SPHERICAL OIL AGGLOMERATION (SOA)/ COLLOIDAL GAS APHRONS (CGA) FLOTATION

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Abstract The main aim of this experimental work was to develop a separation process using micro-bubbles less than 100 μm in diameter. Micro-agglomerates were produced using 10 kg crude oil/tone feed coal treated compared to 250 kg/tone feed coal treated for conventional spherical oil agglomeration. These micro-agglomerates were separated from the unagglomerated minerals by micro-bubble flotation. Colloidal Gas Aphrons, CGA, were produced by using various surfactants. Sodium lauryl sulphate (SLS) surfactant was chosen for the production of CGA because it is thought that its anionic character would depress the flotation of the mineral particles. The combination of micro-agglomeration followed by "Dry CGA" flotation was an effective process in coal de-mineralisation under laboratory conditions. All the particles were-45 μm in size.

Key Words Spherical Oil Agglomeration (SOA), Colloidal Gas Aphrons (CGA), d_{mmf}, Recovery, Grade, Micro-agglomerates, Spinning Disc, Aphrons Generator, Cleaner, Rougher, Scavenger.

چکیده هدف اصلی از این پژوهش دست یابی به یک روش جداسازی با استفاده از حبابهای میکرونی، کوچکتر از ۱۰۰ میکرومتر است. با بکارگیری ۱۰ کیلوگرم نفت خام، بجای ۲۵۰ کیلوگرم که در آگلومراسیون کروی معمولی بکار می رود، میکرو آگلومرتیها تولید شده اند. این میکرو آگلومرتیها از مواد معدنی آگلومریت نشده در پالپ بوسیله میکرو حبابها شناور گردیدند. با استفاده از سرفکتانتهای مختلف (کف سازها)، CGA، افرونها تولید شدند. سدیم لاریل سولفیت (SLS) برای تولید CGA، بخاطر خاصیت آنیونی SLS که فکر شد از شناور شدن مواد معدنی جلوگیری خواهد کرد بکار برده شد. ترکیبی ازلخته سازی میکرونی و شناورسازی با CGA در شرایط آزمایشگاهی یک فرایند موثر در تغلیظ ذغال سنگ از مواد معدنی دیگر بود. تمام ذرات مورد آزمایش ابعادی کوچکتر از ۴۵ میکرون داشتند.

INTRODUCTION

The environmental protection authorities require that the emission of the sulphur dioxide be controlled in flue gases. Flue gas de-sulphurisation is a costly process. One possibility to overcome this problem, is the use of clean natural gas. However, due to possible government taxation, to conserve the reserve, natural gas usage seems to be economically undesirable, in the long term.

It is possible that coal, because of its abundance, ease of production and comparatively low price, could be used as an alternative source of energy. Before coal can plausibly be used, however, consideration has to be taken to the reduction of pollutants both gaseous and particulate.

One scenario for the production of such coal is to treat it as a mineral using fine grinding to liberate the minerals and froth flotation to separate coal from minerals.

Froth flotation is perceived to have difficulties in separating particles less than 10 μmwhich is the likely size required for complete liberation. Spherical oil agglomeration has been suggested as an alternative. However, SOA has the disadvantage that producing mechanically strong agglomerates requires a high usage of reagents. If lower amounts of agglomerating reagents are used, micro-agglomerates are produced which will disintegrate under sieving and washing. There is a need for a method of separating micro-agglomerates of coal from the unagglomerated mineral in low shear fields. It is thought that the shear fields in a normal froth flotation cell will disintergrate the micro-agglomerates. Therefore, alternative methods have been concentrated on producing bubbles in low shear fields. Possible candidate processes include Electro Flotation, Dissolved Gas Flotation, and the use of Colloidal Gas Aphrons (CGA) Flotation.

COLLOIDAL GAS APHRONS (CGA)

The surfactant solution in which micron-sized gas bubbles are dispersed has been named "Colloidal Gas Aphrons", (CGA). CGAs have been produced in two ways [1]:

1. Venturi method
2. Spinning disc method

The spinning disc method of CGA production is carried out by accurately weighing a known amount of surfactant into a 3000 cm^3 aphron generator. (See Figure 1).

