Molybdenite is a heavy mineral but has got a property of natural flotability just like graphite. Depending upon the concentration and the associated minerals gravity method may be applied; but usually molybdenite is concentrated by flotation only.

A Molybdenite from Tamil Nadu

Two samples of molybdenite were received from the Director of G.S.I., Tamil Nadu Circle, for bench scale beneficiation studies.

Sample No. 1

The sample was marked L_2 and consisted of lumps of about 25 mm size. Complete chemical analysis of the sample was as follows:

Constituent	*Assay %*
Mo	0.168
Cu	0.130
Pb & Zn	Trace
Fe	3.00
S	0.88
Ni	0.03
SiO_2	75.44
Al_2O_3	7.98
LOI	0.62

Microscopic examination indicated that molybdenite was associated with other metallic minerals like pyrite, chalcopyrite, covellite, chalcocite, marcasite, goethite and magnetite, while the non-metallic gangue which constituted the bulk of the sample was composed of quartz and felspars. Molybdenite was liberated at 65 mesh size from the siliceous gangue minerals. Molybdenite was present as thin stringers and disseminations in quartzite and granite.

Tabling tests with 10 mesh feed was attempted for the pre-concentration but without success as 37% of molybdenum due to its flaky nature was lost in slimes and taillings.

Flotation tests with a grind passing 25.5% through 200 screen, using fuel oil as collector and cresylic acid as frother produced a rougher concentrate analysing 16.2% Mo with 90.5% recovery. When the rougher concentrate was cleaned thrice using sodium cyanide, a final concentrate assaying 54.5% Mo was obtained with 71% recovery. Retreatment of the cleaner tails after regrinding yielded a concentrate assaying 44.7% Mo with an additional 10.6% recovery. When the two concentrates were combined, the product analysed 53.0% Mo with 81.6% recovery. The concentrate was suitable for commercial utilization.

Sample No. 2

The sample was marked L_3 and analysed as follows:

Constituent	*Assay %*
Mo	0.038
Cu	0.090
Pb	Trace
Zn	Not detected
Ni	Minute trace
S	0.61
SiO_2	70.80
Al_2O_3	8.66
Fe_2O_3	2.82
LOI	0.40

Microscopic examination indicated that molybdenite was present in trace amounts and fairly liberated at 150 mesh size from the associated gangue minerals quartz, felspars and mica. Minor amounts of pyrite, chalcopyrite and goethite were seen.

Attempts for the pre-concentration of molybdenite were unsuccessful. Flotation tests with fuel oil, and cresylic acid with a grind passing 28.5% through 200 mesh screen produced a rougher concentrate assaying 5.99% Mo with 83.7% recovery. Addition of sod. silicate during roughing did not improve the grade or recovery of the concentrate.

The rougher molybdenum concentrate after two cleanings with sod. cyanide produced a concentrate analysing 43.0% Mo, and 1.75% Cu with 70.7% Mo recovery. Bulk sulphide flotation followed by selective flotation of molybdenite yielded a concentrate analysing 42.8% Mo, and 3.39% Cu with 67.6% Mo recovery in it. Molybdenite concentrate obtained from this sample does not meet the industrial specifications as such.

B Molybdenite from Bihar

Molybdenite occurs in the copper ores of Rakha Mines area and uranium ore of Jaduguda area. Molybdenite from both the ores was successfully concentrated.

Rakha Mines: Sample No. 1:

The ore was basically a copper ore containing minor amounts of molybdenite. The sample analysed as follows:

Constituent	*Assay %*
Cu	2.73
Fe	5.54
S	3.96
SiO_2	73.08
Al_2O_3	10.14
MgO	0.60
CaO	1.00
Co	0.20
Ni	0.072
Mo	0.025
Au	0.2 dwt/ton
Ag	2.2 dwt/ton
U_3O_8 (Equivalent)	0.030
Te	Nil
As, Sb, Mn, Pb, Bi & Cr	Trace
P_2O_5	0.78

Chalcopyrite, molybdenite, cubanite, violarite, etc. are the various sulphides present in the ore. The chief gangue was quartz-chlorite schist. Molybdenite was liberated from the non-metallic gangue at 150 mesh size, and from chalcopyrite at 400 mesh size.

As molybdenite was very closely interlocked with chalcopyrite, the ore being mainly of copper the bulk of sulphides particularly copper were concentrated and then molybdenite was separated.

Bulk copper concentrate was produced by flotation with 54.8% —200 mesh grind, 0.03 kg/tonne of Pot. Eth. Xanthate and 0.03 kg/tonne of pine oil at pH 8.8 by using lime for pH adjustment. The bulk concentrate analysed 24.31% Cu and 0.32% Mo with 100% molybdenite recovery in it. When the rougher concentrate was cleaned for copper, after grinding to 150 mesh, all the molybdenite was found to be with the copper concentrate only. The refloat concentrate was further treated by differential flotation depressing chalcopyrite with 5 cleanings using 0.2 kg/ton of Na_2S, 0.2 kg/ton of lime, 0.2 kg/tonne of sod. silicate and 0.8 kg/ton sod. cyanide, which produced a molybdenum concentrate analysing 35.95% Mo with 68.4% distribution. In a continuous operation the moly. cleaner tails when recirculated would improve the recoveries to a great extent.

Sample No. 2

The ROM ore sample analysed as follows :

Constituent	*Assay %*
Cu	1.34
Fe	10.14
S	2.03
SiO_2	65.80
Al_2O_3	10.58
MgO	2.94
CaO	0.66
Ni	0.053
Mo	0.016
U_3O_8 (Equiv.)	0.011
P_2O_5	Trace

Microsccpic examination indicated that chalcopyrite, violarite and molybdenite were the chief ore minerals contributing for Cu, Ni & Mo. The non-metallic gangue was composed of quartz, chlorite, biotite, felspar, muscovite etc. Copper minerals were liberated from the gangue at 100 mesh size and the molybdenite mineral was not fully liberated even at —200 mesh size.

