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# EFFECT OF THE PARTICLE SIZE ON FLOTATION PERFORMANCE OF A SILICEOUS LIMESTONE SAMPLE

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### Abstract

Laboratory tests were conducted to decipher the effect of the particle size of minerals that influenced the concentration of limestone in terms of grade and recovery by flotation. Both direct as well as reverse flotation experiments were conducted on five different mean particle size ranges on a low grade siliceous limestone sample having CaO 43.09% and SiO2 18.24% from Jayantipuram mine of Andhra Pradesh, India. Direct flotation experiments were carried out using sodium silicate and sodium oleate as depressant and collector respectively. Reverse flotation process was adopted where silica containing minerals of the gangue (quartz) are floated using cationic collector SOMU Sokem 565C. The direct flotation process using sodium oleate as a collector was found to yield better weight percentage as well as distribution than reverse flotation.

Key words: Limestone, Particle size, Sodium oleate, Sodium silicate, Sokem 565C.

# 1. Introduction

In designing a suitable flow sheet for a flotation process, particle size of the sample is of primordial importance. This is determined on the basis of either the mineralogy and/or a careful design of laboratory flotation tests. The effect of particle size on flotation performance has been widely studied [1-6]. It is well established that the flotation rate constant depends on the particle size and that the rate of floatability increases with the decreasing particle size up to a critical level from where it falls down again. Coarse particles may show low grade recovery because of poor liberation of minerals. Above all, coarse particles are affected by the disruption of bubbleparticle aggregate in the turbulent zone [7, 8], a decrease in buoyancy of the particlebubble aggregate in the pulp [9] and may result in longer induction time which results in deterioration in the floatability [6]. In case of fine particles, the flotation show low recovery owing to large surface areas which lead to adsorption of excess reagents and are prone to poor selectivity

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due to mechanical entrainment as well as entrapment of ore with gangue or vice versa. Hence, particle size and flotation processes are intimately linked together. Grinding liberates mineral grains while flotation separates them into valuable minerals and gangue [10, 11]. Hence, a study was undertaken to know the mean particle size where the recovery and grade are at the best. In this paper, the comparative effect of particle size, on direct and reverse flotation process, on a siliceous limestone sample was investigated. Flotation is the ideal approach for beneficiating low grade limestone materials. It can be carried out either by using cationic or anionic collector to float either silica or calcite. The actual technique to be adopted can be decided based on the surface properties and characteristics of the sample. Direct (flotation of carbonate minerals) as well as reverse flotation process (flotation of gangue minerals) was adopted to reduce silica content and enrich CaO. Choice of direct or reverse flotation, later, would be mainly dictated by the overall economics of the process and other considerations.

## 2. Experimental and procedures

The low grade siliceous limestone sample used in this study was obtained from Jayantipuram mine of Andhra Pradesh, India. The bulk chemical composition of the sample is presented in the Table 1. Oleic acid and sodium hydroxide used for preparation of sodium oleate were of laboratory grade reagents. Sokem 565C and sodium silicate (specific gravity 1.49, Na<sub>2</sub>O: 12.04 % and SiO<sub>2</sub>: 31.30 % and Total solids: 43.34 %) used

were of commercial grade. The as received head sample of -5 mm was subjected to roll crusher to 100% passing -10 # (BSS). Then the -10# sample was subjected to ball milling for different durations. Grinding was carried out using laboratory ball mill with 49 steel balls of varying diameter (having weight 5 kg). One kilogram of low grade siliceous limestone sample was dry ground in each batch. The sample was ground in the ball mill for 10, 15, 20, 25 and 30 minutes. At the completion of grinding the contents of the ball mill was thoroughly cleaned and transferred to a container before the next batch of grinding. The products ground for different durations were subjected to sieve analysis as well as particle size analysis using a Laser Diffraction Particle Size Analyzer (CILAS 1180 Model, France made). Bench scale conventional flotation tests were performed in a D12 Denver Flotation Cell manufactured by the Denver Equipment Company, England. The sequence of addition of reagents in the present exercise is mentioned below:

- a) Running tap water (natural pH  $\approx$  7.0) was added first along with the feed at 50% solids by weight during conditioning.
- b) In case of direct flotation, required dosage of depressant for silicate minerals (sodium silicate in solution form) was added to the above and conditioned for three minutes. Later, sodium oleate as a collector was added to the system and further conditioned for three minutes.
- c) Collector of required dosage was added and conditioned for three minutes for reverse flotation.

