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Efficient coal concentration using a short-chain amine-type compound as collector reagent: Flotation and optimization studies

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This work presents the flotation of a sub-bituminous coal using 1-butylamine as a short-chain amine collector. The flotation results have been obtained with 1-butylamine (coal yield: 100%) and compared with those achieved with a quaternary amine (coal yield: 95.4%), kerosene (coal yield: 100%) and diesel (coal yield: 95.7%). The best results have been obtained with 1-butylamine. Less depressant effect has been observed with 1-butylamine. The FTIR signals have been attenuated when the coal is conditioned with 1-butylamine. Zeta potential measurements have also been changed after conditioning. The contact angle of water and graphite has decreased from 82.3° to 32.2°. An optimization has been performed using a Box-Behnken experimental design. Flotation has significantly affected by time and collector dosage. The *pH* is not a significant factor. The optimal conditions for the best efficiency using 1-butylamine as a collector are 2.0 min of flotation and 10^{-3} mol/L of collector.

Keywords: Box-Behnken design, Butylamine, Collector, Flotation, Sub-bituminous coal

Froth flotation is the most widely used technological separation and concentration method for hydrophobic minerals and mining materials such as coal. Amines are the traditional ionic collectors for the low-rank/oxidized coal flotation, especially for sub-bituminous ones. This is due to the amphiphilic molecular structure that facilitates interaction with the hydrophilic sections of the oxidized coal. The positive charge of the protonated amine groups allows adsorption to the negative surface of the coal. Kelebek *et al.*[1] reported 98.22% recovery of a sub-bituminous coal using a mixture of dodecylamine and kerosene as collector. Amines were found to improve the recovery of this type of coal. Sarikaya & Obzbayoglu[2] reported about 70% recovery of a highly oxidized coal using amines as collector reagents. Hydrophobic collectors have also been used for this kind of coal, but, in general, a high dosage is needed and the selectivity is not achieved[1,3-5].

However, only long-chain amines have been used for coal flotation, and depressant effect has been commonly observed in this kind of molecules because hydrophobic interactions lead to the adsorption of the amine on the non-oxidized sections of the coal surface. This way, the hydrophilic NH_3^+ groups are exposed to the aqueous medium; making the coal particle hydrophilic[6]. This is why amines are not universal collectors for low-rank/oxidized coals: the effect of the reagent is largely conditioned by the degree of surface oxidation; and this makes the use of collectors for low-rank/oxidized coals somewhat experimental[2]. This leads to the possibility of using short-chain amines which could minimize the depressant effect, but there are no reports on studies of short-chain amines as a coal collector.

In the present work, the performance of 1-butylamine as a short-chain amine collector on the flotation of a sub-bituminous coal is investigated. The effect of physicochemical conditions on gangue and coal recoveries, as well as on selectivity, was evaluated by means of a response surface experimental methodology. From it, optimal flotation conditions were stabilized.

The novelty of this paper lies in the presentation of experimental evidence on the coal yield in the flotation of a sub-bituminous coal using 1-butylamine as a short chain amine-type collector. The effect of physicochemical variables on the process and the optimal conditions using this compound are reported here for the first time, being a proposal of efficient and inexpensive reagent for sub-bituminous coal processing.

Experimental Section

Experimental coal and reagents

The sample used in the present work for the flotation experiments was a sub-bituminous coal (SBC) from Mexico. All the reagents used in this work were purchased from Sigma Aldrich in ACS grade. Ultrapure deionized water was used.

Physicochemical characterization of coal

Identification and semi-quantification of the mineral phases were carried out by the X-ray diffraction method (XRD) employing a Bruker instrument. The analysis conditions were: 30 kV, diffraction range from 4º to 90º, and Cu-Kα radiation (λ = 1.5406 Å). Coal size fraction of -37 µm was analyzed.

The *pH* measurements were performed using a Thermo scientific potentiometer with a combined glass *pH* electrode. To determine the *pH* of the coal, 11.5 g of sample were mixed with 25 mL of water under constant shaking for 5 min. After 2 h of sample rest, measurements were made in the remaining solution[7].

For the total organic carbon (TOC) quantification, a sample of coal was first weighed in a crucible and then incinerated at 1100°C for 4 h. Gravimetric calculations were performed to determine mass loss and TOC[7].

