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INDIAN PHOSPHATE AND ROCKS: SCOPE FOR THEIR USE IN FERTILIZER AND CROP PRODUCTION - A REVIEW

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SUMMARY

The enclosed article examines the scope of Indian rock phosphates, including Mussoorie Phos, for use in fertilizer and crop production. The salient points brought out are presented below.

1. None of the Indian rock phosphates conform to the suitability requirements for use in fertilizer manufacture.

2. None of them, including Mussoorie Phos, is of reactive nature as judged from the crystal chemical structure and other chemical properties.

All of them have very low CO_3/PO_4 ratios which further confirms their non-reactive nature.

3. Soil acidity increases the availability of phosphates only with reactive phosphate rocks. Since Indian rock phosphates are unreactive, as indicated above, the soil acidity does not release the phosphate from them when applied to soils.

4. Field experiments on rice and other short duration crops conducted on acid soils with the direct application of powdered Indian rocks including Mussoorie Phos have given yields comparable only to control. It is evident that Indian rock phosphates are not suitable for direct application as a fertilizer in crop production.

INDIAN PHOSPHATE ROCKS: SCOPE FOR THEIR USE IN FERTILISER AND CROP PRODUCTION—A REVIEW

The scope for use of Indian phosphate rocks in fertiliser and crop production has been reviewed based on the composition of ore and crystal chemical structure of the apatite in the ore. The influence of soil acidity and crop species on the dissolution of P and agronomic effectiveness has been also discussed. The review concludes with a few suggestions for future work.

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With intensive cropping, the use of phosphatic fertilisers, in addition to N, has increased. The Government of India is at present going in a big way for increasing the production of pulse and oilseed crops. These crops have higher P requirements. Increasing cultivation of pulse and oilseed crops and other cereals will thus have higher demand for phosphatic fertilisers. In earlier years, the fertiliser industries in India were importing both the raw materials—phosphate rock and sulphur sulphuric acid for the manufacture of phosphatic fertilisers. In recent years, these materials are being increasingly substituted with imports of phosphoric acid. This has resulted in a marked increase in the cost of the nutrient P in the phosphatic fertilisers.

Several deposits of phosphate rocks have been identified in India during the past 25-30 years and the ore from many of these deposits is being mined. Work on the suitability of these Indian phosphate rocks for the manufacture of phosphatic fertilisers as well as their direct use in crop production was started during the mid-1960. Reports of more systematic work are being published from mid-1970s. This review is an attempt to provide reliable information on suitability of Indian phosphate rocks for fertiliser and crop production.

Use of Indian Phosphate Rocks for Production of Phosphatic Fertilisers

Phosphatic fertilisers are produced by the reaction of phosphate rock with sulphuric acid or phosphoric acid in the wet process or by the thermal process. Therefore, the composition of the ore is important in determining the suitability of their use in the phosphate fertiliser industry. Information on the characters and chemical composition of several Indian phosphate rocks is available (2, 4, 10, 13). From the chemical composition of different phosphate rocks, it was reported that none of the sources except one from Madhya Pradesh was suitable for production of phosphatic fertilisers by the wet process, as these contained less than 13 per cent P; the Mussoorie phosphate rock hardly qualified for use in the phosphate industry, while another grade from Madhya Pradesh was considered suitable for use in the thermal process because of its high silica content. The remaining sources were considered suitable for production of elemental P as they contained about 8-10 per cent (10).

Besides the P content of the phosphate rocks, the nature and amount of accessory minerals present in the ore also determined their suitability in the production of phosphate fertilisers by the wet or thermal processes. The nature and amount of different minerals are easily determined by x-ray diffraction analysis (XRD). Both qualitative and quantitative estimates of the accessory minerals in the ore have been reported (2, 7, 13). These phosphate ores contained appreciable quantities of accessory minerals, especially calcite, dolomite and silica. Phosphate rocks having 13 per cent P content or more, CaO P_2O_5 ratio close to 1.6 and lower contents of accessory minerals containing Ca, Mg, Fe, Al, CO_3 are considered suitable for the manufacture of phosphatic fertilisers in the wet process. In other words, suitable rocks should have an apatite content of 80 per cent or more. Higher contents of accessory minerals entail use of large quantities of acid, blind filters on formation of viscous slurry due to excess Mg, result in sludge and scale formation and precipitation of P due to excess Al, Fe etc.; and in the corrosion of the reaction vessel due to presence of excess Cl and F. The desirable composition of phosphate rocks for the wet process production of P fertilisers was 30-32% P_2O_5, 2.5-3.0% F, 2.5-3.0% SiO_2, 3% R_2O_3 and 4.5% CO_2. Data on chemical composition and content of accessory minerals indicated that none of the Indian phosphate rocks conformed to the suitability requirements for use in the wet process.

Phosphate rocks containing higher amounts of SiO_2 are found suitable for production of P fertilisers like defluorinated phosphate or Rhenania phosphate by the thermal process. The phosphate rocks from Meghnagar (chirt based) and Maldeota may be considered suitable for the production of Rhenania type phosphate fertiliser.