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- d) Natural pH was maintained in both direct and reverse flotation experiments.
- e) Float and non-float products were filtered, dried, weighed and analyzed for CaO, SiO<sub>2</sub> and LOI.

In direct flotation, the gangue is depressed and the useful mineral is floated. Sodium silicate and sodium oleate were used as depressant and collector respectively. In case of reverse flotation, experiments were carried out using Sokem 565C.

# 3. Results and discussion

#### 3.1. Head sample analysis

The bulk chemical composition of the low grade siliceous limestone from Jayantipuram mine sample is presented in the Table 1.

**Table 1.** Chemical analysis of the headsample

Elements/Radicals	%
LOI	34.32
CaO	43.09
SiO <sub>2</sub>	18.24
$Al_2O_3$	1.53
$Fe_2O_3$	0.94
MgO	0.61
K <sub>2</sub> O	0.35
Na <sub>2</sub> O	0.14
TiO <sub>2</sub>	0.08
MnO	0.03

Chemical analysis of the siliceous limestone head sample, revealed that it contains lime (43.09% CaO) as the major constituent followed by Loss on Ignition

(LOI: 34.32%) and Silica (SiO<sub>2</sub> 18.24%; Table 1). Alumina and iron oxide form the minor constituents while magnesia, alkalies (soda and potash), TiO<sub>2</sub> and MnO<sub>2</sub> were in traces. The details of the geochemistry of these limestone samples have been reported elsewhere [13]. Rao et al., [13] reported mineralogical studies by petrological microscope as well as by Xray diffraction pattern studies and reported that the limestone sample was crystalline and dominantly composed of calcite and quartz. The limestone samples from the Jayantipuram mine are simple in mineralogy, and yet they have variable silica and lime contents. Their reported complete geochemical analysis results indicated that the limestone from the Jayantipuram mine shows a wide range of variations in LOI (29.94% to 40.64%), SiO<sub>2</sub> (6.14% to 27.18%), CaO (37.93% to 50.78%), Al<sub>2</sub>O<sub>3</sub> (0.49% to 2.27%) and Fe<sub>2</sub>O<sub>3</sub> (0.28% to 2.4%). MgO, K<sub>2</sub>O, Na<sub>2</sub>O, TiO<sub>2</sub> and MnO<sub>2</sub> are present in traces. Rao et al [13] also reported that there exists strong positive correlation of CaO with LOI whereas CaO with SiO<sub>2</sub> shows a strong negative correlation because of mineralogical factors.

Chemical analysis data of this low grade siliceous limestone sample indicated that it can not be used directly for cement industries as it does not meet the required specifications of cement making.

According to Indian cement manufacturers specifications, limestone, for cement making, should have more than 45% CaO;  $Fe_2O_3$  as well as  $Al_2O_3$  1 to 2%; free silica less than 8%; combined Na<sub>2</sub>O+K<sub>2</sub>O less than 0.6% and P<sub>2</sub>O<sub>5</sub> less than 0.6%. Magnesia content in the limestone should ideally be less than 3%,

although as high as 5% MgO can be used by the cement industry. Magnesia, sulphur and phosphorus are regarded as the most undesirable impurities. The presence of phosphorus (as  $P_2O_5$ ) slows down the setting time of Portland cement [12]. In order to attain these specifications, low grade siliceous limestone needs to be processed. The low grade limestone sample used for the present purpose has less CaO and more SiO<sub>2</sub> content and all other elements well fit the specified limits. Hence, in this limestone sample if CaO content is increased to more than 45% and  $SiO_2$  content is decreased below 7%, it can be used in clinker making. The quality upgradation of limestone can be done by many ways. One alternative is to use crushed limestone to -3 mm size and process it in scrubber and classifier, while the other entails grinding of raw feed to less than 200 mesh and carry out beneficiation by froth flotation [14, 15]. Similarly, a low grade siliceous limestone sample containing 45.10% CaO and 15.60% SiO<sub>2</sub> have been investigated for amenability to beneficiation by flotation [16]. The reverse flotation experiments conducted by Rao et al., [16] revealed that the siliceous limestone can be beneficiated for manufacture of cement at natural pH water and a reagent (Sokem 565C) dosage of 0.7 kg/t and at 20% percent solids whereas the direct flotation experiments revealed that this siliceous limestone sample can be utilized for cement making by beneficiating with water pH at 7, with sodium silicate dosage of 1.2 kg/t and sodium oleate reagent dosage of 0.2 kg/t. Similarly, Rao et al., [17] studied the effectiveness of sodium silicates different silica-soda modulus of as depressants on a low grade siliceous limestone sample having CaO 45.10%; SiO<sub>2</sub> 15.60% and LOI 36.03%. Their studies indicated that the sodium silicate having silica to soda modulus of 2.19 was better for the flotation for the low grade siliceous limestone sample.