The acidic and basic surface groups were quantified by volumetry. For the acidic groups, coal sample was treated with NaOH 0.1 mol/L at a ratio 1:100 g/mL under constant shaken for 24 h. After filtration, titration of the solution was performed using HCl 0.1 mol/L. For the basic groups, the same procedure was followed, but in this case, the coal treatment was performed with HCl 0.1 mol/L, and the filtered solution was titrated with NaOH 0.1 mol/L[8].

Microflotations

Microflotation experiments were performed using 1-butylamine as a collector for the SBC. The efficiency of SBC flotation using the proposed collector was compared with that achieved using the hydrophobics: kerosene and diesel; and the long-chain amine benzyl dimethyl hexadecyl ammonium chloride (BDHAC). A modified Hallimond cell was used for the microflotations. For this, 0.5 g of coal were introduced into the cell and pre-wetted during 3 min. Then, 20 mL of the collector were added, and conditioning was performed at 800 rpm for 3 min. The glass tube was filled with collector and the microflotation tests were carried out for 2 min at 400 rpm using a N_2 gas flow of 159 mL/min. The concentrate was dried and weighed[9]. For kerosene and diesel, the collector dosage was 100 µL and the tube was filled with water. The non-optimized amine dosage was 10^{-3} mol/L.

Total Mineral Recovery (RM) and Coal Recovery (RC) were evaluated; they are shown in the Eq. (1) and (2), respectively. RM describes the amount of concentrate relative to the feed, whereas RC describes the coal grade in the concentrate relative to the feed.

$$RM\ (\%) = \frac{m_{concentrate}}{m_{feed}} \cdot 100 \quad \dots (1)$$

$$RC\ (\%) = \frac{(TOC \cdot m)_{concentrate}}{(TOC \cdot m)_{feed}} \cdot 100 \quad \dots (2)$$

where m, is the mass (g) and TOC is the total organic carbon (%).

The concentration index (CI) shown in the Eq. (3) was calculated to indicate the concentration efficiency of the reagents. As CI increases, the reagent evaluated is better for the flotation.

$$CI\ (\%) = RC - RM \quad \dots (3)$$

Depressant effect on graphite flotation

The depressant effect of 1-butylamine on the flotation of high purity graphite (99.99%) was studied with respect to the natural flotation and BDHAC. The ionic collectors were dosed as described for microflotation tests.

Coal surface modification and hydrophobic interactions

Themodification of the coal surface by the collector was studied by Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (FTIR-ATR) and zeta potential measurements. A Thermo Scientific FTIR-ATR instrument with spectral scan of 600-4000 cm^{-1} and resolution of 0.4 cm^{-1} was used. Spectra were recorded before and after the conditioning for coal flotation. Samples were dried at 70°C to remove moisture.

A Zetasizer Nano ZS90 was used for zeta potential measurements before and after the coal being conditioned with the amine. The coal was suspended in water by stirring and then the large particles were decanted. The fine particles were sampled for zeta potential measurements after 5 min of adjusting the *pH* values from 2 to 13.

The ability of the collector to modify hydrophobics interactions was corroborated by contact angle measurements on a graphite surface as a hydrophobic model. A Ramé-Hart instrument was used for software-controlled photography and angle measurements by the sessile drop method.

Response surface methodology for study of physicochemical conditions and coal yield optimization

A Box-Behnken experimental design was used to study the physicochemical conditions and to optimize flotation[10,11]. Seventeen experiments with five central points were carried out. Collector dosage, *pH*, and flotation time were the experimental factors; they were evaluated at three levels. The response variables were RM and RC. The acceptance criterion for statistical significance of a factor on the response variable was defined as p-value < 0.05. A lack of fit test with p-value > 0.05 was considered to check an appropriate mathematical model. Microsoft Office Excel and Origin Pro 9.0 were used for data processing, and Statgraphics Centurion 19 was used for statistical analysis.

Results and Discussion

Physicochemical characterization of coal

Figure 1 shows the diffractogram corresponding to the experimental coal. As can be seen, graphite and other mineral phases such as quartz, muscovite, margarite, albite, kaolinite, akermanite, and orthoclase are identified. These non-carbonaceous phases could have an important contribution to the hydrophilic gangue due to the permanent structural charge[12].

