Some observations on the beneficiation of lean phosphate rock from indigenous resources

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INTRODUCTION :

With the increase in price of chemical phosphatic fertiliser, the cultivators were hesitant to use them. This trend would adversely affect the agricultural production. Nitrogenous fertiliser could be suitably substituted with lean phosphate rock. Because of its nature and low phosphate content, this lean rock phosphate cannot be directly utilised in the manufacture of chemical fertiliser and needs beneficiation. However, its application as a direct phosphatic fertiliser was appreciated in acid soils. One of the leading organisations in India has already put untiring effort for utilising them through various consuming centres in the country.

Total reserves of fertiliser mineral deposits[1] in the country is shown in Table No. 1.

BENEFICIATION STUDIES :

Sample 1. Mussourie Phosphate :

The studies carried out on a sample from Mussourie, U. P. are presented in this paper. The sample was hard and lumpy and black in colour. The overall size was from 25 mm to -200 mesh. The chemical analysis of the sample was P_2O_5 = 18.96%, CaO =42.78%, MgO = 1.65%, SiO_2 =10.47%, Al_2O_3 =1.22%, Fe (lot) =1.05% and LOI = 15.40%. The object was to recover a concentrate having a grade around 22–24% P_2O_5. For this, attrition scrubbing, acid leaching followed by tabling were studied.

Mineralogical Studies :

Mineralogical studies were undertaken on representative sample showed that the collophane was the chief phosphate mineral. The chief gangue minerals were calcite and minor amounts of pyrite, carbonaceous shale, chert, quartz and goethite. Microscopic examination of various –10 mesh sieve fractions indicated that the phosphate mineral was free from the shale, carbonate and gangue at a grind below 65 mesh size but complete separation may not be possible due to their finely dispersed nature in collophanitic matrix and likely needs finer grind of less than 200 mesh.

Gravity separation tests [2] :

Representative portions of – 10 mesh sample ground to different degree of fineness were subjected to attrition scrubbing with solid liquid ratio 1 : 1 for 10 minutes. Though it was observed that slime had lower P_2O_5 content, there was only marginal improvement in the quality of sand fraction. After the attrition tests were carried out and deslimed, the sand fraction was fed for tabling. The results of the tests were recorded in Table No. 2.

The –28 mesh sample gave the optimum value of assay 20.85% P_2O_5 with a recovery of of 79.1% P_2O_5 (Table-2). Another set of tests on the same line and addition of HCl with

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thorough agitation were conducted. The results of the tests were recorded in Table No. 3.

The result showed that the optimum product having an yield of 67% and assay of 22% P_2O_5 and 77.8% distribution of P_2O_5 in it were obtained from a sample of 28 mesh size.

Batch calcination Test [3] :

Attempts were next made to remove the calcitic gangue from the sample by converting it into CaO by calcination at 950°C followed by quenching, attrition, scrubbing and desliming. Calcination studies were carried out to determine the optimum calcination temperature at which the sample was almost free from CO_2 .

500 gms of −12mm size sample was kept in a crucible in an electric muffle furnace after required calcination temparature had been attained. The calcined products were quenched and deslimed. Results are shown in Table No. 4.

The results showed that at 1000°C and soaking time for 3 hours yielded deslimed sand product weighing 59 % and assaying 25.5 % P_2O_5 with a recovery of 85.2% P_2O_5 in it.

Sample 2 : Purulia phosphate [4] **:**

The sample of low grade phosphate from Beldih mines, Purulia was taken up for beneficiation studies. Previously, the sample was treated for upgrading by magnetic separation or flotation methods. Presently National Metallurgical Laboratory investigated the possibilities of upgrading by a cheaper method only in the range of 23% P_2O_5 grade. Chemical analysis of the phosphate from Purulia was : P_2O_5 = 18.69 %, SiO_2 = 9.86 %, Fe=20%, CaO=23.29%, MgO=1.39, Al_2O_3 = 5.86%, and LOI=6.83%.

The lump ore ranged from 5.5 cm to −100 mesh and was brownish grey to black in colour with varied shades of whitish and yellowish tinges. The apatite in this sample was associated with hydrated iron oxide minerals. Besides this, the ore contains hematite, chert and quartz.

Study on various sieve fractions of −10 mesh representative sample showed a grind below 65 mesh which would be required for the liberation of apatite from associated gangue minerals.

Experimental methods :

Although flotation of apatite from siliceous gangue employing fatty acid is a well established technique, the same was not adopted here. A batch of 500 gm −28 mesh sample was vigorously stirred in a Fagergren cell and deslimed. The results were shown in Table No. 5.

Tests were also conducted employing certain dispersants like :—

(i) Supplied by MMC and (ii) Archimite 600 and the slurry was allowed to settle for a short period and the slime was separated. The sand thus obtained was washed thoroughly. Results are given in Table No. 6.

