

AN ANION EXCHANGE RESIN METHOD FOR SOIL PHOSPHORUS ESTIMATION

D.M.D.I. Wijebandara and L.L.W Somasiri

Coconut Research Institute, Lunuwila

ABSTRACT

Soil phosphorus extracted by anion exchange resin method has shown good correlations with plant response for a wide range of soil types. In this method anion exchange resin and soil is suspended in water at the ratio of 4:5:100 and shaken for 16 hours (overnight) after which P sorbed by resin is eluted and estimated. Long extraction times (overnight shaking) is not convenient for routine laboratory work in Sri Lanka due to frequent power failures particularly in the night which disrupt the smooth operation of shakers. The possibility of using a shorter extraction time for soil P extraction by anion exchange resin method is discussed in this paper.

Anion exchange resin (IRA 400) in Cl form (4 ml), 2 mm sieved air dried soil (5 g) and distilled water (100 ml) in polypropylene containers (250 ml) was shaken for 4, 8 and 16 hours respectively using an end-over-end shaker. Sorbed P by the resin was eluted with 1M NH_4Cl and the elute was analyzed for P by the molybdenum blue method. Pueraria was grown in polypropylene pots (1L) filled with 20 different soils and placed in a green house. Each soil was treated with a basal dose of ammonium sulphate (0.16g), muriate of potash (0.2g) and magnesium sulphate (0.1g) and there were two P treatments for each soil; i.e. triple super

An Anion Exchange Resin Method for Soil Phosphorus

phosphate application at the rate of 1g per pot (+P) and no P application (-P). The percentage response of Pueraria in each soil was worked out by the ratio of total dry matter weight of -P treatment to +P treatment.

The linear correlation coefficients among resin P extracted at different shaking times were highly significant ($p < 0.001$). The extracted P increased with increasing extraction time. Percentage yield of Pueraria P and resin extracted P of soil fitted well to the Cate Nelson model with r^2 of 0.676, 0.592 and 0.616 for extraction time of 4, 8 and 16 hours respectively. It follows that 4 hours extraction time is sufficient for anion exchange resin method instead of 16 hours extraction. This provides a convenient laboratory method for soil P extraction by anion exchange resin method. The suggested modifications results in reducing operation cost and time.

INTRODUCTION

Plant nutrient status of advisory agricultural soil samples can be rapidly determined by performing suitable chemical tests. Therefore soil chemical testing is regarded as one of the essential tools in a sound fertilizer recommendation programme. Many chemical tests are performed for the estimation of available soil phosphorus (Bray & Kurtz, 1945; Olsen, 1954; Vander Paauw, 1971, Schofield, 1955; Anon, 1985). None of those tests are universally applicable due to the great diversity of chemical properties of soils that control availability of phosphorus. The principle in most of those chemical tests is to dissolve a certain fraction of soil phosphorus, depending on the chemical constituents in individual soils. For the determination of soil phosphorus, the anion exchange resin method is distinct in its action of extraction. In

this method, a soil- water suspension is shaken with an anion exchange resin for a pre-determined time and thereafter the resin is separated from the suspension and sorbed phosphorus on to the resin is eluted with a suitable solution.

The P content in the elute is determined by a colorimetric method (Amer et. al. 1955). The mechanism of phosphorus desorption from soil in this method is different from that of other conventional chemical methods because the resin takes up phosphorus from the solution phase. As a result, the phosphate concentration in the solution phase is lowered leading to dissolution of more solid phase P. This process may go on until the system reaches the state where there is no net sorption of phosphorus by the resin. Therefore this process closely simulates the P uptake by plant roots (Amer et. al. 1955, Cooke and Hislop, 1963).

Anion exchange resin method was found to be suitable for the estimation of phosphorus of wide range of soils (Cooke and Hislop 1963; Dalal, 1985; Van Raj et. al., 1986; Bache and Ireland, 1980; Barrow and Shaw, 1979; Bache and Rogers, 1970; Elrashidy et. al., 1975). Wijebandara and Somasiri (1994) showed that the anion exchange resin method could be successfully used for the determination of phosphorus status of different soils of the Intermediate zone - Low country of Sri Lanka.

