BENEFICIATION OF FLUORSPAR

FLUORSPAR is an essential mineral in the metallurgical industry and is used as a flux. It is also used in the manufacture of synthetic cryolite, and is the only source for the production of hydrofloric acid for the chemical industry.

Deposits of fluorspar are located in Madhya Pradesh, Rajasthan and Gujarat. The Gujarat Mineral Development Corporation have set up a plant for the beneficiation of fluorspar and before that, they used to hand-pick the flourspar for marketing.

The usual associates of fluorspar are quartz and calcite which are the chief gangue minerals. These two are separated from fluorspar by flotation and subsequent cleanings only.

A good number of low grade fluorspar samples received from different States have been studied for their beneficiation and agglomeration.

A. MADHYA PRADESH FLUORSPAR

A Sample of low grade fluorspar from Chandidongri Mines was received through the Director of State Mining & Geology Department for beneficiation tests. The sample consisted of 125 mm to 150 mm lumps and analysed as follows :

Constituent	*Assay %*
CaF_2	46.25
SiO_2	49.68
Fe_2O_3	0.33
S	0.12
Pb	0.68
Al_2O_3	1.05
$CaCO_3$, Zn & Ba	Trace.
Cu	Nil

Microscopic examination indicated that quartz formed the bulk of the gangue followed by minor amounts of galena which were liberated at 100 mesh size.

Tabling tests with deslimed and sized —8 mesh feed produced a combined fluorspar concentrate analysing 83.81% CaF_2 with 35.6% CaF_2 recovery in it.

Flotation test with —35% mesh ground feed and using 0.5 kg/tonne of sod. carbonate 1.0 kg/tonne of sod. silicate and 1.0 kg/tonne of 1 :1 oleic acid and sod. hydroxide mixture yielded a concentrate analysing 83.28% CaF_2 with 80.5% CaF_2 distribution. When distilled water was used for the test the concentrate analysed 73.43% CaF_2 with 98.3% distribution. Flotation tests at elevated temperature produced a grade of 77.14% CaF_2 with 96.2% distribution with the reagent addition of 0.5 kg/tonne Na_2CO_3, 1.0 kg/tonne sod. silicate and 0.2 kg/tonne oleic acid and sod. hydroxide mixture.

Tests conducted to remove galena by xanthate flotation followed by fatty acid flotation to reject the silicate produced a concentrate analysing 76.58% CaF_2 with 89.7% CaF_2 distribution. The lead concentrate analysed 51.0% CaF_2 with 7.7% CaF_2 distribution in it. When the concentrate was cleaned for four times, the final concentrate analysed 96.6% CaF_2, 0.06% Pb with 77.9% CaF_2 distribution in it.

B. RAJASTHAN FLUORSPARS

A large number of samples of fluorspar from different areas of Rajasthan were investigated for producing acid and metallurgical grade concentrate.

1. Bhagatwali Mine

The sample was composed of lumps ranging in size from 400 mm to 50 mm and analysed as follows :

Constituent	*Assay %*
CaF_2	56.48
SiO_2	34.70
$CaCO_3$	3.58
Pb	0.29
Fe_2O_3	0.96
Al_2O_3	2.46
S	0.06
Ba, Zn, W & Cu	Trace.

Mineralogical examination revealed that quartz, felspar with minor amounts of calcite, micas, galena and zircon were the gangue minerals associated with fluorspar. Gangue was liberated fairly well at 100 mesh size.

Flotation tests under the optimum conditions of 0.75 kg/tonne of sod. silicate, 0.65 kg/tonne of sod. oleate with 75.5% —200 mesh grind feed produced a concentrate analysing 86.92% CaF_2 with 93.2% CaF_2 distribution. Use of warm water during flotation improved the grade to 91.7% CaF_2 with 91.2% CaF_2 distribution.

Rougher concentrate after four cleanings using 0.5 kg/tonne of sod. silicate for each cleaning, analysed 97.66% CaF_2 with 67.3% CaF_2 distribution. With three cleanings only the product analysed 96.84% CaF_2 with 78.2% CaF_2 distribution.

2. Ramorwali Mine

The sample was composed of lumps ranging in size from 300 mm to 50 mm and analysed as follows :

Constituent	*Assay %*
CaF_2	21.14
Insolubles	75.36
SiO_2	58.80
$CaCO_3$	2.10
Al_2O_3	10.02
Pb	0.31
S	0.12
Zn, Ba, Cu, W	Trace.
Fe_2O_3	0.76

Microscopic examination of the sample revealed that the gangue was composed of quartz, felspar and mica with minor amounts of galena, calcite, zircon and pyrite. Fluorspar was liberated from the gangue at 100 mesh size.

Under the optimum flotation condition of 92% —100 mesh feed with 1.5 kg/tonne sod. silicate, 0.5 kg/tonne H_2SO_4 and 0.25 kg/tonne of oleic acid emulsion produced a concentrate assaying 60.37% CaF_2 with 92.7% CaF_2 distribution. With a deslimed feed, the concentrate analysed 82.69% CaF_2 with 81.3% CaF_2 distribution.

When galena was first floated off with 0.1 kg/tonne of pot. ethyl xanthate followed by the flotation of fluorspar under optimum conditions, the concentrate analysed 82.88% CaF_2 with 83.6% CaF_2 distribution. Use of 0.75 kg/tonne of sod. sulphide and 0.04 kg/tonne pine oil during lead flotation followed by fluorspar flotation, the concentrate analysed 77.94% CaF_2 with 85.8% CaF_2 distribution. Use of warm water in a similar test had given a grade of 70.09% CaF_2 with 88.3% CaF_2 distribution.

Reflotation tests conducted with galena free rougher fluorspar concentrate with 2.25 kg/tonne of sod. silicate after four cleanings, yielded a concentrate analysing 96.53% CaF_2 with 49.4% CaF_2 distribution. After three cleanings the product analysed 96.2% CaF_2 with 63.1% CaF_2 distribution.