Distilled water is added to the aphron generator (A), containing a surfactant, and the volume in the beaker is made up to 1000 cm^3 mark. The surfactant solution is then agitated with a mechanical spinning disc agitator. CGAs produced after a few seconds of agitation could then be pumped by a peristaltic pump (pump F) into the aphron separation unit (column B). Once in this unit the aphron bubbles rise to the top, clear liquid from the bottom is re-circulated (using pump E) back into the aphron generator.

CGAs are very homogeneous in size when first proudced, but some bubbles, grow at the expense of others with time. According to the Laplace Equation [3], ($\delta p = \frac{2\gamma}{r}$) the pressure in a bubble is inversely proportional to its radius (where γ= surface tension, r = bubble radius). Thus smaller bubbles are at a higher internal pressure than the larger ones which will grow by mass transfer at the expense of the smaller ones following a collision. This occurs entirely by a process of diffusion through the water,

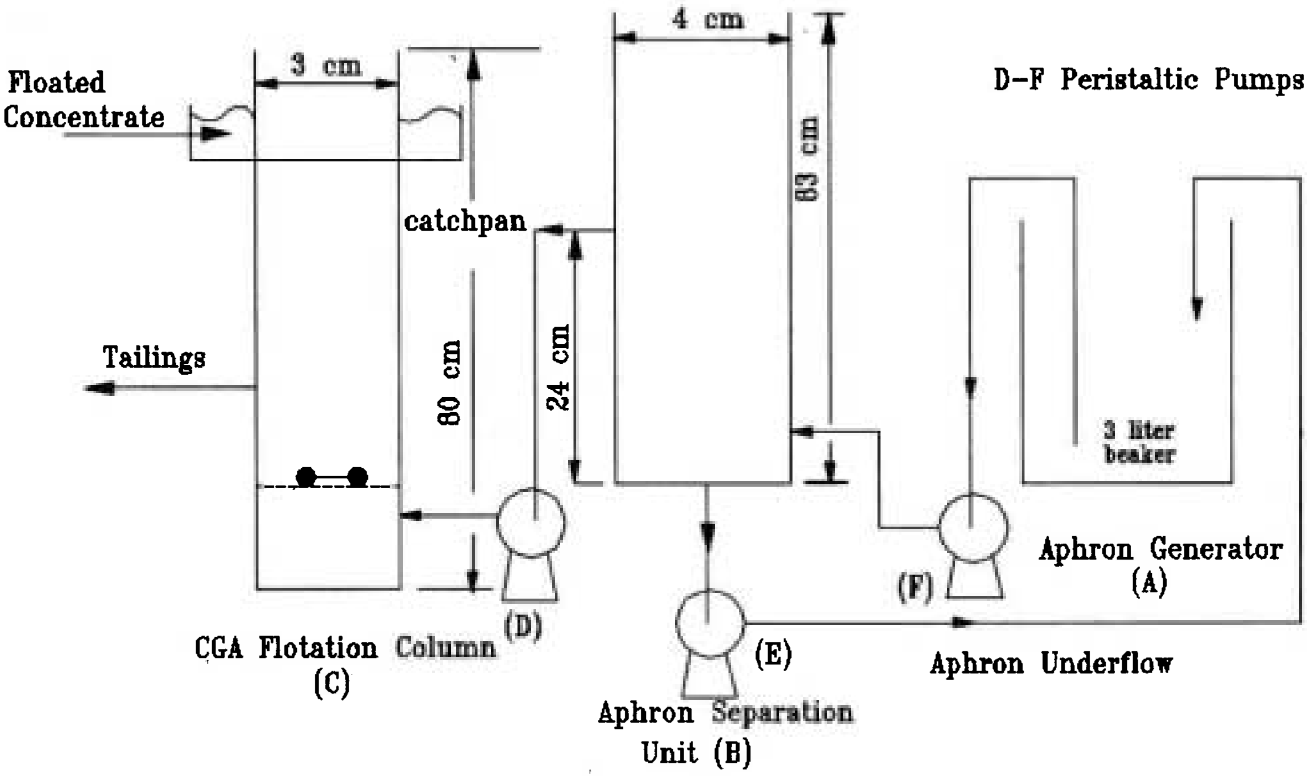

Figure 1. Flowsheet for CGA flotation.

even when the bubbles are not in contact. The barrier to coalescence produced by the soap film is, however, very great and the CGA lifetime is considerable.