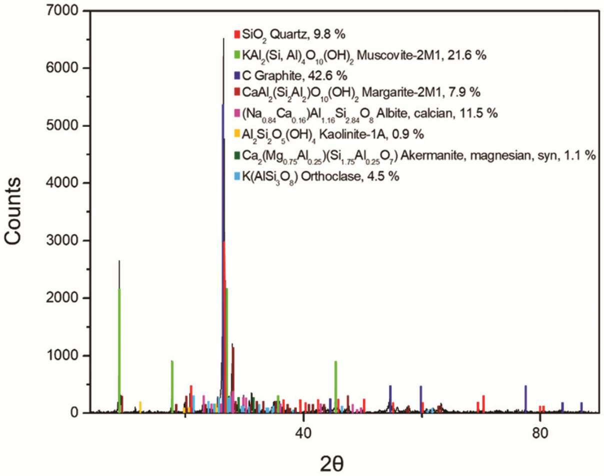

Fig. 1 — SBC X-ray diffraction pattern.

Table 1 shows the analytical physicochemical parameters quantified for SBC. The *pH* of the coal is practically neutral, which indicates that soluble compounds such as carbonates, do not contribute to this parameter. A low moisture content was quantified. TOC is similar to the semi-quantitative value reported by XRD for graphite (Fig. 1), indicating a high degree of coal mineralization. All calculations for the microflotation tests were made considering this TOC value. The predominance of basic organic groups in the sample may be associated with carbonyl groups and oxygen ether-type atoms in the coal[13,14].

Microflotations

Figure 2 presents the results of the coal flotation without collector reagent, also known as natural flotation (SBC-NF), and the results using 1-butylamine (SBC-Butylamine), benzyl dimethyl hexadecyl ammonium chloride (SBS-BDHAC), kerosene (SBC-Kerosene) and diesel (SBS-Diesel). As can be observed, the highest RC value corresponds to 1-butylamine and kerosene; both reached the coal yield of 100%. However, the concentration index (CI) is significantly higher for amine (14.9%), which indicates that this collector allows recovering all the coal content with less amount of gangue in the

Table 1 — SBC analytical physico-chemical parameters.

Experimental parameter	Result ± SD
pH	7.0±0.02
Moisture (%)	0.58±0.02
TOC (%)	46.2±0.4
SAG (mEq/g)	0.48±0.1
SBG (mEq/g)	1.9±0.6

TOC: Total Organic Carbon, SAG: Surface Acidic Groups, SBG: Surface Basic Groups, SD: Standard Deviation

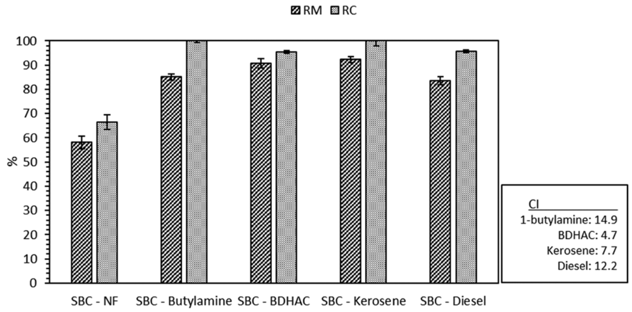

Fig. 2 — SBC flotation results: no reagent (NF), kerosene, diesel and benzyl dimethyl hexadecyl ammonium chloride (BDHAC) as traditional collectors, as well as the proposed collector 1-butylamine.

concentrate than kerosene (7.7%). Diesel has a CI value (12.2%) similar to the one obtained for 1-butylamine, but the hydrophobic collector did not float all the coal in SBC; its RC value is 95.7%. BDHAC is a long-chain quaternary amine, note that this compound was not more efficient than the short-chain amine. RC of the quaternary amine is 95.4% and RM is 90.7%, leading to a CI of 4.7%, a value significantly lower than that obtained for 1-butylamine.

