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Application of Dispersion and Selective Flocculation Techniques in the Reduction of Ash from High Ash Coals

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Abstract

The present paper describes the results of dispersion and selective flocculation studies to reduce ash from high ash (≥ 35%) coals from Kuju mines and Chitra mines which represents coking and non-coking coals, respectively.The effect of process parameters namely pH , pulp density(PD), regent dosage on the suspension index(of ash) were studied. In the dispersion studies, the suspension of fine raw coal particles was destabilized to effect the separation by density difference of coal and ash constituents. Sodium hexa meta phosphate, tetra sodium pyro-phosphate and dispersant N6- a low mol.wt. poly-acrylamides supplied by SNF Floerger INC., were used as dispersant. In the selective flocculation studies, destabilized / conditioned samples (using the dispersant mentioned above) at desired Ph was selectively flocculated using starch and modified starch or poly- ethylene oxide. The suspension was fractionated into lower part and upper part. The particles were dried and analyzed for ash. When no dispersant was added in the suspension, no separation9ash content of upper part and lower part remaining same) was noticed at normal Ph, where as there was marginal separation at high Ph. In case of dispersant N6 or inorganic dispersants STPP and SHMP, with the increase in the PD from 1to 4%, an increase in the yield (with same grade) has been observed, whereas at 7% PD, a decrease in the yield has been observed (same grade). An increase in both the yield and grade has been observed with the increase in Ph from low to high. The performance of N6 was better than the STPP and SHMP. There is an increase in yiled (with same grade) with increase in dispersant doses from 0.5 %(wt/wt) to 1.0 %(wt/wt). Cleaning test/Retrieving test improves the yield with appreciable grade. While similar (to Kuju) trend of separation was observed in case of Chitra coal, the separation results (using the dispersants) in this case were inferior to those of Kuju coal. Poly ethylene oxide (PEO), has performed better as selective flocculant than oxidized starch. In general, complexity arises in separation following flocculation due to hetro-coagulation coating of slime on the particle surface.

Keywords: Dispersion, Selective Flocculation, Coal, Ash, Recovery, Grade

1. Introduction

The problem of generation of fine coal particles during mining, comminution and processing has becoming increasingly important in view of the material loss, apart from the severe environmental problems arising out of handling and safe disposal of (these) fines. Equally important is to utilize the high ash coking / non-coking coals in view of their present demand in metallurgical (iron & steel making) and thermal power plants. R&D on various techniques of beneficiation to recover carbonaceous material from these fines as well as reduction of ash from the low grade (high ash) coals have drawn considerable attention of the researchers.

The process of dispersion utilizes the differences in surface chemical properties of various particles in the mixed suspension. It is based on the preferential adsorption of a selective dispersant on the particular minerals to be dispersed, leaving the remainder of the particles in suspension, which is settled under gravity. Much of the earlier work has been carried out in the presence of inorganic dispersants, like sodium silicate and sodium hexametaphosphate (SHMP). In separation system involving clays, SHMP was found to be a better dispersant than sodium silicate due to its higher adsorption that leads to a higher surface charge. Attia & Deason proposed to use of polymeric dispersant such as PEO in enhancing separation selectivity in systems. Certain low molecular weight polymeric dispersant adsorbs at the interface and prevent coagulation due to entropic and mixing interaction. Bhagat et al, have observed that low molecular additives help in improving grinding efficiency, also the separability, of certain types of minerals by reducing hetero-coagulation.

The process of selective flocculation utilizes the differences in the physic-chemical properties of various particles in the mixed suspension. Selective flocculation is based on the preferential adsorption of an organic flocculant on the particular mineral, thereby flocculating them, while leaving the remainder of the suspension particles dispersed.

The process of selective flocculation, in general, consists of following four major steps:

(a) General dispersion of mineral particles, in which all the particles are stably and uniformly distributed in the suspension.
(b) Selective adsorption of the flocculant and floc formation.
(c) Floc conditioning, which aims at obtaining flocs of desired properties for their subsequent separation and with minimum entrapment of dispersed particles.
(d) Floc separation from the suspension.

