A Comparative Study on the Emulsification of Flotation Reagents*

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This paper presents the results of a comparative study of three coal flotation systems, one currently being used, the others being suggested in this work for the treatment of superfine coal matrix. The matrix is a very fine rich tailings coal, i.e. 54.8 percent ash with 60% of the sample passing through a 46 microns screen. The suggested systems are simply pre-emulsification of the collectors in the cell of froth flotation, prior to the feed being introduced into the flotation unit.

INTRODUCTION

The froth flotation process has been researched and employed in many countries throughout the world, to dress coal. From the reported literature it would appear that coal fines cleaning is almost exclusively by froth flotation.

The complete area of fine coal cleaning is fraught with difficulties. The raw feed to coal flotation plant typically contains large quantities of super fine clays. By the mechanism of slimes coating, these reduce reagent adsorption and result in increased coal losses to the tailings (1). At the same time, clay slimes are entrained in the produced froth and give rise to high ash levels in the coal concentrate; high reagent levels are also apparent.

One means of surmounting the problem of super fine material, is to remove it from the flotation feed by a size separation, at say, 30 microns. This should reduce flotation capacity requirements and improve efficiency (2). However, whilst the efficiency of separation of the -500 microns $+30$ microns fraction is improved, the -30 microns product from the initial separation stage will still contain significant proportion of combustible material. This must be recovered. Two possible methods of achieving this are: The use of oil agglomeration as a complementary process to flotation or; by introducing the reagents to the flotation cells in an emulsion form (i.e. reagent/water emulsion) (3, 4, 5).

The previous work on the emulsification of reagent in coal flotation, although promising in respect of improved flotation efficiency, (i.e. increased flotation machines capacities and some reduction in reagent consumption) was disappointing due to the very high operating and chemicals costs.

Generally, emulsification of oils are carried out by the means of a Jet emulsifier or the use of surfactants (i.e. chemical emulsification) (5, 6, 7).

In the course of the last years, significant advances have been made in the rheology of coal preparation slurry and its effects on the treatment operation (3, 8, 9). In the light of these and from the context above it is apparent that regardless of the efficiency of froth flotation, the final success of this process is mainly dependant upon adequate dispersion of the reagents of flotation. This speci-

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ically the case where slurries of high densities are to be treated, or a shortage of water is to be faced. As such, any improvements in the system of emulsification of reagents could be of vital importance. The present paper describes one simple possible method of producing these significant improvements.

Material and Experimental Procedure

The coal used was very fine rich tailings and had an overall ash of 54.8 percent (dry basis). A 4 percent of this sample was greater than 212 microns in size, while the superfines material (passing 46 microns) comprised 60 percent.

The reagents which were put forward to perform this work, were those which are used industrially in coal cleaning (10, 11, 12, 13). They are Pine Oil as a frother and Oleic Acid, Fuel Oil and Kerosene as collectors.

The froth flotation tests were carried out in a Denver laboratory flotation machine with a capacity of 2 litres.

Hence the initial objectives of this work were to study the effects of emulsification of flotation reagents prior to feeding the coal into the cell with the intention of this system reducing the cost of flotation of very fine coal.

To verify this proposal, it was required that the basic flotation process (i.e. Normal flotation process) should be established in order to identify the standard values of the flotation variables and for comparison with the suggested system. Two modifications for the flotation are proposed here in addition to the basic flotation process were investigated. These methods are emulsified reagent/dry coal and emulsified reagent/slurried coal. The difference between these systems lies in the form in which the coal is fed to the flotation cell. In the former, a dry coal is introduced into the cell following the completion of the emulsification process, while in the latter the feed is slurried coal instead of the dry coal.

The results of the standard conditions for the froth flotation basic process are summarized in Table 1 below.

With regard to the emulsification of the collectors; it should be noted that the flotation cell was initially 3/4 filled with fresh water and then the collector was added, while the impeller was rotating at 1500 rpm and with the air inlet closed. After a half minute the process of emulsification was completed, this was easily determined through the observation of the water color which did become milky. Then the coal and the frother were poured off together into the cell. Mixing was conducted within the cell at 1200 rpm for the conditioning period. This period was found to be shortened to

Table 1. Normal Flotation Conditions

Variable	Value	Variable	Value
Solid Concentration	10% w/v	pH of Pulp	7.2
Conditioning Time	5 mins	Frother Dosage	0.1 lb/ton
Collector Dosage	lb/ton		
1. Oleic Acid	0.15-2	3. Kerosene	0.75-30
2. Fuel Oil	1-30		
Flotation Time	8 mins	Impeller Speed of Conditioning	1200 rpm
Impeller Speed of Flotation Stage	900 rpm		

2 minutes instead of 5 minutes which is established for the basic flotation process. The impeller speed was then reduced to 900 rpm to allow the flotation to proceed. The air inlet was opened. Determination of weight yield and combustible recovery were obtained through filtration, drying and ash analysis of the products.