Criteria for Direct Use of Phosphate Rocks in Crop Production

While the amounts of apatite present in the phosphate rocks determine their suitability for use in the production of phosphatic fertilisers, the crystal chemical structure of apatite in the phosphate rocks determines their suitability for direct use in crop production.

Crystal chemical structure of Indian phosphate rocks

The pure fluorapatite may be represented by the empirical formula $Ca_{10}(PO_4)_6F_2$. Sedimentary phosphate rocks contain fluorapatites which are affected by isomorphous replacement **of their constituent ions like Ca^{+2}, PO_4^{3-} and F^- by (Na^+, K^+, Mg^+), (CO_3^{2-} + F^-) and (OH^-, Cl^-) respectively.** This isomorphous replacement affects the unit cell dimensions (a_0 and c_0 axes) of hexagonal apatite crystal. The unit cell dimensions are determined by XRD and a computer programme (12). From elemental analysis and stoichiometry, the CO_3 substitution (x) of PO_4 (x/6-x) could be determined (12) where the theoretical and observed limits on 'x' was such that the value of x/6-x was less than or nearly equal to 0.3. Higher substitution would disrupt the apatite structure. From the information on a_0 obtained from XRD, apatite of sedimentary phosphate rocks would be represented by the general formula (12):

$$Ca_{10-a-b}Na_aMg_b(PO_4)_{6-x}(CO_3)_xF_{2+y}$$

the values of x, a, b and y being calculated from the relations:

a_0 (obtained from

$$XRD) = 9.374 - 0.204\ \frac{x}{6-x}$$

$$a\ (Na) = 1.327\ \frac{x}{6-x}$$

$$b\ (Mg) = 0.515\ \frac{x}{6-x}$$

$$y\ (\text{average value}) = 0.4\ x$$

These sophisticated facilities of XRD with a related computer programme are not available in many laboratories, especially in the developing countries. From the data on the NH_4-citrate extractable P of the rock in the 1, 2, 1 +2, 1 to 5 and 1 to 8 extract, the a_0 values were calculated (6) with the help of the corresponding regression equations. The calculated a_0 values for each of the extracts, along with the a_0 values obtained from XRD (7) are presented in *Table* 1. Statistical comparison of the a_0 values obtained from XRD and those from calculated from different NH_4-citrate extracts show that the a_0 values calculated from cumulative P in the 1 to 5 or 1 to 8 extract, expressed either as per cent of rock or as per cent of P in the rock were statistically on par with those obtained from XRD, while those obtained in 1, 2 or 1+2 citrate extracts were different. The crystal chemical parameters, a_0, CO_3/PO_4 and F/P_2O_5 of apatite in the Indian phosphate rocks could be calculated from equations (7):

$$a_0 = 9.368 - 0.0040\ x_1$$

$$= 9.367 - 0.00049\ x_2$$

$$CO_3/PO_4 = 0.0197 + 0.0027\ x_2$$

$$F/P_2O_5 = 0.0957 + 0.0027\ x_1$$

$$= 0.0364 + 0.00034\ x_2$$

where x_1 = cumulative P in the first five successive NH_4-citrate extract of phosphate rocks expressed as per cent of the rock

Table 1—Comparison of 'a_0' values of Indian phosphate rocks obtained from x-ray data and from citrate soluble P in successive extracts

Phosphate rock	a_0 Å x-ray	a_0 Å through citrate soluble P (% of rock) in extracts					a_0 Å through citrate soluble P (% of total P) in extracts					Mean
		1	2	1+2	1 to 5	1 to 8	1	2	1+2	1 to 5	1 to 8	
Mussoorie	9.3648	9.3591	9.3601	9.3596	9.3616	9.3636	9.3560	9.3573	9.3566	9.3578	9.3592	9.3591
Jhamarkotra	9.3762	9.3624	9.3624	9.3625	9.3651	9.3676	9.3608	9.3607	9.3608	9.3628	9.3652	9.3630
Kasipatnam	9.4496	9.3627	9.3619	9.3625	9.3655	9.3679	9.3614	9.3604	9.3610	9.3637	9.3666	9.3633
Purulia	9.3052	9.3597	9.3615	9.3607	9.3637	9.3663	9.3575	9.3568	9.3585	9.3612	9.3636	9.3612
Meghnagar I	9.3767	9.3628	9.3621	9.3626	9.3651	9.3676	9.3616	9.3611	9.3614	9.3636	9.3661	9.3634
Meghnagar II (Lime stone based)	9.3777	9.3635	9.3643	9.3640	9.3670	9.3693	9.3623	9.3633	9.3628	9.3654	9.3676	9.3650
Meghnagar III (Chirt based)	9.3732	9.3634	9.3630	9.3633	9.3660	9.3683	9.3620	9.3614	9.3618	9.3639	9.3661	9.3639
't-test'		2.2391 •	2.2187 •	2.2192 •	1.9703 ns	1.7510 ns	2.3932 •	2.3656 •	2.3783 •	2.1719 ns	1.5675 ns	2.1684 ns

• = Singnificant at 5% level.

ns = not significant.