Magnetic separation :

Further work was carried out using wet high intensity magnetic separator on − 65 mesh, − 100 mesh and − 150 mesh samples. The results were given in Table No. 7.

Work on high intensity wet magnetic separation at −65 mesh size resulted a concentrate with an yield of 40% assaying 31.4% P_2O_5 and 67.5% distribution in it. Tests at −100 mesh and −150 mesh gave better yield with a lower grade.

Discussion :

Mussourie phosphate samples of −14 mesh, 28 mesh and 35 mesh were subjected to attrition scrubbing. The sand portion, having relatively high P_2O_5 content when subjected to tabling, showed marginal improvement in the quality of sand fraction. The − 28 mesh sample gave the optimum value having an assay 20.85% P_2O_5 with a recovery of 79.1 % P_2O_5 in it (Table No. 2). Similar set of tests were carried out with addition of HCl and the results were to a certain extent better. The optimum product

obtained, had an assay of 22.0 % P_2O_5 with a recovery of 77.8% at −28 mesh size, (Table No. 3). However, calcination test at 1000°C temperature for 3 hours followed by desliming yielded a sand product of 59 % by weight assaying 25.5% P_2O_5 with a recovery of 85.2% in it. (Table No. 4).

Phosphate rock from Purulia ground to − 28 mesh size when subjected to vigourous agitation and desliming gave a sand product having an yield of 69.8% and assay of 23.30 % P_2O_5 and 87.5% recovery in it. Use of dispersants did not yield better results. Wet high intensity magnetic separation at − 65 mesh, yielded a product with 40 % yield, assaying 31.4 % P_2O_5 and 67.5 % recovery. At − 100 mesh, the product assayed 27.9 % P_2O_5 and 75.5 % recovery in it. In the test with − 150 mesh, the grade did not improve. The phosphate concentrate assayed nearly 10 % Fe and 11.8 % SiO_2 which is not objectionable when the same is used as a direct feed to the soil.

Conclusion :

Use of lean phosphate rock due to depletion of high grade material is extending into fertiliser industry. Mussourie phosphorite deposit is low grade and calcareous. Though calcination of the ore and its subsequent quenching and desliming were found to be a useful technique, the same involves thermal energy. A proper method should be chosen to treat lean phosphate rock where both capital and running costs are comparable to the cost of end product [5]. However, a cheaper method of beneficiation composing of grinding, acid scrubbing followed by gravity separation could produce a concentrate having an analysis of 22.8% P_2O_5 with a free CaO of 20.58%

Deposits of Purulia phosphate can be treated by magnetic separation. The non-magnetic fraction[6] would be a high quality phosphate product suitable for direct introduction into the soil. However involvement of huge investment for the high intensity magnetic separator, would rather be considered uneconomical in this case. Tests conducted with a coarse grind sample with vigourous agitation gave a yield of 69.8 % sand product assaying 23.3 % P_2O_5 with a recovery of 87.5 % in it. Separate tests using dispersant were also carried out but without success.

Keeping in view of restricted world production [3], we must be self-sufficient of indigenous phosphatic resources. The low grade phosphate rock can be used in Terai soils in Jalpaiguri, part of brown forest soil of Darjeeling and West Dinajpur which are acid soil regions. Most of the soils in Kerala, Tripura, Assam and Manipur are also covered under the acid soils in India.

Field demonstration of Mussourie phosphate in different states gave encouraging results. In Assam, Bihar, Orissa, West Bengal and Kerala trial runs gave Khariff 1975-76 and subsequent Rabi crop in order to assess the residual effect of Mussourie phosphate. Besides the field tests in acid soils, attempts were made to utilise Mussourie phosphate in neutral soils through Punjab Engineering University, Ludhiana, Haryana Agricultural University, and Agricultural University of Ranchi. The large scale demonstration programme as well as apatite resources were shown in Tables No. (8) and (9) respectively.

Acknowledgement :

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Table — 1 : Total reserves of phosphatic fertiliser minernl deposits in the country.

Sl. No.	Deposits	Locations	Total Reserve (in mT)	Proved Reserves	Average grade of $P_2 O_5$
1	JHAMAR KOTRA	Rajasthan	17.0 (High Gr.)	53.00	30.0 %
			36.0 (Med. Gr.)	—	22.0 %
2	JHABUA	M. P.	5.45	1.50	15 - 30%
3	MUSSOURIE	U. P.	45.00	10.39	16 - 18%
4	MATON	Rajasthan	9.20	4.00	26%
5	PURULIA	W. Bengal	7.50	1.95	10%
6	KANPUR	Rajasthan	4.0	—	10 - 12%
7	KASHIPATNAM	A. P.	1.72	0.64	35 - 42%
8	SINGHBHUM	Bihar	1.0	—	15%
9	LALITPUR	U. P.	4.0	0.5	18%
10	OTHERS	—	5.0	—	12 - 20%
		Total	135·87	71.98	