Selective flocculation is one of the most promising techniques for separation fine/ultrafine minerals. Since the feed to the selective flocculation is fine/ultrafine, the degree of particle liberation is high, and therefore it is theoretically possible to obtain a high grade product by selective flocculation. The principle of selective flocculation as well as underlying surface chemistry, have been reviewed earlier by several authors. However various problems exist both at basic and applied level, which need to be solved before the potential of selective flocculation is fully realized. This is evident from the fact that while good process, the results are not reproducible when actual ore/caol fines are studied. Naturally, the surface contamination, slime coating and change in surface charge etc during the grinding process predominates. In this regard study on actual samples draws a greater significance.

The need for cleaning and recovery of fine coal fraction has increased in recent years, due to possibility of realizing a substantial recovery of ultra-clean coal improving overall BTU recovery and providing extra revenue for the coal industry. The de-ashing of caol by physical beneficiation processes is limited by the degree to which ash components are liberated from coal. The methodology proposed in the present work is dispersion and selective flocculation of the fine coal particles which could address the need for de-ashing the coals, which could be applicable below 100 µ particle size. The product obtained through the combined beneficiation techniques, studied herein, has the potential of its use as coal-water slurry fuel (CWSF) which could replace fuel oil.

2. Materials and Methods

2.1. Material Characterization

Samples from Kuju mines and Chitra mines which represent coking and non-coking coals, respectively were taken for study Table 1 shows the proximate analysis of these coals. X-ray diffraction analysis of the ash from these coal samples show that it consisted of Fe2O3 and qurtz.Zeta potential of the sample (Table 2) shows that these were highly negatively charged having -55 to -58mV at neutral pH.

Sample Name	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
Kuju Coal	1.32	20.85	34.97	44.18
Chitra Coal	3.39	24.60	40.67	34.73

Table 1: Proximate Analysis of Feed Samples

Sample Name	Zeta Potential at different pH					
Kuju Coal (-26)µ	pH	2.3	3.3	5.4	7.5	10.0
	Zeta Potential (mV)	-26.9	-33.2	-47.6	-55.0	-63.5
Kuju Coal (-45+26)µ	pH	2.1	3.1	5.7	9.2	11.3
	Zeta Potential (mV)	-27.9	-36.5	-46.9	-62.4	-71.9
Chitra Coal	pH	2.2	5.3	6.9	8.1	9.2
	Zeta Potential (mV)	-27.0	-53.1	-58.5	-63.1	-77.5

Table 2: Zeta Potential of Coal Samples at Different pH

2.2. Dispersion Studies

In the dispersion studies, the suspension of fine raw coal particles was destabilized to effect the separation by density difference of coal and ash constituents. Following reagents were used:

1. Sodium hexa meta phosphate (SHMP), AR grade
2. Tetra sodium pyro phosphate (STTP) , AR grade
3. Dispersant N6- a low mol. Wt. poly- acrylamides

400 ml suspension of -45µ coal particles desired PD level was stirred for 5 min at high shear. Thereafter, the particles were allowed to settle for 5 min under gravity. After settling, the suspension was separated as upper part (300 ml) and lower part (100 ml). The particles were dried and analyzed for ash. In the cleaning test same procedure was used with additional step of cleaning the upper part and lower part generated in the single stage dispersion.

2.3. Flocculation Studies

In the selective flocculation studies, 400 ml of coal samples (-45µ) was destabilized/conditioned µfor 5 min using the dispersant mentioned above at desired Ph Thereafter, selective flocculant was added and the suspension was conditioned for 5 min while slow stirring. The suspension was fractionated into lower and upper part. The particles were dried and analyzed for ash. Following flocculants were used:

1. Starch and modified starch, AR grade
2. Poly ethylene oxide, MW: 1000000; Viscosity of 2% aq. solution at 25 deg. C supplied by Alfa Aesar

3. Results and Discussions

3.1. Dispersion Studies

3.1.1. Effect of Process Parameters

The effect of pH, initial solid concentration (pulp density) and dosage of dispersant on the separation parameters was studied. The separation parameters are defined as follows:

Yield (%): The ratio of amount of cleaner (upper) portion of the coal fines following Dispersion and settling of particles for 5 min to the total coal fines treated and Expressed as percentage.

