

SHORT NON-REFEREED PAPER

EVALUATION OF A REDUCED EXTRACTION TIME TO IMPROVE THE EFFICIENCY OF ROUTINE ANALYSIS OF RESIN EXTRACTABLE PHOSPHORUS AND SULPHUR

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Introduction

The Fertiliser Advisory Service (FAS) at the South African Sugarcane Research Institute uses the anion exchange resin (AER) extraction method to estimate plant available amounts of phosphorus (P) and sulphur (S). Several studies comparing different extraction methods have consistently found that AER better predicts a crop response to P over a wide range of crops and soil types (Silva and van Raij, 1999; van Raij *et al.*, 2009). However, the 16-hour overnight extraction step has been recognised as the key constraint of the method for routine testing purposes (Qian *et al.*, 1992; Sousa and Coutinho, 2009). An extraction time that allows for completion of the analysis within a single day is considered highly advantageous to the operational workflow of a commercial laboratory.

Conventionally, the 16 h extraction time is considered adequate to allow equilibrium between the AER and soil to be attained and allows for overnight extraction. However, some studies have considered the potential to reduce extraction time. For example, Qian *et al.* (1992) found that extraction times as short as 15 minutes were linearly related (all times tested $R^2 > 0.96$, $p < 0.001$) to the 16 h extraction time. Sousa and Coutinho (2009) found a significant curvilinear ($R^2 = 0.99$, $p < 0.001$) relationship between several different extracting times for their soils, concluding that a 2 h extraction appeared to offer a viable alternative to the 16 h extraction. This study evaluates if a 2 h extraction time (considered to be the longest practical time to permit analysis within a single day) can be used to estimate the 16 h extraction value using regression methods on a wide range of soils collected from the South African sugarcane growing regions.

Materials and Methods

One hundred and thirty-four soils collected from sugarcane growing fields over a wide range of site types were investigated. Samples ranged in pH_{CaCl_2} from 3.5 to 6.9, clay content from 4 to 62% and total carbon from 0.4 to 9.7%.

Phosphorus and S were extracted in triplicate using an AER strip procedure. Anion exchange resin strips, in bicarbonate form, were placed into centrifuge tubes along with 2.5 g of soil and 50 mL of deionised water. The tubes were shaken on a horizontal shaker for 2 or 16 hours. After the extraction time had elapsed, the strips were removed, rinsed with distilled water and eluted with 40 mL of 1M HCl to strip off the P and S held on the exchange sites. The resulting solution was analysed for P and S using inductively coupled plasma optical emission spectroscopy. As FAS reports result on a volume basis (mass of element per volume soil), these results were converted to a mass basis using soil sample densities (g/mL).

To compare whether the 2 h extraction could predict the 16 h reference values, various regression models were evaluated using the 2 h extraction as the independent variable. In all

instances, simple linear regression provided the best fit. Due to concern over the predictive ability of the models at lower test values (near or below current recommendation thresholds) the sample points with 16 h extraction values with <30 mg P/L or 40 mg S/L were also evaluated for quality of fit using regression analysis. In addition, as certain soil properties play a key role in the P-sorption processes, $\text{pH}_{\text{CaCl}_2}$, organic matter (OM) and clay content were evaluated to determine whether the grouping of data would improve model quality. These parameters were selected as they are included in routine fertility testing and could thus readily be included in regression models developed.

Results

The 16 h extraction values for P ranged from 3.7 to 268 (median: 15.9) mg/L and S ranged from 6.7 to 164 (median: 19.0) mg/L. The 2 h concentrations for P ranged from 1.1 to 164 (median: 6.0) mg/L and S ranged from 2.0 to 172 (median: 13.6) mg/L. Regression analysis using the full dataset (134 soils) showed that 2 h P had a strong, highly significant linear relationship with 16 h P ($R^2=0.96$, $p<0.001$). However, exclusion of a single extreme outlier (16 h P=268 mg/L) resulted in a highly significant ($p<0.001$) but reduced fit model ($R^2=0.93$) (Figure 1a). Examination of the reduced range regressions for P (Figure 1c) show that the linear models had a poorer, but still highly significant, fit than the full model. The P model tends to under-predict P values in the range between 12 and 25 mg/L (16 h extraction). Correlation between the soil properties and the amount of P extracted at either 2 h or 16 h showed no useful relationships (correlation coefficients (r) all <0.5). Weak negative correlations (r about -0.6) between the clay and OM content and the proportional recovery of the 2 h were found, suggesting that these attributes may be affecting the desorption equilibria differently between the samples. Splitting the data according to texture (<15% clay and >15% clay) for samples with <30 mg P/L marginally improved predictive ability in the lower range. The <15% clay model ($16 \text{ h P} = 1.7 (2 \text{ h P}) + 2.4$, $R^2=0.93$, $p<0.001$) was markedly better than the full model (Figure 1c), while for the >15% clay model ($16 \text{ h P} = 2 (2 \text{ h P}) + 3.67$, $R^2=0.8$, $p<0.001$) the improvement was marginally better than the full model.

The 2 h S had a very strong and highly significant linear relationship with 16 h S (one outlier removed; $R^2=0.98$, $p<0.001$) (Figure 1b). In the case of the reduced model (<40 mg/L), there was a small decrease in R^2 , but generally, the regression fit remained excellent (Figure 1d). However, unlike P, there were no useful relationships between S extracted, S recovered and soil properties evident and thus no groupings were applied.