Source: **(7).**

and x_5 = cumulative P in the first five successive NH_4-citrate extract of phosphate rocks expressed as per cent of total P in the rock.

The empirical formula of the apatite, calculated from a_0 of the chemical-statistical model, theoretical P % and ACS are also presented in *Table 2*.

Table 2—Empirical formula and absolute citrate solubility (ACS) of Indian phosphate rocks obtained from a_0 values calculated from P in 1-5 citrate extract (Table 1) by the chemical-statistical model

Phosphate rock	Empirical formula	Theoretical P%	ACS*
Mussoorie	$Ca_{9.87} Na_{0.09} Mg_{0.04} (PO_4)_{5.61} (CO_3)_{0.39} F_{2.15}$	17.4	5.6
Jhamarkotra	$Ca_{9.91} Na_{0.07} Mg_{0.02} (PO_4)_{5.72} (CO_3)_{0.28} F_{2.11}$	17.7	4.1
Kasipatnam	$Ca_{9.92} Na_{0.06} Mg_{0.02} (PO_4)_{5.74} (CO_3)_{0.26} F_{2.11}$	17.8	3.9
Purulia	$Ca_{9.90} Na_{0.07} Mg_{0.03} (PO_4)_{5.68} (CO_3)_{0.32} F_{2.13}$	17.7	4.6
Meghnagar I	$Ca_{9.92} Na_{0.06} Mg_{0.02} (PO_4)_{5.73} (CO_3)_{0.27} F_{2.11}$	17.8	4.0
Meghnagar II	$Cs_{9.93} Na_{0.05} Mg_{0.02} (PO_3)_{5.76} (CO_3)_{0.24} F_{2.09}$	17.9	3.3
Meghnagar III	$Ca_{9.92} Na_{0.06} Mg_{0.02} (PO_4)_{5.74} (CO_3)_{0.26} F_{2.10}$	17.8	3.8

*ACS = 341 (9.376 − a_0).

From the XRD of different phosphate rocks (*Figure* 1.), diffraction peaks for 2-theta values in the range 25.0-34.0°, corresponding to 'd' spacing of 3.46, 3.08, 2.82, 2.79, 2.72 and 2.63 Å indicate the presence of apatite in the phosphate rocks (7). Assuming all the P in the ore is apatite bound, a comparison between theoretical P content of the rock calculated from the empirical formula (Table 2) and that actually obtained (6) would show that the calculated apatite in the rock was in the range 48-68 per cent, which was more or less comparable to that obtained from XRD (7).

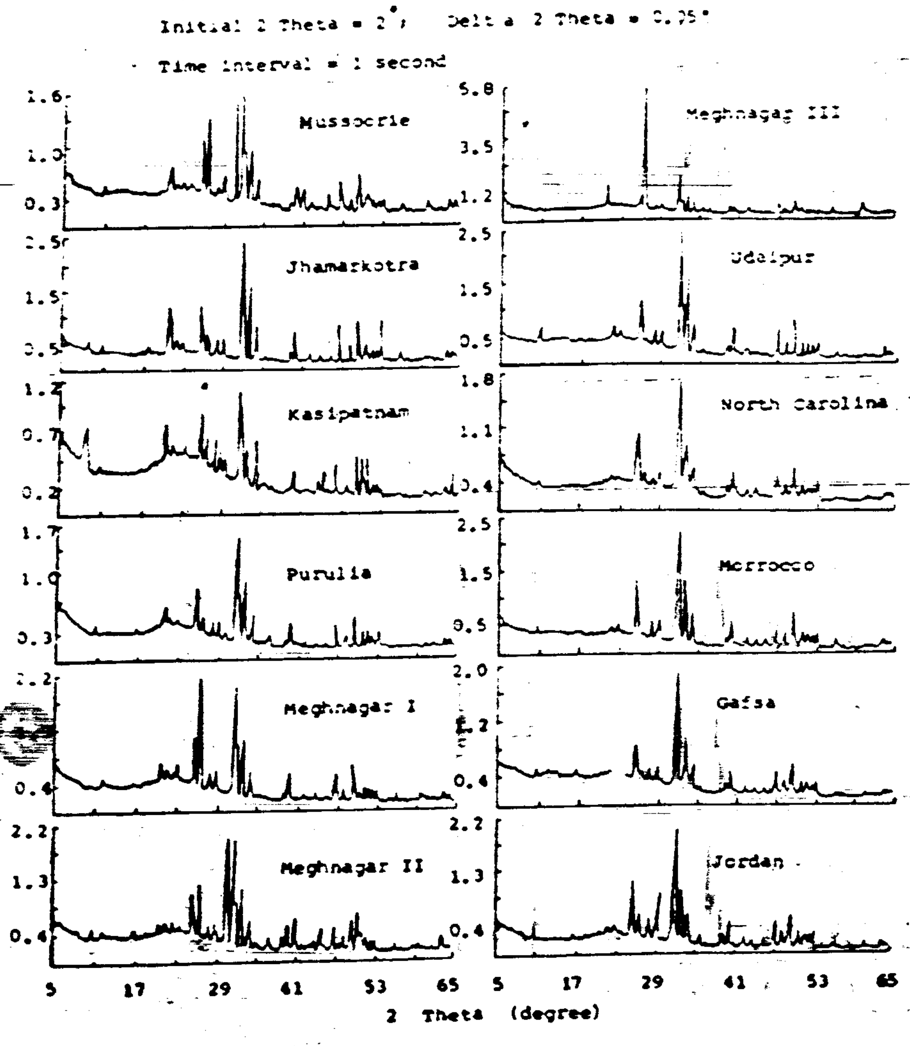


Figure 1—X-ray diffractogram of Indian and exotic rockphosphates.

