FLOTATION OF FINE BRAZILIAN NATIONAL COAL USING SELECTIVE BY-PRODUCT FROM COKING PLANT OF COMPANHIA SIDERURGICA NACIONAL

S.P. MADHUSUDANAN NAIR[1]
SEBASTIÃO VITOR BALIZA[2]
ARMANDO FELTES[3]
CLÁUDIO B. ZILLI[4]

ABSTRACT

Presently by the use of new mechanized mining methods in the coal mining area, there is a tremendous increase in the fine coal generation and the beneficiation of these fines is one of the paramount importance for the economical utilization of any coal reserve. One of the main beneficiation methods adopted for these coal fines is the froth flotation process, where in one of the main economical factors of the process, is the type of the reagents used and their cost aspect. The reagents that are currently employed in the flotation plants of pre-washeries of Carbonífera Próspera are MIBC of Pine oil, as frother and Diesel oil as colector. Through the lab and industrial tests it has been demonstrated in this paper the possibilities of using the by-products from the coke-oven plant namely Disinfectant oil and Creosote oil substituting the regular frothers cited above. The main advantages of this substitution are: (a) the proposed reagents are especially the products of C.S.N.; (b) higher recovery of metallurgical coal in the float with admissible % of ash and sulphur in it; (c) in terms of cost, these reagents are cheaper than the conventional reagents namely MIBC and Pine oil.

[1] M.Sc. Mineral Process Engineering, Process Engineer. Mineração Taboca S/A

[2] Chemical Technician - Researcher - Research Center - C.S.N.

[3],[4] Mining Engineers - Carbonífera Próspera

1.0 INTRODUCTION

The main objective of this project was to study the viability of substituting the reagents that are currently used in the flotation process of fine metallurgical coal by some by-products of coke oven plants of C.S.N.

Presently by the development of new mechanized mining methods in the coal mining area it has been observed that there is a increase in the generation of fines.

It has become of prime importance the processing of these fine coal by froth flotation process or oil agglomeration for the recovery and use in metallurgy. Currently in the coal mines of Carbonifera Próspera about 6000 t to 7000 t/M of fine coal are generated and these are processed by froth flotation process in the flotation plants of the pre-washeries.

In the flotation plants of pre-washeries, for processing these fine coal, the reagents used are Diesel oil as colector and MIBC (Metyl-Iso-Butil Carbinol) as the frother. Through this work it has been demonstrated the possiblity of employing the by-products of coke-oven plant of C.S.N., substituting the frothers that are used currently, namely, MIBC or Pine oil.

2.0 LABORATORY SCALE EXPERIMENTS

2.1 Equipments

The principal equipment used for the development of this work was lab flotation cell. This flotation cell was developed and fabricated in the workshop of the research center of C.S.N. with a capacity of 2.5 liters.

Through lab tests it has been found that the perfomanace of this cell was comparable to any other flotation cell.

2.2 CHARACTERISATION OF THE COKE-OVEN BY-PRODUCTS OF C.S.N.

These reagents used in this work were the by-products of coking plant of C.S.N. and are generally known as the disinfectant oil and creosote oil. The basic constituents of these reagents are aromatic hydrocarbons namely Phenol and Cresols.
The studies on characterisation and identification of various chemical components of these by-products were realized in the research center and were able to identify most of those components of the disinfectant oil and the creosote oil.
Execution of this type of analysis studies was difficult as it depended on various standards and various complimentary analysis techniques like crematography, infra-red, mass spectrometer and other physical tests.
The full chemical analysis of these two reagents are shown in Table I. It may be possible that components like phenantron and xylenol and other elements may be present in these two reagents but could not be identified because of the absence of good standards and procedures.

2.3 SAMPLES

All the samples of fine coal used for the tests were collected from the pre-washeries of Siderópolis. The samples were transported from the pre-washeries to the research center in closed drums with water in order to avoid oxidation. These samples were the underflow of hydrocyclones which was the feed to flotation process, with a size range of 28 x 0 mesh.

2.4 METHODOLOGY OF SAMPLE PREPARATION FOR TESTS

All the samples from the drums were thoroughly mixed, cone and quartered and stocked in plastic bags for evicting oxidation.

2.5 CONDITIONS FOR TESTS

a) Weight of the sample for each test - 250g.

b) Pulp Density - 11% by weight.

c) Type of water used - common tap water.

d) Impeller velocity - 1500 to 1700 rpm.

e) Natural aeration.

f) Conditioning time of feed - 5 minutes simple agitation.

g) Conditioning time of reagents - two minutes for colector and two minutes for frother.