# **3.2.** Flotation studies at different particle size range

# Grinding and particle size analysis:

The as received sample of -5 mm was subjected to roll crusher to 100% passing -10 #. This 100% passing -10 mesh sample was sieved and each sieve fraction was then analyzed (Table 2).

Then the -10 mesh sample was subjected to ball milling for different durations. Their sieve analysis and their distribution in different sieve fractions are presented in Table 3. The particle size analysis was determined by a Laser Diffraction Particle Size Analyzer and the complete size distribution data of all 5 samples (for 10, 15, 20, 25 and 30 minutes ground samples) are presented in Fig. 1.

The conditions of reverse flotation (Table 4) carried out for the present experiments are reagent Sokem 565C (10% solution); Conditioning time=3 minutes; 500 grams of limestone in 1 liter of water; Flotation time= 10 minutes.

The conditions of direct flotation are described in Table 5; sodium silicate (10% solution); sodium oleate (10%); conditioning time=3 minutes; amount of limestone: 500 grams in 1 liter of natural pH water; flotation time=10 minutes. The head sample assay CaO=43.09%; SiO<sub>2</sub>=18.24%; LOI=34.32 w%.

Sieve s	size, mesh	Weight 0/	Assa	ıy %	Distribution %		
(BSS)		weight %	CaO	SiO <sub>2</sub>	CaO	SiO <sub>2</sub>	
-10+18		34.18	43.22	18.01	34.37	34.02	
-18+25		20.42	42.81	18.22	20.34	20.56	
-25+36		6.76	42.87	18.32	6.74	6.84	
-36+52		6.90	42.52	18.84	6.83	7.18	
-52+72		3.96	41.93	19.35	3.86	4.23	
-72+100		3.04	41.17	19.70	2.91	3.31	
-100+150		2.07	40.13	21.34	1.93	2.44	
-150+200	)	0.87	39.93	21.47	0.81	1.03	
-200+240	)	0.69	37.93	24.59	0.61	0.94	
-240+300	)	1.20	39.94	23.44	1.12	1.55	
-300+350	)	1.20		40.02 23.65		1.57	
-350		18.71	44.47	15.78	19.36	16.33	
Head	Assay	100	43.09	18.10			
	Calc.		42.98	18.10			

 Table 2. Assay and distribution percentage of the -10 mesh sieved limestone sample

 Table 3. Sieve analysis of the different time ground sample

Sieve size, mesh	Weight % distribution of samples ground for different times						
(BSS)	10 minutes	15 minutes	20 minutes	25 minutes	30 minutes		
+72	24.43	10.62	3.17	1.28	1.05		
-72+100	10.66	9.59	4.65	2.70	1.68		
-100+150	7.56	10.41	10.77	7.34	7.97		
-150+200	2.17	4.02	5.70	4.26	3.46		
-200+240	2.28	3.61	6.44	5.11	4.30		
-240+350	32.09	25.15	38.75	45.28	18.87		
-350	20.81	36.60	30.52	34.03	62.61		
Total	100	100	100	100	100		
	10 minutes	15 minutes	20 minutes	25 minutes	30 minutes		
Total percentage of -200 mesh	55.18	65.36	75.71	84.82	85.78		
d <sub>80</sub> in microns	53	45	40	40	38		
Mean Particle size in microns	43.96	33.02	25.34	24.89	23.66		



*Figure 1.* Particle size distribution of all the 10, 15, 20, 25 and 30 minutes ground samples