Discussion and Conclusions

The findings for this study suggest that it is feasible to predict 16 h AER extractable P and S from a 2 h extraction time, though further refinement at lower soil test P concentrations is required to ensure accurate fertiliser recommendations. This concurs with the findings by Qian *et al.* (1992) and Sousa and Coutinho (2009). For P, current soil test thresholds range between 10 and 15 mg/L depending on soil texture, while for S a single 15 mg/L value is applied. The reported models for P do not allow accurate predictions in this range, but appear usable for S. In this study, only two extraction times were compared and a linear relationship established. Sousa and Coutinho (2009) evaluated several extraction times and found that, on average, the rate of extraction decreased with longer extraction times, resulting in a highly significant curvilinear relationship. Sibbensen (1978) reported a similar finding using resin beads in the chloride form. In both studies, the authors found that soils with higher clay content had curvilinear responses but that sands tended to be linear. In this study, minor improvements in P prediction were possible by splitting sandy and clay soils, but this was not the case for S. This may be a consequence of only two extraction times being compared. Further improvements to the models may be possible if additional times are investigated to establish whether curvilinear responses are evident, thus potentially reducing model error.

Furthermore, it is necessary to evaluate the effect of the model error on fertiliser prescriptions to minimise risks associated with over or under application.

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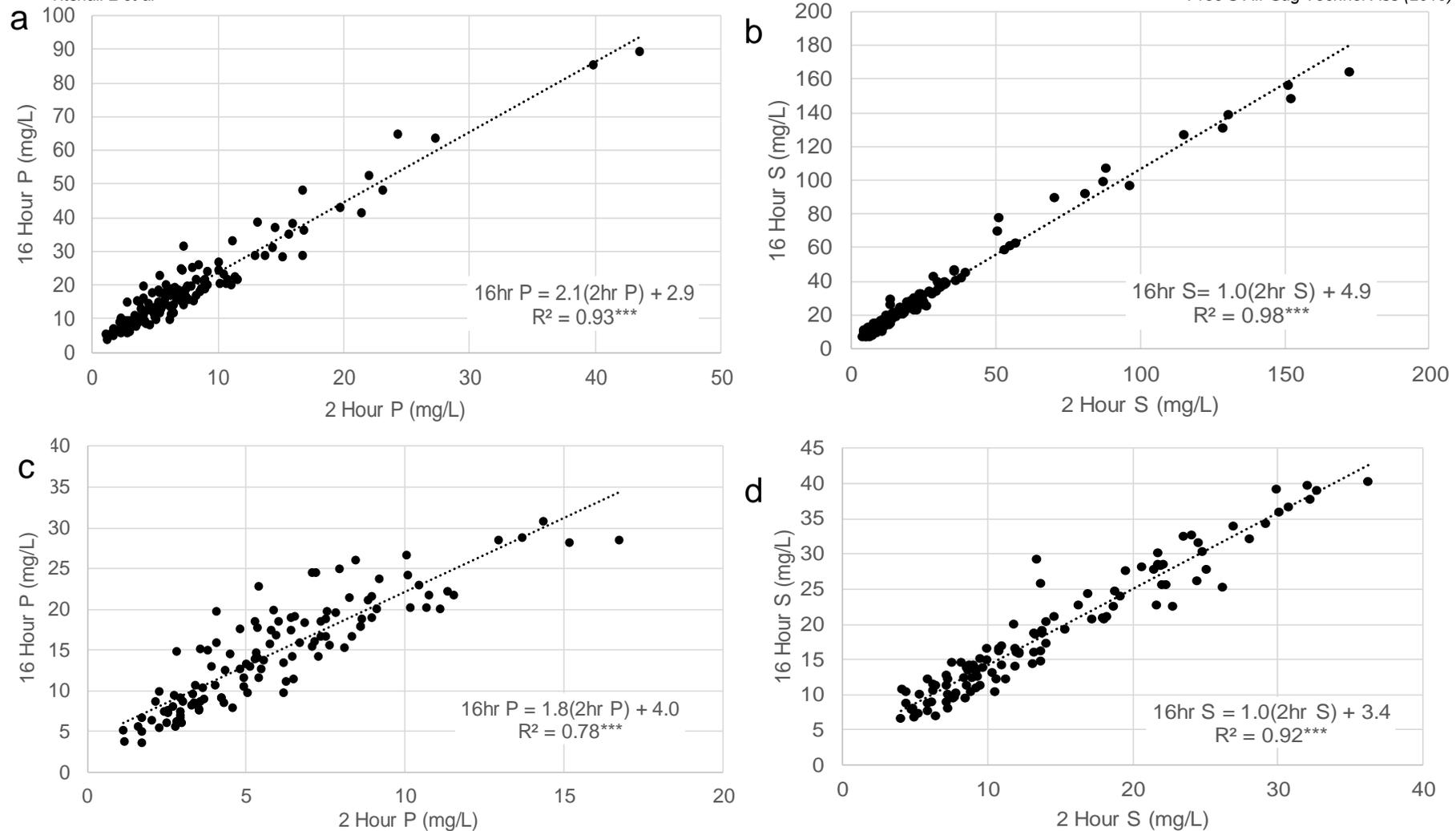


Figure 1: Linear regression relationships between anion exchange resin extracted a) phosphorus (P) for all soils, b) sulphur (S) for all soils tested, c) P with 16-hour values <30 mg/L, and d) S with 16-hour values below 40 mg/L. *** indicates highly significant fits (p<0.001).