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Optimization of LDO Dosage in Flotation Studies on a Fly Ash Sample for Its Utility & Sustainability

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Abstract

The objective of the investigation was to carryout flotation studies on fly ash sample analyzing 26.62 % LOI. 19.92% FC, 67.17 % ash and 2.17 % Moisture, containing mainly silicates and carbonaceous matter with subordinate to minor amounts of iron oxides and alumina minerals, to separate carbon from fly ash, with the non float fly ash concentrate assaying 5% max. fixed carbon (F.C.)/ 7% max LOI focusing on reagent consumption reduction from present 10kg/t 1:1:1mix of LDO, Kerosene and MIBC to as low dosage as possible. Flotation test on – 100 Mesh size ground fly ash sample [D_{80} 100 microns] at29% Solids with 2 stages of cleaning by reverse flotation using1.5 kg/ t LDO as collector and 1.5 kg/t MIBC as frother, yielded a II cleaner carbon float assaying 51.9% LOI with 86% LOI recovery at wt % Yield of 51.3 which can be reused as fuel , while the composite ash concentrate [Non float] assaying 6.62% LOI, and 4.25 %FC with 14% LOI recovery at wt % yield of 48.7 meeting the specifications. Dewatering studies [1] on carbon float yielded unit thickener area of $0.16m^2$/t/day at 0.4 kg/t SUFFLOC–A 1155 [2] on non float ash concentrate yielded unit thickener area of $0.26m^2$/t/day at 1.5 kg/t SUFFLOC – A 1155. The pressure filtration studies at 50% solids on both concentrate and tails thickened pulps yielded cakes of 18% moisture with $0.5t/h/m^2$. The work index of the sample was 9 Kwh/short ton. The sample is amenable to processing as the non float may be used in concrete block pavement industries and the float may be re used back after cleaner flotation in columns to reduce ash content. The evolved process mitigates the problem of vexed disposal of fly ash by reprocessing reusing and recycling besides, reducing reagent consumption during processing. The bulk concentrate produced was found suitable for tile-brick manufacture after evaluation.

Keywords

Fly Ash, Flotation, LDO and Optimisation of Fly Ash Process

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1. Introduction

Fly ash is a finely divided residue resulting from Combustion of pulverized coal or sub-bituminous coal/lignite in thermal power plants and sponge iron plants. Every year about 80 million tons of fly ash is being generated from various thermal power stations and sponge iron plants and their utilization / disposal is of greater concern to the nation. Various studies conducted on fly ash have proved beyond doubt that the ash material has intrinsic properties to emerge as a valuable raw material for high wear resistant ceramic tiles, foam insulation products. light weight refractory, fly ash metal composites/ash alloys continuous casting powders for steel plants, castable synthetic wood, railway sleeper distemper, domestic cleaning powder, ceramic fiber, mosaic titles or glazed facing tiles, fire abatement materials, adsorbent for toxic organics oil well cement, fire bricks, mineral wool and decorative glass. In India, utilization of fly

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ash has increased from 3% in 1994 to 40% in 2011. Mainly it is used in cement brick, road pavements, mine back fill and agriculture sectors as enumerated by works of Nayak and Mishra. (2005). The review of literature on fly ash processing for use in some of the above industries are enumerated by the works of Cao et.al (2002), Surabhi et.al.(2005) and Aruna et.al. (2011) and Ying Huang et.al. (2005). Most of the above work, concentrated on flotation. The objective of the investigation was to carryout flotation studies on fly ash sample to separate carbon from fly ash, with the non float fly ash concentrate assaying 5% max. Fixed carbon (F.C.)/ 7% max LOI with minimum reagent consumption. The present reagent consumption was 15kg/t of collector frother mix of LDO, Kerosene and MIBC.

2. Experimental

2.1. Material

About 80 kg of fly ash sample was received from Tumkur area, Karnataka state with the aim of reducing the reagent consumption in flotation to as minimum as possible and producing a non float ash concentrate assaying max 7% LOI / 5% FC. Light Diesel Oil [LDO] of Indian Oil Co., Ltd., make was used as collector. Methyl iso butyl carbionol [MIBC] of technical grade from NOICL, Vadodara was used as frother. Flocculants from Suyog chemicals, Nagpur were used.

2.2. Equipment

MPE lab mill [175 mm dia x 350 mm] with 10 kg ball charge, Insmart lab flotation machine, Leaf filter and Larox pressure filters, Bond's work index ball mill, Elico pH meter were used.