CGAs once produced have a lifetime of 10-15 minutes, the limiting factor being their tendency to "cream" because of bubble buoyancy, and thus to form a closed-packed concentrate in which the spherical bubbles gradually form a polyhedral structure. The CGA as formed has a 65% incorporation of gas in water at which point the monodispersed spheres are almost at their closest packing density. At this point the CGA is relatively fluid and can be pumped from its generator to a point of application a considerable distance away.

MECHANISM OF CGA FLOTATION

In CGA there is a distinct separation in properties between the bubble and the water in which it is suspended and there is thus an external surface to the encapsulating soap film. Sometimes small particles can get trapped on this outer surface, similar to the cases where a particle can be seen on the outer surface of a large bubble. Particles trapped in this manner will be buoyed to the surface, but clearly in separation techniques such attachments are not useful. More successful flotation is when particles are entrapped only at one point. Sometimes a mass of solid, larger than a bubble, floats to the surface. The explanation for this is that very small, almost invisible, bubbles coat the solid and provide additional buoyancy needed to raise the solid [3]. In CGA flotation, unlike conventional froth flotation where the hydrophobic particles must penetrate the surface of the bubble and be retained there by the surface acting along the perimeter of contact with the gas within the bubble, the minute bubbles actually attach themselves to the total surface of the solid and cause the buoyancy. Sometimes larger particles have been observed to be rising slowly and then halt and reverse thier direction and start to sink [4]. This occurs because the small bubbles tend to diminish as they do so thier contribution to the buoyancy decreases. This implies that the time taken in CGA flotation is a very important variable.

When bubbles loaded with particles have reached the surface the particle below the surface is usually entrapped by the oncoming bubbles. These bubbles should be stable enough to produce a bed of concentrate which can be then removed over a launder or a catchpan.

In CGA flotation, in contrast to classical froth flotation where a turbulant agitation provides enough momentum for solid and the bubble to rupture the thin film separating the two, a quiescent condition should exist and if agitated the particle is detached from the bubble and, therefore, a decrease in separation efficiency is resulted. It is this reason which necessitates the production of the CGA in a separate generator which is then fed into the flotation column at a controlled rate. This gives a quiet column of bubbles.

Another advantage of the CGAs is thier tendency to clump and produce clusters with a velocity much greater than that of individual bubbles in comformity with Stokes' Law ($v = 2gr^2d/9\eta$) where η = viscosity of the suspension through which the bubble rises, v= the bubble velocity, r = bubble radius, g = acceleration due to gravity, d=water and gas density difference. This high velocity in turn minimises the foaming.

VARIABLES DETERMINATION IN SOA/ CGA FLOTATION AND RESULTS

Significant benefit is achieved by conventional spherical oil agglomeration [5], (See Table 1). The final product consists of de-mineralised coal containing a substantial amount of crude oil.

It is generally asserted that the cost of the agglomerating reagent is the chief disadvantage of the spherical oil agglomeration technique. In this experiment the principles adapted in the practical work on con-

TABLE 1. Conventional Spherical Oil Agglomeration. The Effects of Crude Oil Dosage (g/g Feed) on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%).

Experimental conditions:

solids concentration	= 10% wt
mechanical agitator speed	= 3500 rpm
agglomeration time	= 5 minutes
slurry pH	= natural (6.90)
feed ash content	= 51.25% wt
weight of feed coal	= 50 g
weight of ash in feed coal	= 25.62 g
weight of d_{mmf} coal in feed	= 24.38 g

crude oil dosage (g/50 g feed coal)	concentrates					tailings			∑ C&T
	wt of conc. (g)	ash in conc. (%wt)	wt of d_{mmf} coal in con. (g)	grade (%)	recov. of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (% wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.843	9.53	21.07	7.52	58.89	30.85	38.70	61.03	15.03	22.55
1.69	14.49	18.09	11.87	64.70	48.68	34.18	67.01	11.28	23.15
2.53	19.24	11.00	17.12	78.54	70.24	30.29	77.39	6.85	23.97
3.37	19.79	10.21	17.77	80.08	72.89	29.89	78.60	6.40	24.17
4.22	20.33	9.81	18.34	80.86	75.21	29.43	80.00	5.89	24.23
5.06	21.09	9.38	19.11	81.70	78.39	28.73	82.10	5.14	24.25
5.90	22.11	9.11	20.09	82.22	82.43	27.52	85.50	3.99	24.08
6.74	24.57	9.52	22.23	81.42	91.19	25.27	92.00	2.02	24.25
7.597	25.28	9.93	22.77	80.62	93.39	24.39	93.85	1.50	24.27
8.43	24.61	9.21	22.34	82.03	91.76	25.11	93.00	1.76	24.23
10.96	24.76	8.59	22.63	83.24	92.83	25.03	93.59	1.60	24.23
12.65	25.32	7.98	23.30	84.43	95.57	24.43	96.20	0.93	24.23
15.17	25.08	9.80*	22.62	80.88	92.79	24.70	93.50	1.61	24.23
16.86	24.41	12.10	21.46	76.39	88.01	25.31	89.00	2.78	24.24
25.29	18.70	16.43	15.63	67.94	64.10	26.00	72.40	7.18	22.81