After this separate conditioning with colector and the frother the air valve was opened and flotation was effected.
The two fractions float and tails were collected dried in low temp, cooled in room temp and weighed. From the weights the recovery was calculated and samples were analysed for % ash and % sulphur.

3.0 DISCUSSION OF THE RESULTS OF LAB TESTS

The two by-products of coking plant namely desinfectant oil and creosote oil had demonstrated satisfactory results as good frothers. The two derivatives of coaltar are light oils which are basically composed of phenols, cresols and xylenols. The frother properties of the two reagents from coke-oven plant were confirmed mainly by three series of tests. In each series of these tests were separately tested creosote oil or desinfectant oil with three different concentrations of diesel oil varying 0,125 kg/t, 0,25 kg/t and 0,5 kg/t. The main objective of these series was to obtain the optimam concentration of diesel oil for these two reagents as frother.

From these series of tests it has been found that addition of 0.25kg/t of diesel oil would be the optimam concentration. The next objectivie was to find out the optimam frother concentration for disinfectant oil and creosote oil, keeping diesel oil constant at 0.25kg/t. The table II shows the results of the six tests where the concentration of diesel oil was kept at 0.25 kg/t at the same time the concentration of disinfectant oil was varied from 0.125 kg/t up to 1.6 kg/t. Among these six tests the fourth test using 0.8 kg/t of disinfectant oil was most promissing with around 67% recovery of coal in the float and the floated coal with 15.03% and 1.92% of ash and total sulphur respectively.

The table III shows the results of the six tests of second series where the concentration of creosote oil was varied from 0.125 kg/t to 1.6 kg/t at same time keeping steady the concentration of diesel oil to 0.25 kg/t. From the results shown in this table it may be deduced that the fourth test with 0.8 kg/t concentration of creosote oil was the most promissing with 65% recovery in the float and the floated coal with 15.05% and 2.18% of ash and total sulphur respectively.

The fig. 1 demonstrates the parameters Δ ash and "E" as recovery curve for disinfectant oil. These were obtained from table II.

The Δ ash was calculated according to the following formula.

$$\Delta \text{ ash} = \frac{(\%\text{ ash in the feed} - \%\text{ ash in the floated coal}) \times 100}{\%\text{ ash in the feed}}$$

From these values it could be observed the percentage reduction of ash by this process, for the improvement of the quality of floated coal.

The recovery curve is obtained directly from the recovery values for each test. "E" is the efficiency of the flotation process. Generally the efficiency of the flotation process depends on the mineral present in the gangue. In the case of flotation of coal fines the process efficiency is determined by the % recovery of coal in the float and its quality by the reduction of ash and at the same time the quality of tails.

In this way the value "E" can be calculated with the following formula.

$$E = \frac{R \times C_R}{C_F}$$

Where:

R = % Recovery of coal in the float.

C_R = % Ash in the tails.

C_F = % Ash in the float.

In fig. 1 it could be observed that there is a constant increase in the efficiency and recovery with the increase of concentration of disinfectant oil from 0.125 kg/t to 0.8 kg/t at the same time there is decrease in the percentage reduction of ash in floated coal. In other words the increase in concentration of frother (disinfectant oil) helps higher recovery but sacrifices the quality of coal indicating the reduction in the selectivity of the process.

It could be observed that the recovery curve and the efficiency curve attain saturation point when the addition of frother reaches 1.2 kg/t and these two values become constant even if concentration of the frother increases beyond 1.2 kg/t. It clearly demonstrates that after reaching the equilibriam point with frother, any extra addition of this reagent is not economic for the process because the reagent get lost in the system.

The fig. 2 shows the ash curve, the recovery curve and "E" curve for creosote oil. By comparing these two Figures (fig. 1, fig. 2) for disinfectant oil and creosote oil, it can be observed that these two reagents are similar in their frother characteristics. A small difference exists between these two products is that in terms of recovery of floated coal, disinfectant oil is slightly better than creosote oil . This is well shown in fig. 3.

Apart from this, the chemical analysis of these two products (table I) shows a higher % of naphthalene in the creosote oil which would give problem for its industrial use as frother, because, naphthalene when present more than certain percentage would solidify at low temp and clog the installations of the dosage system. Because of this reason it is very important that the quality of disinfectant oil for industrial use as frother should be maintained strictly with the % of naphthalene. The maximum % of naphthalene could be around 10%.