By adopting the above mentioned emulsification system, it was surprising to find out that the time spent in flotation for the standard method, was reduced from 8 minutes to approximately 4 minutes.

Results and Discussion

The results of the tests are given in Tables 2 to 4 and shown by the comparison of the combustible recoveries (Figs. 1 to 3). In these Tables, columns 4, 5, 6 and 7 show the effects of pre-emulsification of collectors, while columns 2 and 3 give the comparative results for normal reagent additions. It was intended to describe the results of each collector individually. However during the course of the work there was an almost natural progression to update suggestions concerning the effects of pre-emulsification on the process performance when the results of each reagent did become available,

Table 2. Results of Oleic Acid

Oleic Acid Dosage lb/ton (1)	Normal System		Efficiency Emulsified System Dry Feed		Slurried Feed	
	Ash %	Combustible Recovery %	Ash %	Combustible Recovery %	Ash %	Combustible Recovery %
	(2)	(3)	(4)	(5)	(6)	(7)
0.15	21.0	50.30	15.0	83.4	18.0	64.5
0.30	23.0	52.90	14.2	85.8	17.5	67.0
0.60	24.5	56.60	13.5	88.4	17.0	70.0
0.90	26.0	56.48	13.0	88.8	16.8	72.0
1.25	33.5	60.80	13.5	90.5	18.0	72.6
1.50	32.0	60.00	13.0	91.6	19.0	73.5
2.00	34.5	61.50	12.0	92.4	20.0	75.0

Table 3. Results of Fuel Oil

Fuel oil Dosage lb/ton (1)	Normal System		Efficiency Emulsified System Dry Feed		Slurried Feed	
	Ash %	Combustible Recovery %	Ash %	Combustible Recovery %	Ash %	Combustible Recovery %
	(2)	(3)	(4)	(5)	(6)	(7)
1.0	16.0	50.0	13.5	70.0	17.0	56.0
1.25	17.0	53.0	12.8	74.0	16.5	58.0
2.50	18.0	56.2	13.8	89.5	15.0	60.1
5.00	18.5	52.6	12.5	92.7	14.0	63.0
10.00	19.5	53.6	12.0	93.0	13.5	67.0
15.00	19.0	55.0	11.5	93.2	15.0	69.0
20.00	20.0	58.0	11.2	93.2	14.5	71.0
25.00	22.0	60.0	10.2	94.0	15.0	73.0
30.00	22.5	63.3	9.0	96.4	14.5	76.0

Table 4. Results of Kerosene

Kerosene Dosage lb/ton (1)	Normal System		Efficiency Emulsified System			
	Ash % (2)	Combustible Recovery % (3)	Dry Feed		Slurried Feed	
			Ash % (4)	Combustible Recovery % (5)	Ash % (6)	Combustible Recovery % (7)
0.75	15.0	51.2	13.2	89.4	14.5	70.0
1.50	16.0	51.8	13.0	91.0	14.0	73.5
2.50	16.5	52.0	12.5	92.0	13.7	75.0
5.00	15.0	51.5	12.0	93.0	13.5	77.0
7.50	16.0	52.0	11.8	93.0	13.0	78.0
10.00	14.0	52.0	11.0	93.3	12.8	79.2
12.50	17.0	53.6	10.0	94.3	12.2	80.0
15.00	18.0	53.8	9.2	94.6	12.0	80.5
20.00	16.0	52.4	9.0	94.6	11.5	81.0
25.00	16.5	52.5	8.5	95.1	11.0	81.0
30.00	15.0	52.0	8.0	95.2	10.5	82.0

which have shown similarity, i.e. all curves follow the normal pattern with regards to reagent dosage, increased reagent gives increased combustible recovery, hence a basic model describing the various effects is proposed.