% Ash Reduced: The ratio of amount of ash reduced (ash content in feed-ash content in Concentrate) to the content in feed and expressed as percentage.

Table 3 shows the results of separation of Kuju coal fines (-45µ size) at 1% and 4% pulp density (PD) without and with using different kinds of organic and inorganic dispersants at neutral and alkaline pH. The dispersant dosage is maintained at 25×10(-4) g per g of solid.

Suspension Conditions	Dispersant Used	Yield (%)	Ash Reduced (%)
Pulp Density: 1% Suspension pH: 7	Without Dispersant	32.8	4.9
	N6 [25*10(-4) g/g]	19.5	18.5
	SHMP[25*10(-4) g/g]	27.0	18.4
	STTP[25*10(-4) g/g]	33.3	16.6
Pulp Density: 4% Suspension pH:7	Without Dispersant	--	--
	N6 [25*10(-4) g/g]	34.7	15.8
	SHMP[25*10(-4) g/g]	36.8	14.0
	STTP[25*10(-4) g/g]	30.5	13.4
Pulp Density: 4% Suspension pH:11	Without Dispersant	31.9	16.0
	N6 [25*10(-4) g/g]	44.9	17.2
	SHMP[25*10(-4) g/g]	34.0	16.0
	STTP[25*10(-4) g/g]	40.8	15.8
Pulp Density: 7% Suspension pH: 7	N6 [25*10(-4) g/g]	20.3	10.4

Table 3: Dispersion Results without and with Using Inorganic and Organic Dispersants

It is apparent from Table 3 that in case of dispersant N6, the increase in PD FROM 1% to 4 % increases the yield (with same grade) from 20% to 35% at 7.0 pH. With further increase in the PD to 7.0%, the yield decreased significantly with marginal decrease in grade. In case of dispersants STPP and SHMP, with the increase in PD from 1% to 4%, marginal increase in yield has been observed. Also grade (% of ash reduced) of the concentrate reduced marginally.

The function of organic dispersant N6 seems to be at par or marginally better compared to the inorganic ones at neutral pH and 4% PD. However, at higher pH (11), significant improvement in the yield was observed when N6 was added, compared to blank condition. The separation in this case was marginally better (yield of the concentrate was higher with similar grade) than those cases when inorganic dispersants were added (Table 3).

Table 4 shows the effect of suspension pH on the separation of Kuju coal fines (-45µ) at 4% PD using various dispersants.

Suspension pH	N6[25*10(-4)ml/g]		STPP[25*10(-4)ml/g]		SHMP[25*10(-4)ml/g]	
	Yield (%)	Ash Reduced(%)	Yield (%)	Ash Reduced (%)	Yield (%)	Ash Reduced (%)
3.1	4.75	13.4	0.4	13.4	0.4	5.4
7.0	34.7	15.8	36.8	14.0	30.5	13.4
11.0	44.9	17.2	40.8	15.8	34.0	16.0

Table 4: Effect of Suspension pH on Separation of -45 µ Kuju Coal Fines at 4% PD

It is apparent from Tables 3 and 4 that pH has significant role in separation. When no dispersant was added in the suspension, no separation (ash content of upper part and lower part remaining same) was noticed at neutral pH and 1% PD, whereas there was separation at 11 pH and 4% PD (Table 3). In case of acidic suspension (pH 3.1), no separation was observed. Yield of the concentrate was nil in case of STTP and SHMP, whereas it was extremely low (4.75%) in case of the organic dispersant (N6). At higher pH (alkaline suspension).the separation was extremely significant. At this pH value, the performance of dispersant N6 was better than those in case of the inorganic dispersants, STTP and SHMP (Table 4). Table 5 shows the effect of dispersant dosage at 4% PD and 7.0 pH.