For comparison of pine oil as frother with these by-products, 5 tests were conducted with pine oil as frother and diesel oil as colector. The table III shows the results obtained in lab scale tests with diesel oil as colector, the concentration of which varied from 0.125 kg/t to 1.00 kg/t and pine oil as frother the concentration of which varied from 0.06 kg/t to 0.50 kg/t.

The fig. 4 shows the curves for ash, recovery and efficiency for pine oil as frother and diesel oil as colector for various concentrations. By analysing these results it was evident that with a small concentration of pine oil as 0.125 kg/t, a high recovery was achieved. But at the same time the quality of coal floated was not good, with high % of ash. This demonstrates that pine oil is a good frother but at the same time a type of coal like brazilian national coal which is easily floatable, a strong frother like pine oil will adversley affect the flotation process. This is because a strong frother like pine oil will try to float all the mineral matter also along with coal. In this aspect the by-products of coking plant namely disinfectant oil and creosote oil have a deffinite edge over pine oil as these two will keep a low ash in floated coal, at the same time maintaining good recovery.

These results from lab tests indicated that an industrial test using disinfectant oil was viable.

4.0 DISCUSSION ON INDUSTRIAL TESTS

The industrial tests were conducted in the flotation plants of Carbonífera Próspera situated in Criciúma and Siderópolis with the by-products of coking plant. Disinfectant oil was the first one to test in the pre-washeries of Criciúma where operated a modern flotation plant using a battery of 4 flotation cells of Wemco model. The feed to the flotation process was from the underflow of hydrocyclones with a size range of 28x 0. The feed rate was about 36 t/h (dry coal basis) with a pulp density of 7 to 8 % solids. Reagents used at that time were diesel oil as colector and MIBC as frother. The reagents addition rates were 2.4 to 3 liters of MIBC and 14 liters of diesel oil per hour. At this rate of addition , the specific consumption of reagents would be 83 g/t of coal for MIBC and 388 g/t of coal for diesel oil. Average production of floated coal using these reagents was 20 t/h to 21 t/h which was representing around 30 to 40% recovery in relation to feed.

So in the place of MIBC the disinfectant oil was used in order to prove its characteristics as frother in the industrial flotation cells. This industrial test was conducted for 5 days in Criciúma where the rate of addition of disinfectant oil varied from 10.8 liters per hour and 7.4 liters to 11.6 liters per hour of diesel oil. By this rate of addition the average consumption of the reagents were 14.5 liters per hour of disinfectant oil and 10.8 liters per hour of diesel oil. The quality of floated coal with these reagents was 16.8% and 2.03% of ash and total sulphur respectively. The tails contained 51.00% and 1.89% of ash and total sulphur respectively. The average recovery of coal in the float was 50% to 60%. With this industrial experiment it has been proved that disinfectant oil can very well substitute MIBC as a frother reagent.

The industrial tests conducted in the flotation plant in Siderópolis with disinfectant oil demonstrated the same results that achieved in Criciúma.

5.0 CONCLUSIONS

1. The lab tests showed clearly that the two by-products of coke-oven plant of C.S.N., namely disinfectant oil and creosote oil have the characteristics of frother and could be substituted for pine oil or diesel oil which are currently used in the flotation process.
2. The properties of these two by-products were similar but disinfectant oil demonstrated a little bit superiority over creosote oil in terms of recovery of coal and chemical quality.
3. Lab tests showed that the optimam concentrations of disinfectant oil and diesel oil were 0.6 kg/t and 0.25 kg/t respectively. This has been proved in the industrial tests also.
4. One of the most important resultsof 5 days industrial tests in Criciúma and Siderópolis was that a better recovery was achieved with disinfectant oil. The recovery improved from 30 to 40% to 50 to 60%. This shows that a strong frother like MIBC or pine oil is not necessary for a well flotable coal like Brazilian national coal.
5. Apart from improved recovery usage of disinfectant oil as frother reduced, the consumption of diesel oil.
6. Another important fact is that this is first time in Brazil a kind of studies is done using the by-products of coke-oven plant for the beneficiation of national coal, which are the internal products of C.S.N. This process of using the by-products of coke-oven plant as frother has been registered for Brazilian patent.