Bulk flotation tests with a grind of 60% —200 mesh, 1.6 kg/tonne of lime, 0.06 kg/tonne of Sodium isopropyl Xanthate and 0.09 kg/tonne of pine oil produced a concentrate assaying 13.25% Cu, 0.308% Ni, and 0.15% Mo with 95.7% Cu, 58.6% Ni and 94.4% Mo. recoveries. But the separation of molybdenite from the bulk concentrate was found to be difficult. Hence differential flotation of molybdenite from the ore followed by Cu & Ni minerals separation was adopted. A rougher Cu-Mo concentrate was produced with a grind of 66% —200 mesh, 0.1 kg/tonne NaCN, 0.16 kg/tonne fuel oil and 0.03 kg/tonne cresylic acid at pH 7.0-8.5 and 14-16% solids. The rougher concentrate analysed 1.24% Mo with 85.2% distribution. When the rougher concentrate was cleaned for few times after regrinding to 99% —200 mesh using 0.2 kg/tonne sod. cyanide, 0.2 kg/tonne sod. silicate, 0.04 kg/tonne fuel oil and 0.02 kg/tonne cresylic acid at natural pH and 5% solids resulted in a moly. concentrate assaying 33.7% Mo and 1.4% Cu with a recovery of 60.2% Mo in it.

Pilot plant scale investigation was also conducted with this sample.

Sample No. 3

This copper ore from Rakha mines area was drawn for the development work of phase-II. The sample was treated both on bench and pilot plant scales. Complete analysis of the sample was as follows :

Constituent	*Assay %*
Cu	0.920
Ni	0.025
Mo	0.015
Fe	9.50
S	1.35
SiO_2	67.25
Al_2O	7.90
MgO	2.36
CaO	2.50
U_3O_8(Equiv.)	0.013
P_2O_5	0.95

Chalcopyrite was the chief copper bearing mineral. Other metallic minerals observed in the ore were magnetite, pyrite-pyrrhotite, Molybdenite, pentlandite etc. Quartz, chlorite, tourmaline minerals formed the non-metallic gangue which formed the bulk. Chalcopyrite was liberated at 100 mesh size from the rest of the gangue whereas molybdenite was liberated at 200 mesh size.

Batch scale tests indicated that a bulk sulphide concentrate may be produced analysing 16.0% Cu, 0.32% Ni, and 0.254% Mo with respective recoveries of 94.5%, 60.0% and 90.0%. The optimum conditions for the bulk flotation were feed ground to 54% —200 mesh, 0.03 kg/tonne of 1 :1 mixture of Pot. Ethyl. & Pot. Iso-Propyl Xanthates and 0.04 kg/tonne of pine oil at natural pH.

After regrinding the bulk sulphide concentrate, it was cleaned once again for bulk concentrate and subjected to eight cleanings under the optimum condition of 82% —200 mesh grind, 0.5 kg/tonne of NaCN, 0.25 kg/tonne of Na_2S, 0.25 kg/tonne of Pot. Ferro cyanide and 0.08 kg/tonne of Fuel oil at pH 7.5. The final molybdenum concentrate analysed 38.2% Mo and 1.13% Cu with 63.29% Mo recovery in it.

The pilot plant scale investigations were also conducted on the sample.

(4) Jaduguda Uranium Ores

The low grade uranium ores occurring at Jaduguda area adjacent to the Rakha Copper deposit contain as much of Mo & Ni minerals as that of copper ore. At the instance of the Uranium Corporation of India limited bench scale and pilot plant scale investigations were taken up at NML for the recovery of Cu, Mo & Ni minerals.

Batch No. 1

Two different samples were received in this batch and analysed as follows :—

Constituent	*Assay %*	
	No. 1	No. 2
Cu	0.17	0.24
Ni	0.17	0.28
Mo	0.032	0.032
SiO_2	77.10	not determined

It was desired to conduct the batch scale tests with the No. 1 sample and produce a bulk concentrate on pilot plant scale tests from the sample No. 2.

Microscopic examination indicated the presence of chalcopyrite, pyrite, millerite, pentlandite, violarite, molybdenite, magnetite and ilmenite in a matrix composed of quartz—chlorite shicstose rock. Nickel minerals were free from chalcopyrite at 400 mesh and molybdenite from the silicates at 200 mesh. Bench scale tests indicated that under the optimum conditions of 0.05 kg/tonne of Pot. Amyl Xanthate, and 0.04 kg/tonne of pine oil at pH 7.9 with a feed ground 55% to pass 200 mesh, a bulk Cu-Mo-Ni concentrate analysing 0.94% Cu, 1.84% Ni, and 0.50% Mo was produced with respective recoveries of 32.6%, 63.8% and 92.2%, The bulk concentrate after grinding to 200 mesh, steam boiled and with three cleanings using lime and sod. cyanide produced Molybdenum concentrate analysing 36.21% Molybdenum 2.0% Cu and 0.39% Ni with 34% Mo recovery in it.

Under the conditions mentioned above 200 kg of bulk Cu-Mo-Ni concentrate was produced in the Pilot Plant and was handed over to UCIL for further testing at BARC, Bombay.

Batch No. 2

Three different samples were received and blended together to form a composite sample which analysed as follows :

Constituent	*Assay %*
Cu	0.180
Ni	0.200
Mo	0.036
Total Fe	7.440
Fe_3O_4	6.630
S	0.560
P_2O_5	1.360
SiO_2	66.850
Al_2O_3	12.540
CaO	3.220
MgO	6.470
LOI	1.910
TiO_2	Trace

Quartz-chlorite schist was the host rock in which the metallic sulphides chalcopyrite, pyrite, millerite, pentlandite, violarite, molybdenite etc. were formed. Metallics were liberated at a size finer than 200 mesh.

A bulk sulphide concentrate analysing 1.7% Cu with 35.7% recovery, 3.0% Ni with 57.1% recovery and 0.77% Mo with 82.6% recovery was produced with a grind 55% —200 mesh, 0.05 kg. pot. ethyl. xanthate and 0.04 kg pine oil/tonne of ore at natural pH and 30% solids with a flotation time of 25 minutes. After one cleaning, the cleaner concentrate was ground to pass 200 mesh and steam boiled; with further three more cleanings, using 0.3 kg/tonne each of Sod. cyanide and sod. silicate, a molybdenum concentrate analysing 33.4% Mo. 1.0% Cu and 1.47% Ni with only 17.6% Mo recovery in it.

Alternately, tests to produce a rougher molybdenum concentrate indicated that with a feed ground 55% —200 mesh employing 0.5 kg/tonne fuel oil, 0.026 kg/tonne cresylic acid at 30% solids and 5 mts flotation time a rougher molybdenum concentrate assaying 3.5% Mo, 6.95% Cu and 10.0% Ni was produced with 89.3% Mo, 35.5% Cu and 46.4% Ni recoveries in it; after grinding the rougher concentrate to 10% —200 mesh and three cleanings with 0.15 kg/NaCN, a final Mo concentrate assaying 30.33% Mo, 2.57% Cu, and 0.6% Ni with 47.2% Mo recovery was produced. Grade of the molybdenum concentrate could not be improved due to the presence of graphite and organic matter along with molybdenite. Due to the fine dissemination of the copper mineral in the gangue, attempts to produce a marketable grade of copper concentrate was quite difficult.