Table 4.	Chemical	analysis of th	ne concentrate	e and tails of the	reverse flotation studies
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Test Grind		Sample	Reagent	Weight		Assay %	Distribution %		
No.	minutes	Description	dosage	%	LOI	CaO	SiO <sub>2</sub>	CaO	SiO <sub>2</sub>
		Concentrate	$0.9 \ln \alpha/t$	34.20	38.68	49.13	9.94	38.50	16.97
1	10	Tails	0.8 kg/t	65.80	31.91	40.79	22.73	61.50	83.04
		Head (Calc.)		100.00	34.22	43.64	18.36		
		Concentrate		54.20	40.72	51.64	5.18	64.77	15.28
2	15	Tails	0.8 kg/t	45.80	25.98	33.25	34.02	35.23	84.77
		Head (Calc.)		100.00	33.97	43.22	18.39		
3	20	Concentrate		55.10	41.17	51.92	4.31	66.98	12.41
		Tails	0.8 kg/t	44.90	24.65	31.41	37.33	33.02	87.57
		Head (Calc.)		100.00	33.75	42.71	19.13		
		Concentrate		65.30	40.85	51.40	3.79	77.57	14.48
4	25	Tails	0.8 kg/t	34.70	21.75	27.96	42.13	22.43	85.52
		Head (Calc.)		100.00	34.22	43.27	17.09		
5		Concentrate		61.70	41.06	51.60	3.75	74.31	12.70
	30	Tails	0.8 kg/t	38.30	22.64	28.74	41.49	25.69	87.30
		Head (Calc.)		100.00	34.00	42.84	18.20		

	Time		Reagent dosage Sodium Sodium		Reagent dosage     Weight     Assay %				Distribution	
Test	of	Sample						ssay /0		%
NO.	minutes	Description	silicate	oleate	%	LOI	CaO	SiO <sub>2</sub>	CaO	SiO <sub>2</sub>
		Concentrate	2 1 / -	0.2  trac/t	49.60	41.25	52.46	4.50	59.95	12.20
6	10	Tails			50.40	27.09	34.48	31.78	40.05	87.20
0	10	Head	2 kg/t	0.2 kg/t	100.00	34.11	43.40	18.25		
		(Calc.)								
		Concentrate			51.80	40.77	52.03	5.60	61.95	15.70
7	15	Tails	2 kg/t	0.2 kg/t	48.20	27.19	34.34	32.33	38.05	84.30
/	15	Head			100.00	34.22	43.50	18.48		
		(Calc.)								
		Concentrate	2 kg/t	0.2 kg/t	66.80	40.39	51.75	6.16	79.30	22.24
Q	20	Tails			33.20	21.24	27.19	43.34	20.70	77.77
0	20	Head			100.00	34.03	43.60	18.50		
		(Calc.)								
		Concentrate	$2 \ln \alpha/t$	0.21rg/t	72.80	39.77	50.38	6.96	84.72	29.06
0	25	Tails			27.20	18.56	24.32	45.46	15.28	70.94
,	23	Head	2 Kg/t	0.2Kg/t	100.00	34.00	43.29	17.43		
		(Calc.)								
		Concentrate	2 kg/t		74.70	39.70	50.33	6.93	86.66	29.77
10	20	Tails		0.21 + 10	25.30	17.93	22.89	48.27	13.34	70.23
	50	Head		0.2 kg/l	100.00	34.19	43.39	17.39		
		(Calc.)								

Table 5. Chemical analysis of the concentrate and tails of the direct flotation studies

The reverse flotation experimental data reveal that the grade of CaO in the concentrate is 49.13% for a 10 minutes grinding time, whereas the 15, 20, 25 and 30 minutes ground sample shows (Fig.2) more or less similar (51.64%; 51.92%; 51.40% and 51.60%) result but the weight percentage (65.30%) and distribution (77.57%) of CaO is highest in case of a 25 minutes grinding time. In case of a 30 minutes grinding time, weight percentage (61.70) and distribution (74.31%) of CaO content in the concentrate falls drastically.

Such a case may be due to over grinding which results in consumption of excess reagent and or as to poor selectivity due to mechanical entrainment as well as entrapment of ore with gangue or vice versa. Similarly the silica content in the concentrate shows a decreasing trend with respect to the time of grinding. The assay percentage of SiO<sub>2</sub> in case of 10, 15, 20, 25 and 30 minutes grinding time is 9.94%; 5.18%; 4.31%; 3.79% and 3.74 % respectively. Based on this it can be inferred that the 25 minutes grinding time leads to the best result in terms of CaO and SiO<sub>2</sub> content in general and recovery in particular.