2.3. Method

Standard feed preparation and sampling methods, laboratory testing methods, mineralogical and assay methods enumerated by hand books were followed. The experimental work has been categorized as characterization, flotation studies, and other auxiliary tests.

3. Results and Discussion

3.1. Characterization Studies

The as received fly ash sample was blackish gray powder with 30% each sand [+0.1mm] and slime [-0.045mm] content respectively. The top size was 6mm and D_{80} was 4mm. The specific gravity was 2.4. The sample assayed 26.62 % LOI. 19.92% FC, 67.17 % ash and 2.17 % Moisture. Mineralogical studies on revealed that silicates and coal (carbonaceous matter) are the major minerals present in the sample with sub-ordinate amounts of hematite and magnetite and alumina minerals. The amenability tests involving desliming, sink – float, magnetic separation indicated that the desired grade ash content could not be produced by sizing, gravity and magnetic concentration. In view of the above, based on works of Surabhi et.al. (2005) and Aruna et.al. (2011) and current suggested practice of flotation, it was decided to concentrate on flotation of carbonaceous matter by collector - frother mix of LDO and MIBC to obtain nonfloat ash concentrate with LOI < 7% / FC <5% with stress on reagent dosage reduction.

3.2. Rod Mill Grindability Tests

The representative portion of 500 Gms of as received sample was stage ground in rod mill with10 kg rod load at 67%S to -30, -100 and -200 mesh size. The size analysis data of as received and stage ground -30,-100 and -200 mesh is shown in Fig 1.

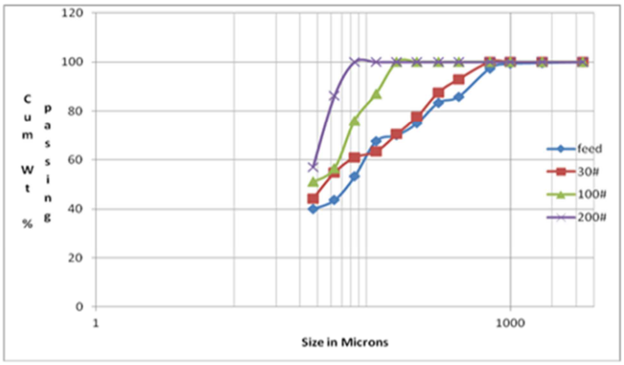

Fig. 1. Size analysis of MOG Products.

3.3. Flotation Tests

Flotation tests were conducted varying MOG, pH, choice of collector, % Solids, collector dosage, number of cleanings to optimize the flotation parameters and initial collector and frother conditioning was done at thick %S of 60.

3.3.1. Flotation Tests Varying Mesh of Grind

Flotation tests were conducted varying mesh of grind -30, -100 and -200 mesh size [D_{80} 220, 92 and 48 microns]. The test conditions and results are given in Table 1. The results indicate that MOG of -100 mesh, D_{80} 92 microns gave optimum results. Flotation at coarser MOG did not yield ash with stipulated LOI/FC grade. Flotation at fine MOG of -200 mesh yielded concentrates with slightly inferior quality probably due to slimes interference. Aruna et.al. [2011] opined that flotation at MOG of -150 mesh yielded better results than flotation of as received sample of -25 mesh size. Similarly Surabhi et.al. [2005] obtained better results at -100 microns. Cao et.al. [2012] recommended - 125 microns as optimum size for flotation of fly ash in columns. float as compared to crystalline graphite. Hence the poor performance of SOKEM collector may be attributed to refractory nature of slimy un burnt carbon to flotation ascompared to crystalline graphite. Aruna et.al [2011] opined that equi-mix of LDO and kerosene gave better results as compared to individual reagents probably due to synergetic effect. Cao et.al. [2002] and Surabhi et.al. [2005] obtained similar results and recommended the use of LDO in combination of frother.

Table 1. Effect of MOG.

Conditions and results: 500 gm Sample Stage ground to varying MOG -30#/-100/-200#

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 Stages	250	1200	LDO MIBC	0.3+0.3+0.3+0.3 0.4+0.4+0.4+0.3	10' each 3' each	3' each

MOG	Products	Wt%	%LOI	
			Assay%	%Dist
-30# D_{80} 220 microns	C. Float(rej)	77.8	29.44	81.8
	Nonfloat(conc)	22.2	22.91	18.2
	Head(Cal)	100.0	27.93	100.0
-100# D_{80} 92microns	C. Float(rej)	80.2	31.62	96.7
	Non float(conc)	19.8	4.38	3.3
	Head(Cal)	100.0	26.23	100.0
-200# D_{80} 48 microns	C. Float(rej)	93.3	27.68	98.6
	Non float(conc)	6.7	5.31	1.4
	Head	100.0	26.18	100.0

Table 2. Choice of collector.