The main differences between the two sets of results (i.e. of normal and emulsified systems) are that pre-emulsification has improved the efficiency of the process. The general improvements are seen in all of the results. The most prominent feature of these results is in the very dramatic fall in ash content of the concentrate in all collectors systems without any exception. For example, at 1.25 lb/ton Oleic acid, the normal addition gives concentrate with ash content of 33.5%, the same dosage, when the oleic acid was pre-emulsified, produces an ash of concentrate of about 13%, while simultaneously the combustible recovery was increased from 60.8 to 90.5% at the same reagent level. It is apparent also from Figs. 1 and 3 that fuel oil and kerosene, as expected, exhibited selectivity

Figure 1. Fuel Oil

Figure 2. Results of Oleic Acid

Figure 3. Results of Kerosene

towards the coal in a coal/shale matrix. It was not anticipated however that oleic acid (anionic reagent) would also produce a low ash concentrate, since the principal constituent of shale is kaolinite (14). Kaolinite has a plate like structure consisting of negatively charged faces and positively charged edges (15, 16). Anionic reagent such as oleic acid will be attracted to the edges producing a hydrophobic surface on shale, subsequently the shale will report with the froth floats. The results shown in Table 2 do not support this. This is probably due to the fact that shale being completely wetted preferentially by water and in the aqueous phase (i.e. water only) the high shearing rate enables good dispersion of collectors and hence improved coal collection. The mobility of emulsified oil is greater and has improved spreading power leading to pick up the hydrophobic super fine coal particles coupled with improved shale rejection due to the hydrophilic nature that shale exhibits.

In view of the similarities between the three test series; again emulsified oils are more mobile and have improved spreading power rendering coal particles more compatible with oils. Surprisingly

Plate 1. A Proposed Flowsheet for Pre-emulsification Froth Flotation Process

for both systems of kerosene (i.e. normal and emulsified) the selectivity of coal occurred and a good separation was obtained. It is considered that this may have resulted from kerosene being characterised by very low kinematic viscosity, which, of course, will affect the rheology of the flotation pulp. For example, the viscosity of fuel oil is 0.5 stokes, while of kerosene 0.016 at 20°C (16).

These values indicate that the viscosity of fuel oil is 31 times of kerosene at the same temperature.

Finally, some comments with regard to the more highly pronounced selectivity and improved recovery of the dry feeding system for all series of tests of the three collectors. The results of slurried feed indicate ash content of the product being higher than those of dry-feeding system. The corresponding combustible recoveries were low, suggesting that wetting of coal particles prior to the addition of oils had decreased the spreading power of these oils onto the surface of coal particles; hence the coal collection belittled clearly. Prime reasons for this, are the higher resistance offered by the finer coal particles for the replacement of water by collector after being wetted, and the higher moisture retained by capillary attraction, at the same time, within the coarser coal particles. With the dry feeding system, one must assume that both coal and shale particles are very thirsty, when these particles are to be found in an oil/water emulsion, a selective attachment of oil to the coal occurred, shale is not similarly wetted by oil, but being wetted preferentially by water. This should improve selectivity and rise combustible recovery.

Summary

To summarise the results of this work, the successes achieved with pre-emulsification of the collectors are employed to concentrate the fine coal particles, (being a component of a rich tailings fine coal matrix.) are thus:

With the introduction of collectors into the fresh water within the flotation cell, prior to the addition of coal material, (dry or slurried.) the process produced a clean coal with an average ash of 13%, giving a combustible recovery of over 90% for dry feed and of over 70% for the slurried feed.

At economical reagent levels, i.e. 0.15 to 2.5 lb/ton, an increase of between 20% and 34% greater recovery than that obtained from the normal system, was present. At the same time, a decrease in the ash content of the concentrate, varied from 5% to 12%.

Emulsification also increased the flotation rate; i.e. the total period to float this coal was reduced by 50%, allowing

an increase in the capacity of the flotation machines, equal to twice that of the normal standard flotation process.

An emulsification flotation flowsheet (see plate 1) is therefore suggested. As far as practical implication are concerned, the validity of the proposed technique, particularly, relating to the method of emulsification is unquestionable. While oil being emulsified in an aqueous phase by a simple mixer, reagent dispersal will be optimum and energy savings possible.

It is also expected that a reduction in reagent consumption by half, would occur and simultaneously improve the performance of the process.

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