DOI: https://doi.org/ 10.25100/iyc.v20i1.6180

CHEMICAL ENGINEERING

Evaluation of the flotation of a refuse tailing fine coal slurry using release analysis

INGENIERÍA QUÍMICA

Evaluación de la flotación de lodos finos de carbón usando análisis reléase

Jorge Piñeres[1§], Juan Barraza[2], Edward García[2], Sebastián Sandoval[2]

[1]Facultad de Ingeniería, Programa de Ingeniería Química, Universidad del Atlántico. Barranquilla, Colombia. [2]Escuela de Ingeniería Química, Universidad del Valle. Cali, Colombia.
jorgepineres@mail.uniatlantico.edu.co, juan.barraza@correounivalle.edu.co, edward.garcia@correounivalle.edu.co, sebastiansp@familia.com.co

(Recibido: Abril 25 de 2017 - Aceptado: Octubre 31 de 2017)

Abstract

Performance of flotation when processing a refuse tailing fine coal slurry from a Colombian coal cleaning plant was studied using release analysis to evaluate the potential cleaning in terms of both, low ash and high recovery. Results from release analysis showed, that this was a good estimate for obtaining ideal flotation data, as well as exploring the feasibility of cleaning this material.

The results showed that the best combination of collector–frother was diesel oil-MIBC, producing recoveries on the order of 65%, with cumulative ash value less than 10%. It was also tend that the type of frother significantly affected the behavior of a refuse tailing fine coal slurry in a mechanical flotation cell.

Keywords: *Collector, frother, refuse tailing fine coal slurry, release analysis.*

Resumen

Se usó el análisis release para estudiar el desempeño de la flotación de lodos finos de carbón de una planta Colombiana de limpieza de carbón, en términos de obtener bajos contenidos de cenizas y altas recuperaciones másicas. Los resultados del análisis release mostraron una buena estimación para obtener datos ideales de flotación, así como explorar la viabilidad de la limpieza de este material.

Los resultados mostraron que la mejor combinación de colector-espumador fue el Diesel oil-MIBC, produciendo recuperaciones del orden del 65%, con valores de cenizas acumulado inferior al 10%. También se observó que el tipo de espumante afectó significativamente el comportamiento de los lodos finos de carbón en una celda de flotación mecánica.

Palabras claves: *Análisis reléase, colector, espumante, lodo fino de carbón.*

1. Introduction

Several tons of refuse tailing fine coal slurry are produced in Colombian coal cleaning plants, which are usually discarded in refuse ponds. Any strategy to recover and utilize the organic matter present in the refuse tailings slurry could represent an important energy source having environmental and economic benefits (1).

Froth flotation as a process separation is widely accepted a process for cleaning coal fine than about 0.5 mm. During the flotation, hydrophobic particles (organic matter of coal) adhere to air bubbles and they are taken in the froth, while hydrophilic particles (mineral matter of coal) are obtained in tailing of flotation cell (2). This process takes advantages of the differences in surfaces properties (hydrophobicity) between mineral and organic matter present in coal. The behavior of coals during the flotation varies according to its coal rank, oxidation time and type of mineral matter. The floatability of coal depends mainly on the amount of hydrophilic sites, oxygen functional groups in their surface and its petrographic constituents (3, 4). The hydrophobicity of coal surface is related to the carbon content and the contact angle, which measures the coal wettability. Prolonged contact with oxygen results in coal surface oxidation and causes dramatic changes in its surface properties and floatability.

The observed variations in the coal hydrophobicity with the carbon content undoubtedly depend on the rank, reflected in the differences in chemical composition and oxygen functional groups on coal surface. The high content of carboxylic oxygen [-COOH], carbonyl [-C=O] and phenolic in lignite and sub-bituminous coals change surface hydrophobicity. These groups are ionizable and have an appreciable effect on the surface charge, which control the electrokinetic process and in turn floatability (4-7).

Radicals of the oxygen functional groups on coal surface that are electrochemically active in water are of particular importance due to the change on surface concentration of the reactive functional group, resulting in changes in the polarity degree of the surface and on the structure of the double layer of the solution coal-water. These functional groups are determined by Fourier transform Infrared (FT.i.r) analysis of coal (5, 6, 8).