Conditions and results: 500 gm Sample Stage ground to -100 mesh, Flotation pH 8, 29%S

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 Stages	250	1200	Collector MIBC	0.3+0.3+0.3+0.3 0.4+0.4+0.4+0.3	10' each 3' each	3' each

Collector	Products	Wt%	%LOI	
			Assay%	%Dist
1.2 kg/t LDO 1.5kg/t MIBC	C. Float(rej)	80.2	31.62	86.7
	Nonfloat(conc)	19.8	4.38	3.3
	Head(Cal)	100.0	26.23	100.0
1.2 kg/t Kerosene 1.5kg/t MIBC	C. Float(rej)	90.1	26.76	92.0
	Non float(conc)	10.9	19.33	8.0
	Head(Cal)	100.0	26.22	100.0
1.2kg/t SOKEM 705C 1.5kg/t MIBC	C. Float(rej)	82.1	30.29	93.1
	Non float(conc)	17.9	10.24	6.9
	Head	100.0	26.70	100.0

3.3.2. Effect of Pulp Density / % Solids

Flotation tests were done varying flotation 16, /29/, 40 %S at -100 mesh, pH8, 1.2kg/t LDO and 1.5kg/t MIBC. The results are given in Table 3. The results indicate that increase in %S increases the yield of float and removal of carbonaceous

3.3.3. Effect of Collector LDO Dosage Variation

Tests were done varying collector dosage 0.8/1.2/1.8/5.12 kg/t at -100 mesh MOG, pH 8, 29%S and 1.5kg/t of MIBC as frother. Since this was the main objective of experiment, tests were done in duplicate to confirm the results. The test condition and results of effect of LDO collector dosage is given in Table 4. The results indicate that increase in collector dosage reduces the carbon content and yield of Non float. Optimum results were obtained at 1.2kg/t of LDO. Poor results at dosage <0.8 kg/t is attributed lack of collector inrelation to more carbonaceous impurity. Aruna [2011] recommended Collector and frother dosage of 10kg/t and 5kg/t in an effort to float coarse -1mm un burnt coal particles avoiding grinding. They further found that flotation after regrinding to -150 mesh yielded better results Incidentally Cao et al [2002] used 1.2 kg/t of LDO and 0.6 kg/t of frother as optimum value at size of -0.125 mm.. Hence, it may be attributed that reduction of size to normal flotation size of -100 mesh slightly with minimum slime generation may reduce the requirement of frother and collector dosage.

Table 3. Effect of %S.

Conditions and results: 250/500/750 gm Sample Stage ground to -100 mesh, Flotation pH 8

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 Stages	250	1200	LDO MIBC	0.3+0.3+0.3+0.3 0.4+0.4+0.4+0.3	10' each 3' each	 3' each

% Solids	Products	Wt%	%LOI	
			Assay%	%Dist
16	C. Float(rej)	73.6	33.93	94.2
	Nonfloat(conc)	26.4	5.78	5.8
	Head(Cal)	100.0	26.50	100.0
29	C. Float(rej)	80.2	31.62	96.7
	Non float(conc)	19.8	4.38	3.3
	Head(Cal)	100.0	26.23	100.0
40	C. Float(rej)	92.8	28.25	98.8
	Non float(conc)	7.2	4.30	1.2
	Head	100.0	26.53	100.0

Table 4. Collector dosage variation.

Conditions and results: 500 gm Sample Stage ground to -100 mesh, Flotation pH 8, 29%S
Collector dosage varied and added in 4 equal stages for 4 stages of flotation

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 tages	250	1200	LDO MIBC	0.8/1.2/1.8/5.2 0.4+0.4+0.4+0.3	10' each 3' each	- 3' each

Collector dosage kg/t	Products	Wt%	%LOI	
			Assay%	%Dist
0.8	C. Float(rej)	60.0	36.50	82.8
	Nonfloat(conc)	40.0	9.50	17.2
	Head(Cal)	100.0	25.71	100.0
1.2	C. Float(rej)	80.2	31.62	96.7
	Non float(conc)	19.8	4.38	3.3
	Head(Cal)	100.0	26.23	100.0
1.8	C. Float(rej)	95.8	26.42	99.3
	Non float(conc)	4.2	4.55	0.7
	Head	100.0	25.50	100.0
5.2 As per project data	C. Float(rej)	98.4	25.24	99.9
	Non float(conc)	1.6	0.74	0.1
	Head	100.0	24.86	100.0