Dispersant Dosage [25*10(-4)] g/g	N6		STPP		SHMP	
	Yield (%)	Ash Re-duced(%)	Yield (%)	Ash Re-duced(%)	Yield (%)	Ash Re-duced(%)
1.0 Unit	34.7	15.8	36.8	14.0	30.5	13.4
0.5 Unit	25.5	9.8	18.1	2.0	18.0	2.0

Table 5: Effect of Dispersant Dosage on Separation of -45µ Kuju Coal Fines at 4% PD

From Table 5 it is apparent that a significant decrease in yield with significant to marginal decrease in grade was observed when the dispersant dose was decreased from 25×10(-4) g/g to half of it.

3.2. Two Stage Dispersion

Two stage dispersion tests were conducted using dispersant N6 and STTP in order to scavenge the tail part and clean the concentrate further based on the scheme shown in Fig 1. Table 6 shows the results at 11 pH at 25×10(-4) g/g dosage.

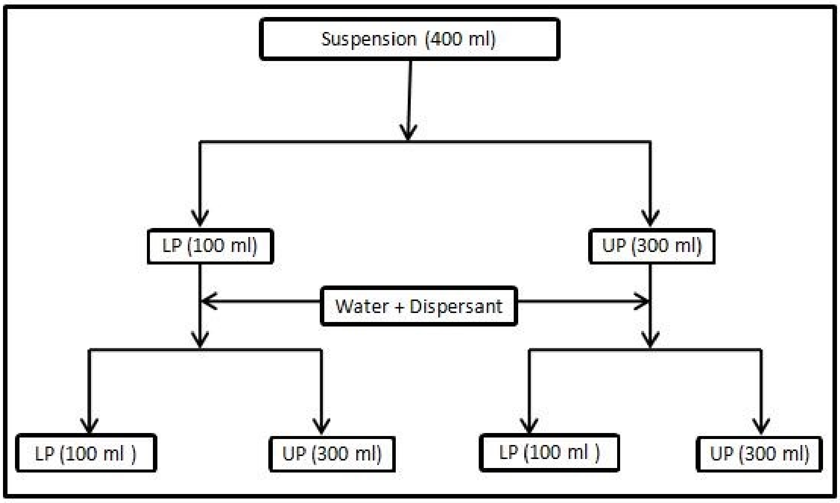

Figure 1: Effect of Two Stage Dispersion on the Separation of Kuju Coal (-45 micron)

Dispersant	1st stage of Dispersion	2nd stage of Dispersant	Wt (%)	Ash (%)	% Ash Removed	Cum. Weight (%)	Cum. Ash (%)
N6	Upper Part(UP)	UP (Stream 1)	20.2	30.3	2.2	51.0 (Str. 1+2+3)	31.3
		LP (Stream 2)	15.4	32.0			
	Lower Part(LP)	UP (Stream 3)	15.4	31.9	20.4	49.0 (Stream 4)	40.1
		LP (Stream 4)	49.0	42.7			
	Total		100.0				
STPP	Upper Part(UP)	UP (Stream 1)	17.5	32.3	1.7	46.6 (Str. 1+2+3)	32.1
		LP (Stream 2)	8.4	34.0			
	Lower Part(LP)	UP (Stream 3)	20.7	31.1	19.9	53.4 (Stream 4)	41.8
		LP (Stream 4)	53.4	41.8			

Table 6: Effect of Two Stage Dispersion on the Separation of -45 µ Kuju Coal Sample

From Table 6, it is apparent that the grade of the concentrate does not improve significantly while cleaning the upper part (that is concentrate) of the first stage of dispersion. One stage scavenging, the lower part (that is trails) of the first stage of dispersion resulted in 25% additional recovery from the tails with the similar (to the upper part) grade.

Typical dispersion experiments were conducted at pH 11 with N6 as dispersant [dosage 25×10(-4) g/g] by splitting the coal sample into two size fractions and treating them separately. Table 7 shows the results at 4% PD and using N6 as dispersant. From this table, it is apparent that better separation results were achieved by splitting the sample in to two size fractions. However, the effect was better in case of finer size fraction.