B I B L I O G R A P H Y

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TABLE I - ANALYSIS OF THE TWO BY-PRODUCTS

	DISINFECTANT OIL	CREOSOTE OIL
CRESOLS (o, m, p)	76,0	N.E.
NAPHTHALENE	4,5	29,5
PHENOL	1,5	9,0
1 METYL NAPHTHALENE	N.E.	4,8
2 METYL NAPHTHALENE	N.E.	17,8
ANTRACENE	N.E.	4,6
β NAPHTHAL	N.E.	4,8

TABLE II - DIESEL OIL + DISINFECTANT OIL

FEED 28 Mx0			FLOAT								TAILS		
TEST	* ASH (%)	S (%) TOTAL	REAGENTS DIESEL	REAGENTS DISINF. Kg/t	RECOVERY (%)	ASH (%)	S (%) TOTAL	Δ ASH (%)**	E**	RECOVERY (%)	ASH (%)	S (%) TOTAL
1	34,05	2,13		0,125	42,2	12,35	1,66	63,7	171	57,8	49,92	2,45
2	34,92	2,33		0,25	47,7	12,13	1,93	65,3	219	52,3	55,70	2,70
3	35,15	2,38	0.25	0,50	55,3	13,94	2,04	60,3	244	44,7	61,38	2,80
4	32,70	2,28	Kg/t	0,80	67,1	15,03	1,92	54,0	307	32,9	68,72	3,00
5	32,05	2,26		1,2	79,3	19,90	2,13	37,9	313	20,7	78,60	2,77
6	31,24	2,31		1,6	82,9	20,78	2,28	33,5	327	17,1	81,94	2,48

TABLE III - DIESEL OIL + CREOSOTE OIL

FEED 28x0			FLOAT								TAILS		
TEST	* ASH (%)	S (%) TOTAL	REAGENTS DIESEL	REAGENTS CREOS. Kg/t	RECOVERY (%)	ASH (%)	S (%) TOTAL	Δ ASH (%)**	E	RECOVERY (%)	ASH (%)	S (%) TOTAL
1	33,85	1,96		0,125	43,50	13,85	2,05	59,1	155	56,50	49,25	1,87
2	33,36	2,24		0,25	45,30	11,89	11,87	64,4	197	54,7	51,73	2,54
3	35,67	2,61	0,25	0,50	53,70	13,86	2,22	61,1	236	46,3	60,94	3,06
4	32,73	2,41	Kg/t	0,80	63,10	15,05	2,18	54,0	264	36,9	62,95	2,80
5	32,22	2,25		1,2	71,7	19,55	2,54	39,3	236	28,3	64,32	1,52
6	33,08	2,31		1,6	73,5	19,03	2,64	42,5	278	26,5	72,04	1,38

TABLE IV - DIESEL OIL + PINE OIL

FEED 28x0			FLOAT								TAILS		
TEST	* ASH (%)	S (%) TOTAL	REAGENTS DIESEL Kg/t	REAGENTS PINE Kg/t	RECOVERY (%)	ASH (%)	S (%) TOTAL	Δ ASH (%)**	E**	RECOVERY (%)	ASH (%)	S (%) TOTAL
1	33,65	3,02	0,125	0,06	57,8	19,4	3,12	42,3	159	42,2	53,2	2,90
2	34,84	2,46	0,25	0,125	83,9	26,98	2,50	22,6	235	16,1	75,66	2,22
3	31,80	2,53	0,50	0,25	89,5	26,26	2,60	17,4	269	10,5	78,80	1,60
4	35,07	2,80	0,75	0,35	86,7	27,65	2,99	21,2	262	13,3	83,47	1,55
5	31,57	2,61	1,00	0,50	88,0	24,38	2,74	22,0	304	12,0	84,36	1,69

* CALCULATED VALUES

** REFER PAGES 05, 06 FOR DEFINITION OF THESE PARAMETERS

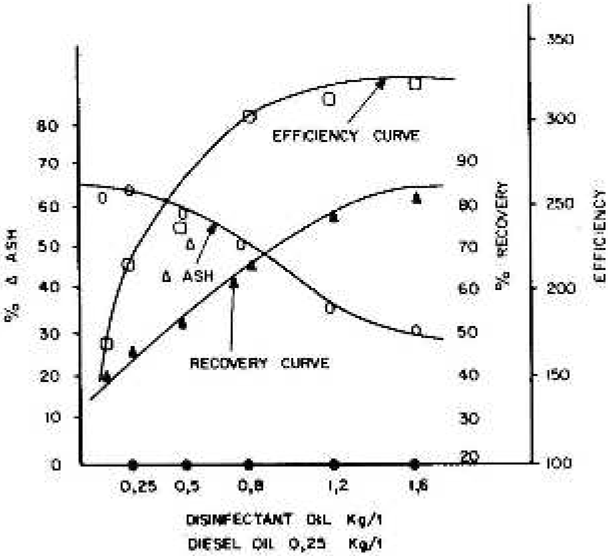

FIG I - CURVES OF RECOVERY, EFFICIENCY AND Δ ASH USING DIESEL OIL AND DISINFECTANT OIL AS REAGENTS.