3.3.4. Effect of PH

Flotation tests were done at natural pH of 8 and 9.5 using Sodium silicate / sodium carbonate as modifiers. The results are given in Table 5. The results indicate that concentrate quality was better at pH 9.5 than natural pH though the yield was low. Aruna et.al. [2011] recommended the use of sodium silicate if the FC content of the float has to be increased though its use is insignificant was low. Aruna et.al. [2011] recommended the use of sodium silicate if the FC content of the float has to be increased though its use is insignificant in further reduction of FC in non float ash concentrate

3.3.5. Test Under Optimum Conditions with Cleanings for Rougher Float

Flotation tests were done at optimum conditions of -100 mesh MOG, pH 8, 29%S pulp density, 1.2kg/t of LDO as collector and 1.5 kg/t of MIBC [in 4 stages]. The rougher float was cleaned thrice employing 0.08kg/t MIBC each in cleaner stage. The results are given in Table 6a The results indicate that the composite non float of rougher, I, II and III cleaners assayed 9.41% LOI at wt% yield of 64.1 failing to meet the specifications of 5%FC or 6.6% LOI. However the Rougher flotation with 2 cleanings in open circuit will yield a composite non float assaying 6.62% LOI at wt% yield of 56.7. The reagent consumption is 1.2kg/t LDO as collector and 1.66kg/t MIBC as frother just meeting the requirements of industry. Aruna et.al. [2011] obtained 66.8 wt% yield concentrate assaying 3.85% FC and 7.45% LOI. meeting the specifications although using 9kg/t LDO, 5kg/t MIBC. The results are significant in reduction of collector –frother mix from 14kg/t to 2.8kg/t by just grinding 1/3 of feed to -100 mesh. Cao et al [2002] obtained using column flotation which is equivalent to 2 cleanings of rougher float in Counter current configuration, a concentrate assaying 2.13% LOI from fly ash sample assaying 15.52% FC. Similar results were obtained by Ying Huang et.al. [2002] who used column flotation to obtain nonfloat with FC <1.5% fly ash with 6% FC feed. The above data clearly indicate that FC content and yield depends on the feed FC also. Test without sodium silicate use is chosen Table 6 a as optimum.

Table 5. Effect of pH.

Conditions and results: 250 gm Sample Stage ground to -100 mesh, pH 8/9.5, 16%S

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 Stages	250	1200	Na_2SiO_3	0/1.5	10'	-
			LDO	0.3+0.3+0.3+0.3	10' each	-
			MIBC	0.4+0.4+0.4+0.3	3' each	3' each

pH	Products	Wt%	%LOI	
			Assay%	%Dist
8	C. Float(rej)	73.6	33.93	94.2
	Nonfloat(conc)	26.4	5.78	5.8
	Head(Cal)	100.0	26.50	100.0
9.5	C. Float(rej)	89.6	29.18	98.1
	Non float(conc)	10.4	4.80	1.9
	Head(Cal)	100.0	26.65	100.0

Table 6. Test with cleanings under optimum conditions.

Conditions and results: 500/250 gm Sample Stage ground to -100 mesh, pH 8/9.5, 29/16%S

Stage	Cell	Rpm	Reagent	Dosage kg/t	C.T(min)	F.T(min)
RF4 Stages	250	1200	Na_2SiO_3	0/1.5	10'	-
			LDO	0.8+0.4+0.3	10' each	-
			MIBC	0.6+0.3+0.2	' each	3' each
ICl F	250	1200	MIBC	0.2	3'	3'
IICl F	250	1000	MIBC	0.2	3'	3'
IIICl F	250	1000	---------	-	-	3'

Products	[a]Test at 29%S, pH 8			[b] Test at 16%S, pH 9.5		
	Wt %	% LOI		Wt %	% LOI	
		Assay %	% Dist		Assay %	% Dist
R. Tails	19.8	4.38	3.3	10.4	4.58	1.8
I Cl. Tails	26.4	7.55	7.3	19. 6	5.97	4.4
II Cl. Tails	10.5	8.49	3.4	10.0	6.21	2.3
III Cl. Tails	7.4	30.83	8.7	8.7	10.51	3.4
III Cl. C Float reject	35.9	56.26	77.3	51.3	45.85	88.1
Head [Cal]	100.0	26.23	100.0	100	26.65	100.0
R+ICl+IICl Tails Conc [C]	56.7	6.62	14.0	40.0	5.66	8.5
R+ICl+IICL+IIICl Tails [C] conc	64.1	9.41	22.7	48.7	6.43	11.9
II Cl C float Co- product[C]	43.3	51.9	86.0	60.0	40.64	91.5