The coal hydrophobicity increases with the carbon content due to the decrease in the number of hydrophilic groups. However, for high carbon contents, such as that presented in the anthracite coal, the hydrophobicity is lower. This decrease may due attributed to the increase in the number of π bonds of the aromatic carbon rings of the anthracite coal. The petrographic composition affects the hydrophobicity of coal. Through contact angle measurements in different US coals, it has been found that the order of decreasing hydrophobicity of macerals is: liptinite, vitrinite, inertinite, indicating its dependence on the coal type. (5, 7, 9).

Different material, chemical, operational and equipment parameters affect the flotation yield, ash value, combustible recovery, separation efficiency and flotation kinetics. Chemical parameters include collector type and concentration, frother type and concentration and different mixed frothers, whereas equipment parameters can be represented by the kind of machine such as mechanical conventional cell and column flotation (4).

Release analysis is the counterpart froth flotation to float and sink analysis in gravity concentration. It provides an ideal separation of a sample via flotation. Froth flotation washability data are required for finding the froth flotation cleaning potential and helping to diagnose the causes of any difficulty in the flotation circuit (10, 11).

The objective of this study was to obtain washability curves of a refuse tailing coal slurry using release analysis for three types of frother and collector respectively to evaluate its potential cleaning in terms of both, low ash and high recovery percentage.

2. Metodology

2.1. Equipment

The release analysis runs were carried out in a Denver laboratory flotation machine. The impeller speed and solids content were kept constant at 1,500 rpm and 20% (by weight), respectively in the first stage. Three types of frother and collector respectively were used. The three frothers used were MIBC (neutral), Genapol 28% (anionic) and Dodigen (cationic), whereas the three collectors were creosote oil, kerosene and diesel oil. The concentrations used were: 10 lb / ton dry solid for the collector, and between 2 and 5 lb/ton dry solid for the frother.

2.2. Materials

One 55-gallon drum of a refuse tailing fine coal slurry was supplied from a Colombian coal preparation plant. In order to obtain sample dry of a refuse tailing fine coal slurry, for sieve analysis, proximate analysis and analysis of major elements, the coal slurry sample was dewatered in a filter press device and subsampled by coning and quartering to obtain representative samples. Additional material was also dried at room temperature in order to use at the flotation runs.

2.3. Experimental procedure

The release analysis for a refuse tailing coal slurry was carried out according to the procedure described by Dell (10, 11). These tests were conducted in a cell flotation machine, with slurry concentration of 20% (w/w) (1,000 ml of tap water and 200 g of coal), and impeller speed of 1,500 rpm. After conditioning, the air inlet valve was opened fully. A high pulp level was maintained throughout this stage of the separation to maintain a high recovery. Extra frother was added as needed to maintain a stable froth throughout the separation. The tailing material was saved and the froth product was repulped (2 to 3 times) for additional cleaning (some amounts of collector and frother were used). The resulting products from this first stage of the release analysis procedure consisted of a clean coal product containing all the truly floatable material and a combined refuse product containing all of the non-floatable material.

The second stage of the release analysis procedure involved the separation of the floatable material into components having various degrees of floatability. In this second stage, the product from the first stage of the procedure was repulped and flotation was initiated with an impeller speed of 1,200 rpm and aeration rate approximately 25% of full range (was this done by adjusting the air valve). These weak flotation conditions allowed only the most floatable material to report to the froth product. After the froth became barren, the collection basin was changed, the impeller speed was increased to 1,300 rpm and the aeration rate was increased (50% of full range) until a second froth was formed. This froth was removed as the second froth product. The procedure of increasing the impeller speed and aeration rate (until 100% of full range) was repeated seven more times to produce a total of nine froth products. A diagram of the release analysis procedure is shown in Figure 1.