Metallurgical grade fluorspar assaying 84.9% CaF_2 with 87.2%. Distribution may be produced with one cleaning of the rougher concentrate.

3. Thurwali Mine

The sample was received from the Director of State Mining and Geology. In its "as received" state the sample consisted of 125 to 50 mm lumps and analysed as follows :

Constituent	*Assay %*
CaF_2	22.50
SiO_2	64.65
$CaCO_3$	1.75
Al_2O_3	4.30
Fe_2O_3	2.70
Pb	0.04
S	0.20
Zn, Ba, CU & W	Trace

Microscopic examination of the sample indicated that felspar followed by small quantities of quartz, calcite, mica and traces of pyrite, chalcopyrite formed the gangue minerals. Fluorite was liberated fairly at 100 mesh size.

Flotation tests under the optimum condition of 60% —200 mesh grind, 0.75 kg/tonne of sodium silicate, 0.3 kg/tonne of H_2SO_4 and 0.4 kg/tonne of sodium oleate produced a rougher concentrate analysing 70.0% CaF_2 with 95.0% CaF_2 distribution. Addition of frother like MIBC and Butyl Alcohol helped in reducing the collector to 0.25 kg/tonne with slight improvement in the distribution of CaF_2 in the concentrate.

Reflotation of the rougher concentrate with 0.36 kg/tonne of sod. silicate and 0.75 kg/tonne of collector after three cleanings, the final concentrate analysed 92.15% CaF_2 with 74.8% CaF_2 distribution; with only two cleanings the concentrate analysed 89.4% CaF_2 with 86.1% CaF_2 distribution.

3. Composite Sample

50 tonne composite sample prepared by mixing the low grade fluorspar samples from Ramourwali, Matamgari and Bhagatwali mines was received from the Director of State Mining and Geology for pilot plant tests. The sample consisted of 50 to 75 mm lumps with small qunatity of fines and analysed as follows :

Constituent	*Assay %*
CaF_2	21.2
$CaCO_3$	6.6
SiO_2	67.1
Al_2O_3	1.6
Fe_2O_3	1.8
Pb, Zn, S, Ba	Trace.

Microscopic examination of the sample indicated that bulk gangue was composed of quartz, felspar and calcite with minor amounts of mica and zircon. About 90% fluorspar was liberated at 100 mesh size.

Preconcentration of the sample by H.M.S. tests at sp.gr. 2.6 with —25 mm +6 mesh and —19 mm +6 mesh rejected 36.0% and 40.7% of the feed with 22.5% and 30.4% CaF_2 distribution in the rejects.

Batch tests conducted under different conditions indicated that 75.5% —200 mesh grind, 0.75 kg/tonne of sod. silicate and 0.5 kg/tonne of oleic acid emulsion were optimum wherein a concentrate analysing 66.51% CaF_2 with 94.1% CaF_2 distribution was produced. The rougher concentrate after two cleanings with 0.38 kg/tonne of Katha (Kutch extract/qubracho) yielded a final concentrate assaying 93.1% CaF_2 with 71.2% CaF_2 distribution. Use of Katha for sod. silicate in rougher flotation and also in the cleaning stages improved the grade of final concentrate to 93.4% CaF_2 with 66.7% CaF_2 distribution.

PILOT PLANT TESTS

Continuous pilot plant tests were conducted with the sample. The ore crushed to 7 mesh was fed into a ball mill operating in closed circuit with a spiral classifier. The classifier overflow was sent to the conditioner where 0.75 kg/tonne of sod. silicate and 0.5 kg/tonne of oleic acid emulsion are added to the pulp. After conditioning the pulp was treated in a battery of 6 Nos. of Denver No. 7 cells and the tails were rejected. The concentrate was conditioned with 0.25 kg/tonne sod. silicate and treated in a battery of three cells and the cleaner concentrate was cleaned once again in two cells with an additional 0.13 kg/tonne of sod. silicate. The recleaner tails from both the circuits were recirculated to the conditioner. The concentrate analysed 71.0% CaF_2, 18.63% $CaCO_3$, and 8.13% SiO_2 with 96.0% CaF_2 distribution.

In the second series of the tests 0.13 kg/tonne of Katha was added to each cleaner circuit. The concentrate obtained with 0.45 kg/tonne collector, 0.85 kg sod. silicate and 0.22 kg Katha per tonne analysed 90% CaF_2.

In the third series both sod. silicate and katha were added to the rougher flotation and katha for cleanings. With a total reagent additions of 1.0 kg collector, 0.7 kg sod. silicate and 0.35 kg Katha, the concentrate analysed 93% CaF_2.

As the concentrates produced under different conditions did not come upto the acid grade specification one more cleaning was done with the final concentrate with 0.13 kg/tonne of Katha and the concentrate analysed 97.3% CaF_2, 1.2% $CaCO_3$ and 1.1% SiO_2 with 30.8% CaF_2 distribution.

The tailings of this test analysed 83.1% CaF_2, 8.6% $CaCO_3$ and 5.3% SiO_2 with 48.7% CaF_2 distribution which meets the stipulation of the metallurgical grade of fluorspar.

4. Kahila Sample

The sample was received from the State Industrial Development Corporation for the beneficiation and production of acid grade concentrate. The sample consisted of lumps upto 110 mm down to fines and analysed as follows:

Constituent	*Assay %*
CaF_2	24.60
SiO_2	62.34
$CaCO_3$	0.74
Al_2O_3	5.41
Fe_2O_3	1.14
P_2O_5	0.04
S	0.59
Ba	0.21
Pb & Zn	Trace.

Mineralogical examination indicated that the sample was siliceous in nature, with quartz as the chief gangue constituent, followed by calcite, dolomite, muscovite, barite, ferruginous material etc. The siliceous gangue was liberated from fluorspar at about 100 mesh size.