3.3.6. Final Test

Flotation test on – 100 Mesh size ground fly ash sample [D_{80} 100 microns] at 29% Solids as a 2 stages of cleaning by reverse flotation using 1.5 kg/ t each of light diesel oil as collector and t MIBC as frother, yielded a II cleaner carbon float assaying 51.9% LOI with 86% LOI recovery at wt % Yield of 51. , while the composite ash concentrate [Non float] assaying 6.62% LOI, and 4.25 %FC with 14% LOI recovery at wt % yield of 48.7. The test results are given in Table 6 a.

3.4. Auxiliary Tests and Utility of Fly Ash Produced

The auxiliary tests comprised of thickening tests, limited filter tests and Bond's work index test.

3.4.1. Dewatering Tests

Dewatering studies indicated that use of flocculent increased the settling rate. SUFFLOC -A 1155 gave the best results amongst the 6 flocculants tested. Tests on [1] carbon float yielded unit thickener area of $0.16m^2$/t/day at 0.4 kg/t SUFFLOC-A 1155 [2] on non float ash yielded unit thickener area of $0.26m^2$/t/day at 1.5 kg/t SUFFLOC - 1155. A leaf filtration study at 50% solids, 500 mm vacuum on both concentrate and tails thickened pulps yielded cakes of 18% moisture with $0.5t/h/m^2$ using TF24 filter cloth of Dinesh mills.

3.4.2. Bond's Work Index

The Bond's ball mill work index was found to be 9Kwh/ short ton.

3.4.3. Utility of Fly Ash

The bulk fly ash thus produced was tested for its utility at CASHUTEC Raichur and was found suitable. The specifications are given in Tables 7 and 8 respectively &products are shown in Figure 2.

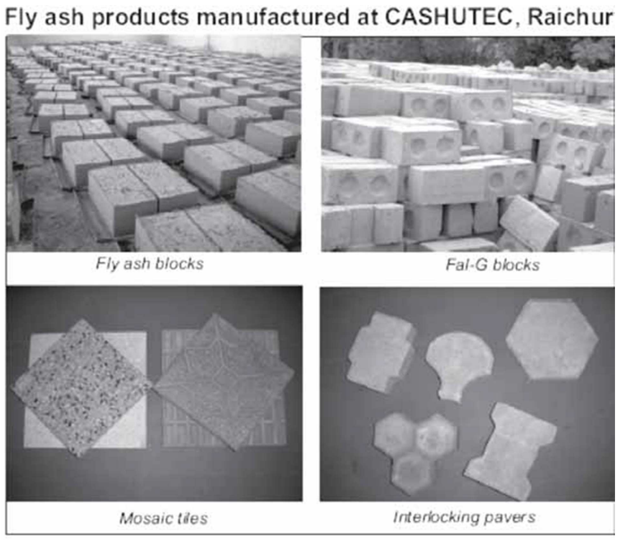

Fig. 2. Bricks, Blocks, Pavement and Mosaic Tiles from Fly ash concentrate.

Table 7. Assay of final concentrate and specifications for tiles & bricks.

Radical	% assay	% assay and specs
SiO_2	27.20	30-62
Al_2O_3	13.62	10 – 30
Fe_2O_3	15.30	6.3 – 14.3
MgO	0.02	0.01 - 0.05
CaO	0.50	0.2 - 0.8
Na_2O	0.10	0.07 – 0.14
FC	4.25	1-5
LOI	6.62	0.2 - 7
pH	8	6 - 8
Wet transverse strength N/mm^2	4.5	>3
Water absorption %	3.3	<10
Abrasion resistance	1.75	<2

4. Conclusions

A fly ash sample when subjected to flotation at -100 mesh [D_{80} 100 microns], 8 pH, 29%S with 2 cleanings, using 1.5kg/t each of LDO and MIBC, yielded a concentrate assaying 6.62%LOI/4.25%FC at 56.7wt% yield meeting the specifications. The process mitigates the problem of disposal of fly ash, reduces LDO consumption and produces products for reuse. The bulk fly ash thus produced was evaluated at CASHUTEC Raichur and were found suitable.

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