It may also be seen from the empirical formula, calculated from the chemical-statistical model (Table 2), that there is very little substitution of CO_3 + F for PO_4 in the apatite of the Indian phosphate rocks. The IR spectra (*Figure* 2) of Indian and North Carolina phosphate rocks show typical absorption bands of P-O for apatite minerals at 575, 602 and 1040 cm^{-1}. The nature of IR spectra of carbonate-apatite, carbonate-fluorapatite and hydroxy-apatite, are already characterised and North Carolina phosphate rock has been established to contain carbonate-apatite, which has absorption bands at 1420 and 860 cm^{-1}. A comparison of absorption bands of eight Indian phosphate rocks with those of North Carolina phosphate rock (*Figure* 2) indicated that the former were close to fluorapatite with little or no substitution of CO_3 + F for PO_4 in the apatite crystal (2, 7, 13). Hence, if from field geology or from IR spectra, an unknown phosphate rock is established to contain carbonate-apatite, in the absence of sophisticated XRD and computer facilities, it may be possible to compute the crystal chemical parameters, a_0, CO_3/PO_4 and F/P_2O_5 of apatite present in the

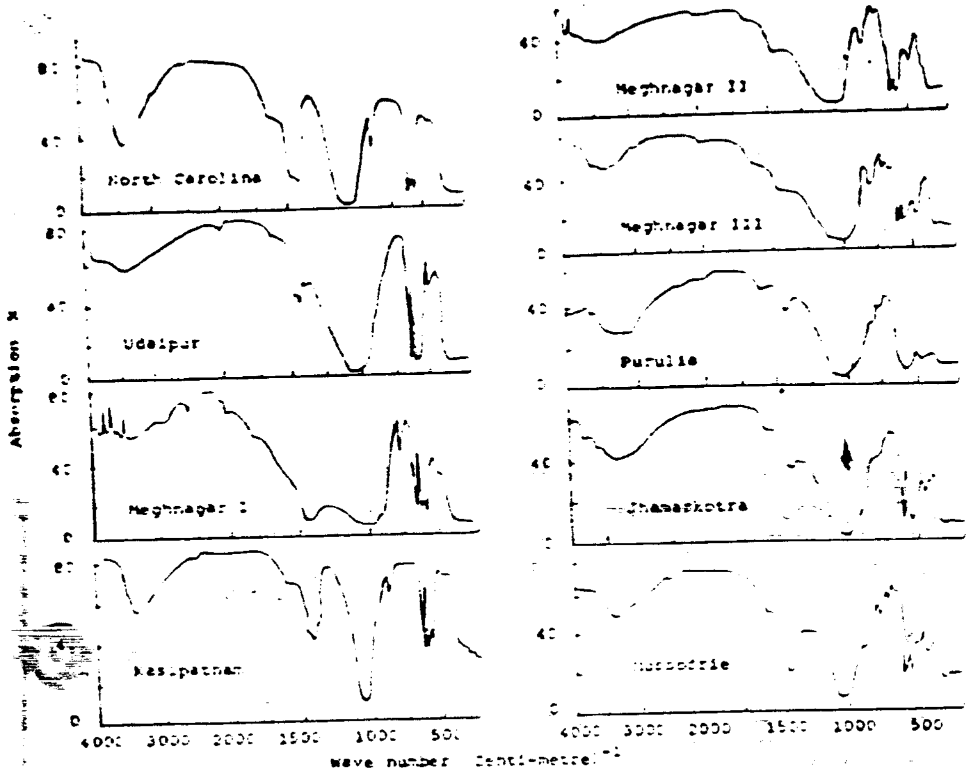


Figure 2—IR spectra of Indian and North Carolina rockphosphates.

rock with a reasonable degree of accuracy, from accurate information on cumulative P in the 1 to 5 successive extract of the phosphate rock in the NH_4-citrate, with the chemical-statistical model (7).

Reacticity of Indian phosphate rocks in relation to their crystal chemical structure

The effectiveness of phosphate rocks from different geologic and geographic origins, when used directly for crop production, has been reported to be variable. This is attributed to their differences in reactivity. The reactivity is measured in the laboratory from the amount of P extracted from the phosphate rock by NH_4-citrate, 2 per cent citric acid, 2[illegible]ent formic acid or 1 per cent la[illegible]acid or NaEDTA (12).