Batch No. 3

100 tonnes of ROM was received for the concentration of Cu, Mo & Ni minerals on pilot plant scale, and the investigation were conducted.

The beneficiation studies of copper and nickel minerals in the above complex ore samples are separately treated in the copper and nickel ores. Optimum test results are given in tabular form in Table No. 44.

References

(1) Beneficiation of low grade molybdenite sample (L-2) from Karadikuttam, Madurai Dist., Tamil Nadu (NML.IR.NO. 633/71).

By C. Satyanarayana, S. K. Banerjee & G. P. Mathur.

(2) Beneficiation of low grade molybdenite sample (L-3) from Karadikuttam, Madurai Dist., Tamil Nadu (NML.IR.NO. 636/71).

By M. V. Ranganathan, S. K. Banerjee & G. P. Mathur.

(3) Flotation studies on a copper ore sample from Rakha mines, Bihar of NMDC Ltd., (NML.IR. NO. 353/66).

By K. N. Rakshit, P. V. Raman & P. I. A. Narayanan.

(4) Batch as well as pilot plant beneficiation studies for the recovery of copper, nickel and molybdenum from low grade copper ore from Rakha mines (Phase-I) and detailed proposal for setting up of a 1000 tonnes per day beneficiation plant (NML.IR.NO. 498/69).

By K. Vijayaraghavan, S. K. Dhar, P. D. Prasada Rao, P. K. Sinha, G. S. Ramakrishna Rao & P. I. A. Narayanan.

(5) Batch and pilot plant studies of the recovery of copper, molybdenum and nickel minerals from the low grade copper ore from Rakha mines (Phase-II) of Hindusthan Copper Ltd. (NML. IR.NO. 754/73).

By Dr. M. S. Prasad, P. N. Pathak, S. N. Prasad, C. Satyanarayana & G. P. Mathur.

TABLE 5.3—BENEFICIATION RESULTS OF MOLYBDENITE

State & Locality	Feed Assay % Mo & Cu	Beneficiation Method	Concentrate		Remarks
			Assay % Mo/Cu	Recovery % Mo	
Tamil Nadu					
(1) Karadikuttam	0.168 Mo	Rougher flotation followed by 3 cleanings.	53.0 Mo	81.6	
(2) Karadikuttam	0.038 Mo	Bulk flotation followed by selective Mo flotation.	42.8 Mo	67.6	Poorer conc.
Bihar					
(3) Rakha mines	0.025 Mo 2.730 Cu	Bulk Cu-Mo-Ni flotation followed 5 cleanings with NaCN.	35.95 Mo	68.4	
(4) Rakha mines	0.016 Mo 1.340 Cu	Rougher Mo concentrate followed by regrinding and four cleanings with NaCN.	33.7% Mo 1.4% Cu	60.2	Pilot plant investigation
(5) Rakha mines	0.015 Mo 0.920 Cu	Bulk sulphide flotation followed by regrinding and eight cleanings.	38.2% Mo 1.13 Cu	63.29	,,
(6) Jaduguda (a)	0.032 Mo 0.170 Cu	Bulk sulphide conc. followed by steam boiling, regrinding and three cleanings.	36.21% Mo 2.0% Cu	34.0	Batch test for pilot plant operation with (b).
(b)	0.032 Mo 0.240 Cu	—	—	—	Pilot plant test to produce 200 kg. bulk conc. only
Jaduguda (2)	0.036 Mo 0.180 Cu	Bulk conc. cleaning, regrinding, steam boiling & three cleanings.	33.41% Mo 1.0% Cu	17.6	Conc. of Cu minerals was difficult.
		Rougher moly. conc. followed by regrinding & 3 cleanings.	30.33% Mo 2.57% Cu	47.2	
Jaduguda (3)	0.021 Mo 0.140 Cu	4 types of flowsheets on pilot plant scale operations.	31.54 Mo 1.10 Cu		Pilot Plant operation.

(6) Flotation studies for the recovery of Molybdenum, Copper & Nickel minerals from Uranium ores from Jaduguda Part-I. (NML.IR.NO. 488/68).

By R. Ganesh, G. S. Ramakrishna Rao & P. I. A. Narayanan.

(7) Flotation studies for the recovery of Molybdenum, Copper & Nickel minerals from Uranium ores from Jaduguda. Part-II. (NML.IR.NO. 523/69).

By R. Ganesh, G. S. Ramakrishna Rao & PIA Narayanan.

(8) Flotation studies for the recovery of Molybdenum, Copper & Nickel minerals from Uranium ores from Jaduguda. Part III. (NML.IR.NO. 543/69).

By R. Ganesh, K. Vijaya Raghavan, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan.

Cassiterite (Tin Stone) SnO_2

Cassiterite is the only ore of Tin and the deposits in India are very poor. Two samples of alluvial cassiterite were received from M/s. National Cement, Mines and Industries Ltd., Ranchi to produce a 60% grade of concentrate.

(1) Sample No. 1

The sample was −14 mesh in size and analysed 5.18% Sn. Cassiterite being a heavy mineral, the sample was treated on shaking table. As all the cassiterite was found to be coarse-grained in nature, the table concentrate was ground to 20 mesh and then cleaned again on table. The cleaner concentrate analysed 63.02% Sn with 95.5% Sn distribution.

(2) Sample No. 2

The sample analysed 0.205% Sn and was associated with quartz, tourmaline and limonite clay. Sizing and analysis indicated that 85.9% of the Sn was distributed in +4 mesh sand. The sample contained about 39% +4 mesh portion in it.

The sample was crushed to 4 mesh and then sized on 10 mesh screen. The +10 mesh portion was treated in a jig and the concentrate was ground to 10 mesh and cleaned on a table, which yielded a grade of 62.15% Sn with 40.9% Sn distribution.

The +10 mesh fines after tabling yielded a concentrate assaying 65.13% Sn with 26.1% Sn distribution.

The combined concentrate assayed 63.27% Sn with 67.0% distribution.

Tabling tests with 20 mesh sized feed yielded a concentrate assaying 57.45% Sn with 74.5% Sn distribution in it.

Jigging tests with −10+20 mesh portion followed by tabling of the same after crushing to 20 mesh along with −20 mesh portion and cleaning yielded a cleaner concentrate assaying 63.92% Sn with 63.6% Sn distribution in it.