Table — 2 : Results of attrition scrubbing of crushed material

Feed size	Product	Wt. %	Assay % $P_2 O_5$	Dist % $P_2 O_5$
(a) — 14 mesh	Table conc.	70.2	19.50	73.5
	Table tail	22.0	17.00	20.1
	Slime	7.8	15.00	6.4
	Head (Calc)	100.0	18.60	100.0
(b) — 28 mesh	Table conc.	73.5	20.85	79.1
	Table tail	17.7	15.55	15.0
	Slime	8.8	13.04	5.9
	Head (Calc)	100.0	19.31	100.0
(c) — 35 mesh	Table conc.	68.5	21.31	77.0
	Table tail	21.2	16.02	17.9
	Slime	10.3	9.29	5.1
	Head (Calc)	100.0	18.95	100.0

Table — 3 Results of tabling tests on acid leached sample with attrition scrubbing

Feed Size	Product	Wt %	Assay % P_2O_5	Dist % P_2O_5
(a) — 14 mesh	Table conc.	68.5	21.31	77.0
	Table tail	21.2	16.02	17.9
	Slime	10.3	9.29	5.1
	Head (Calc)	100.0	18.95	100.0
(b) — 28 mesh	Table conc.	67.0	22.00	77.8
	Table tail	19.8	13.08	13.7
	Slime	13.2	12.21	8.5
	Head (Calc)	100.0	18.94	100.0
(c) — 35 mesh	Table conc.	58.5	22.80	69.8
	Table tail	23.5	14.50	17.8
	Slime	18.0	13.16	12.4
	Head (Calc)	100.0	19.11	100.0

Table No. — 4 : Results of the calcinatisn tests.

No. of test	Temp °C	Time in hours	Loss Wt %	Slime Wt % P_2O_5	Slime Assay %	Sand Wt % P_2O_5	Sand Assay %	Dist % P_2O_5
(i)	950	3	14.2	12.6	0.13	70.8	23.8	91.1
(ii)	1000	3	16.0	25.0	10.5	59.0	25.5	85.2
(iii)	1050	3	18.0	31.0	10.97	50.9	26.9	80.1

Table No. — 5 : Desliming followed by scrubbing; condition : settling time before desliming = 2 mins; Scrubbing time = 5 mins;

Condition	Product	Yield	Assay % P_2O_5	Dist % P_2O_5
No reagent, but with scrubbing	Sand	69.8	23.33	87.6
	Slime	30.2	7.68	12.4
	Head (Calc)	100.0	18.60	100.0

Table No. — 6 : Desliming followed by scrubbing.
Condition : Settling time before desliming = 2 mins;
Scrubbing time = 5 mins;
Reagent quantity 1% (V/V) solution 400 cc/400 gm sample.

Condition	Product	Yield	Assay % P_2O_5	Dist % P_2O_5
MMC reagent	Sand	83.33	20.20	90.8
With scrubbing	Slime	16.67	10.18	9·2
	Head (Calc)	100.00	18.50	100.0
Archimite 600	Sand	78.00	21.98	92.7
With scrubbing	Slime	22.00	6.16	7.3
	Head (Calc)	100.00	18.84	100.0
MMC reagent	Sand	74.00	22.24	86.8
With scrubbing	Slime	26.00	9.65	13.2
	Head (Calc)	100.00	18.96	100.0
Archimite 600	Sand	71.4	22.81	86.7
With scrubbing	Slime	28.6	8.68	13.3
	Head (Calc)	100.0	18.76	100.0

Table No. — 7 : Wet magnetic separation using Eriez high intensity magnetic separator :

Grind size	Product	Wt %	Assay % P_2O_5	Dist % P_2O_5
– 65 mesh	Non mag.	40.0	31.4	67.5
	Mag. I	28.5	10.0	15.0
	Mag. II	25.0	10.4	14.0
	Mag.III	6.5	10.0	3.5
	Head (Calc)	100.0	18.6	100.0
– 100 mesh	Non mag.	50.0	27.9	75.5
	Mag. I	18.1	9.0	8.8
	Mag. II	16.3	9.0	7.9
	Mag.III	15.6	9.2	7.8
	Head (Calc)	100.0	18.48	100.0
– 150 mesh	Non mag.	54.1	27.2	77.3
	Mag. I	24.6	8.2	11.2
	Mag. II	13.1	9.8	7.1
	Mag.III	8.2	9.8	4.4
	Head (Calc)	100.0	19.25	100.0

Table — 8 : Details of the large scale demonstration programme with Mussoorie phos. in different states of India.