All these results show that, for this experimental SBC, the successful collector is the short-chain amine, rather than the hydrophobics and the long-chain amine. The reason why there are no efficient universal collectors for coal flotation lies in the complex surface composition, which is conditioned by the type of coal and the degrees of oxidation and graphitization. Mainly, the oxidation degree and the surface groups strongly depend on the geological origin, the environmental and mineralogical characteristics of the rock. This makes the surface composition very heterogeneous, even for coals of the same rank. For this reason, it is not ruled out those chemical substances outside the traditional flotation schemes can be more efficient and selective, as in the case presented here.

To study the depressant effect, amines were evaluated with respect to the natural flotation of high purity graphite (Fig. 3). Due to the hydrophobic property of graphite, recovery of 96.3% was achieved when no reagents were used. A lower recovery is observed when the amines are used (1-butylamine:95.5%, BDHAC: 87.6%), this indicates that the amphiphilic molecular structure of the collector makes the graphite surface hydrophilic. However, the depressant effect is accentuated in the case of the long-chain amine, while the recovery using 1-butylamine is not far from the value obtained in natural flotation.

El-Eswed[15] uses the octanol-water partition coefficient ($\log K_{ow}$) as a quantitative parameter to evaluate the hydrophobic nature of amines. The hydrophobicity is higher as the $\log K_{ow}$ value increases. $\log K_{ow}$ of 1-butylamine is 0.86 whereas it is 3.91 for BDHAC[16]. This means that BDHAC is 4.5 times more hydrophobic than the short-chain amine and this is consistent with the magnitude of the depressant effect observed for each. This demonstrates that high hydrophobicity is not always an advantageous property for coal flotation when amines are used, because non-oxidized sections of the surface can become depressed. The adsorption of amines on hydrophobic coal surfaces has been described as a complex interaction where electrostatic, acid-base, ion-exchange, hydrophobic, and π interactions may be present, and the predominance of one over the other depends largely on *pH*, ionic strength, hydrophobic nature of the amine and aromaticity[15]. All this makes the amine-coal interaction variable and leads to the non-universal character of these compounds as a coal collector.

Figure 4 shows the FTIR spectra for SBC before and after conditioning with the 1-butylamine. Few signals are recorded for this coal; this has been described for some sub-bituminous coals[17]. The C-O-C stretching signal is characteristic of sub-bituminous and other low-rank coals[17-19]. Two signals have been associated with silicates[17-19], which coincides with the phases identified by XRD. The signals associated

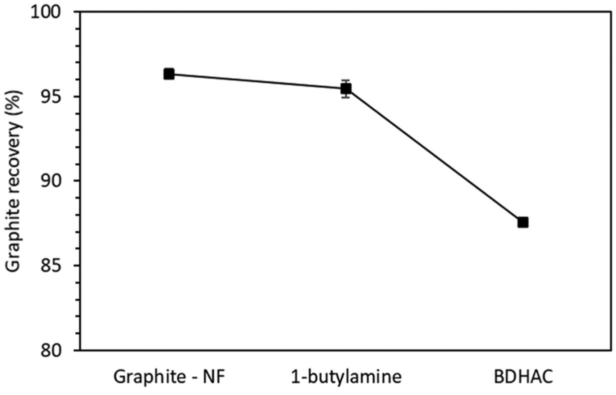

Fig. 3 — Graphite flotation results: no reagent (NF), 1-butylamine and benzyl dimethyl hexadecyl ammonium chloride (BDHAC) as collectors.

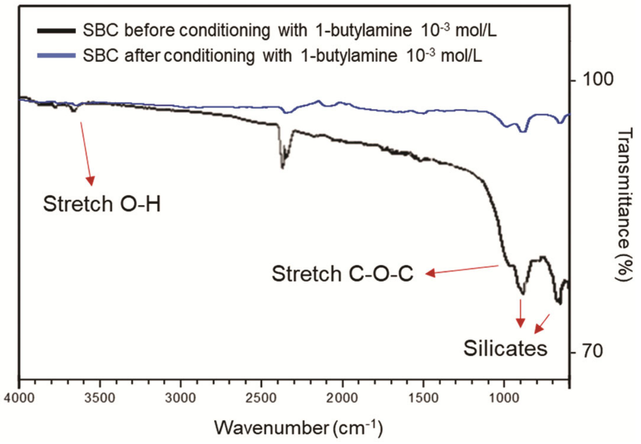

Fig. 4 — SBC FTIR spectra before and after conditioning with 1-butylamine.

with 1-butylamine are not present in the spectra, but an attenuation of the main peaks is observed, and this is a preliminary indication of the surface group's modification, which can be corroborated by zeta potential measurements as a safer experimental tool.