Rougher flotation tests under optimum, conditions with 60.2% —200 mesh grind, 1.4 kg sod. silicate and 0.8 kg oleic acid emulsion/tonne of ore, a concentrate analysing 75.03% CaF_2, 1.02% $CaCO_3$ and 7.63% SiO_2 with 96.6% CaF_2 distribution was produced. When the rougher concentrate was cleaned for four times and with 0.4 kg/tonne sodium silicate, the final concentrate analysed 97.04% CaF_2 0.61% $CaCO_3$ and 1.14% SiO_2 with 85.3% CaF_3 distribution. This product was suitable for the acid industry.

Use of 0.1 kg/tonne of Katha during third and fourth cleanings, the grade of the concentrate was improved to 98.0% CaF_2, 0.40% $CaCO_3$ and 1.0% SiO_2 with 82.4% CaF_2 distribution.

After 3 cleanings of the rougher concentrate, the product analysed 97.14% CaF_2, 0.61% $CaCO_3$ and 1.5% SiO_2 with 87.7% CaF_2 distribution.

5. Mixed Sample

The sample was prepared by blending the fluorspar samples from Kahila and Mandokipal deposits and was received from the Industrial & Mining Development Corporation. The sample analysed as follows:

Constituent	*Assay %*
CaF_2	18.58
SiO_2	64.80
$CaCO_3$	3.30
Al_2O_3	6.77
Fe_2O_3	1.50
P_2O_5	0.09
S	0.10
Ba	0.17
Pb & Zn	Trace.

The sample contained more of calcite than Kahila sample. Fluorspar was liberated at 150 mesh size.

Rougher flotation concentrate obtained under optimum conditions of 70% —200 mesh grind, 1.5 kg. sodium silicate and 0.8 kg. oleic acid emulsion/tonne analysed 54.8% CaF_2 with 95.1% CaF_2 distribution. When the rougher concentrate was cleaned for six times using 0.41 kg. sod. silicate and 0.1 kg. Katha/tonne, the final concentrate analysed 87.00% CaF_2, 3.0% $CaCO_3$ and 5.93% SiO_2 with 81.3% CaF_2 distribution. Similar test with 93% —200 mesh reground feed yielded a concentrate analysing 90.24% CaF_2, 2.5% $CaCO_3$ and 2.13% SiO_2 with 83.5% CaF_2 distribution.

Addition of 0.5 kg/tonne katha during rougher flotation followed by regrinding and six cleanings with 0.3 kg/tonne katha yielded a concentrate assaying 94.04% CaF_2, 2.5% $CaCO_3$ and 1.6% SiO_2 with 80.3% CaF_2 distribution. Further attempts to improve the grade over 95% CaF_2 and also to reduce the $CaCO_3$ content were not successful.

6. Fluorspar from Mandokipal area (Received from Rajasthan State Industrial & Mineral Development Corporation)

The 15 tonnes run-of-mine fluorspar sample composed of 65 mm to fines and analysed as follows:

Constituent	*Assay %*
CaF_2	8.72
SiO_2	72.25
Al_2O_3	6.77
$CaCO_3$	3.10
Fe_2O_3	1.40
P_2O_5	0.09
S	0.20
Cu, Pb, Zn	Trace
Ba	0.18

Microscopic examination of the sample revealed that fluorspar was the economic mineral and quartz and calcite formed the gangue and were liberated from fluorspar at 100 mesh. Iron oxide coating was found on both fluorspar and gangue minerals.

Preliminary flotation tests indicated that under the optimum conditions of 58% —200 mesh feed employing 1.5 kg/tonne of Sodium silicate and 0.6 kg/tonne of Oleic acid emulsion in three stage produced a concentrate assaying 31.52% CaF_2 with 90.5% CaF_2 distribution in it. Six cleanings of this product using 0.1 kg/tonne of sodium silicate in each stage produced a concentrate assaying 85.5% CaF_2 with 78.5% CaF_2 distribution.

When 0.5 kg/tonne of Katha (Tannin extract) was used for the 5th & 6th cleanings, the grade of the concentrate improved to 91.6% CaF_2 with 63.0% CaF_2 distribution in it. Regrinding the rougher concentrate to 200 mesh followed by six cleanings using Katha in the 5th & 6th stages, further improved the grade to 94.48% CaF_2, 0.9% CaO and 1.8% SiO_2 with 61.5% CaF_2 distribution in it.

7. Fluorspar from Kahila Area (Received from Rajasthan State Industrial & Mineral Development Corporation)

The 10 tonnes sample comprising of 65 to 0 mm sized lumps analysed as follows:

Constituent	*Assay %*
CaF_2	17.56
SiO_2	68.42
Al_2O_3	5.61
$CaCO_2$	0.90
Fe_2O_3	1.10
P_2O_5	0.40
S	0.49
Cu, Pb, Zn	Trace
Ba	0.20

Mineralogy of the sample was similar to that of Mandokipal sample and the liberation size was about 48 mesh size.

Preliminary flotation tests indicated that 58% —200 mesh grind, using 1.5 kg/tonne of sodium silicate and 0.6 kg/tonne of oleic acid emulsion were optimum wherein the rougher concentrate analysed 60.2% CaF_2 with 95.6% CaF_2 distribution in it. Six cleanings of the concentrate with 0.1 kg/tonne of sodium silicate in each stage improved the grade to 94.1% CaF_2, with 83.8% CaF_2 distribution in it.

Addition of Katha in the last two stages of cleaning produced a concentrate assaying 96.1% CaF_2 with 74.6% of CaF_2 distribution. Regrinding of the rougher concentrate followed by six cleanings with Katha in the last two stages yielded a grade of 97.58% CaF_2, 0.4% $CaCO_3$ and 1.2% SiO_2 with 71.2% CaF_2 distribution in it.

Flotation tests with a mixed sample of Kahila and Mandokipal in 1:1 ratio and 2:1 ratio with roughing followed by regrinding and six cleanings with Sodium Silicate and Katha respectively produced concentrates 95.45% CaF_2, 0.5% $CaCO_3$ and 1.8% SiO_2 with 58.7% CaF_2 distribution and 96.12% CaF_2, 0.65% $CaCO_3$ and 1.4% SiO_2 with 61.7% CaF_2 distribution in it.