Feed Size	Yield (%)	Ash Removed (%)
(-45+26)µ	35.0	14.2
-(26)µ	56.7	18.3
Composite Sample	44.9	17.2

Table 7: Effect of Feed Size on the Separation at 4% PD and N6 as Dispersant

3.3. Flocculation Studies

Table 8 shows the results of lowering of ash content using N6 as dispersant and oxidized starch as selective flocculent with dispersant dosage was at [25×10(-4) ml/g].

Pulp Density (%)	Suspension pH	Dosage of starch g/g of solid $*10^{(5)}$	Upper part		Lower Part	
			Yield (%)	Ash (%)	Yield (%)	Ash (%)
2	10	12.5	57.1	34.4	42.8	41.4
4	10	12.5	47.5	33.2	52.5	39.4
7	10	7.14	56.8	33.1	43.1	39.6
4	7	12.5	33.3	36.5	66.7	36.2

Table 8: Results of Flocculation Test Using Modified Starch

Table 9 Shows the results of lowering of ash content using N6 as dispersant and poly ethylene oxide (PEO) as selective flocculant.Dispersant dosage was maintained at [25*10(-40 g/g].

Pulp Density (%)	Dispersant Dosage g/g of solid$\times 1^{0(4)}$	Dosage of PEO g/g of solid$\times 10^{(5)}$	Upper Part		Lower Part	
			Yield (%)	Ash (%)	Yield (%)	Ash (%)
4	25	12.5	45.5	36.8	54.5	37.7
4	50	12.5	26.4	37.7	73.6	35.3
7	12	7.14	53.8	35.5	46.2	36.4
7	20	7.14	28.9	35.5	71.1	36.3
7	25	12.5	55.0	34.8	44.9	35.9

Table 9: Results of Flocculation Test Using PEO

From Table 8 it is apparent that ash content in the upper part was lower than that in the lower part and the results were similar to those with the addition of dispersant, N6,only (see Table 6). These facts suggest that oxidized starch, as flocculent, was not effective. In contrast, Table 9 suggest that there was no significant difference between the ash content of the upper part and lower part. These results suggest that there was selectivity in separation in case of PEO, but because of two opposite forces (coal being lighter flocculated to make them settled) acting at a time, we could not get desired result. In case of PEO as flocculent, its dose remaining same, the yield of concentrate (upper part) decreases with increase in the dispersant dose [1-13].

4. Conclusions

I. In case of blank condition (without any dispersant) no separation was noticed at normal pH, whereas there was marginal separation at high pH. When dispersant was added, an appreciable separation was noticed at all pH levels. With increase in pH from low (acidic) to high (basic) an increase in both yield and grade of the concentrate was ha been observed.

II. An increase in PD from 1% to 4% resulted in increase in the yield (with same grade) in case dispersant N6. A further increase in the PD however decreases the yield of the concentrate. In case of dispersants STPP and SHMP, with the increase in the PD from 1% to 4% an increase in yield has been observed. However, the grade was poorer than that observed in case of organic (N6) dispersant. The performance of N6 was better than that of STPP and SHMP.

III. There is an increase in yield (with same grade) with increase in dispersant doses from 0.5 %(wt/wt) to 1.0 %(wt/wt). Cleaning test/retrieving test improves the yield with appreciable grade. Splitting of coal samples (-45µ) into two size fractions and separately treating them resulted in better separation.

IV. While similar (to kuju) trend of separation was observed in case of chitra coal, the separation results (using the dispersants) in this case were inferior to those of kuju.

V. Oxidized starch was not affective as selective flocculant, whereas selectivivity in separation was observed in case of poly ethylene oxide (PEO). In case of PEO as flocculant, keeping the dose same, the yield of concentrate (upper part) decreases with increase in the dispersant dose.

VI. In general, complexity arises in separation following due to hetero coagulation coating of slime on the particle surface. The selection of flocculants as well as optimization of the process parameters need to be carried out through further experiments and using different polymers.

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