The direct flotation experimental results indicate that the CaO content of the concentrate shows a decreasing trend with increasing the grinding time. The 10, 15, 20, 25 and 30 minutes ground shows 52.46%; 52.03%; 51.75%; 50.38% and 50.33% respectively (Fig.3).



Figure 2. Particle size versus grade of the concentrate for the reverse flotation



Figure 3. Mean particle size versus grade of the concentrate of direct flotation

But, the  $SiO_2$  content increases when the grinding time increases till 25 minutes and falls down at a 30 minutes grinding time. The direct flotation experimental results indicate that the recovery and distribution of CaO in the concentrate increase with increasing the grinding time (Fig.4).



*Figure 4.* Mean particle size versus weight percentage of the direct flotation concentrate

The weight percentage of the CaO content in the concentrate shows 49.60%, 51.80%; 66.80%; 72.80% and 74.70% for 10, 15, 20, 25 and 30 minutes of grinding time respectively. The CaO distribution of the concentrate shows 59.95%; 61.95%; 79.30%; 84.72% and 86.66% for 10, 15, 20, 25 and 30 minutes respectively. Based on this it can be inferred that the 25 minutes grinding gives rise to the best result in terms of CaO and SiO<sub>2</sub> content in general and recovery in particular. Of course, the 30 minutes grinding time leads

to the best recovery and distribution but the CaO and  $SiO_2$  content in the concentrate is more or less similar to that of the 25 minutes ground sample. As grinding is costlier unit operation, the 25 minutes ground sample is better than the 30 minutes ground sample.

A comparison between direct and reverse flotation process indicates that the direct flotation process using sodium oleate as a collector was found to yield better recovery (Fig.5) and grade of CaO in the concentrate (Fig.6).



*Figure 5.* Weight % distribution versus mean particle size for both direct and reverse flotation



*Figure 6.* Grade of CaO obtained from direct and reverse flotation versus mean particle size for both direct and reverse flotation

#### 4. Conclusions

Flotation experiments were conducted at different particle size (grind) and the present data provide information about the influence of particle size on the flotation of a low grade limestone sample. The data clearly reveal that the separation of calcite from gangue minerals is better when the sample was ground to fine size with a  $d_{80}$ value close to 40 micrometers. A 25 minutes grinding time and reverse flotation using Sokem 565C lead to the best results in terms of CaO and SiO<sub>2</sub> content in general and recovery in particular. Based on the direct flotation data it is clear that the 25 minutes grinding time leads to the best result in terms of CaO and SiO<sub>2</sub> content in general and recovery in particular. Of course, direct flotation following the 30 minutes ground gave the best recovery and distribution but the CaO and SiO<sub>2</sub> content in the concentrate is more or less similar to that of the 25 minutes ground sample. As grinding is costly, the 25 minutes ground sample is better for direct flotation than the 30 minutes ground one. It may be noted that the reagents optimization study may be undertaken for a 25 minutes grinding time or a  $d_{80}$  close to 40 micrometers of sample for better flotation results.

Direct flotation using 2 kg/t of sodium silicate and 0.2 kg/t of sodium oleate has yielded a concentrate having 50.38 % of CaO and 6.96% of SiO<sub>2</sub> with a CaO recovery of 72.80 and 84.72% of CaO distribution. Reverse flotation using 0.8 kg/t Sokem 565C has yielded a concentrate having 51.40 % of CaO and

3.79% of SiO<sub>2</sub> with a CaO recovery of 65.3% and 77.57% of CaO distribution. From the results, it is apparent that the direct flotation process using sodium oleate as collector was found to yield better weight percentage as well as distribution/recoveries. Of course, the direct flotation concentrate has relatively poorer grade than the reverse flotation but the main objective was for utilisation of the sample for meeting the required specifications of cement making. In this regard though the grade of direct flotation is poorer than that of reverse flotation but concentrate of the direct flotation fulfils the specifications of cement making with high recoveries. Hence, direct flotation can be a better choice over reverse flotation in this present case. The relevant cost factors for using the above flotation reagents are also essential parameter for predicting the economic viability of the direct or reverse flotation process.

The study also indicates that the low grade limestone resources can be utilized by adopting flotation. These beneficiation studies has a direct bearing on the Indian cement industry as it presents a scope for extending the life of limestone resources.

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