Sl. No.	Name of the State	Season	Crop	Location		Area under Mussoorie phos. demonstration	Remarks
				District	Village		
1	ASSAM	Kharif 75-76	Paddy	Sibsagar Dibrugarh do	Narhiloidari Meripathar Melergial, Betjam	130 Hq	The Kharif (1975-76) has been harvested the observations are collected.
2	BIHAR	Kharif 75-76	Paddy and Potato	Ranchi	Sprong Panchayat Bijulia Panchayat Pali Panchayat	do	The Rabi crop has been sown to observe the residual effect of Mussoorie phos.
3	ORISSA	do	Ground Nut Agril, Moong and Urad	Dheb Kanal	Sobolabhanga, Shyam sunderpur, Kumarsigha, Shradhapur, Chenndipada, Kukurpeda	do	do
4	W. BENGAL	do	Paddy	Midnapur	Khudiasol, Lapuria	do	do
5	U. P.	Rabi 75-76	Wheat and Sugar cane	Dehradun	Panchnoldanga, Chandbella, Pathrisol, Teliwala Bullawala, Kurkawala of Markhamgrant Gram Sabha	100 Hq	Demonstration during Rabi 1975-76, have been started in wheat crop and large scale demonstrations will be started from spring 1976.
6	KERALA	Rabi 75-76	Paddy	Trichur do Palghat Ernakulam Kottayam Alleppey do do	Chathankole, Anthikad Arimboor Tri Thala, Koodatoor Keezhillam, Asseamannoor Changanssery, Parrippu Alleppey, Thankazhy Thalavay, Pathiyoor Thiruvella	402 Hq	The programme was started during Rabi 1975-76, and the crop will be harvested during March and April 1976.

Table — 9 : Apatite resources in India

State	Area	Nature of Occurance	Associated Rock	Tonnage		% P_2O_5	Remarks
				Prebable	Possible		
BIHAR	Singhbhum Dist.	Veins and Lenses	Apatite, magnetite biotite, chlorite rocky in phyllites and schists	1,094,165	—	15.48% (14—19%	In leased and non leased area. Amenable to beneficiation upto 37% P_2O_5 with a recovery of 82.37%. Feasibility of under ground mining to be studied.
WEST BENGAL	Kutni Purulia Dist.	Lenses, layers, stringers and vug filling	Sheared brecciated cherty and quartzite metabasic and phyllite	—	46,000	10.52%	Required up gradeing
	Chirugora Purulia Dist.	Lenses and thin layer	Phyllite and quartzites		40,000 20,000	6.72% 16.16%	Require beneficiation
RAJASTHAN	Udaypur Dist. a) Maton	Phosphatic stromatolites	Dolomite and chert	22,80,000	31,20,000	21.29%	Under production with HZL
	b) Jhamarkotra	do	do	71,80,000	3,20,00,000	+30%	Under production with RSMM
U. P.	Dehradun and Tehri Dist.						
	a) Kappu window			1,10,000	80,000	16.49%	
	b) Masrana			19,50.000	1,10,000	19.0%	
	c) Maldeota East				80,000	10.0%	
	d) Maldeota West				15,00,000	20.0%	

As far as NML's investigations are concerned.

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DISCUSSION :

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Question 1 : Paper does not speak any thing of magnetite association with the phosphatic deposit of Singhbum district. If the ore is associated with magnetite, what procedure should be adopted to separate it? Does the mineralogy, concentration and textural relationship of the phosphatic deposit with the gangue minerals change with the continuation of depth persistence?

Author : Purulia phosphate (as well as Singhbhum) having ferruginous impurities i.e. P_2O_5 18.69%, SiO_2 9.86%, Fe 20% on magnetic separation yielded 40% with 31.4% P_2O_5 & 67.5% recovery. The product gave 10% and 11.8% SiO_2 which is not objectionable when the sample is used as a direct feed in the soil. With scrubbing operation, the product can be used in neutral soil. The party has good response in Tea garden area/ Teria soil and Braswa forest area in Jalpaiguri and W.Dinajpur.

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Question 2 : Would you like to infrom us whether beneficiation tests have been conducted by you on the phosphorite deposits of Bhawnathpur, Dist.Palamau Bihar. There are good reserves of Phosphorite in Bhawnathpur but the P_2O_5 content is low. The samples of phosphorite have been tested at Agricultural University at Ranchi and by their direct application in the field, the production of soyabean, was reported to be doubled.

Author : So far we have not received any samples from Bhawanathpur/Palamau. However low grade samples of phosphate from Singhbhum Dist. were carried out. The ferruginous variety of ore having 10% to 15% P_2O_5 was treated by wet HIMS and subsequent flotation, which yielded a grade of 35% P_2O_5. We are keeping in touch with the sponsor to get current information from M/s. PPCL and M/s. WBMDC. However we are not in a position to furnish it at the moment.

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