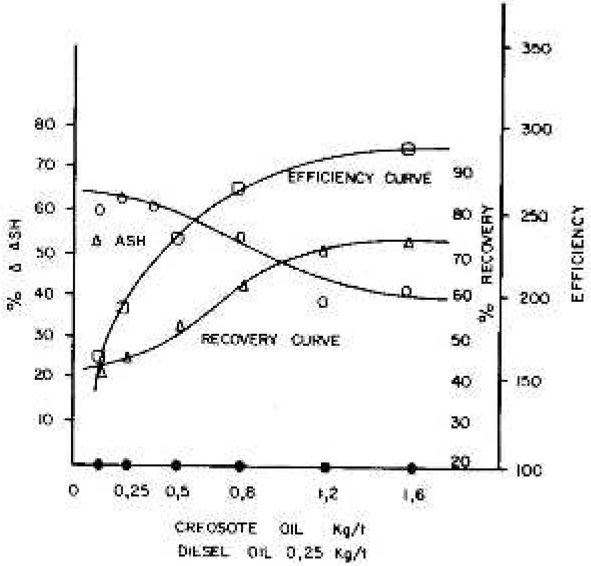

FIG. 2 - CURVES OF RECOVERY, EFFICIENCY AND Δ ASH USING DIESEL OIL AND CREOSOTE OIL AS REAGENTS

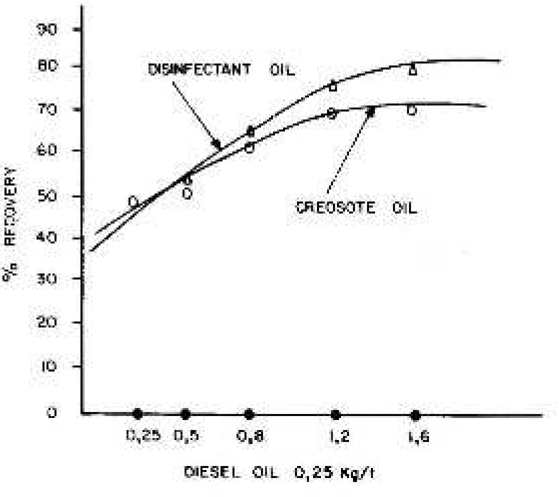

FIG. 3 - COMPARISON OF RECOVERY CURVES OF DISINFECTANT OIL AND CREOSETE OILS

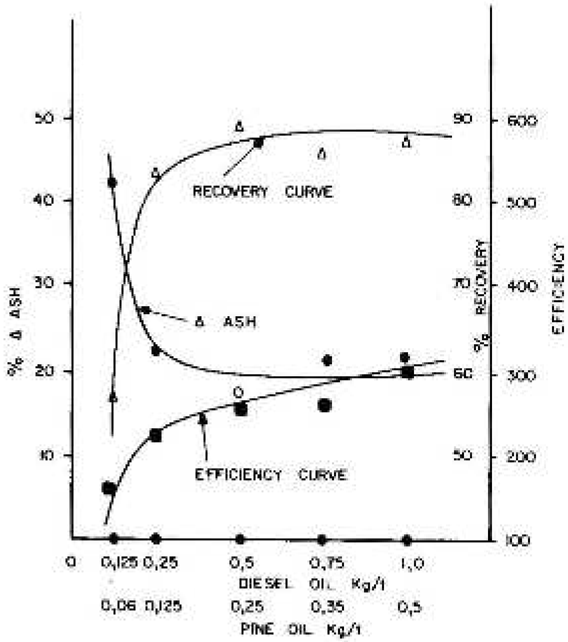

FIG. 4 - CURVES OF RECOVERY, EFFICIENCY AND Δ ASH USING DIESEL OIL + PINE OIL AS REAGENTS