C. GUJARAT FLUORSPAR

Four different samples including one for large scale pilot tests were received from GSI and Gujarat Mineral Development Corporation for beneficiation studies. All these samples were drawn from the Ambadongar mine in Baroda District.

1. Sample No. 1

This sample was prepared by mixing three different samples from different pits No. 1, No. 3 and No. 8 but similar in mineral composition and liberation size and marked A. Like-wise another sample B was prepared by mixing the samples from pit No. 7 & No. 9. These samples were received from the Director, GSI Rajasthan circle, for preliminary tests for their feasibility to produce acid grade or metallurgical grade concentrates.

Sample A

The sample analysed as follows:

Constituent	*Assay %*
CaF_2	56.30
SiO_2	37.60
Al_2O_3	0.21
Fe_2O_3	3.20
$CaCO_3$	2.10
Pb	0.04
Ba & S	Trace.

Mineralogical examination indicated presence of violet and green coloured fluorspar crystals in

association with quartz, felspar with traces of calcite, apatite, and clinopyroxenes. Fluorite was liberated from the gangue at 65 mesh size.

Flotation test with 75% —200 mesh using 0.5 kg. sodium silicate and 0.75 kg. oleic acid emulsion/tonne, a rougher concentrate analysing 82.83% CaF_2 with 96.7% CaF_2 distribution was produced. After two cleanings with 0.5 kg. sod. silicate, the concentrate analysed 98.01% CaF_2, 0.88% SiO_2 with 77.3% CaF_2 distribution.

Sample B

The sample analysed as follows:

Constituent	*Assay %*
CaF_2	57.20
SiO_2	32.60
Al_2O_3	1.80
Fe_2O_3	3.91
$CaCO_3$	2.20
Pb	0.02
Ba & S	Trace.

Fluorspar was liberated from the gangue composed of quartz, felspar and minor amounts of calcite at a size below 270 mesh.

Rougher flotation tests with 90% —270 mesh feed using 0.5 kg/tonne sodium silicate and 0.75 kg/tonne oleic acid emulsion and followed by one cleaning yielded a concentrate assaying 90.72% CaF_2 with 91.2% CaF_2 distribution. With two more cleanings, the product analysed 97.1% CaF_2 with 75.7% CaF_2 distribution.

2. Fine Grained Fluorspar

One tonne of sample consisting of fines and lumps upto 50 mm was received from G.S.I. for beneficiation tests. The sample analysed as follows:

Constituent	*Assay %*
CaF_2	34.7
SiO_2	56.0
Al_2O_3	1.72
$CaCO_3$	1.70
S	0.30
Fe_2O_3	3.30
Pb	Trace.

Mineralogical examination revealed that quartz followed by minor amounts of felspar , calcite and iron oxides formed the gangue. Traces of galena were also observed. Fluorite was liberated at 150 mesh.

Flotation tests under optimum conditions of 60.7% —200 mesh grind, 0.8 kg sodium silicate and 0.7 kg. oleic acid emulsion yielded a rougher concentrate assaying 63.1% CaF_2 with 95.9% CaF_2 distribution. This product after regrinding to 96% —200 mesh size followed by three cleanings with 1.2 kg. sod. silicate/tonne produced a concentrate assaying 89.9% CaF_2 and 2.0% SiO_2 with 84.8% CaF_2 distribution. One more cleaning of this concentrate with 0.4 kg/tonne sod. silicate yielded a concentrate assaying 95.5% CaF_2 and 1.1% SiO_2 with 69.4% CaF_2 distribution.

Similar test with 0.2 kg/tonne katha for each cleaning in place of sod. silicate and with 4 cleanings produced a concentrate analysing 97.9% CaF_2, 0.16% SiO_2 and 0.7% $CaCO_3$ with 54.0% CaF_2 distribution. The combined tails of 3rd and 4th cleanings analysed 82.4% CaF_2, 6.0% SiO_2 and 3.8% $CaCO_3$ with 28.1% CaF_2 distribution. It may be seen that the combined 3rd and 4th cleaner tails form the metallurgical grade concentrate and the 4th cleaner concentrate forms the acid grade conc. In this case 1st and 2nd cleaner tails were rejected.

Complete replacement of sodium silicate with katha only 0.4 kg. during primary flotation and 0.2 kg. during each cleaning after six cleanings produced a concentrate assaying 96.6% CaF_2, 0.8% SiO_2 and 0.4% $CaCO_3$ with 56.7% CaF_2 distribution. The combined 4th, 5th and 6th cleaner tails analysed 87.0% CaF_2, 6.2% SiO_2 and 1.2% $CaCO_3$ with 22.8% CaF_2 distribution in it. In this case 1st, 2nd and 3rd cleaner tails were rejected.

3. Coarse Grained Fluorspar Sample

The sample was received from GSI Maharastra-Gujarat circle and was composed of lumps upto 75 mm with little fines. Complete chemical analysis of the sample was as follows:

Constituent	*Assay %*
CaF_2	70.0
SiO_2	24.0
$CaCO_3$	1.5
Al_2O_3	0.4
Fe_2O_3	2.1
S	0.2
Pb	Trace.

The gangue was composed of quartz, felspar, and minor amounts of barites and calcite, Most of the fluorspar was free at coarser sizes, but complete liberation was observed only at 150 mesh size.

Jigging tests with the sized —6 mesh feed produced a combined concentrate analysing 88.33% CaF_2 with 19.5% CaF_2 recovery.

Flotation tests under optimum conditions of 75.44% —200 mesh feed, with 0.3 kg/tonne of oleic acid emulsion produced a rougher concentrate analysing 83.8% CaF_2 with 99.6% CaF_2 recovery. After three cleanings the product analysed 96.23% CaF_2 and 2.38% SiO_2 with 95.2% CaF_2 recovery.

