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**THE FORM AND AVAILABILITY OF SLOWLY  
AVAILABLE PHOSPHORUS IN DEPLETED  
VERTOSOLS**

Submitted by

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I dedicate this thesis to my grandparents, Ronald  
and Helen McLaren, and Jim and Olive Nelson.

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102

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108 minerals in the dark: P forms in Vertosols. *Cotton Science Forum*, Narrabri, NSW

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121 **Publications**

122

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128 useful indicator of the supply of slowly available phosphorus in Vertosols. *Soil Science Society of  
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132 extractant in a Vertosol soil using XANES. *Soil Science Society of America Journal* (submission  
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135 Rapid, non-destructive total elemental analysis of Vertosol soils using portable X-ray fluorescence.  
136 *Soil Science Society of America Journal*, **76**(4), 1136-45

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138 **McLaren, T.I.**, Guppy, C.N., Tighe, M. (2012). A rapid and non-destructive plant nutrient analysis  
139 using portable X-ray fluorescence. *Soil Science Society of America Journal*, **76**(4), 1146-53

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141

## 142 **Abstract**

143

144 Vertosols are an important soil type used for cotton production in the northern grains region (NGR)  
145 of eastern Australia. Historically, cotton has received minimal phosphorus (P) input due to the high  
146 fertility of Vertosols. Over the past 30 years, P fertiliser use in the cotton industry has increased  
147 six-fold, due to the perceived decline in soil fertility in the NGR. However, when P is applied to  
148 cotton systems, the amount of P fertiliser recovered by the crop (PUE) has been low and  
149 unpredictable (0 – 67 %), and few studies have investigated why this is so. The unpredictability of  
150 PUE in cotton systems refers to the lack of cotton response when P fertiliser has been applied in  
151 some soils, which was based on recommendations from the commonly used Colwell soil P test.  
152 Long-term field trials suggest the quantity of readily available P measured using the Colwell  
153 extractant is being replenished by other soil phosphates not measured by the Colwell extractant.  
154 This may be due to the large amounts of Ca phosphates typically found in Vertosols, which may  
155 supply or replenish labile soil P pools in the 0 – 10 cm and 10 – 30 cm layers. Recent studies have  
156 used a dilute acid test (colloquially referred to as the BSES extractant) to measure Ca phosphates in  
157 Vertosols, however, little is known about the P pools removed by this extractant. The aim of this  
158 project was to understand the ability of Ca phosphates to supply or replenish plant available P in  
159 the cotton systems of the NGR.

160 In Chapter 2, the response of faba bean and cotton to subsoil P when moisture was limiting  
161 in the topsoil was investigated in glasshouse studies. The growth and P uptake of faba bean and  
162 cotton was related to Colwell-P concentrations in the subsoil, demonstrating the potential  
163 importance of subsoil P pools in crop P uptake under dry topsoil conditions. This study confirms  
164 the Colwell soil P test is a good indicator of plant available P in Vertosols of the NGR. The  
165 quantity of BSES-P was not related to faba bean and cotton P uptake over one crop cycle, although  
166 this was unsurprising give the majority of these soils contained BSES-Ca/P ratios above 74:1  
167 (Chapter 3).

168 This study confirmed that cotton is well suited to low soil P environments. Cotton was less  
169 responsive to increasing subsoil Colwell-P concentrations when compared to faba bean. It is  
170 possible that the concentration of P in the soil solution needed to trigger cotton root proliferation is  
171 low in comparison to other crop species. This is also supported by the lack of cotton response to  
172 increasing volumes of soil P enrichment in the subsoil. However, caution is needed applying these  
173 results to cotton systems as the majority of cotton is irrigated and this would increase the amount  
174 of P sourced from topsoils. We recommended that Colwell-P be measured in both the topsoil and  
175 subsoil layers, and BSES-P used periodically (e.g., every ~ 10 years) to monitor soil P rundown in  
176 Vertosols.

177 In Chapter 3, this study demonstrated the ability of BSES-P to supply Colwell-P in the  
178 Vertosol soils of the NGR. In approximately half the Vertosols tested Colwell-P was being  
179 replenished by BSES-P and this occurred when soils contained BSES-P concentrations greater than  
180 61 mg P/kg. This experiment also found that BSES-P was more likely to supply Colwell-P when  
181 the ratio of Ca (mg/kg) to P (mg/kg) in the BSES extract was greater than 74, suggesting the  
182 presence of Ca phosphates of low solubility.

183 In Chapter 4, the soil phosphates removed by the 0.1 M NaOH and 1 M HCl extractants  
184 were investigated using P K-edge X-ray absorption near-edge structure (XANES). This study  
185 aimed to understand the difference between the soil phosphates removed by the 1 M HCl and  
186 BSES extractants (Chapter 4). An important aspect of this study was the application of P K-edge  
187 XANES for soil P studies in untreated soils, although the high method detection limit of the Soft  
188 X-ray beamline at the Australian Synchrotron made accurate identification of soil phosphates  
189 difficult.

