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LIGNITE CLEANING IN NaCl SOLUTIONS BY THE REVERSE FLOTATION TECHNIQUE

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Abstract: A cleaning possibility of low (BD) and high ash content (IM) lignites in NaCl solutions using a reverse flotation technique was studied. Preliminary test results indicated that BD lignite cannot be cleaned effectively in the traditional manner because gangue was floated first rather than a combustible material. Further studies indicated that the lignite cleaning in NaCl solutions using the reverse flotation technique is possible. However, a large quantity of dodecyl amine hydrochloride (DAH) was required. A lower ash content in a concentrate can be obtained by addition of NaCl. A concentrate yield can be improved by addition of starch. Under the test conditions, for BD lignite with the ash content in the feed 15.19%, the ash content in the concentrate 11.44% and the concentrate yield 67.38% were obtained. Similarly, for IM lignite with the ash content in the feed 57.40%, the ash content in the concentrate 32.90% and the concentrate yield 25.08% were obtained. The flotation rate constant k and maximum ash recovery ε_{∞} significantly increased with the NaCl concentration.

Keywords: lignite, reverse flotation, NaCl, kinetics, coal preparation

Introduction

Flotation, an important and versatile mineral processing technique, is a selective separation process for which the separation efficiency depends primarily on differences in surface properties of various mineral particles (Anderson, 1916; Taggart, 1920; Subrahmanyam and Forssberg, 1988; Nguyen et al., 1998). Lower rank coals (e.g. lignite) are more hydrophilic, and therefore difficult to float in the traditional manner, even using a high reagent dosage. It is due to the presence of higher oxygen functional groups, such as hydroxyl, carbonyl and carboxyl (Arnold and Aplan, 1989; Atesok and Celik, 2000; Jia et al., 2000; Cebeci, 2002; Zhang and Tang, 2014).

In contrast to traditional flotation, previous studies have reported that a reverse flotation technique can be used for coal separation, whereby gangue moves to the flotation froth and a combustible material does not (Stonestreet and Franzidis, 1988, 1989, 1992; Pawlik and Laskowski, 2003). In many studies of coal reverse flotation, cationic amine surfactants were used to float gangue since these surfactants act as ash collectors, coal depressants and frothers (Dey, 2012). Ding and Laskowski (2006) investigated reverse flotation of subbituminous coal. In their study, the ash content decreased from 34.6 to 16.7% for a concentrate yield of 50.4%. In addition, Patil and Laskowski (2008) for bituminous coal using reverse flotation obtained the concentrated ash content of 16.5% and 55% concentrate yield for a feed ash content of 34.7%. In this case, the reverse flotation technique is most likely better suited for lignite flotation because of its strongly hydrophilic character compared with a higher rank coal.

There is a correlation between the flotation recovery, kinetics, bubble size distribution and stability profile of froth with addition of salt solutions (Marrucci and Nicodemo, 1967; Weissenborn and Pugh, 1996; Pugh et al., 1997; Harvey et al., 2002; Marcelja, 2006). Many studies have demonstrated that electrolyte ions can enhance the flotation performance (Klassen and Mokrousov, 1963; Laskowski, 1965; Yoon, 1982; Hampton and Nguyen, 2009; Ozdemir et al., 2009, 2013). Paulson and Pugh (1996) found that the ionic strength is an important factor in scaling the overall recovery of graphite, which was linked to the dissolved gas concentration gradients in the electrolyte solutions. Bournival et al. (2012) investigated the stability of bubbles through measurements of coalescence time between bubble at the tip of capillary using high-speed video imaging. They demonstrated that the bubble became more stable as the salt concentration increased in the solution. The results also suggested that a relatively larger concentration of salt was required to achieve stability in the froth zone.

In this study, to evaluate the possibility of lignite cleaning in NaCl solutions using the reverse flotation technique, two lignites (BD with low ash content and IM with high ash content) were selected as raw samples. The effect of NaCl, dodecyl amine hydrochloride (DAH) and starch on the reverse flotation performance was investigated. In addition, the flotation performance, as a function of NaCl, was estimated by analysing the flotation kinetics.