The reactivity of sedimentary phosphate rocks, as measured by extraction with NH_4-citrate is considerably influenced by the amounts of accessory minerals like calcite, dolomite, gypsum etc. present in the sample. Higher contents of these minerals consume a part of the added citrate, as a result of which the release of P into the extractant is reduced in the first extraction. Hence, for the determination of reactive P, the first extract is discarded and the second extract is taken into consideration (12). But citrate was found to interfere in the colorimetric determination of P by the vanado-molybdate colour method (6). A modification of the more sensitive sulphomolybdate-$SnCl_2$ blue colour method was used for determination of very small amounts of NH_4-citrate extractable P of phosphate rocks in presence of 0.1 meq of NH_4-citrate (6).

Petrological examination of different phosphate rocks showed that the apatite in most sedimentary ores was homogeneous (12). Most of the P forms in a given deposit consist of a single type of apatite, whose physical and chemical characteristics are relatively uniform. However, the concentration of apatite in the ore varies considerably from one location of the deposit to another. This is known as the grade of phosphate rock. Hence, the reactivity of the phosphate rocks for a given deposit cannot be related to the grade of the ore. Hence the solubility of P in the phosphate rocks may be expressed in terms of the grade of the apatite, which is relatively uniform in a given deposit, instead of the grade of the phosphate rock which was not constant (12) and a reactivity index (aboslute citrate solubility, ACS), was suggested in preference to conventional citrate solubility. These two indices have been defined as:

$$\text{Conventional citrate solubility} = \frac{\text{citrate soluble P}}{\text{total P in the rock sample}} \times 100$$

$$ACS = \frac{\text{citrate soluble P}}{\text{theoretical P \% apatite}} \times 100$$

Information on the unit cell length a_0 is therefore essential to know the theoretical P content of the apatite for determination of ACS. It is also related to a_0 by the equation:

$$ACS = 341 (9.376 - a_0)$$

and gives a more reliable measure of the reactivity of phosphate rock than NH_4-citrate soluble P alone.

Bhujbal and Mistry (2), with the use of Indian phosphate rocks from Udaipur, Jhamarkotra, Purulia, Jhabua, Maldeota, Durmala, Mussoorie and Kasipatnam and exotic phosphate rocks from North Carolina, Central Florida and Tennessee, established a significant linear relationship between NH_4-citrate soluble P and mole ratio CO_3 PO_4 of the rocks (*Figure* 3). In these studies, the Indian phosphate rocks CO_3 PO_4 of less than 0.06, had much less than 1.0 per cent of NH_4-citrate soluble P, whereas the phosphate rocks from Tennessee, Central Florida and North Carolina having CO_3 PO_4 of 0.065, 0.140 and 0.252 had citrate soluble P of about 1.0 per cent, 1.5 per cent and 3.0 per cent respectively.

The relationship between ACS and CO_3 PO_4 for eight Indian (Mussoorie, Jhamarkotra, Kasipatnam, Purulia, Udaipur and three ores from Meghnagar), six US (North Carolina, North Florida, Central Florida, Missouri, Idaho and Tennessee), two African (Morocco and Gafsa) and one middle east (Jordan) phosphate rocks was found to be linear and highly significant (Figure 8). This population had more number of values of CO_3/PO_4, especially in the range 0.06-0.262 as compared to those of Bhujbal and Mistry (2). The eight Indian phosphate rocks and Missouri (US) rock, which had CO_3/PO_4 of less than 0.06, had ACS values less than 6. The Idaho and Tennessee rocks,

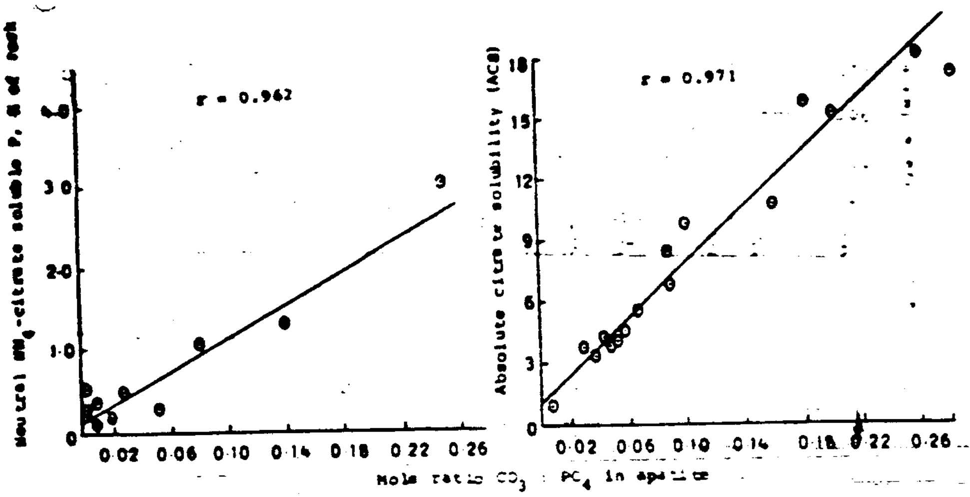


Figure 3—Relations of mole ratio CO_3: PO_4 of phosphate rocks with citrate soluble P and ACS.

