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AN INVESTIGATION OF THE AGRONOMIC VALUE OF FINE GRINDING AND GRANULATING REACTIVE PHOSPHATE ROCKS.

A thesis presented in partial fulfilment of the requirements for the degree of Master of Horticultural Science in Soil Science at Massey University New Zealand.

Sally Jane Officer

1989

ERRATA

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138	1	8	106 mgP/pot(	106 mgP/pot (where the x axis is the mg P dissolved per pot)
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175	1	14	in the ratio to formic acid volume basis	ratio of phosphate rock to extractant solution
193	equation 3		Division line missing over $P^{32}c + P^{32}ex$	
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ABSTRACT

The future trends in the use of reactive phosphate rocks in New Zealand may be dependent on improving the handling characteristics of these fine sand- and powder-like materials. Granulation of these materials has been suggested as one option. The effect of fine grinding and granulating reactive phosphate rocks on their agronomic performance was evaluated using a range of phosphate rocks, in laboratory studies and in field and glasshouse trials.

North Carolina, Arad, Sechura and White Youssafia phosphate rocks, in forms normally imported into New Zealand (sand sized material, majority <2mm particle size), were characterised in terms of origin, composition, particle size, and solubility in 2% formic acid. In a 30 minute formic acid extraction of the imported material, White Youssafia phosphate rock at 44% solubility was found to be less reactive than the other phosphate rocks, which ranged from 47% to 55% in formic solubility

In preliminary field trials a very finely ground North Carolina phosphate rock (100% <42µm particle size) was granulated with K_2SO_4. The ungranulated phosphate rock, and granules of 0.5-1mm, 1-2mm and 2-4mm diameter, were evaluated on permanent pasture on the Tokomaru silt loam, using an inverse isotopic dilution technique in which the field soil, at the 1.5-6cm depth, was uniformly labelled with ^{32}P by a novel injection method. No plant yield response to fertiliser was observed but significant differences in herbage phosphate content and specific activity indicated a phosphate uptake response to fertiliser. Despite careful selection of areas of sward which had a similar plant content and vigour, the large variability in data from replicate treatments limited the amount of information which could be drawn from the results but the data indicated that the agronomic performance of the finely ground North Carolina phosphate rock was not limited by granulating to 0.5-1mm (mini-granules).

A further range of granulation agents, including neutral salts, organic and mineral acids, their salts, and tallow, were tested for their ability to form strong mini-granules from unground North Carolina phosphate rock. The best granulation agent was a 1:0.6 mixture of citric acid and magnesium sulphate, producing 0.5-1mm mini-granules which had an arbitrary crushing strength of 0.8kg/granule. The production of mini-granules involved pre-drying a phosphate rock/granulation agent slurry until it was just unsaturated, followed by cutting the wet mix through a 0.710mm seive, granulation at high speed for 30 seconds, and drying of the granules at 80°C for approximately 2 hours. This granulation process was then used to manufacture granules from unground Sechura and Arad phosphate rocks, as well as ground North Carolina and Arad phosphate rocks. Ground North Carolina phosphate rock was also granulated using tallow, by melting the fat and mixing in the phosphate rock, followed by setting the mix in a mould.

Granulated materials, including a commercially prepared product ("Hyphos"), and ungranulated phosphate rocks (including White Youssafia), were evaluated in a glasshouse pot trial. The fertiliser was applied to the surface of pots of established "Nui" perennial ryegrass, with 7 harvests over three and half months.

In general, at the common application rate of 60kgP/ha, the phosphate rock materials were never more than 70% as effective as mono calcium phosphate. The yeilds derived from unground, ungranlated Sechura, North Carolina, Arad and White Youssafia phosphate rocks were similar, the only significant difference being that the yield derived from Sechura phosphate rock was greater than the yield derived from North Carolina phosphate rock.

The effect of mini-granulation on agronomic performance varied with with the type and particle size of the phosphate rock used to make the granules. For example, mini-granulation of "as received" North Carolina and Sechura phosphate rocks caused no reduction in phosphate

availability from these materials, however, mini-granulated "as received" and works ground Arad phosphate rock caused a significant reduction in phosphate availability.

The agronomic performance of North Carolina phosphate rock was improved by grinding to less than 250μm in particle size but no further improvement occurred if the phosphate rock was more finely ground (<42μm particle size). The agronomic performance of Arad phosphate rock was not improved by grinding.

The sequential fractionation of soil phosphate (1MNaOH followed by 1MHCl) indicated that only approximately 8% of the works ground North Carolina phosphate rock fertiliser had dissolved in the soil at the 5th harvest (10 weeks). A comparison of yields derived from pots fertilised with different rates of K_2HPO_4 sprayed onto chromite (whch had a similar particle size distribution to the unground phosphate rocks) indicated that the dissolved phosphate in the soil from the phosphate rock had a similar agronomic value to the K_2PO_4. The low amount of phosphate rock dissolution and the absence of increased of yield response when works ground North Carolina phosphate rock was applied to soil at rates greater than 40 kgP/ha indicated that soil factors were limiting the dissolution of phosphate rock in this experiment. The extent of the limitation varied depending on the phosphate rock type and also the type of pot used (the black polythene bag used for the majority of treatments was enclosed in a galvanised steel cylindar for an inverse isotopic dilution experiment). The variable effects of grinding and granulation were attributed to the limitation of the phosphate rock dissolution.