Experimental

Two lignite samples were used as flotation feed: BD lignite with low ash content (15.19%) and IM lignite with high ash content (57.40%) on a dry basis. The BD and IM lignite samples were obtained from Alberta (Canada) and Inner Mongolia (China), respectively. The as-received samples were crushed to -2 mm. Next, the crushed samples were ground to -425 μm using a ball mill (ADVAN-TEC, No. 5C). The analysis and particle size distribution of lignite samples on a dry basis are presented in Tables 1 and 2.

Dodecyl amine hydrochloride (DAH, $C_{12}H_{28}ClN$, $\geq 99\%$) was used as a surfactant in preliminary tests (traditional flotation tests). DAH which is known for its beneficial role in flotation of low-rank coal at a small dosage (Kelebek et al., 2008). DAH in large dosages is also used as a collector for gangue in the reverse flotation tests (Stonestreet and Franzidis, 1988; Pawlik and Laskowski, 2003). In this paper kerosene was used as a collector, corn starch ($(C_3H_{10}O_5)_n$, MW=162.15 g/mol, laboratory grade) as a coal depressant, 4-methyl-2-pentanol (MIBC, laboratory grade) as a frother and sodium chloride (NaCl, laboratory grade) was used as a soluble salt. To increase the solubility of corn starch, 1% causticised corn starch solution was prepared (corn starch-to-NaOH = 4:1, wt.%). All experiments were conducted in distilled water.

The reverse flotation tests were performed in a 1 dm³ Denver flotation cell. All tests were performed at room temperature. For each test, 50 g of lignite sample on a dry basis was first mixed with 500 cm³ desired salt solution in the cell, and then agitated for 5 min at an impeller rotation speed of 1600 rpm. Next, starch, DAH and MIBC were added to a slurry. The condition time for starch, DAH, and MIBC was 3, 2, and 1 min, respectively. After conditioning, the desired salt solution was added again to increase the volume of slurry in the cell to 1 dm³, and then, the slurry was stirred for additional 4 min. Subsequently, air was introduced into the cell at a flow rate of 1.35 dm³/min. Either distilled water or desired salt solution was added during the tests to maintain a constant pulp level, and then, the slurry was floated for 20 min. The froth was collected after 2, 6, 12 and 20 min. Finally, the flotation concentrate (combustible material which did not report to the flotation froth) and tailings (gangue which reported to the flotation froth) were filtered, dried, weighed, and then the ash content was determined by applying the ASTM procedure. The ash and combustible recoveries were calculated using equations:

$$\text{Ash recovery (\%)} = [M_T A_T / M_F (100 - A_F)] \times 100 \quad (1)$$

$$\text{Combustible Recovery (\%)} = [M_C (100 - A_C) / M_F (100 - A_F)] \times 100 \quad (2)$$

where M_C , M_T , M_F are concentrate, tailings feed masses (%), respectively, A_C , A_T , A_F are ash contents in concentrate, tailings and feed (%), respectively. The ash contents in the concentrate and tailing were on a dry basis.

The cumulative ash recoveries after 2, 6, 12 and 20 min of flotation time were fitted to the first-order rate equation:

$$\varepsilon = \varepsilon_{\infty} [1 - \exp(-kt)], \quad (3)$$

where ε is cumulative ash recovery at time t , ε_{∞} the maximum ash recovery and k is the first-order rate constant. The non-linear regression software 1stOpt (<http://www.7d-soft.com>) was used to simulate the flotation rate constant (k), maximum ash recovery (ε_{∞}) and correlation coefficient (R^2).