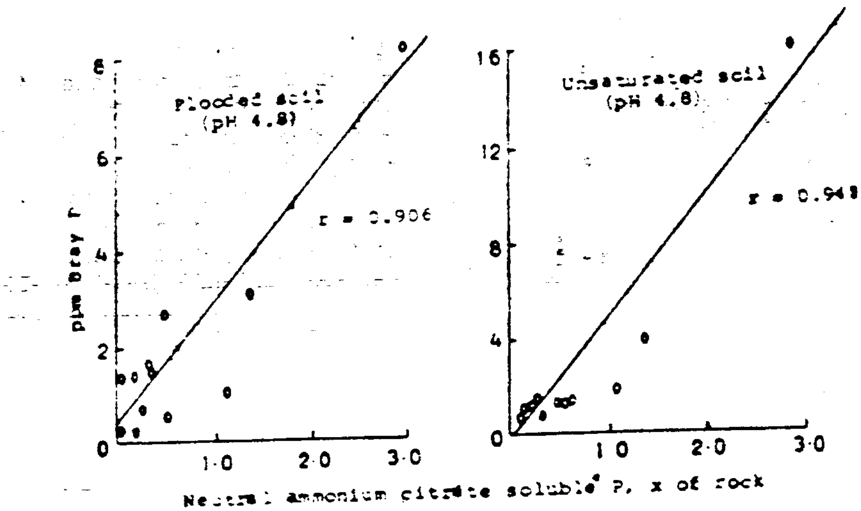
which had CO_3:PO_4 in the range 0.06-0.09, had ACS in the range 6-9. The remaining phosphate rocks with CO_3:PO_4 of 0.10-0.26, had ACS of 9-18. The North Carolina phosphate rock had the maximum possible substitution of CO_3 + F for PO_4 in the apatite crystal to make it stable. With the ACS of North Carolina rock being taken as 100, the phosphate rocks having relative ACS below 40 per cent with reference to North Carolina phosphate rock are unreactive and unresponsive (9). The Indian phosphate rocks had ACS values of 31 per cent or less relative to North Carolina phosphate rock. Hence, these may be considered unreactive.

Agronomic Effectiveness of Indian Phosphate Rocks

From the discussions presented so far, it may be seen that the crystal chemical structure, especially mole ratio CO_3:PO_4 (CO_3 + F substitution for PO_4 in apatite crystal) of the apatite, is an indication of reactivity of phosphate rocks which is measured by neutral NH_4-citrate extractable P. The Indian phosphate rocks had very low CO_3:PO_4 ratios, hence were not very reactive. The dissolution of P from phosphate rocks may be hastened under acid soil conditions resulting in increased available P for crop growth. Further, the types of crops—cereals, legumes, oilseeds etc. may have differential ability to absorb P from a fertiliser-soil system involving phosphate rocks. Thus the three types of driving forces—crystal chemical structure of apatite in the phosphate rock, edaphic factors and the plant factor would determine the efficiency of phosphate rocks for direct application in crop production. Some of these aspects are discussed here.

Soil factors favouring dissolution

Low soil pH in the acid range is likely to dissolve the apatite in the phosphate rock. Bhujbal and Mistry (1), working with eight Indian phosphate rocks and three US rocks obtained significant correlations between citrate soluble P in the rock and Bray I extractable P in flooded or saturated acid soil of pH 4.8. Their data, presented in Figure



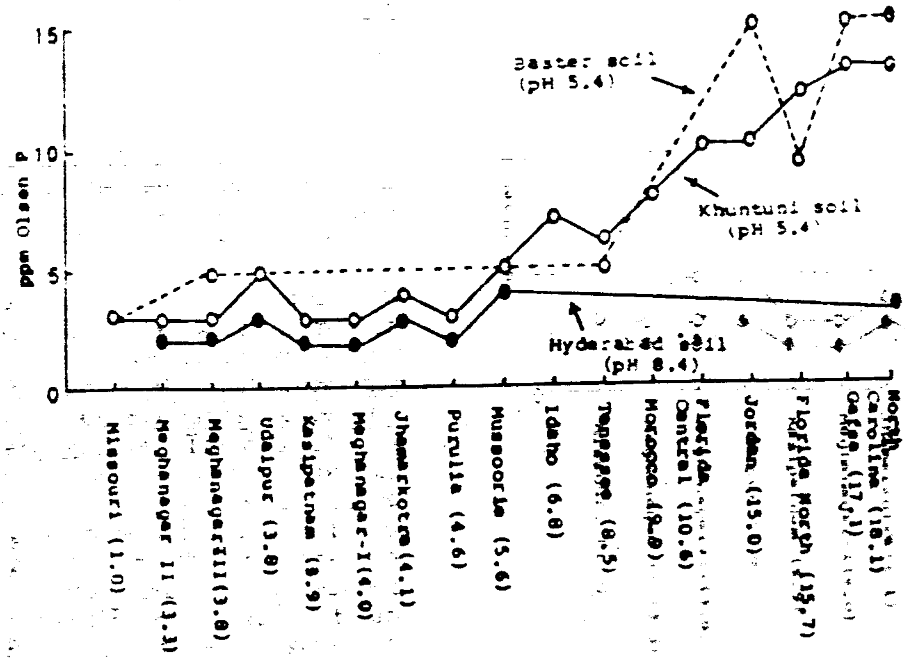


Figure 4—Available P in different soils as influenced by application of phosphate rocks of varying citrate soluble P or ACS.

