol/dm³, 0.35 mmol/dm³, 0.43 mmol/dm³ and 0.54 mmol/dm³. The impeller speed is 1800 rpm, which produced an aeration rate of 1.33 dm³/min. The bubbles were collected after 5mins and the concentrations of surfactants in the flotation cell were then measured using the Eosin Y method.

Reverse flotation test

Reverse flotation tests were carried out in a 1 dm³ XFD laboratory flotation cell. In all flotation tests, a 60 g sample was used. The impeller speed in both conditioning and flotation processes is 1800 rpm, which produced an aeration rate of 1.33 dm³/min. The coal sample was first prewetted in flotation cell for 1 min. Then pH was adjusted with HCl at pH = 5. Dextrin was added to flotation slurry and conditioned for 2 min. Then collector was added and conditioned for 1 min. Each flotation test was performed for 5 min. The dosage of dextrin was 0.8 kg/Mg. The dosages of collectors were 0.83 kg/Mg, 1.08 kg/Mg, 1.33 kg/Mg, and 1.66 kg/Mg corresponding to 0.27 mmol/dm³, 0.35 mmol/dm³, 0.43 mmol/dm³ and 0.54 mmol/dm³.

Results and discussion

Surface activity of DAH and FADs

The results of surface tension (γ) measurements of DAH and FADs are shown in Fig. 3. Surface tension decreases rapidly with increasing concentration and each curve has a level off point at the concentration corresponding to critical micelle concentration CMC, then remains at an almost constant value at higher concentration. Figure 3 shows that the surface activity of the surfactant increased with the increasing of hydrocarbon chain length of the fatty acids. The critical micelle concentration (CMC) and the surface tension at CMC, γ_{cmc} , were showed in Table 4. Generally, increasing the hydrocarbon chain of the surfactant leads to the surfactant molecules being more hydrophobic. As a result, surfactants with longer hydrocarbon chains have a driving force for the aggregation, and thus dramatically reduce the solution CMC. At air/water interface, the minimum surface area per adsorbed FAD molecule, A_{min} was obtained from the excess surface concentration Γ_{max} using the equation:

$$A_{min} = \frac{1}{\Gamma_{max} N_A} \tag{2}$$

where Γ_{max} is the excess surface concentration (mol/cm^2), the excess surface concentration Γ_{max} can be calculated from the Gibbs isotherm, according to the equation below:

$$\Gamma_{max} = -\frac{d\gamma}{RTd(\ln C)} \tag{3}$$

Here γ is the surface tension in N/m , R is gas constant ($8.314 \text{ J}/\text{mol K}$), T is the absolute temperature and C is the molar concentration of surfactant (mol/dm^3). The term $[dC/d(\ln C)]$ is the slope of $\gamma-\ln C$ plot before the CMC. The values of CMC, surface tension at CMC and A_{min} of FAD are listed in Table 4.

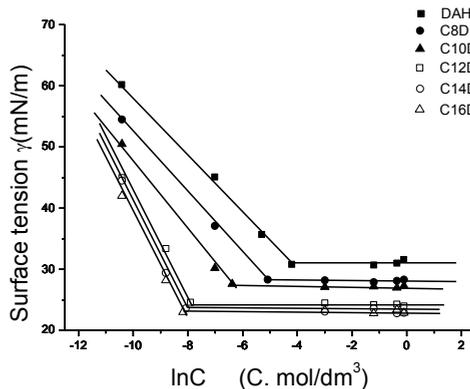


Fig. 3. Surface tension of DAH and FADs solutions in tap water (pH = 5)

From the relation between Γ_{\max} , A_{\min} and FADs, it is evident that the Γ_{\max} in the adsorbed state at liquid/air interface became larger as the length of fatty acid chain increased. It means that more surfactants would adsorb on the liquid/air interface. And the decrease of the A_{\min} indicated that the surfactants molecules adsorbed more closed in the adsorption layer.