Table 1. Analysis of -2 mm lignite samples on dry basis

Samples	Inherent moisture (wt.%)		Ash (wt.%)		Volatile matter (wt.%)		Fixed carbon (wt.%)	
	As received	Dry basis	As received	Dry basis	As received	Dry basis	As received	Dry basis
BD lignite	22.46	—	11.78	15.19	45.86	59.14	19.90	25.67
IM lignite	20.16	—	45.83	57.40	19.48	24.40	14.53	18.20

Table 2. Size and ash content of -425 μm lignite samples on dry basis

Size fraction (μm)	BD lignite				IM lignite			
	Weight (%)	Ash (%)	Cumulative weight (%) finer than size	Cumulative ash (%) finer than size	Weight (%)	Ash (%)	Cumulative weight (%) finer than size	Cumulative ash (%) finer than size
-425+250	16.15	11.49	100.00	15.19	22.7	48.64	100	57.40
-250+150	21.58	11.94	83.85	15.90	21.85	55.27	77.3	59.97
-150+75	21.80	12.80	62.27	17.27	23.86	59.05	55.45	61.82
-75+45	11.91	14.55	40.47	19.68	15.11	62.00	31.59	63.92
-45	28.56	21.82	28.56	21.82	16.48	65.68	16.48	65.68
Total	100.00	15.19			100	57.40		

Results and discussion

Lignite characterisation

The particle size and ash distribution BD and IM lignite samples presented in Table 2 indicate that the ash content of both lignite samples increased as the particle size decreased. The yield of -45 μm size fraction is 28.56%, with ash content of 21.82% for the BD lignite sample, whereas for the IM lignite sample the yield of the -45- μm size fraction is 16.48%, with ash content of 65.68%. The significant increase in the ash content for fine particles (-45 μm) can be explained by a fragile characteristics of lignite, which causes a decrease in the selectivity in froth flotation. In addition, fine lignite particles are easy to slime during the flotation process. Therefore, separation of fine particles is crucial to enhance the flotation performance of lignite samples.

To understand the hydrophilic properties of lignite samples, Fourier transform infrared spectroscopy FTIR (ABB MB3000) was used to analyse the oxygen functional groups. A high rank coal with ash content of 10.89% was selected as a reference sample. For all tests, three samples were ground to -45 μm using a ball mill. The spectra were collected in the range of 500 to 4,000 cm^{-1} at a spectral resolution of 2 cm^{-1} . The FTIR spectra of three samples are presented in Fig. 1. It can be seen that the oxygen

functional groups were abundant in the IM lignite sample, whereas they were scarcely present in high rank coal. The increase in the oxygen functional groups implied the increase in hydrophilicity. The FTIR analysis indicated that BD and IM lignite samples were more hydrophilic than high rank coal.

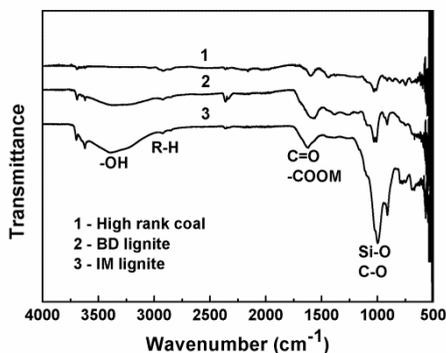


Fig. 1. FTIR spectra of high rank coal (1), BD (2) and IM (3) lignite samples

Preliminary tests

A sample of BD lignite was selected for preliminary tests in the traditional manner. DAH was used as surfactant, kerosene as collector, MIBC as frother, and NaCl as soluble salt. The test conditions were the same as previously described. The preliminary tests were first performed in NaCl solutions without any flotation reagents. Table 3 shows the effect of NaCl concentration on the BD lignite flotation performance. From Table 3 it can be seen that the ash content in the froth products was higher than for the feed and decreased with flotation time, that indicates on the reverse flotation process.

To improve floatability of BD lignite, DAH, which is the cationic amine surfactant, was added together with kerosene and MIBC. Tables 4 and 5 show the effect of DAH and kerosene on the BD lignite flotation performance, respectively. It can be seen that a similar flotation response was reported when compared with that of NaCl solutions, even in the absence of DAH. DAH improved the reverse flotation performance compared with kerosene, and the ash content decreased with increase in the DAH dosage. In the preliminary tests, the ash of BD lignite decreased from 15.19 to 13.71% for the concentrate yield of 54.05% after 20 min of flotation. The preliminary test results suggest that BD lignite cannot be cleaned effectively in the traditional manner and that gangue was floated first rather than the combustible material. Based on the preliminary tests, batch reverse flotation tests of BD and IM lignite were performed in a subsequent experiment to further evaluate the possibility of lignite cleaning in the NaCl solutions by the reverse flotation technique.