Figure 5 shows the zeta potential for different *pH* values before and after conditioning the SBC with 1-butylamine. No isoelectric point (IEP) is observed in the *pH* range for the unconditioned coal; however, when conditioning is performed, an IEP is observed at *pH* 2. This indicates that the IEP of the coal is probably located below *pH* 2 and this is consistent with the result obtained for the surface groups in the physicochemical characterization where the basic ones were predominant (Table 1). The results indicate that a displacement occurs after the treatment because the negative surface charge decreases as a consequence of the collector adsorption. These results corroborate a modification of the coal surface by the amine molecules[20,21].

Figures 6(a & b) show the contact angle for water and 1-butylamine 10^{-3} mol/L solution on a graphite surface as a hydrophobic model. As can be seen, the contact angle between 1-butylamine and graphite is much smaller than that of water/graphite, although it is greater than zero. This is related to the amphiphilic character of 1-butylamine, which allows the molecules to interact with the hydrophilic surface of the oxidized coal through the amino group. In this way, the non-polar carbon chain is exposed to the aqueous medium, making the coal hydrophobic and floatable[1,2].

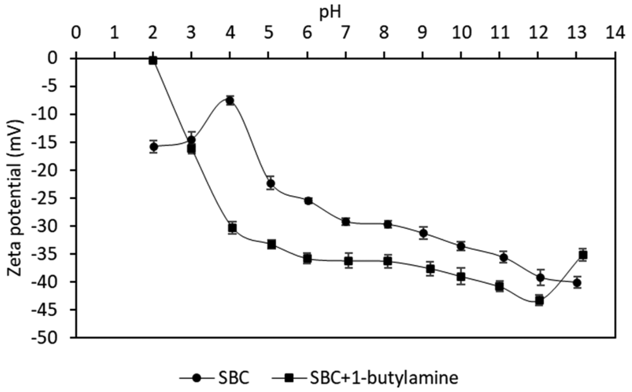

Fig. 5 — Zeta potential at different *pH* values for SBC before and after conditioning with 1-butylamine.

Study of physicochemical conditions and flotation optimization

Table 2 shows the Box-Behnken design carried out to study the conditions and optimize the flotation using 1-butylamine as a collector. The design has 17 experiments with 5 central points.

Table 2 — Experimental design for the study of conditions and optimization of SBC flotation using the 1-butylamine collector

Flotation	*pH*	t (min)	$BtNH_2$	RM (%)	RC (%)
1	5	2	10^{-5}	63.4	63.0
2	7	1	10^{-5}	47.2	49.3
3	5	3	$5.05\cdot10^{-4}$	80.0	92.4
4	5	1	$5.05\cdot10^{-4}$	46.5	47.2
5	9	3	$5.05\cdot10^{-4}$	79.1	92.9
6	7	2	$5.05\cdot10^{-4}$	72.1	74.2
7	7	2	$5.05\cdot10^{-4}$	60.2	70.0
8	7	1	10^{-3}	52.7	54.4
9	5	2	10^{-3}	88.6	100
10	7	2	$5.05\cdot10^{-4}$	62.2	61.4
11	9	1	$5.05\cdot10^{-4}$	46.2	42.1
12	9	2	10^{-5}	63.0	54.0
13	7	3	10^{-3}	95.0	100
14	7	3	10^{-5}	83.4	95.4
15	7	2	$5.05\cdot10^{-4}$	59.7	60.5
16	7	2	$5.05\cdot10^{-4}$	58.7	57.4
17	9	2	10^{-3}	81.6	99.2

Ft: flotation time, $BtNH_2$: concentration of 1-butylamine, RM: Total Mineral Recovery, RC: Coal Recovery