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings

ventional spherical oil agglomeration were exploited. The following variables were experimented and the results are presented in Tables 2 to 6.

1- Screening Results for Choosing a Suitable Surfactant for the Production of CGA Used in SOA/CGA Tests

Three surfactants, sodium lauryl sulphate (anionic), Triton x-405 (non-ionic), and cetyltrimethyl ammonium bromide (cationic) with dosages between 0.00003 to 0.6 g/30 g feed coal (0.0009 to 20 kg/tone) were used in these screening trials. Sodium lauryl sulphate produced the best results under the experimental conditions and was, therefore, used for the CGA production.

2.The Effect of Surfactant Concentration on Ash Content of the Concentrates

From the screening results reasonable reductions in concentrate ash content were achieved, by using sodium lauryl sulphate, at grealtly reduced oil usage.

TABLE 2. The Effects of Surfactant (SLS) Concentration on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%)

Experimental conditions:

solids concentration	= 10% wt
crude oil dosage	= 0.3 g/ 30 g feed
slurry pH	= natural (6.90)
surfactant pH	= natural (4.84)
weight of feed coal	= 30 g
weight of ash in feed coal	= 15.38 g
weight of d_{mmf} coal in feed	= 14.62 g
feed ash content	= 51.25% wt

amount of Surf. used (g/30 g feed)	concentrates					tailings			∑ C&T
	wt of conc. (g)	ash in conc. (%wt)	wt of d_{mmf} coal in con. (g)	grade (%)	recov of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (%wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.0001	11.05	18.92	8.96	63.08	61.28	18.65	73.15	5.01	13.96
0.0003	13.18	16.86	10.96	67.10	74.95	16.60	82.54	2.72	13.68
0.0009	14.63	15.13	12.42	70.48	84.93	15.16	90.91	1.38	13.80
0.0033	16.03	15.09	13.61	70.56	93.10	13.81	95.30	0.65	14.26
0.0053	15.98	16.10	13.41	68.59	91.70	13.90	95.20	0.67	14.08
0.096	15.83	16.29	13.25	68.21	90.64	13.89	94.42	0.78	14.03
0.159	16.00	17.08	13.27	66.67	90.75	13.90	94.25	0.80	14.07
0.315	15.22	18.73	12.37	63.45	84.61	14.59	90.10	1.44	13.81
0.633	14.47	19.56	11.64	61.83	79.61	15.21	85.12	2.26	13.90

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings
SLS = Sodium lauryl sulphate

The following tests were designed to see whether further improvements in beneficiation could be achieved at lower surfactant usage. sodium lauryl sulphate (SLS) concentrations between 0.00015 to 0.633 g/30g feed coal (0.005 to 21 kg/tone) were used. The results are presented in Table 2.

3.The Effects of pH of Surfactant Solution on Ash Content (%wt), Grade (%), and d_{mmf} Coal Recovery (%)

So far all the experimental work, in relation to CGA, has been carried out at a natural pH (4.84). The effects of surfactant pH both for the acidic and alkaline conditions were studied. No significant effects were shown and, therefore, a natural pH was used for the subsequent trials.

4.The Effects of Slurry pH on Ash Content (%wt), Grade (%), and d_{mmf} Coal Recovery (%)

All the experimental work up to this point has been carried out in a natural slurry pH (6.90) regions. The effects of slurry pH, both in alkaline and acidic regions, were further experimented and the results showed no significant effects in the concentrates ash content reductions. A natural pH (6.90) was adapted throughout the study.