Table 4. Surface parameters of DAH and FADs (the values of the CMC, surface tension at CMC, γ_{cmc} , the average area per surfactant molecule, A_{\min} and the excess surface concentration, Γ_{\max})

Reagent	CMC(mol/dm ³)	γ_{cmc} (mN/m)	Γ_{\max} /(mol/cm ²)	A_{\min} /nm ²
DAH	$1.5 \cdot 10^{-2}$	31.2	$1.88 \cdot 10^{-10}$	0.88
C8D	$6.2 \cdot 10^{-3}$	28.1	$1.96 \cdot 10^{-10}$	0.84
C10D	$1.7 \cdot 10^{-3}$	27.3	$2.27 \cdot 10^{-10}$	0.73
C12D	$3.7 \cdot 10^{-4}$	24.1	$3.36 \cdot 10^{-10}$	0.49
C14D	$3.1 \cdot 10^{-4}$	23.7	$3.58 \cdot 10^{-10}$	0.46
C16D	$2.8 \cdot 10^{-4}$	22.9	$3.65 \cdot 10^{-10}$	0.45

Adsorption of DAH and FADs onto coal

The adsorption density of DAH and FADs on clean coals was shown in Fig. 4. As it illustrated, the adsorption density of DAH and FADs on coal increased with the increase of the equilibrium concentration. The shapes of adsorption isotherms of DAH and FADs on coals were similar to each other. At low concentration, the surfactants adsorb as individual molecules through some interactions between the negatively charged sites on the coal surfaces and the positively charged amine cations. With the increasing of the equilibrium concentration near the CMC, the adsorption density increased intensely due to the interactions between the hydrocarbon chains. Meanwhile, the adsorption density of FADs on the coal was higher than that of DAH. It may cause by the interactions between the hydrocarbon chains of DAH and fatty acids.

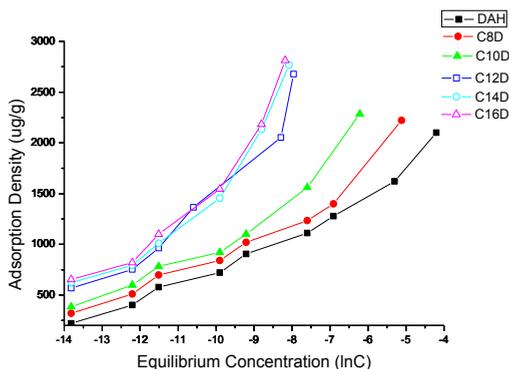


Fig. 4. Adsorption isotherms of DAH and FADs onto coals (pH = 5)

Adsorption of DAH and FADs on bubbles

The adsorption results of DAH and FADs on bubbles are shown in Fig. 5. As it illustrated, the FADs surfactants were much easier to adsorb on the bubbles surface. The adsorption amount of C16D surfactant on bubble surface was almost six times more than that of DAH.

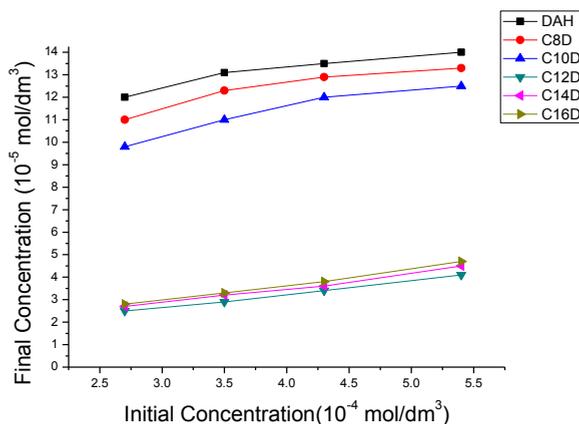


Fig. 5. Adsorption of DAH and FADs surfactants on bubbles (pH = 5)

A probable interaction mechanism of DAH and fatty acid was proposed. Figure 6 shows the schematic representation of the structure of adsorption layer at pH 5. According to the proposed structure, the fatty acid molecules adsorb between the DAH molecules in the adsorption layer and, thus, decrease significantly the average area per surfactant molecule in the adsorption layer. It conformed to the results of surface tension test. Based on the proposed structure of the adsorption layer, we can explain why the FADs showed a stronger collectivity than DAH. The “bubble transfer” hypothesis has been proposed by other researchers (Digre and Sandvik, 1968). They postulated that during a real flotation process, amine molecules are transferred onto particles surfaces by repeated collisions with the amine-coated air bubbles. So air bubbles indeed act as “amine carriers” (Pawlik and Laskowski, 2003a). In FADs solutions, more amine molecules would adsorb onto bubbles surfaces than that in DAH solutions. Thus, it can enhance the adhering of particles onto bubbles surface and prevent the desorbing. As a result, a high froth product yield would be obtained. Zero-condition time concept in reverse flotation was proposed by Laskowski. He pointed that using the DTAB-coated bubbles in coal reverse flotation would lead to a decrease in DTAB consumption and increase the froth product yield. Amine-coated bubbles would be more easily to adhere to the particles in flotation process.