Addition of 0.5 kg/tonne of sodium silicate in the primary flotation followed by four cleanings with 0.2 kg/tonne sodium silicate in each stage produced a final concentrate assaying 97.81% CaF_2, and 1.21% SiO_2 with 66.4% CaF_2 distribution. The combined 2nd, 3rd & 4th tails analysed 88.92% CaF_2 and 5.13% SiO_2 with 16.7% CaF_2 distribution.

Jigging tests followed by flotation of the jig rejects with katha only as mentioned earlier produced combined jig conc. and metallurgical grade conc. (2nd and 4th cleaner tails) analysed 88.5% CaF_2 and 7.25% SiO_2 with 32.9% CaF_2 recovery. The final flotation concentrate which was of acid grade analysed 97.81 CaF_2, and 1.21 SiO_2 with 53.4% CaF_2 recovery. By this method the distribution of CaF_2 in the final rejects was reduced from 16.9% in case of flotation to 13.7% i.e. the recovery of CaF_2 was improved by 3%.

Heavy media separation tests at sp. gr. 2.9 with −25 mm +6 mm portion produced a sink product analysing 88.1% CaF_2, and 9.0% SiO_2 with 57.2% CaF_2 distribution. Flotation tests with untreated —6 mesh fines and the float obtained from HMS tests produced a final float concentrate assaying 96.5% CaF_2, and 1.52% SiO_2 with 26.7% CaF_2 distribution. The metallurgical grade concentrate when combined with the HMS sink portion, the product analysed 87.8% CaF_2 and 8.5% SiO_2 with 67.0% CaF_2 distribution. By this method only 6.3% of the total CaF_2 and 79.7% SiO_2 was rejected in the tails.

4. Ambadongar Bulk Sample

The sample was received from M/s. GMDC Ltd. in five different lots collected from different parts of the deposit. The individual samples were blended to form the composite test sample and analysed as follows:

Constituent	*Assay %*
CaF_2	27.99
$CaCO_3$	2.31
SiO_2	49.21
Al_2O_3	8.25
Fe_2O_3	7.40
Ba	0.16
Cu	Trace.
Zn	Nil
Pb	Trace.

The sample consisted of 25 mm lumps and fines with a lot of clayey matter. Mineralogical examination revealed that quartz, felspar and calcite in their order of abundance formed the gangue. Small amounts of barytes and galena were also observed. Gangue and fluorspar were liberated at 100 mesh size.

Associated clayey matter was initially removed by scrubbing and washing and the washed sample was used for the batch and pilot plant tests. By washing, the grade of the sample improved to 28.85% CaF_2 with 7.8% of total CaF_2 loss in the washing slimes.

Batch flotation tests conducted under the optimum conditions of 66.2% —200 mesh grind, 1.5 kg. sodium silicate and 0.6 kg. oleic acid emulsion produced a rougher concentrate analysing 66.18% CaF_2 with 95.0% CaF_2 recovery. Attempts to reduce the S, content of the sample did not produce satisfactorily results, owing to presence of non-sulphide minerals which contribute to S of the ore.

Rougher concentrate after four cleanings without any reagent addition, analysed 91.0% CaF_2, 0.15% S with 77.5% CaF_2 distribution. Use of 0.2 kg. sodium silicate/ tonne in each stage of cleaning, further improved the grade to 92.1% CaF_2 with 74.2% CaF_2 recovery. When the sodium silicate was replaced by 0.1 kg. katha in the first two cleanings each and 0.05 in the 3rd & 4th cleanings each, produced a concentrate analysing 94.06% CaF_2 and 0.10% S with 65.4% CaF_2 distribution.

Rougher flotation with 0.2 kg. katha and 0.6 kg. oleic acid emulsion followed by four cleanings with 0.3 kg. katha/tonne, produced a final concentrate

assaying 95.05% CaF_2 and 0.1% S with 53.2% CaF_2, distribution. Rougher flotation at 92°—94°C with sod. silicate followed by four cleanings with katha had produced a concentrate analysing 93.15% CaF_2 and 0.08% S with 74.2% CaF_2 distribution.

Reflotation tests conducted with the 93% —200 mesh reground feed and four cleanings with katha as in case of earlier tests produced a concentrate analysing 94.3% CaF_2 and 0.1% S with 70.2% CaF_2 distribution. 4th cleaner tails analysed 85.2% CaF_2 with 13.0% CaF_2 distribution.

Further flotation tests with reground feed using 0.5 kg. sod. silicate in the 1st and 2nd cleanings and 0.15 kg. katha in 3rd & 4th cleanings yielded a concentrate assaying 94.25% CaF_2 and 0.1% S with 74.1% CaF_2 distribution. Use of 0.35 kg/tonne of dextrine in place of katha during 3rd & 4th cleanings yielded a grade of 94.5% CaF_2 with 80.4% distribution.

Rougher flotation with sodium silicate and oleic acid emulsion followed by regrinding and two cleanings with sod. silicate, Six cleanings with dextrine and final two cleanings with katha produced a concentrate analysing 97.22% CaF_2, 0.05% S with 62.6% CaF_2 distribution. Refloat tails of 7th to 10th cleaning tests analysed 91.45% CaF_2 and 0.08% S with 13.6% CaF_2 distribution.

Cleaning tests with the reground feed using sod. silicate in the initial two cleanings and another two cleanings with katha with hot pulp yielded a grade of 96.6% CaF_2 and 0.6% S with 75.4% CaF_2 distribution. Similar tests with 4 cleanings with dextrine instead of 2 cleanings with katha after heating produced a concentrate assaying 96.9% CaF_2 and 0.05% S with 77.4% CaF_2 distribution. The combined 5th and 6th recleaner tails analysed 88% CaF_2 with 6.0% CaF_2 distribution in it.

Regrinding of the rougher concentrate followed by two cleanings with sodium silicate and heating and then two cleanings with dextrine and final two cleanings with katha yielded a concentrate assaying 89.2% CaF_2 and 0.03% S with 70.0% distribution. The combined 5th & 6th cleaner tails analysed 90.9% CaF_2 and 0.06% S with 13.4% CaF_2 distribution.