References

1. Tabling of a low grade cassiterite concentrate from Ranchi, Bihar (NML. IR/19/53)—By G. V. Subramanya & P. I. A. Narayanan.
2. Beneficiation of low grade cassiterite ore from Ranchi, Bihar (NML. IR/20/53)—By G. V. Subramanya & P. I. A. Narayanan.

ROCK PHOSPHATE AND APATITE DEPOSITS

Rock phosphate and apatite are the most important minerals for the production of phosphate based fertilizers. Large deposits of rock phosphates are located in Rajasthan and Uttar Pradesh along with some apatite deposits in Andhra Pradesh and Bihar and phosphatic nodules in Tamil Nadu.

The rock phosphates must be having a minimum of 36% P_2O_5 for the use in the fertilizer industry. The usual associates of rock phosphate which lower the grade of P_2O_5 content are calcite and quartz. These two deleterious mineral constituents are removed only by flotation. Removal of silica is not a big problem; but the carbonates behave like phosphate in the flotation circuit. However, some depressants like *Katha/Kutch extract and quabracho are used to depress carbonates but with partial success. In such cases where they are calcitic in nature the phosphates are calcined to decompose the carbonates and then deslimed before flotation where in the decomposed lime will be taken out along with the slimes.

Apatite and Rock phosphate samples from Bihar, Uttar Pradesh and Rajasthan have been tested on bench and pilot plant scale operation with the following results.

*Note :—Katha/Kutch Extract and Quabraco are all Tanin Extracts from Leather Industry.

A Rock phosphates from Rajasthan

A large deposit of low grade rock phosphate is situated in Maton area in the district of Udaipur. A large number of samples from the same deposit taken from different sectors have been tested in the NML for the production of fertilizer grade of concentrate.

Maton block

Phosphate samples from this area were tested both on bench scale and pilot plant scale studies.

(1) Sample No. 1

The sample comprised of lumps from 100 mm down to fines. Complete chemical analysis of the sample was as follows :

Constituent	*Assay %*
P_2O_5	35.46
SiO_2	10.56
Al_2O_3	1.10
CaO	47.75
MgO	0.28
FeO	0.08
Fe	0.91
CO_2	0.94
F	1.60
SO_3	0.21
Cl & Cu	Trace
LOI	1.66

Microscopic examination of the sample indicated that apatite formed chief phosphate mineral, followed by quartz and calcite. Most of the calcite was liberated at 100 mesh and the quartz at 48 mesh size. However, disseminated quartz, was found in apatite grains even at 150 mesh size.

Flotation tests conducted under different conditions indicated that a grind 86% passing 200 mesh screen, with 1 kg/tonne of sod. silicate to depress the siliceous minerals and 1 kg/tonne of oleic acid emulsion were optimum where in a phosphate concentrate assaying 37.34% P_2O_5 with 99.5% distribution was produced. When the concentrate was further cleaned twice, cleaner concentrate analysed 40.51% P_2O_5, 2.37% SIO_2, 0.1% Al_2O_3 and 0.54% Fe with 75% of P_2O_5 distribution in it.

(2) Sample No. 2 : Kanpur Block

This was lean sample against the earlier sample and analysed as follows :

Constituent	*Assay %*
P_2O_5	12.66
SiO_2	8.20
Al_2O_5	0.26
CaO	39.60
MgO	9.65
Fe_2O_3	0.64
CO_2	24.77
F	1.66
SO_3	0.21
FeO, Cl, Cu	Trace
LOI	27.32

Microscopic examination revealed that collophane and apatite formed the chief phosphate ore minerals while calcite, quartz and minor amounts of opaques formed the gangue. Liberation of the phosphates from the gangue was observed at 150 mesh size.

Flotation tests indicated that with a feed ground to 200 mesh size, 1.0 kg/tonne of sod. silicate as depressant for silicates, 0.2 kg/tonne of quebracho (tannin extract) were optimum reagents while tal oil was the best collector for the sample where in a concentrate analysing 15.55% P_2O_5 with 92.3% distribution in it resulted. Other collectors like oleic acid emulsion etc. effected either on grade or recovery. When the concentrate was cleaned for 4 times using 0.2 kg/tonne of sod. silicate at each stage, a concentrate assaying 30.4% P_2O_5 with 66.5% distribution was produced. Calcination and desliming followed by flotation under optimum conditions after two cleanings produced a concentrate assaying 34.06% P_2O_5 with 58.1% distribution. This was due to the loss of apatite during removal of calcined carbonates as slimes.

(3) Sample No. 3

160 tonne rock phosphate sample, drawn from the Maton block was received from M/s. Hindusthan Zinc Ltd. for pilot plant scale trials to produce a fertilizer grade concentrate. The sample in its "as received" state contained fines and lumps upto 200 mm. Complete analysis of the sample was as follows :

Constituent	*Assay %*
P_2O_5	28.06
SiO_2	17.88
Al_2O_3	1.10
Fe_2O_3	1.80
CaO	41.30
CO_2	5.50
F	1.16
SO_3	1.30
MgO	Trace

Microscopic examination indicated that calcite, gypsum and quartz formed the bulk of the gangue. Apatite and collophane formed phosphate mineral group. Phosphates were liberated from the gangue at 150 mesh. Preliminary batch scale tests indicated that with a grind of 90% —200 mesh, 1.0 kg/tonne oleic acid emulsion, 2.0 kg/tonne sod. silicate were essential to get a rougher concentrate analysing 33.1% P_2O_5 with 88.3% distribution in it. With one cleaning adding an additional quantity of 0.25 kg/tonne of oleic acid emulsion and 0.5 kg/tonne of sod. silicate, a cleaner concentrate assaying 35.1% P_2O_5 with 77.9% distribution was produced. Addition of Katha in the cleaning stages produced a concentrate assaying 38.50% P_2O_5 with 72.1% distribution in it.

Pilot Plant Tests:

After confirming the optimum conditions for the concentration of apatite, large scale pilot plant tests were taken up.

The run of mine ore was initially crushed in jaw crusher (38 mm gap) and after screening the coarser portion in a secondary cone crusher (10 mm gap) which operates in closed circuit with a vibrating screen. The crushed ore (—10 mm) is stored in the bin. The ore from the bin was fed to a belt conveyor through a constant weight feedometer which in turn feeds the ball mill, which is operating in closed circuit with a rake classifier. The classifier over flow along with the reagents was pumped to the conditioner. The conditioned feed (18-20% solids) was fed to two roughing cells of 50 c.f.t. capacity. The concentrate was cleaned in two batteries of cells and finally stored in a thickener and the primary tailings were rejected. The tails from the cleaner cells were recirculated back to the conditioner. As the concentrate did not analyse 36%

P_2O_5, the product was again treated with katha, where in a concentrate of acceptable grade was produced.