TABLE 3. Spherical Oil Agglomeration with Single CGA Flotation. The Effects of Crude Oil Dosage(g/g feed) on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%).

***Experimental conditions*:**

surfactant dosage	= 0.0033 g/30 g feed coal
surfactant pH	= natural (4.84)
solids concentration	= 10% wt
mechanical agitator speed	= 3500 rpm
agglomeration time	= 5 minutes
slurry pH	= natural (6.90)
feed ash content	= 51.25% wt
weight of feed coal	= 30 g
weight of ash in feed coal	= 15.38 g
weight of d_{mmf} coal in feed	= 14.62 g

crude oil dos. (g/30 g feed)	concentrates					tailings			∑ C&T
	wt of conc. (g)	ash in conc. (% wt)	wt of d_{mmf} coal in con. (g)	grade (%)	recov. of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (%wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.03	13.55	21.03	10.70	58.97	73.19	16.25	78.21	3.57	14.27
0.09	14.09	18.13	11.54	64.62	78.90	15.86	82.61	2.76	14.30
0.15	14.11	16.43	11.79	67.94	80.65	15.69	83.46	2.60	14.39
0.21	14.77	15.11	12.54	70.52	85.76	15.13	88.04	1.81	14.35
0.30	14.98	14.53	12.80	71.65	87.57	14.90	89.46	1.57	14.37
0.45	15.25	14.18	13.07	72.33	89.40	14.58	90.95	1.15	14.22
0.60	15.46	13.89	13.31	72.90	91.06	14.79	91.29	1.26	14.57
0.99	15.91	13.91	13.69	72.86	93.69	13.82	93.77	0.86	14.25
1.50	16.09	13.73	13.88	73.21	94.94	13.76	94.88	0.70	14.58

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings

5.Single CGA Flotation-the Effects of Crude Oil Dosage

In this part of the experimental work, by keeping the determined variables constant, the effects of crude oil addition, for dosages between 0.03 to 1.5 g/30 g feed coal (1 to 50 kg/tone), on concentrate ash content, grade and d_{mmf} coal recovery were studied. The results are given in Table 3.

6.Spherical Oil Agglomeration with Multi-stage CGA Flotation

From the results in single stage CGA flotation, it is obvious that some of the carbonaceous constituents of the feed coal are lost in the tailings and some of the non-carbonaceous materials are reported within the concentrates.

In order to recover the coal lost during the CGA flotation and reject the non-carbonaceous constituents trapped within the agglomerates, a plan was devised, (schematically shown in Figure 2), to retreat both the concentrates and the tailings obtained from the first stage, i.e. the Rougher test, in the Cleaner and the Scavenger test, respectively.

7.The Rougher Experimental Results

Three Rougher tests were carried out. The concentrates and the tailings were collected and used as fresh feed in the Cleaner and the Scavenger tests, respec-