PILOT PLANT TESTS

Continuous pilot plant tests were conducted with the fluorspar sample including washing.

Washing

The ROM ore was crushed to 63 mm in a jaw crusher and stored in the bin. The ore from the bin was fed through a constant weight feedometer on to a belt conveyor which in turn fed the gravel washer fitted with lifters. Water was added at the feed end and in the washer too. The lumps were taken separate and the wash waters were fed to a spiral classifier which separates the slimes and sand. The washed sand and the lumps from the vibrating screen formed the feed for flotation tests.

Flotation

The washed ore was crushed to 58 mm in the primary crusher and then to 13 mm in a cone crusher that operated in closed circuit with a vibrating screen. The crushed 13 mm ore was stored in the bin from where it was fed to the ball-mill (150 cm x 75 cm) —operating in closed circuit with a rake classifier—through a constant weight feedometer. The classifier over flow was sent to the conditioner where the silicate and collector were added. The conditioned feed was treated in a battery of 8 Nos. of No. 8 (2.75 cu. ft. volume) Denver Sub A Cells. The concentrate after thickening in a thickener was reground in a ball mill and the tailings were rejected. The reground concentrate was cleaned twice in batteries of 2 cells each of No. 8 Denver cells and the concentrate was conditioned with Dextrine in a conditioner. The conditioned feed was cleaned twice in batteries of 2 cells of No. 8 type. The tailings of 3rd & 4th cleanings were recirculated to the rougher circuit. The 5th, 6th, 7th and 8th cleanings were done in two Nos. of No. 7 (1 Cu. Ft. Vol.) Denver Sub A cells. The tailings of 5th, 6th and 7th cells were fed back to 3rd cleanings. The tailings of 8th cleaning were of metallurgical grade and were sent to thickener. The 8th cleaner concentrate was further cleaned twice in No. 7 cells using katha where in the final concentrate was of almost acid grade. The 9th & 10th cleaner tails were of metallurgical grade and were combined with the 8th tails.

AGGLOMERATION OF METALLURGICAL GRADE FLUORSPAR CONCENTRATES

Briquetting and pelletization tests were conducted with the metallurgical grade fluorspar concentrate.

Briquetting tests were conducted with different binders like cement, starch, bentonite, lime, sodium

silicate, sulphite lye, and molasses both singly and in combination with one or two. 5% of sulphite lye alone produced a compression strength of 315 lbs/sq. inch. A mixture of 3% sulphite lye, 6% lime and 1% cement improved the strength to 425 lbs/sq. inch. But due to the higher cost and the sulphur content of the sulphite lye, it was not recommended. Briquettes prepared with 8% of molasses alone gave 371 lb/sq. inch. compression strength. But these briquettes without heat hardening at 250°C absorbed moisture and crumpled to produce fines. Briquettes prepared with 4% moisture in combination with 2% lime or 1% cement gave the compression strength of 354 lb/sq. inch. and 373 lbs/sq. inch respectively.

Pelletization tests conducted with the fluorspar concentrate indicated that 5% molasses and 11.75% moisture produced best results of 22.7 and 73.5 knocking resistance from 8" drop and 3" drop respectively for green pellets. Addition of lime and cement did not improve the strength of the green pellets.

Drying /heat hardening tests with the green pellets at different temperatures and varying drying/heating time indicated that 250°C temp. and 30 mts. of heating time were optimum, where the pellets produced a knocking resistance of 21.0 from 6 ft. height. Ageing of the green pellets for 48 hours avoided cracking of pellets due to sudden heating reducing the frying time heating time to 15 mts. to produce a knocking resistance of 28.9 from 6 ft. height. The resistance further improved to 36.7 with 30 mts. of heating time. The fried pellets after ageing to 48 hours did not lose their strength even after 144 hours of exposure to atmosphere which actually resulted in a knocking resistance of 27.7.

Of the two processes studied, pelletizing may be considered to be a more efficient process, for agglomerating fine fluorspar flotation concentrate. Molasses was found to be the cheapest and best of the binders tried.

PELLETIZATION STUDIES ON AMBADONGAR FLUORSPAR CONCENTRATE

Detailed pelletization studies were carried out with the metallurgical grade fluorspar concentrate produced from a low grade fluorspar sample from Ambadongar, Gujarat, received from GMDC. The chemical analysis and sieve analysis of the concentrate is as follows :

Constituent	*Assay %*
CaF_2	87.37
$CaCO_3$	1.52
SiO_2	4.54
Al_2O_3	3.10
Fe_2O_3	2.40
S	0.06

Size	*Wt. %*
+ 65 mesh	—
− 65+100 ,,	0.3
−100+150 ,,	1.7
−150+200 ,,	6.0
−200+250 ,,	6.5
−250+325 ,,	15.3
−325 ,,	70.2
Total	100.0

The bulk density of the dry powder with 1% water was 1.5 tonne/cu. meter and with 4.37% water it was 1.13 tonnes/cu. meter.

Initial pelletising experiments were conducted in a laboratory balling drum using different binders, e.g., sodium silicate, sulphite lye, molasses, bentonite and dextrine with acid washed and unwashed concentrate. Better results were obtained with acid washed product. With 4% sulphite lye as binder, the pellets made from unwashed feed and after oven-drying had a strength of 5.8 kg/pellet compared to 21.6 kg/pellet for the washed feed.

The residual oleic acid collector coating present on the concentrate grains obviously inhibited the binder action and consequently the strength. Among the various conventional binders tested with the acid washed feed, good results were obtained using dextrine and molasses which gave strengths of 27.0 and 36.6 kg for oven dried pellets respectively. For unwashed feed 6% molasses was necessary to give sufficient strength comparable to pellets from washed feed with 4% molasses. Using a larger amount of molasses would, however, be preferable compared to the cost and other problems of washing the feed with acid and rinsing with water, etc.