The type of granulation agent (including partial acidulation) had no significant effect on the agronomic performance of the granulated materials, except when tallow was used as a granulation agent and reduced the availability of works ground North Carolina phosphate rock. Unground White Youssafia phosphate rock requires further testing under more rigorous conditions before conclusions can be made about its agronomic availability.

Two isotopic techniques were utilised in the glasshouse experiment in an attempt to quantify the extent of phosphate rock dissolution in the soil. The surfaces of some phosphate rock treatments were sprayed with a carrier free solution of P^{32}, and the inverse isotopic dilution technique used in the field was used again on some treatments.

The use of labelled $K_2HP^{32}O_4$ as a control for the surface labelled experiment provided sufficient information to allow differentiation of phosphate in the plant which was derived from soil and the fertiliser but the model developed could not be directly applied to results from the phosphate rock treatments. The dissolution of different forms of phosphate rock could not be compared using this labelling technique.

The inverse isotopic dilution technique was re-evaluated in the glasshouse trial, by uniformly injecting the pots of ryegrass with a carrier free P^{32} solution. The fertiliser treatments unpredictably stimulated uptake of labelled soil phosphate, so that the changes in herbage specific activity provided little meaningful information.

These two unsuccessful attempts to derive quantitative information from the introduction of the P^{32} isotope into the phosphate rock-soil-plant system demonstrated the difficulties involved in using isotopic dilution techniques to examine phosphate rock dissolution in field soils.

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The Lord is my strength
and my shield;
My heart trusts in him, and
I am helped;
Therefore my heart exults

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CHAPTER 1: INTRODUCTION

Phosphate rock has become the general term for all rocks that contain approximately 2% (or more) phosphorus as orthophosphate (PO_4^{3-}) (Notholt, 1980). The fertilising value of these materials has been recognised for a long time (Morfit, 1873), but usually only in terms of their suitability as raw materials for superphosphate manufacture.

Recently reactive phosphate rocks have been recognised as a distinctive type of phosphate rock which can have a high agronomic performance when applied directly to the soil as fertiliser. These materials have been called "reactive" because they exhibit relatively high solubility in weak organic acid solutions, compared to other phosphate rocks.

The use of reactive phosphate rocks as fertilisers in New Zealand has been limited, but interest in these materials has increased as the cost of superphosphate has risen, and the ore deposits which have been traditionally used for superphosphate manufacture become depleted. There has also been interest generated in reactive phosphate rocks as slow release "natural" fertilisers, more suited to the "organic husbandry" philosophy.

Most reactive phosphate rocks are imported into New Zealand as light, dry, free flowing, sand-like products, which become fine powder when ground. The unground sand sized materials are proving difficult to handle efficiently in current transporting systems. In some cases considerable portions of loads have been lost as the material sifts

through any small openings in the transport (Mr N. Charteris, Ravensdown, pers.comm. 1989). The unground phosphate rocks are also difficult to apply evenly to soil by aerial topdressing in anything but very calm conditions (Gillingham *et al.* 1987), although the small quantities at present being applied allow top-dressing operators to wait for optimum weather conditions (Mr. Bernie Haskill, Jet Spread. pers.comm. 1989). These problems will be accentuated if the phosphate rocks are ground, which is highly probable in view of the strong recommendation to grind these materials to improve their agronomic value. Several researchers have shown that the agronomic performance of these materials is significantly improved by grinding (Khasawneh and Doll, 1978; Rajan *et al.* 1987), and fertiliser regulations in the EEC already require that such materials be ground before application.

The solution to the handling and spreading problems of the reactive phosphate rocks is to granulate these materials, which would also allow the addition of other elements to the fertiliser. Conventional trials of granulated phosphate rocks have found that granulation reduces the amount of phosphate available from these materials (Buchan *et al.* 1970; Chien and Hammond, 1978a), but recent work suggests that on New Zealand pastures, where fertiliser is usually applied to the soil surface, the effects of granulation on agronomic performance may be negligible (Gillion *et al.* 1978; MacKay *et al.* 1981).

The objectives of this thesis are:

1. To examine research literature concerning reactive phosphate rocks, and their use as fertilisers directly applied to the soil (chapter 2), in such a way that a range of rocks can be selected and characterised (chapter 4) for the manufacture of granulated fertilser.

2. To evaluate methods for the granulation of unground and ground reactive phosphate rocks (chapter 6).

3. To evaluate the effects that grinding and granulation have on the agronomic performance of the selected reactive phosphate rocks (chapters 5 and 7).