(4) Phosphate from Karbaria Block

The sample consisted of lumps upto 65 mm with fines and was sponsored by G.S.I., Rajasthan circle. The sample analysed as follows:

Constituent	*Assay %*
P_2O_5	22.74
SiO_2	25.32
Al_2O_3	0.78
CaO	37.62
Fe	2.37
CO_2	7.24
Cl	0.12
SO_3	0.28
Cu	0.35
LOI	8.07
MgO, FeO, F	Trace

Microscopic examination of the sample indicated that collophane and minor amounts of apatite formed bulk of phosphate minerals where as quartz, calcite, gypsum and sericite formed gangue. Phosphates were liberated from the gangue at 100 mesh size.

Preliminary flotation tests with quabracho, sod. silicate and oleic acid emulsion indicated that 75% —200 mesh grind was essential to get a good recovery of the phosphates. Among the various collectors tried, oleic acid emulsion produced best results yielding a concentrate assaying 27.0% P_2O_5 with 94.5% recovery. Additional three cleanings with the concentrate improved the grade to 31.9% P_2O_5 with 78.4% recovery.

When the rougher concentrate was ground to 200 mesh size and then cleaned thrice, the concentrate analysed 34.0% P_2O_5 with 76.9% distribution. Calcination followed by desliming, roughing and three cleanings also produced a concentrate assaying 34% P_2O_5 with 57.1% recovery.

(5) Phosphate from Dakan Kotra Block

The sample was received from GSI-Rajasthan circle. The ore in its "as received" state contained lumps upto 65 mm, down to fines and analysed as follows:

Constituent	*Assay %*
P_2O_5	14.05
SiO_2	6.26
Al_2O_3	0.21
CaO	37.24
MgO	12.12
Fe	0.24
CO_2	22.70
F	1.38
Cl	0.06
SO_3	0.69
Cu	0.05
LOI	27.29
FeO	Trace

Microscopic examination of the sample indicated that collophane and apatite formed the phosphate minerals.

Calcite was the chief gangue mineral followed by quartz. Even at 150 mesh size interlocked calcite-collophane particles were observed.

Preliminary flotation tests revealed that a grind of 90% —200 mesh was necessary 0.2 kg/tonne quebracho and sod. silicate 1.0 kg/tonne were used as depressants and oleic acid emulsion 0.8 kg/tonne produced a concentrate assaying 15.5% P_2O_5 with 92.3% recovery. Other collectors like liquid Tall oil etc. did not produce any better results.

Reflotation tests with sod. silicate, 0.2 kg/tonne at each stage after four cleanings produced a concentrate assaying 30.6% P_2O_5 with 63% recovery. The sample was calcined and deslimed before flotation, and flotation was conducted after adjusting the pH to 8.3 under optimum conditions. This when cleaned twice, the final concentrate analysed 34.5% P_2O_5 with 58.6% distribution in it.

(6) Phosphate from Kataria Hill

The sample was received from the Directorate of Geology and Mining, Rajasthan for beneficiation studies. The ore comprised of 65 mm lump down to fines and analysed as follows:

Constituent	*Assay %*
P_2O_5	9.8
SiO_2	23.8
Al_2O_3	0.5
Fe	1.1
FeO	0.6
CO_2	17.3
CaO	28.8
MgO	11.8
SO_3	0.5
LOI	23.2
Cl & Cu	Trace

Microscopic examination indicated that collophane and apatite were associated with calcite, quartz, chlorite mica etc. and a fair liberation of the phosphates was observed at 150 mesh size.

As the sample was very poor grade, heavy media separation tests were attempted with —12 mm+6 mesh sized ore where in a lighter product i.e. float obtained at sp. gr. 2.8 analysed 4.4% P_2O_5 with 24.5% distribution which was too rich to be rejected.

Flotation studies with a feed ground to 150 mesh size using different collectors like tall oil (0.04 kg/tonne) liquiro (0.04 kg/tonne, sodum luryl sulphate and oleic acid emulsion as collectors indicated that oleic acid emulsion suited best for the sample. Tests employing oleic acid emulsion, sod. silicate and quebracho with 3 cleanings produced a concentrate assaying 14.9% P_2O_5 with 45.5% distribution. Similar test with a calcined feed yielded a concentrate assaying 34.2% P_2O_5 with 69.4% distribution.

(7) Phosphates from Jhamar Kotra Block

The sample was received from the Director of State Mining and Geology Dept. Rajasthan and was composed of between 12 mm to 25 mm. The sample analysed as follows:

Constituent	*Assay %*
P_2O_5	23.00
SiO_2	4.78
Al_2O_3	0.56
CaO	46.33
MgO	4.66
Fe	0.21
SO_3	0.31
CO_2	15.52
Cl	0.17
F	2.33
LOI	18.14
Cu, Mn	Trace

Collophane and apatite constituted the phosphatic minerals while calcite, quartz and gypsum formed gangue minerals. Phosphates were fairly liberated at 150 mesh size. Initial tests indicated that 90% —200 mesh was essential for the flotation of apatites.

Further tests employing 0.2 kg/tonne Katha to depress calcite 1.0 kg/tonne of sod. silicate to depress silicates and 1.0 kg/tonne of oleic acid emulsion a concentrate analysing 24.54% P_2O_5 with 96.3% distribution was produced. After one cleaning the grade improved to 34.6% P_2O_5 with 59% distribution in it. By simple calcination and desliming only the sandy portion analysed 34.8% P_2O_5 with 87% distribution.

Calcination studies on Jamarkotra Phosphate:

Six different phosphate samples drawn from different zones were received for beneficiation. The samples varied in P_2O_5 % from 13.0% to 27.2% and CaO content varied from 38.8% to 50.6%. Representative samples were drawn from each of the samples and were combined to give the following chemical analysis:

Constituent	*Assay %*
P_2O_5	17.49
CaO	40.39
MgO	12.77
Total Fe	0.60
FeO	Trace
SiO_2	2.45
LOI	24.60

The sample as such was high in CaO content. The composite sample was crushed to 13 mm size and the various sized products when analysed for P_2O_5 content, did not show any marked difference. Differential thermal analysis indicated the presence of dolomite as the major gangue with some calcite in the sample.