4 show that the Bray 1 extractable P for the Indian phosphate rocks added at 200 ppm P was less than 2 ppm P, which might be considered deficient for optimal growth of any crop.

Data on Olsen's P as influenced by the application of 60-100 ppm P as Indian, US, African and Middle East phosphate rocks in soils of pH 4.2-5.8 has been arranged in ascending order of ACS (Figure 4). All the Indian phosphate rocks, which had relative ACS of 30 per cent or less, gave Olsen's P value of 5 ppm or less, which might be considered deficient for crop growth, while those from Morocco, Central Florida, Jordan, North Florida, Gafsa and North Carolina, which had relative ACS of more than 60 per cent, gave 8-15 ppm Olsen's P in soil, which might be considered adequate for crop growth. Chaudhary and Mishra (3), working with a soil of pH 5.6, however obtained an increase in Bray P in the range 52-59 ppm with Indian phosphate rocks applied at 500 ppm P, in an equilibration time of 30 days. But no such transformations even with North Carolina phosphate rock was observed (6) in a soil having pH 6.4 (data also given in Figure 4). It may therefore be inferred that soil acidity was possibly one of the edaphic driving forces in increasing P availability from phosphate rocks and further the P availability increased in acid soils to moderate levels only with reactive phosphate rocks.

Crop response to application of Indian phosphate rocks

Dash *et al.* (6) and Dash and Pattnaik (unpublished) have tried to evaluate crop factors by growing rice and *dhaincha* separately on acid soils. In these studies, the phosphate rocks were primed under moist aerobic soil condition before growing rice. Data on dry matter production, P content and uptake for the two crops, arranged in ascending order of ACS, are presented in *Figure* 5. It may be seen that Indian phosphate rocks, though gave slightly higher dry matter production than control, the yields were as poor as control compared to Jordan, Gafsa and North Carolina phosphate rocks, whose yields were comparable to that of superphosphate.

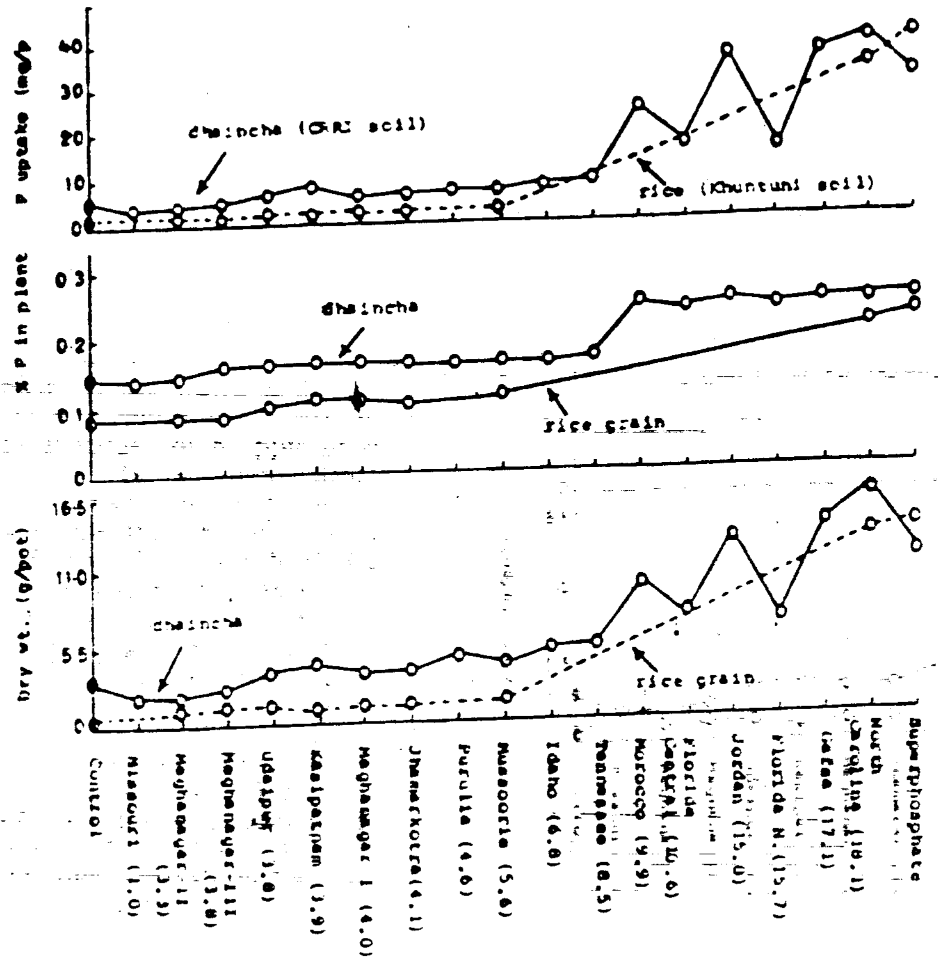


Figure 5—Dry matter production, P content and uptake in rice and dhaincha grown on acid soils with different phosphate rocks arranged in ascending order of ACS.