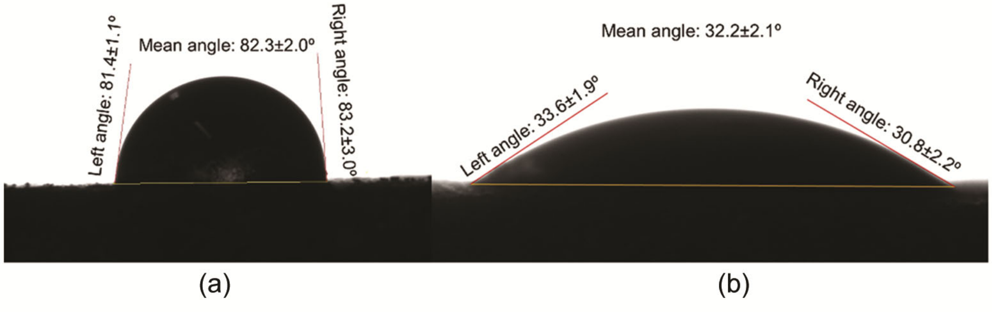

Fig. 6 — Contact angle on a hydrophobic graphite surface (a) Water and (b) 1-butylamine 10^{-3} mol/L.

The analysis of variance (ANOVA) for RM is shown in Table 3. All factors and interactions with p-value less than 0.05 (significance level, α) have statistical significance. As can be seen, the effect of *pH* is not significant on this response variable. However, flotation time and concentration of 1-butylamine have a significant effect. In addition, RM exhibits a quadratic dependance (CC) on 1-butylamine concentration. During the floatation process, *pH* can modify the coal surface and the reagent structure. In the present work, the *pH* has been evaluated from 5 to 9, and as it is shown in Fig. 5, the zeta potential of the coal in this *pH* range varies only 9 mV (*pH* 5: -22.3 mV and *pH* 9: -31.3 mV). This indicates a low modification of the coal surface by the H^+ ions. On the other hand, the pK_a of 1-butylamine[22] is 10.77, so only at *pH* values above this, the amine will deprotonate and the action of the collector can be modified. Time and collector dosage are always important experimental factors in flotation; therefore, both are expected to be of statistical significance[23].

Table 3 — Analysis of Variance for RM for the study of conditions and optimization of SBC flotation using the 1-butylamine collector

Source	SS	DF	MS	F-Value	P-Value
A	9.245	1	9.245	0.31	0.6081
B	2624.5	1	2624.5	87.64	0.0007
C	463.601	1	463.601	15.48	0.0170
AA	25.74	1	25.74	0.86	0.4063
AB	0.09	1	0.09	0.00	0.9589
AC	10.89	1	10.89	0.36	0.5790
BB	18.6127	1	18.6127	0.62	0.4746
BC	9.3025	1	9.3025	0.31	0.6070
CC	348.482	1	348.482	11.64	0.0270
Lack of fit	112.218	3	37.4058	1.25	0.4031
Error	119.788	4	29.947		
Total	3743.92	16			

A: *pH*, B: flotation time (t), C: $BtNH_2$, SS: Sum of Squares, DF: Degrees of Freedom, MS: Mean Square

The lack of fit test indicates a significant statistical fit of the proposed model for RM (p-value > α = 0.05). This model is shown in the equation (4). The determination coefficient (R^2) is 0.9380, which means that the statistical model describes 93.8% of the RM variability.

$$RM = 49.8681 - 8.19958A + 25.4917B - 16616.3C + 0.618125A^2 - 0.075AB - 1666.67AC - 2.1025B^2 + 3080.81BC + 3.71289\cdot 10^7 C^2 \quad \ldots(4)$$

Where A is the *pH*, B is the flotation time, and C is the 1-butylamine concentration

Figure 7 shows the main effects plot for RM. As can be seen, *pH* presents a minimum at value 7, and a maximal response at *pH* 5 but with little significance. Time has a direct effect on RM over the entire range. The concentration of 1-butylamine has a minimum, but its effect is direct in almost all the range.

Table 4 shows the ANOVA for RC. The behavior is similar to that observed for RM: time and 1-butylamine concentration have the most important statistical effects. Collector dosage also has a quadratic effect.