190 There was supporting evidence that the 0.1 M NaOH and 1 M HCl extractants remove soil  
191 phosphates according to the solubility of the P minerals that the reagent is likely to dissolve.  
192 Calcium phosphates were the dominant soil P pool in Vertosols, and XANES spectra characteristic  
193 to Ca phosphate reference materials were removed after 1 M HCl extraction. However, P K-edge  
194 XANES spectra on the soil residue after 0.1 M NaOH and 1 M HCl extraction lacked pre- and post-  
195 edge features because of the high method detection limit of the Soft X-ray beamline, and it is  
196 possible that other soil phosphates may have been removed in addition to Ca phosphates. This  
197 study suggests that the method detection limit of the Soft X-ray beamline at the Australian  
198 Synchrotron is ~ 2200 mg total P/kg, and further difficulties were found in attempting to identify  
199 and quantify the soil phosphates removed by the BSES extractant.

200 A comparison of the solution extracts removed by the 1 M HCl and BSES extractants  
201 indicate approximately 80 % of the P removed by the 1 M HCl extractant is removed by the BSES  
202 extractant. The amount of Ca phosphates removed by the BSES extractant is likely to be lower  
203 when compared to those removed by the 1 M HCl extractant because the later reagent has a lower  
204 pH and could potentially dissolve more thermodynamically stable Ca phosphates. However, it was  
205 unclear from this study what soil phosphates constitute BSES-P and further research is needed to  
206 investigate this.

207 This project significantly advances our understanding of the slowly available P pool in  
208 Vertosols, and demonstrates the ability of the BSES extractant to predict the supply of Ca  
209 phosphates to supply or replenish plant available P in Vertosols of the NGR. Further research is  
210 needed to understand the form and availability of Ca phosphates at differing BSES-P  
211 concentrations and BSES-Ca/P ratios.

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376

## 377 **Abbreviations**

378

379	Al	Aluminium
380	AMF	Arbuscular mycorrhizae fungi
381	ANOVA	Analysis of variance
382	AR	<i>Aqua regia</i>
383	As	Arsenic
384	ASPAC	Australasian Soil and Plant analysis Council
385	Ca	Calcium
386	CaCl <sub>2</sub>	Calcium chloride
387	CaCO <sub>3</sub>	Calcite
388	CCRMP	Canadian Certified Reference Materials Project
389	CO <sub>2</sub>	Carbon dioxide
390	Cr	Chromium
391	Cu	Copper
392	DCP	Dicalcium phosphate
393	DCPD	Dicalcium phosphate dihydrate
394	DL	Definitive level
395	DM	Dry matter
396	ECEC	Effective cation exchange capacity
397	FA	Fluorapatite
398	Fe	Iron
399	FeO	Iron oxide
400	H <sub>2</sub> O	Water
401	H <sub>2</sub> O <sub>2</sub>	Hydrogen peroxide
402	H <sub>2</sub> SO <sub>4</sub>	Sulfuric acid
403	HA	Hydroxyapatite
404	HCl	Hydrochloric acid
405	HClO <sub>4</sub>	Perchloric acid
406	He	Helium
407	HF	Hydrofluoric acid
408	HNO <sub>3</sub>	Nitric acid
409	ICP-OES	Inductively couple plasma optical emission spectroscopy
410	IRMM	Institute for Reference Materials and Measurements
411	K	Potassium
412	KH <sub>2</sub> PO <sub>4</sub>	Potassium dihydrogen orthophosphate

413	LCF	Linear combination fitting
414	LXRF	Laboratory X-ray fluorescence
415	MDL	Method detection limit
416	Mg	Magnesium
417	Mn	Manganese
418	Mo	Molybdenum
419	NAA	Neutron activation analysis
420	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	Sodium pyrophosphate
421	NaHCO <sub>3</sub>	Sodium bicarbonate
422	NaOH	Sodium hydroxide
423	NGR	Northern grains region
424	NHHF	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , and HF acid digest
425	Ni	Nickel
426	NIST	National Institute of Standards Technology
427	OCP	Octocalcium phosphate
428	OVD	Open vessel digest
429	P	Phosphorus
430	Pb	Lead
431	PUE	Phosphorus use efficiency
432	PXRF	Portable X-ray fluorescence
433	Qual	Qualitative screening
434	Quant	Quantitative screening
435	Rec	Elemental recovery
436	Rh	Rhodium
437	RSD	Relative standard deviation
438	S	Sulfur
439	SCD	Sealed chamber digest
440	Si	Silica
441	SiO <sub>2</sub>	Silica dioxide
442	TCP	β-Tricalcium phosphate
443	TFY	Total fluorescence yield
444	Ti	Titanium
445	USEPA	United States Environmental Protection Agency
446	XANES	X-ray absorption near-edge structure
447	XRF	X-ray fluorescence
448	Zn	Zinc
449		