Preliminary calcination tests indicated that when the sample was calcined at 950°C for 2 hours when quenched in water and deslimed, the sand portion analysed 35.69% P_2O_5 with 91.0% P_2O_5 distribution in it.

Continuous calcination tests were conducted with the composite sample in a rotary kiln at a temperature of 950°C with a retention time of 120 minutes. The calcined product after quenching and desliming produced an average P_2O_5 content of 34.65% with 80% P_2O_5 distribution in it. Some more phosphate was recovered from slimes when treated in cyclone which analysed 25.79% P_2O_5. The combined sand and cyclone underflow analysed 32.95% P_2O_5 with 92.6% P_2O_5 distribution in it.

B Uttar Pradesh Deposits:

Rock phosphates from Uttar Pradesh

Large deposits of low grade rock phosphates occur in the lower regions of Himalayas in Mussoorie area of Uttar Pradesh. A phosphate sample from Masrana block of Mussoorie area was received from Director G.S.I., of Northern region for beneficiation tests. The ore was composed of 25 mm lumps down to fines. Complete chemical analysis of the sample was as follows:

Constituent	*Assay %*
P_2O_5	21.50
SiO_2	35.92
CaO	26.34
CO_2	1.08
Fe_2O_3	5.81
Al_2O_3	1.23
MgO	1.84
F	0.59
SO_3	1.01
Cl	Trace

Microscopic examination revealed that collophane and apatite formed the chief phosphate ore minerals where as quartz and minor amounts of chert, dolomite, ferromagnesian minerals formed the gangue. Phosphates were not fully liberated at even 200 mesh size.

Flotation tests conducted with different grinds of feed and reagent combinations indicated that a grind passing 99% —200 mesh screen 1.25 kg/tonne of sod. silicate and 1.5 kg/tonne of oleic acid emulsion produced a concentrate analysing 27.3% P_2O_5 with 93.2% distribution. When the concentrate was cleaned thrice, it analysed 35.8% P_2O_5 with 60.5% distribution. With regrinding of the rougher concentrate to 88.6% —325 mesh and three cleanings, the concentrate analysed 38.4% P_2O_5 with 72.4% distribution.

Phosphate from Durmala (UP)

Rock phosphate sample was received from M/s. PPCL and consisted of 20 mm lump to fines having the following chemical analysis:

Constituent	*Assay %*
P_2O_5	17.99
SiO_2	7.44
Al_2O_3	1.05
Fe_2O_3	2.54
CaO	39.24
CO_2	15.04
MgO	2.82
F	2.15
SO_3	3.16
LOI	21.84

Mineralogical examination of the sample showed collophane as the chief phosphatic mineral in association with carbonates which formed the bulk of the gangue. Minor amounts of quartz, chert, magnetite were also present. Collophane was liberated from the gangue at a very fine size i.e. 325 mesh.

Flotation tests with 1.5 kg/tonne of sod. silicate, 1.0 kg/tonne of oleic acid emulsion, 0.6 kg/tonne of kerosene oil and 0.3 kg/tonne of pine oil with a grind passing 70% through 200 mesh produced a concentrate assaying 19.95% P_2O_5 with 53.44% P_2O_5 distribution in it.

Calcination at 950°C and desliming had improved the grade to 25.07% P_2O_5 with 99.5% P_2O_5 distribution. Flotation tests with the deslimed sand with 0.8 kg/tonne of sod. silicate, 0.04 kg/tonne of kerosene oil, 1.2 kg/tonne of oleic acid emulsion and 0.03 kg/tonne of pine oil yielded a concentrate analysing 25.72% P_2O_5 with 53% P_2O_5 distribution in it.

ROM sample crushed to 6 mm size followed by calcination, quenching, regrinding and flotation yielded a concentrate assaying 27.0% P_2O_5 with 62.2% P_2O_5 distribution in it. Similar test with —10 mesh sample produced a concentrate assaying 28.7% P_2P_5 with 75.7% P_2O_5 distribution in it.

Rock Phosphate from Maldeota

Three different samples sent by M/s. PPCL, from Maldeota mine designated as bed sample, hanging wall sample and foot-wall sample amounting to 158 tonnes were received for beneficiation. A composite sample was prepared by mixing 80% bed ore and 10%

each of hanging and foot-wall samples. The composite sample analysed as under:

Constituent	*Assay %*
P_2O_5	16.80
CaO	42.15
SiO_2	10.21
MgO	1.77
Al_2O_3	0.80
Total Fe	0.86
FeO	0.43
LOI	16.81
Insolubles	12.27

Mineralogical examination of the composite sample indicated the presence of collophane as the chief phosphatic mineral while the carbonates formed the bulk of the gangue. Carbonaceous shale. chert, quartz, goethite formed the other associated gangue minerals. Although bulk of the phosphates were liberated from the gangue at about 65 mesh, complete liberation was only at 400 mesh size.

Chemical analysis of the representative —10 mesh products indicated that the portions finer than 100 mesh assayed less than 4% P_2O_5 with 3.2% P_2O_5 distribution in it. Attrition scrubbing tests with 6 mm crushed ore did not show any improvement in the sand portion. Pneumatic classification tests with 10 mesh material improved the grade marginally.

Flotation tests were conducted with a feed passing 81% through 200 mesh screen. The sample was deslimed partially for flotation tests. Carbonaceous matter flotation with kerosene oil and pine oil followed by Calcite flotation and then phosphate mineral flotation with oleic acid emulsion yielded a grade of 17.8% P_2O_5 which was not much different from the feed. When orthophosphoric acid was used in place of oleic acid emulsion it yielded a grade of 18.21% P_2O_5 with 30% P_2O_5 distribution in it.

Sulphide flotation with xanthate, carbonaceous matters flotation with kerosene oil and pine oil and then calcite flotation and phosphate flotation with oleic acid emulsion using sodium silicate to depress the silicates, produced a grade of 21.0% P_2O_5 with 68.7% P_2O_5 distribution in it. Under similar conditions when amine was used to float the silicates after removing the carbonates, the grade of the concentrate was 20.10% P_2O_5 with 85.0% P_2O_5 distribution in it.

Flotation tests with Hydro-fluo-silicic acid and oleic acid emulsion with a grind deslimed feed passing 70% through 200 mesh followed by regrinding and flotation yielded a grade of 25.6% P_2O_5 with 55.7% P_2O_5 distribution in it.