The lack of fit test indicates that the model generated for RC fits the experimental data with statistical significance. Equation (5) shows the statistical model obtained. The R^2 value is 0.8751, indicating that 87.51% of the variability of the response variable is explained by the equation.

$$RC = 89.6038 - 17.7395A + 19.34B - 32982.1C + 1.02813A^2 + 0.7AB + 2070.71AC - 0.1625B^2 - 252.525\cdot BC + 4.17815\cdot 10^7 C^2 \ldots (5)$$

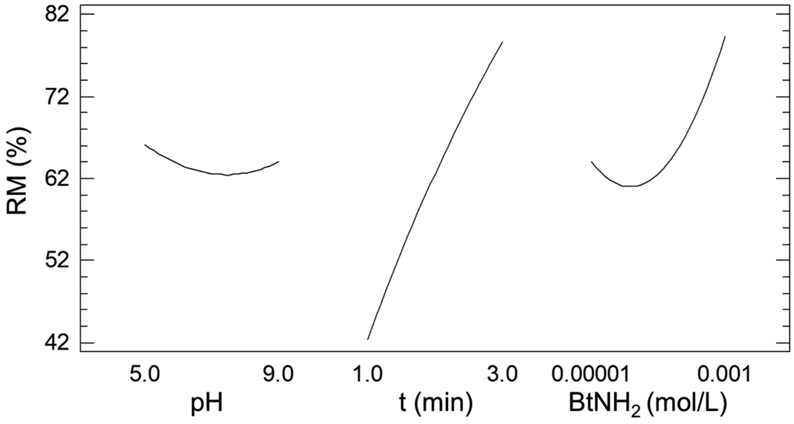

Fig. 7 — Main effects on RM for the coal flotation in SBC using the 1-butylamine collector.

Where A is the *pH*, B is the flotation time and C is the 1-butylamine concentration

Figure 8 shows the main effects plot for RC. As can be seen, factors show a similar behavior as in the case of RM. This result suggests that, from a mineralogical and kinetical point of view, coal is the determining variable for the flotation process with the proposed collector. This is logical if it is taken into account that coal is the main component, as observed in the XRD analysis (Fig. 1).

The response surface for the joint optimization of coal flotation using 1-butylamine as collector is shown in the Fig. 9. Desirability is the joint optimal response followed in the flotation. Here, RC has been prioritized. The *pH* is set to 7 due to its low influence on flotation results. The best desirability (blue color: 0.9-1.0) is reached for 2.6 min and 10^{-3} mol/L, conditions indicated by the experimental design for optimal SBC flotation using the mathematical models shown in the Eq. (4) and (5). However, the design indicated that one experimental point meets the optimal conditions. Table 5 shows the identification of this experiment. Desirability is most likely achieved in flotation test No. 9. This result demonstrates that the optimal conditions for flotation were achieved within the experimental data. Note that time is the only difference between the predicted conditions from Fig. 9 and the optimal point identified in Table 5. The predicted optimal time is 2.5 min, while the experimentally determined time for the best flotation is 2.0 min. Therefore, the flotation time 2.0 min can be taken as the best value for optimal results.

Table 4 — Analysis of Variance for RC for the study of conditions and optimization of SBC flotation using the 1-butylamine collector

Source	SS	DF	MS	F-Value	P-Value
A	25.92	1	25.92	0.52	0.5115
B	4403.91	1	4403.91	88.01	0.0007
C	1055.7	1	1055.7	21.10	0.0101
AA	71.2112	1	71.2112	1.42	0.2988
AB	7.84	1	7.84	0.16	0.7124
AC	16.81	1	16.81	0.34	0.5933
BB	0.111184	1	0.111184	0.00	0.9647
BC	0.0625	1	0.0625	0.00	0.9735
CC	441.29	1	441.29	8.82	0.0412
Lack of fit	662.722	3	220.907	4.41	0.0927
Error	200.16	4	50.04		
Total	6907.47	16			

A: *pH*, B: flotation time (t), C: $BtNH_2$, SS: Sum of Squares, DF: Degrees of Freedom, MS: Mean Square

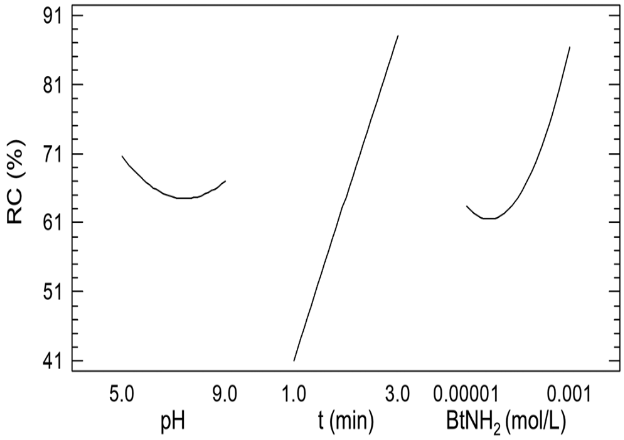

Fig. 8 — Main effects on RC for the coal flotation in SBC using the 1-butylamine collector.