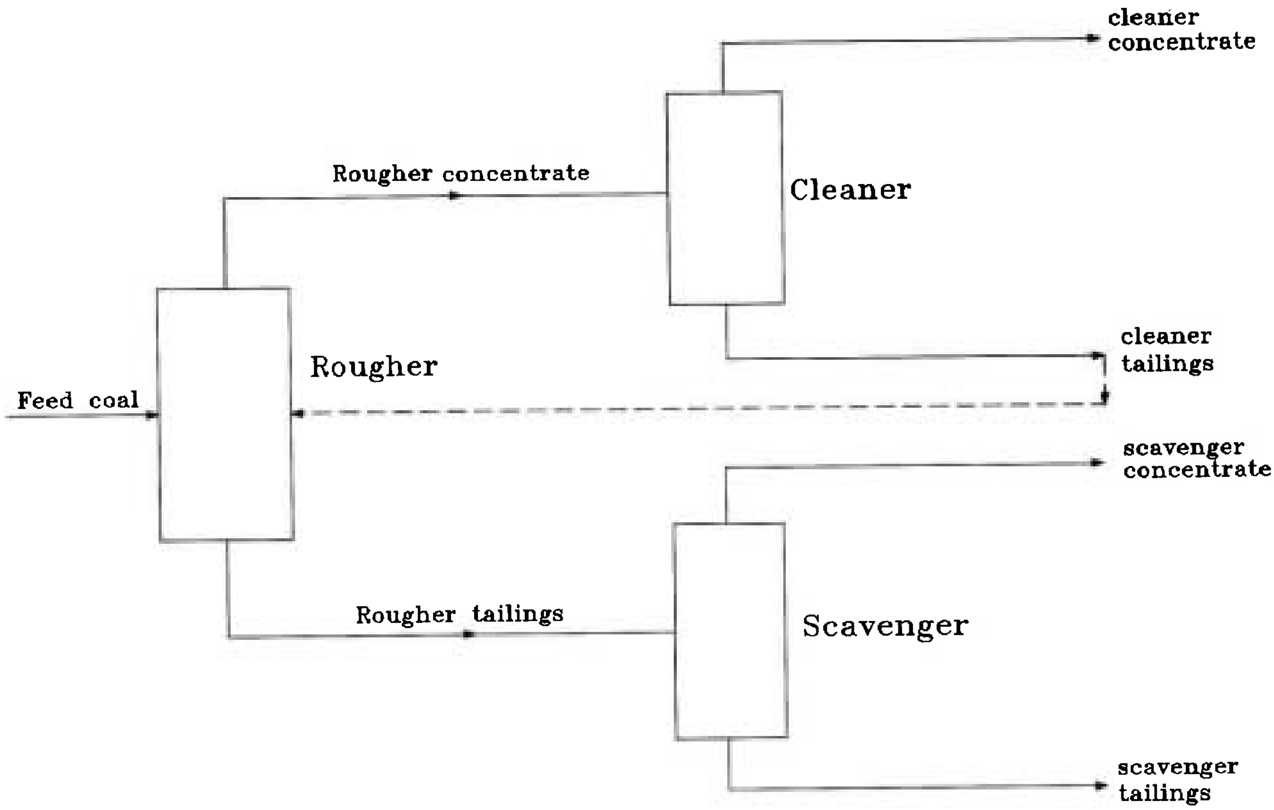

Figure 2. Multistage CGA flotation.

tively.

All the variables determined in the spherical oil agglomeration/CGA flotation were used in this part of the experiment. The Rougher tests results are presented in Table 4. The grade and the d_{mmf} coal recovery are calculated with respect to the mineral content of the feed with an ash content of 51.25% wt.

8. The Cleaner Experimental Results

The concentrates from the Rougher stage were used as fresh feed in the Cleaner test agglomerated with the same amount of the crude oil and surfactant (0.3 and 0.0033 g/30 feed respectively or 10 and 0.1 kg/tone). The results are presented in Table 5.

The grade and the d_{mmf} coal recovery are calculated with respect to the mineral content of the original feed coal with an ash content of 51.25% wt.

9.The Scavenger Experimental Results

The tailings from the Rougher stage were used as fresh feed in the Scavenger test. In Scavenger test, the crude oil dosage was 0.03g/30g feed coal (1 kg/tone) instead of 0.3g/30g feed coal (10kg/tone) as used in the Rougher and the Cleaner experiments. The amount of the surfactant used was also reduced from 0.0033 to 0.0003.30 g feed coal (0.1 to 0.01 kg/tone). The Scavenger test results are given in Table 6. The grade is calculated with respect to the ash content of the tailings in the Rougher tests. The d_{mmf} coal recovey is calculated with respect to the feed coal ash content (51.25% wt).

DISCUSSION

Various variables studied in this experiment. Those which are worth discussing are the amounts of the

TABLE 4. The Rougher Test Effects of CGA Flotation on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%)

Experimental conditions:

surfactant dosage	= 0.0033 g/30 g feed coal
surfactant pH	= natural (4.84)
solids concentration	= 10% wt
mechanical agitator speed	= 3500 rpm
agglomeration time	= 5 minutes
slurry pH	= natural (6.90)
feed ash content	= 51.25% wt
weight of feed coal	= 30 g
weight of ash in feed coal	= 15.38 g
weight of d_{mmf} coal in feed	= 14.62 g

crude oil dos. (g/30 g feed)	Rougher concentrates					Rougher tailings			$\sum$ C&T
	wt of conc. (g)	ash in conc. (% wt)	wt of d_{mmf} coal in con. (g)	grade (%)	reco. of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (%wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.30	15.29	14.18	13.12	72.33	89.75	14.60	91.00	1.31	14.43
0.30	14.93	14.35	12.79	72.00	87.47	15.89	88.81	1.67	14.46
0.30	15.02	14.09	12.90	72.51	88.26	14.81	88.90	1.64	14.54
0.30	15.08	14.21	12.94	72.27	88.49	14.77	89.57	1.54	14.48

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings

reagent and those of the surfactant used. The slurry pH and that of the CGAs did not show any significant effect on the products ash content and, therefore, are not discussed here.