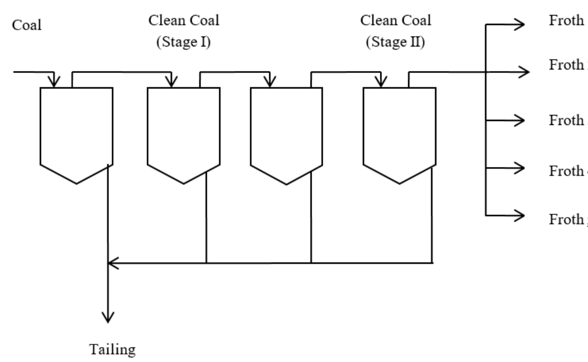

Figure 1. *Diagram showing release analysis procedure.*

The tailing material remain in the cell was combined with the tailing material from the first stage of the release analysis to produce an overall tailing product. Finally the nine clean products and the combined refuse product were filtered, dried and weighed. The dry weights were recorded and the percent ash was determined.

3. Results and discussion

3.1. Characterization of the refuse tailing coal slurry

Table 1 shows the size distribution and ash yield of the size fractions of the dewatered refuse tailing coal sample, it is observed that the fraction containing the highest ash content (72.4%) corresponds to the - 400 mesh, this behavior was to be expected, because ash settles with the finer particles.

Table 1. *Size analysis results of the ash-received refuse tailing fine coal sample.*

Particle size (US mesh)	Ash (%)	Wt (%)
+60	20.5	4.2
-60+120	12.1	8.0
-120+200	18.1	7.3
-200+325	35.6	7.5
-325+400	31.3	1.1
-400	72.4	71.9
Total feed	64.8	100

Table 2 shows the proximate analysis (db) of the dewatered refuse tailing coal. This feed sample had an ash of 64.8 % at a particle size less than 60 mesh, reflected in a low fixed carbon content (21.5%) and low heat value (3,073 Btu/lb). This sample had a low heat value because of the high ash value. The contents of the major elements present in the refuse tailing coal sample, an as received basic, are presented in Table 3. The sample has a higher composition of Al and Fe, whereas, concentrations on the order of ppm occurred in the elements Ca, Mn, Mg, K, Na, while for Ti, the concentrations were in the parts per billion range.

Table 2. *Proximate analysis of the sample.*

Analysis db	Value
Volatile Matter %	19.86
Ash %	58.63
Sulphur total %	1.13
Fixed carbon %	21.52
Heat value Btu/lb	3,073

Table 3. *Major elements of the refuse tailing coal sample.*

Metal	Value
Al % p/p	0.87
Ca, ppm	96.28
Fe % p/p	0.27
Mn ppm	52.83
Mg ppm	0.12
K ppm	0.35
Si ppm	0.20
Na ppm	0.13
Ti ppm	N.D.< 81.90

3.2. Release analysis obtained with Dodigen frother

Figure 2 shows the release analysis curves obtained with Dodigen frother for the three collectors. The observed behavior is very unusual, which may be attributed to than the Dodigen is a cationic frother, which produces little froth deep, which may be attributed to the low decreased surface tension of slurry compared with MIBC and Genapol; therefore produces very low yields and selectivities.

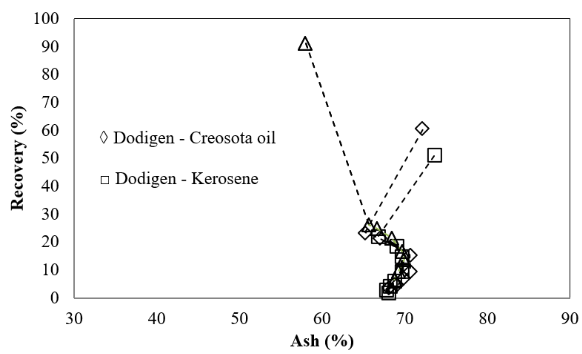

Figure 2. *Release analysis curves obtained with Dodigen frother.*

It is important to highlight a particular behavior observed in the results obtained for the combination

Dodigen - diesel oil, wherein the tailing stream has a lower ash concentration compared with to the overflow stream.