Table 3. Flotation results for BD lignite as a function of NaCl without flotation reagents

NaCl concentration	0.1 M		0.3 M		0.5 M		1.0 M		2.0 M	
	Yield (%)	Ash (%)								
Flotation time (min)										
2	5.97	19.71	10.26	18.54	8.94	18.18	9.07	18.50	10.71	18.81
6	7.01	18.22	11.47	14.45	12.66	15.95	14.40	16.46	17.22	14.90
12	9.31	17.96	13.43	16.23	11.32	13.26	13.13	13.97	15.49	15.73
20	9.90	16.67	9.85	13.91	11.41	13.77	10.61	13.00	13.55	14.46
Sink	67.81	13.88	54.99	14.69	55.67	15.22	52.78	15.02	43.03	14.44
Total	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19

Table 4. Flotation results for BD lignite as a function of DAH (kerosene 3000 g/Mg, MIBC 800 g/Mg)

DAH dosage	0 g/Mg		100 g/Mg		300 g/Mg		500 g/Mg		700 g/Mg	
	Yield (%)	Ash (%)								
Flotation time (min)										
2	32.50	15.90	30.72	16.44	22.56	17.41	27.43	17.49	29.21	17.84
6	9.38	16.40	7.87	16.51	6.81	17.39	7.38	16.15	6.82	16.20
12	4.28	15.78	4.03	15.26	4.97	15.75	3.51	15.34	3.71	14.91
20	4.33	15.31	5.35	14.76	3.15	15.50	6.04	14.91	6.21	14.68
Sink	49.51	14.43	52.03	14.29	62.51	14.09	55.64	13.95	54.05	13.71
Total	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19

Table 5. Flotation results for BD lignite as a function of kerosene (DAH 300 g/Mg, MIBC 800 g/Mg)

Kerosene dosage	1000 g/Mg		2000 g/Mg		3000 g/Mg		4000 g/Mg		5000 g/Mg	
	Yield (%)	Ash (%)								
Flotation time (min)										
2	18.73	18.84	20.11	17.99	22.56	17.41	23.63	17.29	32.37	16.60
6	10.61	16.77	9.28	16.74	6.81	17.39	8.58	16.66	7.33	16.21
12	4.56	15.64	5.68	15.55	4.97	15.75	4.73	15.22	4.57	15.60
20	3.65	15.32	2.07	14.88	3.15	15.50	4.13	15.23	3.29	15.21
Sink	62.44	13.79	62.85	14.04	62.51	14.09	58.93	14.13	52.43	14.14
Total	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19	100.00	15.19

Batch reverse flotation tests

The previous studies indicated that inorganic electrolytes can generate abundant smaller bubbles and that the charge of gas bubbles in the aqueous solution can be re-

duced and even reversed by the addition of multivalent inorganic salts (Li and Somasundaran, 1991; Ozdemir et al., 2009). In this case, inorganic electrolytes may improve floatability of fine particles with a high ash content in the reverse flotation process. The effect of NaCl on lignite reverse flotation was tested in the presence of DAH, starch and MIBC. The results for BD and IM lignite samples are shown in Figs. 2 and 3, respectively. Figures 2 and 3 show that the ash content in concentrate decreased with addition of NaCl for both lignite samples. The concentration of NaCl had only a little effect on the yield of the products of BD lignite, while for IM lignite, the concentrate yield decreased with addition of NaCl. However, it is difficult to explain the effect of NaCl concentration on the product yield and ash contents because of the presence of other chemical additives in the process. This difficulty is attributed to the possibility that the flotation behaviour of either gangue or combustible material frequently varies in the presence of NaCl and DAH during the flotation process. Yoon and Yordan, (1991) investigated the bubble-particle attachment time for the quartz-amine flotation system as a function of KCl concentration at pH 6.75. The induction time was found to decrease with increase of KCl concentration at low DAH concentration ($5 \cdot 10^{-6}$ M). In contrast, the induction time was found to increase with increase in KCl concentration at higher DAH concentration ($5 \cdot 10^{-4}$ M), particularly when KCl concentration was higher than 10^{-1} M.