From Table 1, it is obvious that the amount of the reagent used is far too high which is economically undesirable and this is the only disadvantage of the conventional spherical oil agglomeration technique. Although the agglomerating reagent remains with the concentrates and can be used as a source of energy, the reagent consumption prohibits the industrialists in commercialising the process. The use of the surface active reagents improves the efficiency of the process but this is offset by the high consumption of the surfactant.

From Table 3, it can be easily seen that by reducing the amount of the agglomerating reagent consumption from 250kg to only 10 kg/tone feed coal treated more or less the same grade and d_{mmf} coal recovery are obtained. This is a great advantage to micro-agglomerate formation and CGA flotation.

CONCLUSIONS

By using conventional spherical oil agglomeration technique in coal de-mineralisation at oil to coal ratio of 0.25 to 1 a significant ash reductions has been reported by various authors [6-9, etc.]. Agglomerates produced are strong enough to withstand sieving and washing under the tap water. When surfactant (SLS) was used, a considerable reduction in the agglomerating reagent was made for the same d_{mmf} coal recovery. In order to reduce the high amount of the agglomerating reagent even further, micro-agglomerates (by

TABLE 5. The Cleaner Test Effects of CGA Flotation on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%).

***Cleaner experimental conditions*:**

surfactant dosage	= 0.0033 g/30 g feed coal
surfactant pH	= natural (4.84)
solids concentration	= 10% wt
mechanical agitator speed	= 3500 rpm
agglomeration time	= 5 minutes
slurry pH	= natural (6.90)
feed ash content	= 14.21% wt
weight of feed coal	= 30 g
weight of d_{mmf} coal in feed	= 25.74 g
weight of ash in feed	= 4.26 g

crude oil dosage (g/30 g feed)	Cleaner concentrates					Cleaner tailings			∑ C&T
	wt of conc. (g)	ash in conc. (%wt)	wt of d_{mmf} coal in con. (g)	grade (%)	reco. of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (%wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.30	15.81	9.13	14.37	82.19	30.00	14.02	20.80	11.10	25.47

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings

TABLE 6. The Scavenger Test Effects of CGA Flotation on Ash Content (% wt), Grade (%), and d_{mmf} Coal Recovery (%)

***Scavenger experimental conditions*:**

surfactant dosage	= 0.0003 g/30 g feed coal
surfactant pH	= natural (4.84)
solids concentration	= 10% wt
mechanical agitator speed	= 3500 rpm
agglomeration time	= 5 minutes
slurry pH	= natural (6.90)
feed ash content	= 89.57% wt
weight of feed coal	= 30 g
weight of d_{mmf} coal in feed	= 3.13 g
weight of ash in feed	= 26.87 g

crude oil dosage (g/30 g feed)	Scavenger concentrates				Scavenger tailings				∑ C&T
	wt of conc. (g)	ash in conc. (%wt)	wt of d_{mmf} coal in con. (g)	grade (%)	reco. of d_{mmf} coal (%)	wt of tail. (g)	ash in tail. (%wt)	wt of d_{mmf} coal in tail. (g)	d_{mmf} coal (g)
0.03	2.52	49.00	1.29	45.29	4.15	27.29	94.30	1.56	2.85

All analyses given in moisture and oil-free basis, g/g con.
C = concentrates, T = tailings

the time before they cream. In this study CGAs were withdrawn from about a quarter of the way up the Aphron Separation Unit (B) and this comparatively improved the overall efficiency further.

The other advantage of this study over the previous ones [2] was the maximum beneficiation of the carbonaceous constituents of the slurry, i.e. the tailings from the Cleaner stage were re-fed into the Rougher unit. In this manner the optimal non-gangue separation was achieved.

NOTATIONS

d_{mmf} = mineral matter free coal
d = water and gas density difference
p = pressure inside bubbles
γ = surface tension
r = bubble radius
η = suspension viscusity
v = bubble velocity
g = acceleration due to gravity

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