Using dextrine as a binder, good strength was obtained in pellets made with unwashed feed also (43 kg/pellet), besides very good retention of strength in humid atmosphere (20-25 kg). This was a distinct advantage over molasses, in which case the strength of pellets with molasses was reduced from 40 kgs/pellet to about 5 kg/pellet under similar conditions.

Heat hardening tests were carried out with molasses as a binder at temperature for 200°C to 400°C for duration of 20 to 60 minutes in an electric furnace. Best crushing strength was obtained by heating the air dried green pellets for 30 minutes at 250°C, or 45 minutes at 200° C. At higher temperatures or increasing the retention time beyond the above limits, the crushing strength was hampered due to the burning up of molasses.

After detailed comparative tests with different types of dextrine used as binders dextrine 280 gave the highest strength followed by dextrine 220. Optimum conditions were determined with these binders including drying temp. and heat hardening temp. and given as follows :

Feed Moisture	:	3.5% max.
Binder	:	Prepared from dextrine 280 or 220 in the form 1 :2 or 1 :1.5 solid : water solution.
Quantity	:	2.5%
Moisture content of the final Mix	:	4.5—6.0%
Mixing time	:	4-5 mts/5 kg lots
Moisture content of finished pellets	:	9-10%
Pellet size	:	8-10 mm.
Drying temp.	:	70°-100°C for 25 mts. 100°-150°C for 10 mts.
Heat hardening temp.	:	150°C for 30 mts.

A few large scale pelletisation tests were carried out under optimum conditions on disc pelletiser set at 55° to vertical rotating at 20 rpm. The pellets were heat-hardened in air oven and batch-down-draft grate furnace and aged after drying and heat hardening operation. Dextrine 220 with 2% to 4% gave crushing strength from 25 to 55 kg for oven dried pellets. With the down draft pot-grate heat hardening furnace, the optimum conditions were arrived as follows :

Size of pellets	:	10 mm	12-15 mm
Bed thickness	:	3"	4"
Pressure drop (W.G.) start	:	19"	16"
Average	:	16.5	15
Drying rate 70°-110°C	:	12 mts.	15 mts.
100°-150°C	:	5 ,,	6 ,,
150°-160°C	:	10 ,,	8 ,,
Cooling time	:	1 mt	2 mts.

Tumbling test results showed 73% + $\frac{1}{4}$" fraction for 2% binder which was raised to 96.63% for 4%. Abrasion resistance was correspondingly lowered from 21.4 to 3.26%. Shatter test showed good stability. Bulk density for the pellets varied with size from 1.53 to 1.34 tonnes/cu. meter.

Conclusively pellets with excellent physical properties and storage characteristics could be prepared from metallurgical grade fluorspar concentrate using dextrine based binders and molasses.

Consolidated results of beneficiation on the various fluorspar samples are given in Table 4.3.

TABLE 4.3—SUMMARY OF RESULTS OF BENEFICIATION OF FLUORSPAR SAMPLES

Locality	Head/ROM Assay %	Beneficiation Method (Optimum)	Concentrate obtained Grade %	Recovery %	Remarks
(1)	(2)	(3)	(4)	(5)	(6)
Madhya Pradesh					
1. Chandidungri Mines	CaF_2 = 46.25 SiO_2 = 49.68 Pb = 0.68 (Siliceous in nature) with galena Liberation = 100 mesh.	Fatty acid flotation Sod. silicate 1 kg/tonne Sod. Carbonate 0.5 kg/ton. Oleic acid + NaOH = 1.0 kg/t. Feed = —65 mesh After four cleanings after xanthate flotation.	CaF_2 = 84.07 CaF_2 = 96.6 Pb = 0.06	CaF_2 = 89.5 CaF_2 = 77.9	Galena association. Xanthate followed by fatty acid flotation. Final concentrate Acid grade.
Rajasthan					
1. Bhagatwali Mine	CaF_2 = 56. 48 SiO_2 = 34.70 (Siliceous) Liberation = 100 mesh	Fatty acid flotation Sod. oleate —0.65 kg/t. Sod. silicate —0.75 kg/t. Feed —75% —200 mesh After four cleanings	CaF_2 = 86.92 CaF_2 = 97.66	CaF_2 = 93.2 CaF_2 = 67.3	Metallurgical grade concentrate Acid grade
2. Ramorwali Mine	CaF_2 = 21.14 SiO_2 = 58.8 Pb = 0.31 (Siliceous) with galena pyrite. Liberation = 100 mesh	Xanthate flotation followed by fatty acid flotation. Feed —100 mesh = 97% Rough concentrate: Sod. Silicate = 1.5 kg/t. Oleic acid emulsion = 0.25 kg/t. Sod. sulphide = 0.75 kg/t. Fine oil = 0.04 kg/t. One cleaning Four cleanings	CaF_2 = 77.94 CaF_2 = 84.9 CaF_2 = 96.53	CaF_2 = 85.8 CaF_2 = 87.2 CaF_2 = 49.4	 Metallurgical grade Acid grade
3. Thurwali Mine	CaF_2 = 22.5 SiO_2 = 64.65 (Siliceous) Liberation = 100 mesh	Fatty acid flotation Feed = 60% —200 mesh Sod. silicate = 0.75 kg/t. H_2SO_4 = 0.3 kg/tonne Sod. oleate = 0.4 kg/ton. Rough conc. Two cleanings. Three cleanings	CaF_2 = 70.0 CaF_2 = 89.4 CaF_2 = 92.15	CaF_2 = 95.0 CaF_2 = 86.1 CaF_2 = 74.8	Metallurgical grade
4. Composite sample 50 tonne (Ramorwali, Matamgari and Bhagatwali)	CaF_2 = 21.2 SiO_2 = 67.1 $CaCO_3$ = 6.6 Siliceous with some calcite. Liberation = 100 mesh pilot plant test	Fatty acid flotation Feed = 75% —200 mesh Sod. silicate = 0.75 kt/t. Oleic acid = 0.5 kg/ tonne. Rough conc. Two cleanings with Katha 0.38 kg/tonne Rough conc. Final conc. Re. cl. Tails	CaF_2 = 66.51 CaF_2 = 93.1 CaF_2 = 71.0 $CaCO_3$ = 18.63 SiO_2 = 8.13 CaF_2 = 97.3 $CaCO_3$ = 1.20 SiO_2 = 1.1 CaF_2 = 83.1 $CaCO_3$ = 8.6 SiO_2 = 5.3	CaF_2 = 94.1 CaF_2 = 71.2 CaF_2 = 96.0 CaF_2 = 30.8 CaF_2 = 48.7	Batch and pilot plant tests. Acid grade Metallurgical grade
5. Kahila	CaF_2 = 24.6 SiO_2 = 62.34 Siliceous with carbonates Liberation = 100 mesh	Fatty acid flotation Feed = 60.2% —200 mesh Sod. silicate = 1.5 kg/t. Oleic acid emulsion = 0.8 kg/tonne Rough conc. four cleanings	CaF_2 = 75.03 CaF_2 = 97.04	CaF_2 = 96.6 CaF_2 = 85.3	 Acid grade
6. Mixed sample (Kahila + Mandokipal)	CaF_2 = 18.58 SiO_2 = 64.8 Siliceous with calcite Liberation 150 mesh	Fatty acid flotation Feed = 70% —200 mesh Sod. silicate = 1.5 kg/t. Oleic acid emulsion = 0.8 kg/ton. Rough conc. Six cleanings with Katha	CaF_2 = 54.8 CaF_2 = 94.04	CaF_2 = 95.1 CaF_2 = 80.3	 Acid grade