The mechanism of action in NaCl solutions using the reverse flotation technique requires further investigation, particularly the interactions in this system, which were implemented with the simultaneous addition of NaCl, DAH, starch and MIBC. Under the test conditions, although for IM lignite the concentrate yield decreased by addition of NaCl, lower ash content is obtained, which can be substantially reduced from 57.40 to 28.82% by adding 0.4 M NaCl.

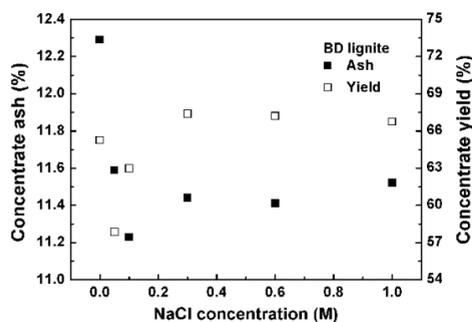


Fig. 2. Effect of NaCl on reverse flotation of BD lignite (DAH 5000 g/Mg, starch 2500 g/Mg, MIBC 200 g/Mg)

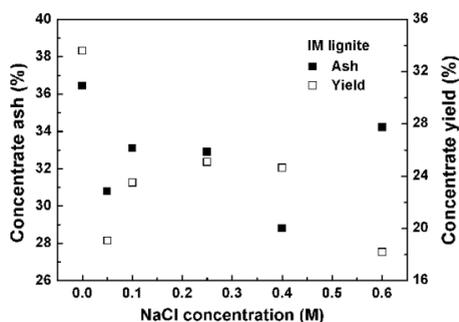


Fig. 3. Effect of NaCl on reverse flotation of IM lignite (DAH 5000 g/Mg, starch 3000 g/Mg, MIBC 200 g/Mg)

The excellent ash-collecting ability of quaternary amines in coal reverse flotation has been reported by Stonestreet and Franzidis (1988, 1989, 1992). The effect of DAH on reverse flotation of lignite is shown in Figs. 4 and 5. Figures 4–5 show that for BD

and IM lignite samples, both concentrate yield and ash content decreased with increasing DAH dosages. The ash content in the concentrate for BD lignite decreased slightly from 12.56 to 11.26% while for IM lignite the ash content decreased from 42.45 to 27.68%, when the DAH dosage increased from 2000 to 6000 g/Mg. The results clearly indicate that BD lignite was difficult to clean.

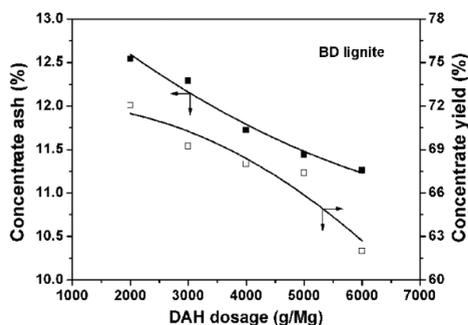


Fig. 4. Effect of DAH on reverse flotation of BD lignite (NaCl 0.3 M, starch 2500 g/Mg, MIBC 200 g/Mg)

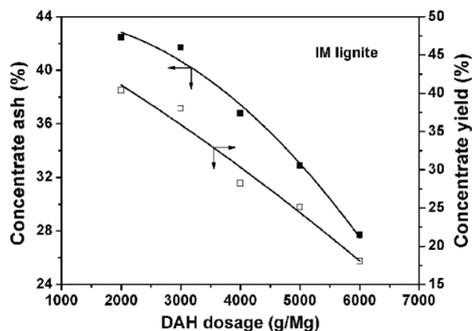


Fig. 5. Effect of DAH on reverse flotation of IM lignite (NaCl 0.25 M, starch 3000 g/Mg, MIBC 200 g/Mg)