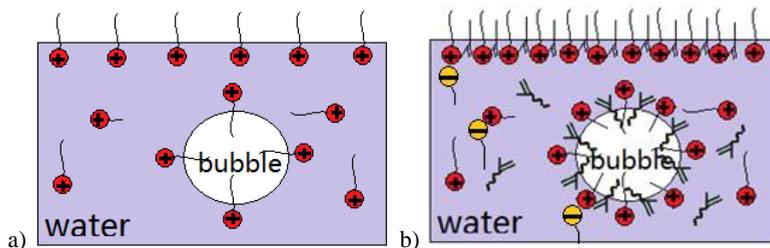


Fig. 6. Schematic presentation of structure of adsorption layer at pH 5
 (a) Bubbles in DAH solution, (b) Bubbles in FADs solutions

Reverse flotation results

The reverse flotation results were shown in Fig. 7. It can be seen in Fig. 7a that the FADs showed stronger collectivity than DAH. Only 28% froth product yield was obtained in the presence of 1.66 kg/Mg DAH. However, when 0.83 kg/Mg C16D surfactant was used, the froth product yield reached 29%. About 50% surfactant was saved under the same froth product yield.

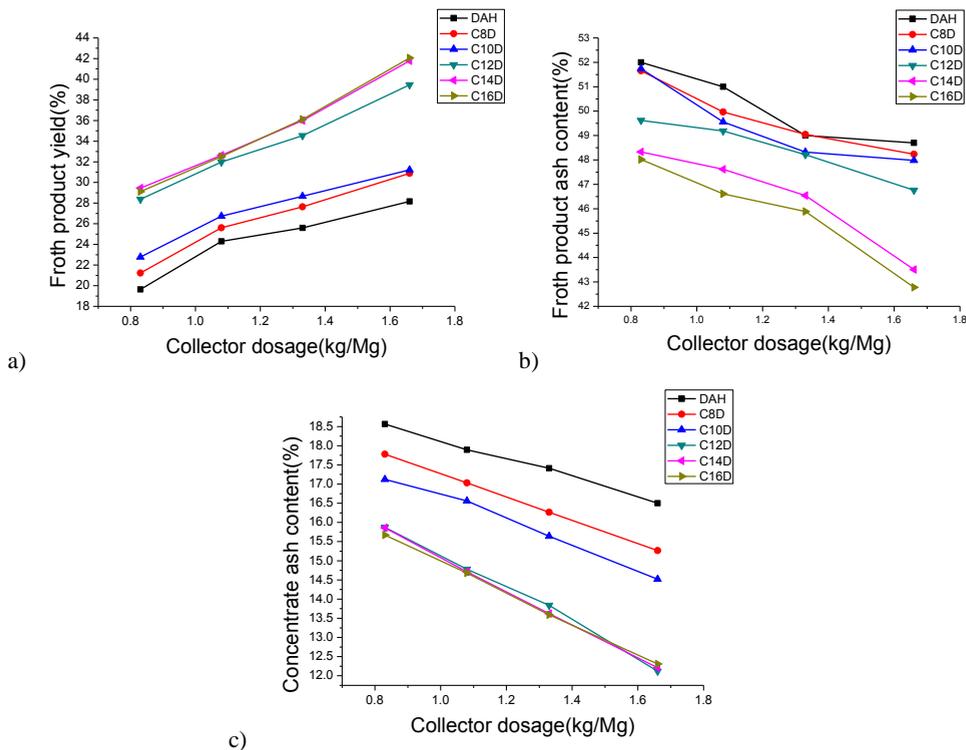


Fig.7. Reverse flotation results in the presence of DAH and FADs (pH = 5)
 (a. Froth product yield b. Froth product ash content. c. Concentrate ash content in the presence of DAH and FADs)

Figure 7b showed that the selectivity of the FADs decreased with the increasing of hydrocarbon chain length of the fatty acids. This was consistent with the adsorption test results in Fig. 4. The adsorption amount of FADs on coals increased with the increasing of hydrocarbon chain length of the fatty acids.