TABLE 4.3—SUMMARY OF RESULTS OF BENEFICIATION OF FLUORSPAR SAMPLES *(Contd.)*

Locality	Head/ROM Assay %	Beneficiation Method (Optimum)	Concentrate obtained Grade %	Concentrate obtained Recovery %	Remarks
(1)	(2)	(3)	(4)	(5)	(6)
Gujarat					
1. Sample A	CaF_2 = 56.3 SiO_2 = 37.6 $CaCO_3$ = 2.1 Siliceous with calcite. Liberation = 65 mesh	Fatty acid flotation Feed = 75% —200 mesh Sod. silicate = 0.5 kg/t. Oleic acid emulsion = 0.75 kg/t. Rough conc. Two cleanings	CaF_2 = 83.83 CaF_2 = 98.01	CaF_2 =96.7 CaF_2 = 77.3	Metallurgical grade Acid grade
2. Sample B	CaF_2 = 57.2 SiO_2 = 32.6 $CaCO_3$ = 2.1	Fatty acid flotation Feed = 90% —270 mesh Sod. silicate = 0.5 kg/t. Oleic acid emulsion = 0.75 kg/t.			
	Siliceous with calcite. Liberation = = 270 mesh	one cleaning two cleanings	CaF_2 = 90.72 CaF_2 = 97.1	CaF_2 = 91.2 CaF_2 = 75.7	 Acid grade
3. Fine grained sample	CaF_2 = 34.7 SiO_2 = 56.0 $CaCO_3$ = 1.7 Siliceous Liberation = 150 mesh	Fatty acid flotation Feed = 60% —200 mesh Scd. silicate = 0.8 kg/t. Oleic acid emulsion = 0.75 kg/t. Rough conc. Regrind and 3 cleanings 4 cleanings 6 cleanings and Katha cleaner tails	CaF_2 = 63.1 CaF_2 = 89.9 CaF_2 = 95.5 CaF_2 = 96.6 CaF_2 = 87.0	CaF_2 = 95.9 CaF_2 = 84.8 CaF_2 = 69.4 CaF_2 = 56.7 CaF_2 = 22.8	 Metallurgical grade Acid grade Metallurgical grade
4. Coarse grained sample	CaF_2 = 70.0 SiO_2 = 24.0 $CaCO_3$ = 1.5 Siliceous Liberation = coarse size.	Fatty acid flotation Feed = 75.44% —200 mesh Oleic acid emulsion = 0.3 kg/t. Rough conc. Three cleanings **Reflotation:—** Sod. silicate 0.8 kg/t. four cleanings Final conc. Cleaner tails	CaF_2 = 83.8 CaF_2 = 96.23 CaF_2 = 97.81 CaF_2 = 88.92	CaF_2 = 99.6 CaF_2 = 95.2 CaF_2 = 66.4 CaF_2 = 16.7	 Acid grade Metallurgical grade
5. Ambadongar bulk sample	CaF_2 = 27.99 SiO_2 = 49.21 $CaCO_3$ = 2.31 Siliceous with calcite and clay Liberation = 100 mesh	Scrubbing Fatty acid flotation Grind = 66.2% —200 Sod silicate 1.5 kg/t. Oleic acid emulsion = 0.6 kg/t. Rough conc. 4 cleanings Rough float regrind and two cleanings and six cleanings Refloat tails	CaF_2 = 66.18 CaF_2 = 91.0 CaF_2 =97.22 CaF_2 = 91.45	CaF_2 = 95.0 CaF_2 = 77.5 CaF_2 = 62.6 CaF_2 = 13.6	 Acid grade Metallurgical grade
6. Ambadongar Pilot plant studies. 190 tonne sample	CaF_2 = 27.99 SiO_2 = 49.12 $CaCO_3$ = 2.1 Siliceous with calcite	Scrubbing washing fatty acid flotation. Roughing and Regrinding 10 cleanings. Final concentrate 9th & 10th cleaner tails	CaF_2 = 96.1 CaF_2 = 87.37	CaF_2 = 62.6 CaF_2 = 13.6	Acid grade Metallurgical grade
7. Ambadongar Flotation conc.					
Agglomeration : Briquetting studies. Pelletisation studies.	Conducted successfully using various binders. Drying and heat hardening studies also were undertaken.				

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