The ash content in the concentrate decreased with DAH concentration. It result was observed by Ding and Laskowski (2006) and Kelebek (2008) for coal reverse flotation. It could be explained by the effect of adsorption density of amine on coal and gangue. Pawlik and Laskowski (2003) reported that the adsorption density of amine on hydrophilic-oxidised and subbituminous coals was much higher than on bituminous coal and silica. Yoon and Yordan (1991), who studied the induction time and the flotation recovery of quartz particles at pH 6.6, found that the induction time first decreased and then increased with the increase in DAH concentration, whereas the flotation recovery first decreased and then increased concomitantly. The shortest induction time and the highest flotation recovery were obtained at approximately 10^{-3} M DAH concentration, which was largely consistent with the investigated range of DAH dosage (which corresponds to $4.5 \cdot 10^{-4}$ M – $1.4 \cdot 10^{-3}$ M) in this study. The results supported the hypothesis that cationic amine surfactants can be used as collectors for ash and as depressants for coal (Dey, 2012).

The effect of starch on reverse flotation of lignite is shown in Figs. 6 and 7. It can be seen that the increase in starch dosage increased the concentrate yield and ash content for BD and IM lignite. The results indicate that addition of starch cannot improve the selectivity in this process, however improves the concentrate yield.

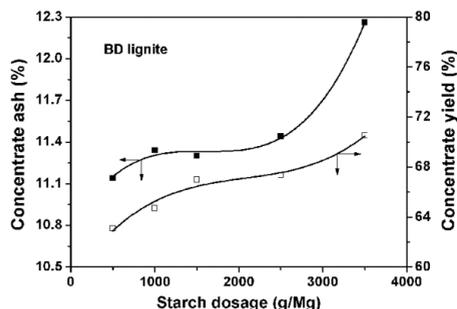


Fig. 6. Effect of starch on reverse flotation of BD lignite (NaCl 0.3 M, DAH 5000 g/Mg, MIBC 200 g/Mg)

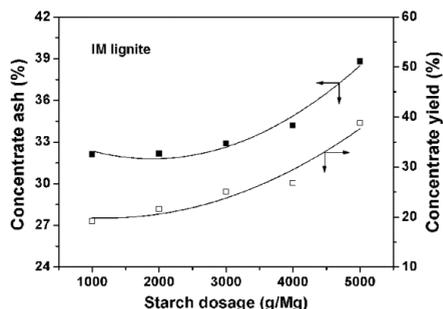


Fig. 7. Effect of starch on reverse flotation of IM lignite (NaCl 0.25 M, DAH 5000 g/Mg, MIBC 200 g/Mg)

Kinetics of lignite reverse flotation as a function of NaCl solutions

The results clearly indicate that cleaning of BD and IM lignite samples is possible by using the reverse flotation technique with the ambient pH value in NaCl solutions. For BD lignite with the ash content in the feed of 15.19%, the ash content in the concentrate 11.44% and the concentrate yield 67.38% were obtained, whereas for IM lignite with the ash content in the feed of 57.40% the ash content in the concentrate and yield were 32.90 and 25.08%, respectively.

Sodium chloride influences the kinetics of flotation (Table 6). It is evident that the flotation rate constant k and maximum ash recovery ε_{∞} significantly increased with NaCl concentration. The flotation rate constant k for BD lignite increased from 0.15 min^{-1} (in distilled water) to 0.62 min^{-1} in 0.01 M NaCl solution.