This indicates that this combination (Dodigen-Diesel Oil) favors flotation by entrainment, which may be attributed to the lack of froth is likely due to an insufficient amount of floatable material being present which causes very quick drainage of the water layer around the bubbles leading to bubble coalescence and froth instability. This indicates that this combination provides a process of forming micelles reverse between reactants, the bubble surfaces and coal, as a result of the cationic nature of the frother (2). In this case, the chemical nature of Dodigen creates a favorable environment for the flotation of the mineral matter, seeing reflected in the results of ash contents, which show orders of 70%.

Clearly the low chemical affinity of this reagent with the prevailing conditions in the flotation of refuse tailing coal, which may be attributed to the strong affinity of the Dodigen cations with the ions present in the mineral matter of the refuse tailing coal.

3.3. Release analysis obtained with Genapol frother

Figure 3 shows the release analysis curves obtained with Genapol frother for the three collectors. Tests with this reagent had certain characteristics, however, frother performance of this compared with Dodigen was much better due to the constant production of froth.

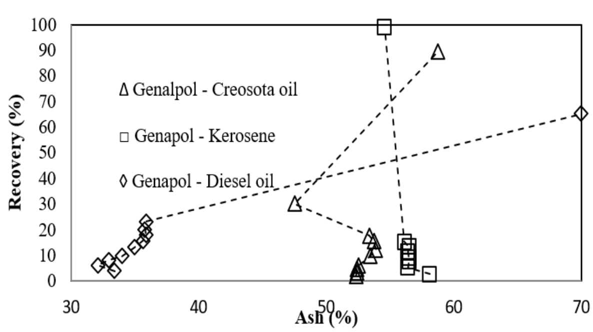

***Figure 3.** Release analysis curves obtained with Genapol frother.*

Genapol is an anionic frother, which interacts well with coal organic matter, producing a stable froth. Nevertheless, during testing a decrease in the froth loading occurred as the concentration of frother was increased.

The flotation behavior for frother concentrations of 2lb/ton of dry solid were satisfactory for producing froth with good organic loading and good stability. However, as the frother concentration was increased, the froth became unstable, with low loading of floated material, resulting from the excess of polar heads added by the frother molecule (2).

Excessive froth production at high concentrations of Genapol decrease in the hydrophobicity of air bubbles, which may be attributed to the increasing population of polar heads of sodium lauryl ether sulfate ions (Genapol) at the bubble surface, which are strongly hydrated, indicating that the air–water interface becomes less hydrophobic, producing a detrimental effect on the selectivity of the flotation of the organic material present in the refuse tailing coal.

On the other hand, the frother one acts on refuse tailing coal surface through hydrophobic interactions with the ions of sodium lauryl ether sulfate, by means of the sodium ion and functional groups on refuse tailing coal surface, creating an environment favorable for the adhesion refuse tailing coal particles–air bubble, therefore forming a stable froth (2, 12, 13). The best results with low concentrations Genapol relate to increasing the hydrophobicity of the bubbles as a result of the association of water molecules with the polar component of the frother.

3.4. Release analysis obtained with MIBC frother

Figure 4 shows the release analysis curves obtained with MIBC frother for the three collectors. The use of MIBC (methyl isobutyl carbinol, neutral frother) as frother produced positive results, having a considerably higher performance (low ash percentage and high recovery) compared with the frothers Dodigen and Genapol. With MIBC similar behavior was found to that reported in the literature

(5, 6), which data obtained formed a sharp elbow for diesel oil, creosote oil and Kerosene.

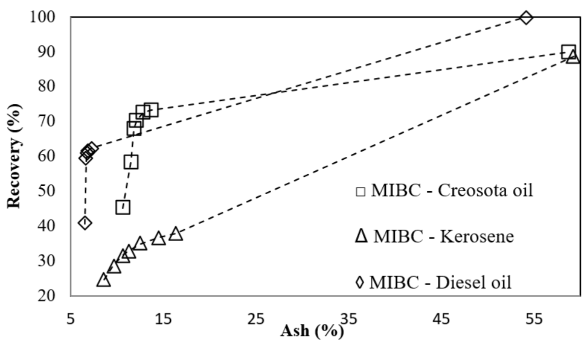

Figure 4. *Release analysis curves obtained with MIBC frother.*

Using MIBC ash value below 10% with recoveries of organic material of about 60 % and 70% were obtained. Experiments with MIBC produced good yields and it was observed for all concentrations of MIBC, a good froth production with small diameter bubbles were observed with great stability and good load of floated material, reducing the flotation time.