Finally the sample was calcined at 1000°C for 2 hours followed by quenching and thorough scrubbing was taken for flotation. Carbonaceous matter was floated with Kerosene oil and pine oil and then the pulp was conditioned with Katha (Tanin extract), sod. silicate and oleic acid emulsion produced a grade of 28.33% P_2O_5 with 79.0% P_2O_5 distribution in it.

C BIHAR APATITE DEPOSITS

Large phosphate deposits are being worked in Patharghar area of Singhbhum district. Some lean ore bodies are also present. These deposits are associated with magnetites; in addition to these, apatite is present as accessory mineral in the Singhbhum copper belt. Beneficiation studies on the copper ores and low grade apatite were taken up at N.M.L.

(1) Dalbhum Phosphate

Low grade apatite sample from Samaidih was received from M/s. Dalbhum Phosphate Co. Ltd. for the production of fertilizer grade concentrate. The sample was composed of lumps from 45 mm to fines and analysed as follows :

Constituent	*Assay %*
P_2O_5	23.26
SiO_2	19.31
Fe	11.32
Al_2O_3	9.15
CO_2	0.40
CaO	30.00
F	2.10
Cl	0.24
Cu, SO_3, & Org. matter.	Trace

Microscopic examination of the sample revealed that apatite was the only phosphate mineral and the gangue was composed of chlorite, biotite, felspar, quartz and magnetite. Magnetite and siliceous gangue were free from apatite at 28 mesh and 48 mesh respectively. Flotation tests indicated that with a feed ground to 76% —200 mesh size and 0.69 kg/tonne of oleic acid emulsion alone a concentrate

analysing 41.0% P_2O_5 with 95.5% distribution was produced. Tests with addition of sod. silicate and pine oil did not show any improvement but lowered the recovery. When the rougher concentrate was cleaned once, the concentrate analysed 41.7% P_2O_5 with 84.4% recovery. The recovery would improve if the cleaner tails are recirculated to the flotation cells.

(2) Recovery of apatite from Ghatsila Copper Tailings

The copper ore deposits of singhbhum area contain small quantity of apatite and at present it is being rejected in the copper tails. The ROM analysed 1.36% P_2O_5 while the copper tailings analysed 1.49% P_2O_5. Attempts have been made to recover the apatite from the tails by flotation method. Other minerals present in the tailings are tourmaline, calcite, ferromagnesiam minerals etc.

When the tailings were floated employing 0.36 kg/tonne sod. silicate and 0.75 kg/tonne of oleic acid emulsion a rougher concentrate analysing 8.11% P_2O_5 was produced with 76.4% recovery. On further cleaning with an additional quantity of 0.25 kg/tonne of sod. silicate, the concentrate analysed 28.21% P_2O_5 with 50% distribution. Better recoveries are expected when the middlings are recirculated.

(3) Apatite from I.C.C. tailings

The copper mill tailings from Mosabani concentrator analysing 1.36% P_2O_5 were received from M/s. Indian Copper Complex for the recovery of apatite. Although different samples were received, only a 'fresh tailing' sample was investigated in detail.

Preliminary flotation tests with 0.6 kg/tonne of sod. silicate and 0.5 kg/tonne of oleic acid emulsion with the sample in its 'as received state' produced a concentrate assaying 6.6% P_2O_5 with 80% P_2O_5 distribution in it. Further two cleanings of the concentrate improved the grade to 12.9% P_2O_5 with 78% P_2O_5 distribution in it.

Grinding the sample to 55% —200 mesh size followed by flotation with 3 cleanings produced a concentrate analysing 16.68% P_2O_5 with 78.5% P_2O_5 distribution in it. Grinding of the sample to 80% —200 mesh and flotation has improved the grade to 27.3% P_2O_5 but the P_2O_5 recovery was only 40.0%. Roughing followed by regrinding and three cleanings produced a grade of 24.3% P_2O_5 with 37.5% P_2O_5 distribution in it

Attempts to recover apatite from the fresh mine sample after taking out sulphide float with xanthate followed by apatite flotation with 3 cleanings also only produced a grade of 15.85% P_2O_5 70.0% P_2O_5 distribution in it.

The results of the above samples are given in a Tabular form in Table No. 45.

References

(1) Beneficiation of phosphate rock sample from Singhbhum Dist., Bihar—NML.IR.NO. 332/65.

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(3) Beneficiation of phosphate rock from Masrana block, Mussoorie area—Uttar Pradesh. NML.IR.NO. 493/68.

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(4) Pilot Plant beneficiation studies on a low grade phosphate sample from Maton block, Udaipur Dist., Rajasthan. NML.IR.NO. 541/69.

By Joga Singh, G. Radha Krishnan, P. D. Prasada Rao, B. L. Sengupta, G. P. Mathur & P. I. A. Narayanan.

(5) Beneficiation of phosphate rock sample from Kanpur block of Udaipur Dist., Rajasthan. NML.IR.NO. 517/69.

By K. N. Rakshit, S. K. Banerjee & P. I. A. Narayanan.

(6) Beneficiation of phosphate rock sample from Maton block of Udaipur Dist. Rajasthan. NML.IR.NO. 458/68.

By K. N. Rakshit, P. V. Raman & P. I. A. Narayanan.

(7) Beneficiation of low grade rock phosphate sample from Dakan Kotra area—Udaipur Dist. Rajasthan. NML. IR.NO. 594/70.