Table 5. Reverse flotation results

Surfactant name	Dosage (kg/Mg)	Froth product yield (%)	Froth product ash content (%)	Concentrate yield (%)	Concentrate ash content (%)	Mineral matter recovery (%)	Combustible recovery (%)
DAH	0.83	20	52.00	80	18.56	41	87
	1.08	24	51.00	76	17.17	49	84
	1.33	26	49.00	74	17.41	49	82
	1.66	28	48.70	72	16.50	54	81
C8D	0.83	21	51.65	79	17.78	44	86
	1.08	26	49.97	74	17.03	50	83
	1.33	28	49.04	72	16.27	54	81
	1.66	31	48.24	69	15.27	59	79
C10D	0.83	23	51.74	77	17.12	47	85
	1.08	27	48.46	73	16.56	52	82
	1.33	29	48.32	71	15.64	55	80
	1.66	31	47.98	69	14.52	60	78
C12D	0.83	28	49.62	72	15.87	55	81
	1.08	32	49.19	68	14.78	61	78
	1.33	35	48.21	65	13.84	65	76
	1.66	39	46.76	61	12.12	72	72
C14D	0.83	29	48.33	71	15.86	56	80
	1.08	33	47.62	67	14.71	61	77
	1.33	36	46.54	64	13.62	66	74
	1.66	42	43.51	58	12.21	72	68
C16D	0.83	29	48.02	71	15.67	56	80
	1.08	33	46.61	67	14.68	60	77
	1.33	36	45.89	64	13.59	66	74
	1.66	42	42.78	58	12.31	72	68

Lower concentrate ash content can be obtained in the presence of FADs. As Fig.7c showed that 12.31% concentrate ash content was obtained when C16D was used comparing with 16.50% of that in the presence of DAH only. The reverse flotation results of DAH and FADs were shown in Table 5. It can be seen that the mineral matter and combustible recoveries in the presence of 1.66 kg/Mg DAH were 54% and

81%, while 55% mineral matter recovery and 81% combustible recovery were obtained in the presence of 0.83 kg/Mg C12D. And the highest mineral matter recovery 72% in these experiments was obtained in the presence of 1.66 kg/Mg C14D. However, taking the both evaluation indicators into consideration, C12D surfactant showed a better performance that high mineral matter recovery near C14D and higher combustible recovery than C14D and C16D. In addition, less weight of C12 would be used under the same molar ratio (DAH : Fatty acid = 1:1) due to its lower molecular weight than C14 and C16.

Conclusion

This study investigated the properties of DAH and fatty acids/DAH mixtures. In addition, their effects on coal reverse flotation were also investigated. Based on the results, the following conclusions can be presented.

1. Surface tension measurements indicated that the fatty acids/DAH mixtures can obtain a much lower CMC and γ_{cmc} than DAH and the surface activity of the FADs increased with the increasing of hydrocarbon chain length of the fatty acids.
2. Adsorption experiments showed that the adsorption density of DAH and FADs on clean coal increased with the increase of the equilibrium concentration and the adsorption density of FADs on the coal was higher than that of DAH.
3. Adsorption of DAH and FADs on bubbles revealed that the FADs surfactants were more easily to adsorb on the bubbles surface. The adsorption amount of C16D surfactant on bubble surface was almost six times more than that of DAH and a probable interaction mechanism of DAH and fatty acid in solution was proposed.
4. Reverse flotation results showed that the FADs surfactants have stronger collectivity than DAH and that C12D showed better performance with a high mineral matter recovery similar to that with C14D as well as higher combustible recovery than with C14D and C16D.
5. For some difficult-to-float coals such as oxidized coal and lignite, forward flotation is not suitable. Many scientists have studied on improving the flotation behavior using both forward flotation and reverse flotation methods (Xia and Yang, 2013a; Zhang and Tang, 2014; Zhang and Liu, 2015). In most of the works it was observed that the trend of reagent consumption is towards the higher side (Xia and Yang, 2013a; Zhang and Liu, 2015; Ding and Laskowski 2006a). This paper proposed a new way to reduce about 50% collector consumption in steam coal reverse flotation and make reverse flotation to be an alternative way for upgrading difficult-to-float coal.
6. Kaolinite and quartz are usually treated as gangue minerals in many other mineral flotation processes. Therefore, the mixture of DAH/fatty acids used in this study may also be used in the reverse flotation process of other minerals such as iron ores and bauxite.

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