Figures in parentheses indicate the ACS values.

The relative agronomic effectiveness (RAE) of phosphate rocks for rice on acid soils has been evaluated with triple superphosphate (TSP) as 100. The relative economic effectiveness (REE) is the product of RAE and the price ratios of P_2O_5 in TSP/RP. So phosphate rock may be directly used in place of TSP only when REE exceeds 100.

From the foregoing discussions, it may be seen that raw powdered Indian phosphate rocks may not be suitable for direct application as a fertiliser in crop production. There are, however, reports with different crops which show the efficiency of Indian phosphate rocks, especially from Mussoorie and Purulia, approaching superphosphate (8, 11, 15). The driving forces favouring such increased crop production with application of unreactive Indian phosphate rocks could not be explained in the light of rock's reactivity. So this needs further study. Panda (14) has suggested that depending on soil acidity and cropping system, the cost of phosphate fertilisation could be reduced by adding small portions of water soluble P as superphosphate to large portions of Indian phosphate rocks. Tandon (16) has listed this as a low priority item, possibly because there will still be need to manufacture superphosphate which required additional imports of sulphur (sulphuric acid).

Scope for the use of partially acidulated phosphate rock as a P fertiliser

Hydrochloric acid effectively substitutes H_2SO_4 or H_3PO_4 for acidulation of phosphate rock to produce phosphatic fertilisers (5). Phosphate rock acidulated with HCl, H_2SO_4 or H_3PO_4 to the extent of 50% of com-

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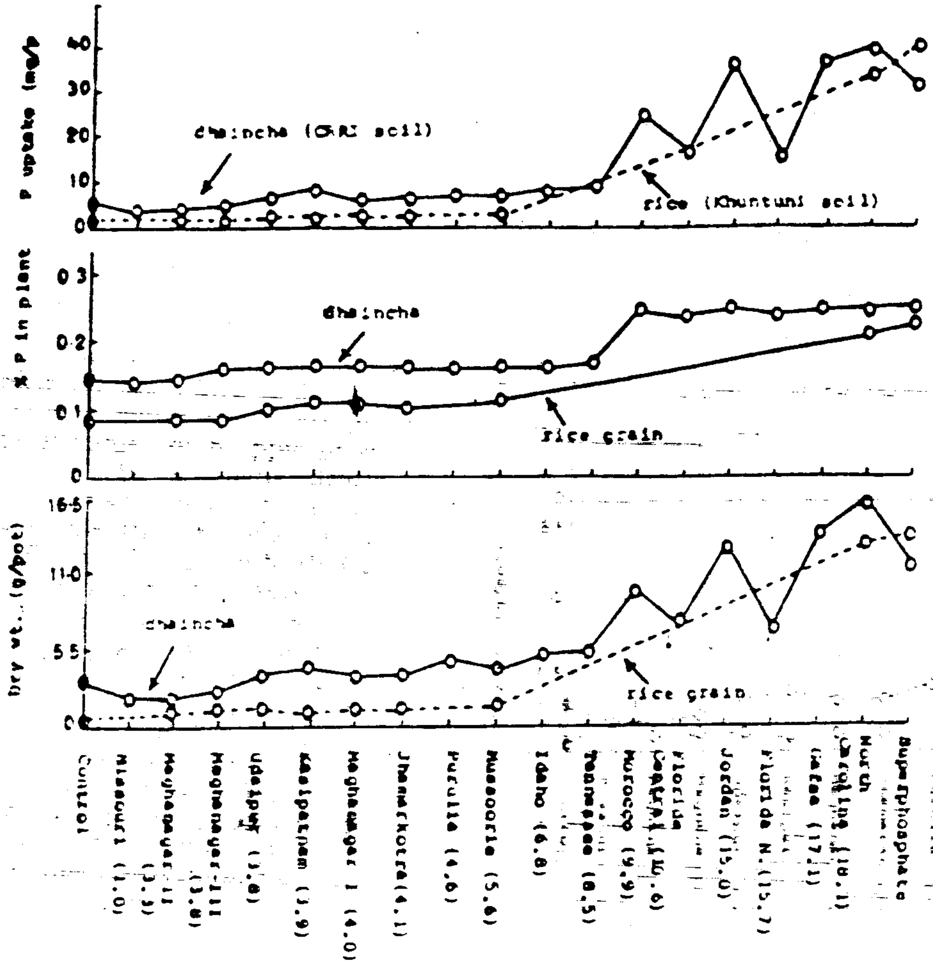

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