TABLE 5.4—BENEFICIATION RESULTS OF PHOSPHATE SAMPLES

State & Locality	Assay % of ROM % P_2O_5	Beneficiation methods	Assay % of conc. % P_2O_5	Recovery % P_2O_5	Remarks
1	2	3	4	5	6
Rajasthan					
Maton block (1)	35.46	Flotation followed by two cleanings	40.51	75.0	
Maton block (2) (Kanpur)	12.66	Calcination followed by flotation & cleanings	34.06	58.1	
Maton block (3)	28.06	Flotation and one cleaning with kutch	38.5	72.1	Pilot plant scale tests were also conducted.
Karbaria	22.74	Flotation, regrinding and three cleanings.	34.0	76.9	
Dakan Kotra	14.05	Calcination and flotation with 2 cleanings.	34.5	58.6	
Kataria Hill	9.8	Calcination, flotation followed by 3 cleanings.	34.27	69.4	
Jamar Kotra	23.0	Flotation followed by 1 cleaning.	34.8	87.0	
	Composite of Six Samples	Calcination at 950°C, Quenching, desliming. Sand	35.69%	91.0%	
	P_2O_5 = 17.49				
	CaO_2 = 40.39	Continuous calcination tests :			
	LOI = 24.60	Sand	34.65%	80.0%	
	MgO = 12.77				
	SiO_2 = 2.45	Cycloning slime	25.79%	12.6%	
		Combined conc	32.95	92.6%	
Uttar Pradesh					
Mussoorie	21.5	Flotation followed by regrinding three cleanings.	38.4	72.4	
	P_2O_5 = 17.99%	**Flotation:**			
	SiO_2 = 7.44%	Grind = 70% — 200 mesh	19.95%	53.44%	
		Flotation of deslimed sand	25.07%		
		0.8 kg/ton. sod. silicate			
		0.04 kg/ton. Kerosene oil			
		0.03 kg/ton. Pine oil	25.72%	53%	
		1.2 kg/ton. Oleic acid emulsion.			
		Calcination at 6 mm size, Quenching, regrinding and flotation.	27.0%	62.2%	
		Calcination at —10 mesh size, Quenching, regrinding and flotation.	28.7%	75.7%	
	Composite sample:	**Flotation:**			
	P_2O_5 = 16.8%	Grind = 81% —200 mesh			
	CaO = 42.15%	Deslimed.			
	SiO_2 = 10.21%	Sulphide flotation xanthate.			
	MgO = 1.77%	Carbonaceous matter flotation			
	LOI = 16.81%	with kerosene oil and pine oil	21.0%	68.7%	
		Calcite flotation and phosphate flotation with oleic acid emulsion sod. silicate depressant	25.0%	55.7%	
		Regrinding and flotation using hydrofluoric acid and oleic acid emulsion	28.33%	79.0%	
		Calcination and quenching, and flotation.			
Bihar					
Samaidih	23.26	Straight flotation	41.0	95.5	Cleaning improve the grade to 41 only.
Mosabani copper tailings	1.49	Flotation followed by cleaning	28.31	50.00	
Apatite from I.C.C.		Flotation as received			
Tailings—HCL.	P_2O_5 = 1.36%	0.6 kg/ton. sod. silicate			
		0.5 kg/ton. Oleic acid emulsion.	6.6%	80.0%	
IR. NO. 841/75	Liberation 400 mesh.	After two cleanings	12.9	70.0%	
		Grinding to 55% —200 mesh			
		Flotation with three cleanings	16.68%	78.5%	
		Grinding to 85% —200 mesh and flotation	27.3%	40.0%	
		Flotation of fresh sample	15.85%	70.0%	

By M. V. Ranganathan & G. P. Mathur.

(8) Beneficiation of low grade phosphate rock from Kataria Hill Mine, Udaipur Dist. Rajasthan. NML. IR. NO. 512/69.

By S. K. Dhar, P. V. Raman & P. I. A. Narayanan.

(9) Beneficiation of a low grade phosphate rock (Sample No. B) from Jhamar Kotra area Udaipur Dist. Rajasthan. NML. IR. NO. 628/71.

By S. Prasad, S. K. Banerjee & G. P. Mathur.

(10) Beneficiation of studies on a low grade rock phosphate sample from Karbaria block Udaipur Dist. Rajasthan NML. IR. NO. 647/71.

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(11) Beneficiation of phosphate rock from Durmala (U.P.) NML. IR. NO. 832/75.

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(12) Bench scale flotation studies for recovery of apatite as a by-product from the I.C.C. flotation tailing samples.

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(13) Calcination studies on Jhamarkotra Rock Phosphate.

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(14) Beneficiation studies on Maladeota rock phosphate sample from Mussoorie, U.P.

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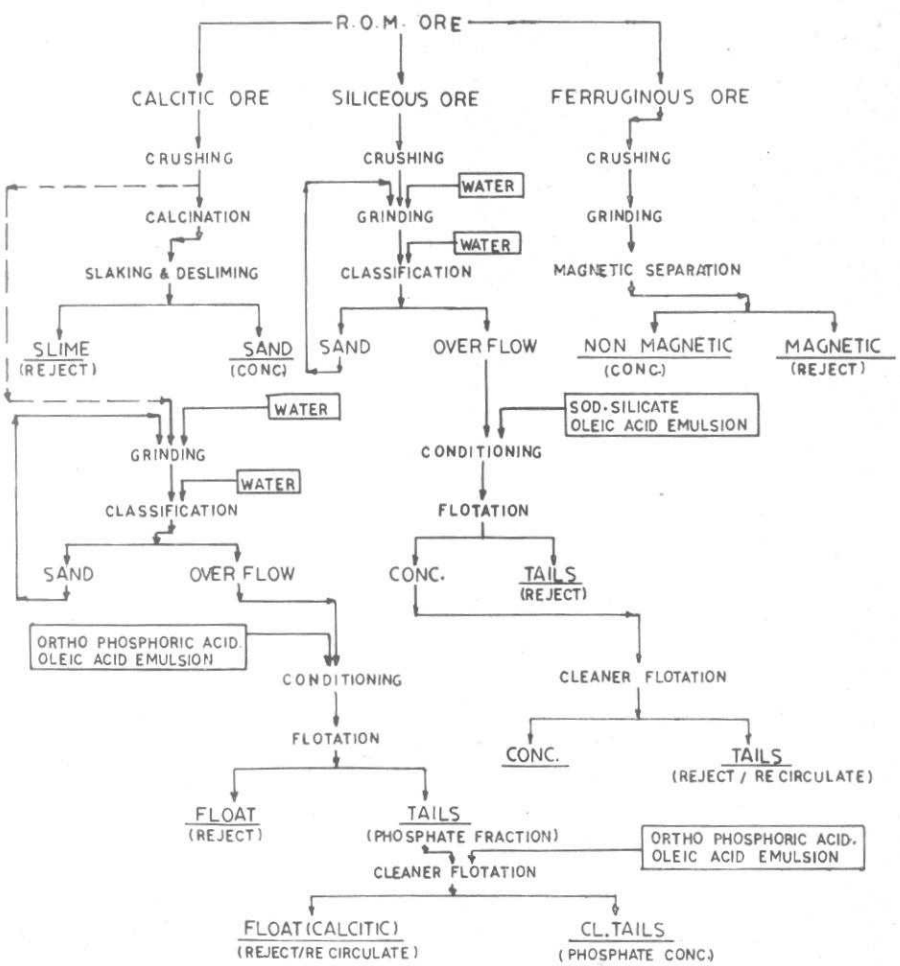

Fig. 5.2—General Flowsheet for Beneficiation of Rock Phosphate