The best results have been found with MIBC, which may be attributed by the strong hydrophobic interaction between this frother and the air bubble, generating small diameter bubble, increasing the contact surface (area available) generating good stability of the interface bubble-particle. The above is reflected in a strong adhesion as a result of interactions with the MIBC and the conditions of the slurry, which favors an increase in the recovery of combustible material and therefore greater separation performance comparated to the other two frothers.

In general terms, release analysis curves were grouped by frother type. It was noted the close relationship of the results with the type of frother, which may be attributed to the chemical nature of the frother, using one cationic, a neutral and an anionic frother, while the collectors have very similar chemical characteristics, being hydrophobic organic compounds which are products of petroleum refining. However the three sets of release analysis curves indicate good performance in all cases for the diesel oil, which may be attributed to the compatibility of the collector with the coal surface present in the refuse tailing coal slurry. The release analysis curve provides a measure of the maximum separation of a coal sample by froth flotation. For this reason it is considered a suitable technique for characterizing the performance of flotation equipment for processing coal samples. On the other hand, the coal potentiality to float depends also on the associations between the mineral matter and the organic matter, as well as, the type and occurrence mode of the mineral matter, syngenetic or epigenetic. Normally, epigenetic minerals are liberated with milling process and they can be separated by physical cleaning. In this work, given that small particle sizes were used, it could be assured that most mineral matter was liberated and therefore separated during flotation process.

4. Conclusions

The results show that the type of frother significantly affects the behavior of the refuse tailing coal in the flotation cell.

In general terms it was observed that MIBC frother mixed with any of three collectors used produced the best floats separation efficiencies.

The behavior of the Genapol frother was intermediate, whereas the Dodigén frother produced the lowest separation.

Release analysis showed that the best combination was MIBC (frother) - diesel oil (collector), producing organic recoveries on the order of 65%, with cumulative ash value below 10%.

5. References

1 Barraza J, Guerrero J, Piñeres J. Flotation of a refuse tailing fine coal slurry. Fuel Process. Technol. 2013; 106: 498-500.

2 Piñeres J, Barraza J. Effect of pH, air velocity and frother concentration on combustible recovery, ash and sulphur rejection using column flotation. Fuel Process Technol. 2012; 97:30-7.

3 Vamvuka D, Agridiotis V. The effect of chemical reagents on lignite flotation. Int. J. Miner. Process. 2001; 61(3): 209-24.

4 Tao D, Li B, Johnson S, Parekh B. A flotation study of refuse pond coal slurry. Fuel Process. Technol. 2002;76(3):201-10.

5 Stachurski J, Tora B. The investigation on coals flotation. Memorias V Congreso Nacional de Ciencias y Tecnología del Carbón; 2001; Valledupar, Colombia. p. 325-34.

6 Esen B, Selma S, Sabriye P. The effect oxidation on the flotation properties of a Turkish bituminous coal. Fuel Process Technol. 1998;55(2):101-5.

7 Wieslaw S. New trends in coal preparation technologies and equipment. Philadelphia (PA): Gordon and Breach Publishers; 1994.

8 Polat M, Polat H, Chander S. Physical and chemical interactions in coal flotation. Int J Miner Process. 2003;72:199-213.

9 Arnold B, Aplan F. Hydrophobicity of coal macerals. Fuel. 1989 May;68(5):651-58.

10 Dell C. Determination of froth-flotation washability data. J Institute Fuel. 1957; 30:523-26.

11 Dell C. An Improved release analysis procedure for determining coal washability. J Institute Fuel. 1964;37:149-50.

12 Forrest W, Adel G, Yoon R. Characterizing coal flotation performance using release analysis. Coal Prep. 1994;14(1-2):13-27.

13 Piñeres J, Barraza J. A comparative study between release analysis and column Flotation. International Conference on Coal Science and Technology. 2007 Jul-Aug 28-3. Nottingham, UK: The University of Nottingham; 2007.