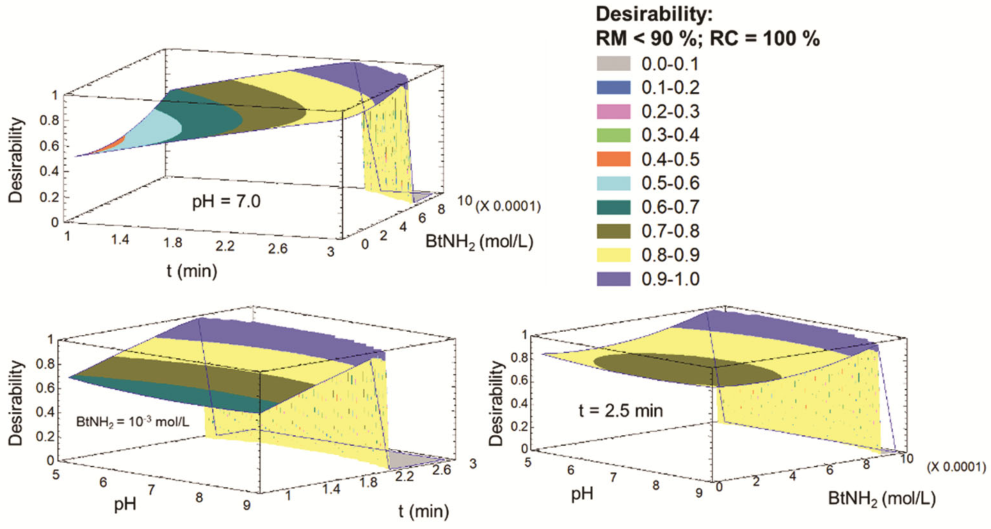

Fig. 9 — Response surface for the joint optimization of RM and RC in the SBC flotation using the 1-butylamine collector.

Table 5 — Optimal experiment for SBC flotation using the 1-butylamine collector

Flotation test	RM (%)	RC (%)	Desirability	
			Predicted	Observed
1	63.4	63.0	0.72346	0.666183
2	47.2	49.3	0.447386	0.508479
3	80.0	92.4	0.919699	0.906274
4	46.5	47.2	0.496021	0.493829
5	79.1	92.9	0.901979	0.903597
6	72.1	74.2	0.670731	0.770989
7	60.2	70.0	0.670731	0.684268
8	52.7	54.4	0.634327	0.564395
9	88.6	100.0	0.920638	0.992192
10	62.2	61.4	0.670731	0.651416
11	46.2	42.1	0.452619	0.46488
12	63.0	54.0	0.689275	0.614817
13	95.0	100.0	0.0	1.0
14	83.4	95.4	0.870762	0.940234
15	59.7	60.5	0.670731	0.633496
16	58.7	57.4	0.670731	0.611862
17	81.6	99.2	0.892911	0.948374

RM: Total Mineral Recovery, RC: Coal Recovery

Conclusion

1-butylamine is a better flotation collector for a sub-bituminous coal than diesel, kerosene and benzyl dimethyl hexadecyl ammonium chloride. A lower depressant effect has been observed for the short-chain amine than for the long-chain. This amine significatively modifies the coal surface.

The *pH* does not affect the total mineral recovery or coal yield with statistical significance, but 1-butylamine concentration and flotation time do have a significant effect. Both response variables increase with increments in floatation time. As collector concentration increases, there is a minimum of the response variables followed by an improvement. The optimal conditions for flotation are 2.0 min of flotation and 10^{-3} mol/L of 1-butylamine at any *pH* value from 5 to 9.

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