Table 6. Flotation kinetic parameters for BD and IM lignite as a function of NaCl

NaCl Concentration	BD lignite			NaCl Concentration	IM lignite		
	K (min^{-1})	ε_{∞} (%)	R^2		K (min^{-1})	ε_{∞} (%)	R^2
0.00 M	0.15	48.43	0.9943	0.00 M	0.30	77.60	0.9992
0.05 M	0.51	51.85	0.9788	0.05 M	0.31	90.80	0.9970
0.10 M	0.62	51.74	0.9959	0.10 M	0.55	83.41	0.9920
0.30 M	0.35	47.70	0.9950	0.25 M	0.54	83.19	0.9978
0.60 M	0.42	48.24	0.9962	0.40 M	0.63	90.18	0.9984
1.00 M	0.50	47.36	0.9934	0.60 M	0.78	87.58	0.9988

For IM lignite the maximum ash recovery ε_{∞} increased from 77.60% (in distilled water) to 90.80% in 0.05 M NaCl solution. It can be explained by differences in the bubble size and bubble coalescence. As shown in Fig. 8, abundant smaller bubbles

were generated as the NaCl concentration increased. This phenomenon was consistent with the findings of Craig et al. (1993), who showed that the bubble coalescence was inhibited in certain salt solutions and that a number of smaller bubbles was generated. In addition, Ozdemir et al. (2009) reported that the flotation efficiency of fine particles increased in bore water because of the high number of bubbles in the flotation system.

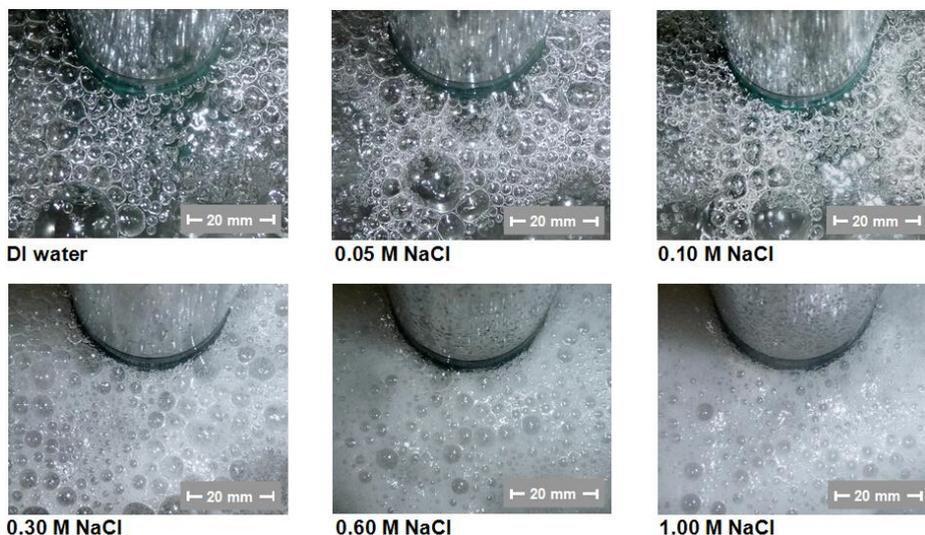


Fig. 8. Bubbles in distilled water (DI) and NaCl solutions (1 dm³ Denver cell, impeller speed of 1600 rpm, air flow rate of 1.35 dm³/min)

Conclusions

This study investigated the flotation performance of low (BD) high (IM) ash content lignite samples in NaCl solutions using the reverse flotation technique. Based on the results, the following conclusions can be presented.

1. FTIR spectra indicated that BD and IM lignite samples were more hydrophilic than high rank coal. A sample of BD lignite cannot be cleaned effectively in the traditional manner since gangue was floated first rather than the combustible material.
2. Cleaning of lignite in NaCl solutions using the reverse flotation technique is possible. A high concentration of dodecyl amine hydrochloride (DAH) was required to float gangue from lignite by reverse flotation. Lower ash content in the concentrate can be obtained by addition of NaCl. The concentrate yield can be improved by addition of starch. Under the ambient pH value in NaCl solutions in the reverse flotation technique, for BD lignite with the ash content in the feed of 15.19%, the ash content in the concentrate 11.44% and the concentrate yield 67.38% were obtained, whereas for IM lignite with the ash content in the feed of 57.40% the ash content in the concentrate and yield were 32.90 and 25.08%, respectively.

3. The flotation rate constant k and maximum ash recovery ε_{∞} significantly increased with the increase in the NaCl concentration primarily because of abundant smaller bubbles generated by presence of NaCl.

Acknowledgments

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