Sibbesen (1977) suggested a convenient routine laboratory method for the anion exchange resin extraction. In the method, anion exchange resin (4g) (as beads), included in a mesh bag, is shaken in a soil - water suspension (5g : 100ml) for 16 hours (preferably overnight). The resin bag is recovered after extraction

and washed free of soil residues and shaken with 1M HCl (100ml) to elute resin sorbed P. Previous workers showed that the anion exchange resin extractable P varied rapidly with shaking time during the initial 4 hours and then gently reached to the maximum extractable quantity after a long extraction time which is variable from 16h to 100h (Amer et. al, 1955; Elrashidi et. al., 1975; Evan and Jurinak, 1976; Sibbesen, 1978). El-Nennah (1978) showed that the variation of extractable P with the extraction period was not consistent and the time required to achieve maximum desorption depends on soil types. However, Sibbesen (1977) recommended 16h as a practical extraction time for routine analysis. The main disadvantage of this method is that the 16 hours (over night) shaking time is too long and not very practicable in Sri Lankan laboratories due to the occurrence of frequent power failures particularly during the night. Therefore, the objective of the present study was to investigate the possibility of employing a shorter extraction time for anion exchange resin procedure for soil P extraction.

MATERIALS AND METHODS

A green house pot experiment was carried out to obtain a plant index for soil P status of each soil. Ten soil series were sampled from the Intermediate zone-Low country of Sri Lanka for the study; four from the coastal plain, two from an alluvial flood plain and four from the mantled plain. Soil samples were collected from both A and B horizons of each series separately. Sampling depths correspond to A and B horizons of each series and common soil properties of each soil are given in Table 1. Soil (both A and B horizons of each soil series separately) were passed through 6 mm sieve at their field moist state, filled into polypropylene pots (each soil into four pots), and packed up to 1.5l mark by gently tapping

Table 1. Common properties of soils and the sampling depth

Soil series	Samplin g depth (cm)	pH 1:5 Soil:H ₂ O	EC umho/ cm	Organic matter (%)	% sand	% silt	% clay
Weliketiya	0-15	5.2	34.9	0.22	94.6	1.9	3.4
Weliketiya	15-30	5.1	44.7	0.11	89.3	3.9	2.0
Madampe	0-10	5.7	6.2	0.45	90.0	3.6	2.1
Madampe	10-20	5.2	35.6	0.22	87.4	5.0	3.4
Rathupasa	0-18	5.7	9.2	0.34	92.6	4.1	2.2
Rathupasa	18-36	5.7	22.1	0.24	93.7	3.8	2.2
Sudu	0-8	5.8	18.3	0.22	95.0	1.0	3.8
Sudu	8-16	5.5	32.6	0.17	96.0	1.2	3.6
Welipelessa	0-11	5.3	28.1	0.34	87.6	4.2	4.0
Welipelessa	11-22	5.8	28.1	0.29	88.0	6.6	1.9
Pahugaswewa	0-14	5.6	28.8	0.25	88.5	3.8	4.0
Palugaswewa	14-28	5.8	34.1	0.28	74.9	11.3	7.9
Tambarawa	0-15	5.4	39.4	0.47	89.1	3.7	4.1
Tambarawa	15-30	5.4	36.4	0.34	87.1	4.1	4.0
Andigama	0-15	5.90	45.1	1.27	60.3	8.4	26.6
Andigama	15-30	5.79	29.6	0.98	60.0	8.4	27.8
Kurunegala	0-10	5.03	46.8	1.40	74.2	5.9	17.6
Kurunegala	10-20	4.99	32.7	0.81	71.3	5.3	18.7
Melsiripura	0-13	5.52	44.6	2.1	49.0	10.7	32.2
Melsiripura	13-26	4.62	19.8	0.8	54.2	10.5	30.2

Anion Exchange Resin Method for Soil Phosphorus

each pot against a table-top until no further change in soil volume occur. A constant soil volume in pots was preferred to a constant soil weight because the amount of nutrients available to plants in the field depends on the available soil volume rather than the available soil weight. Each soil was treated with two phosphorus levels viz., two pots with no phosphorus application (-P) and two pots with phosphorus application at the rate of 1 g triple super phosphate (46% P₂O₅) per pot (+P). Each pot was treated with a basal dose of 0.16 g ammonium sulphate (20.6% N), 0.2 g muriate of potash (60% K₂O) and 0.1 g magnesium sulphate (16% MgO).

For each +P and -P treatment of each soil, pots were prepared in duplicate and altogether 80 pots were prepared. Pots were kept in a green house for two weeks after treatment application and moisture status of each pot was maintained at 90% field capacity. After two weeks, pre-germinated *Pueraria phaseoloides* seedlings were planted in each pot. Pots were kept in a green house and arranged in a row. Different soils receiving +P and -P treatments were randomized within the row. Duplicates were kept in a separate row and randomized. Three months after planting, *Pueraria* pots were dismantled and fresh and dry weights of vegetative parts and roots were recorded.

Relative yield (RY%) of the crop in each soil was calculated as,

$$\text{RY \%} = \frac{\text{Yield at -P treatment}}{\text{Yield at +P treatment}} \times 100$$

The cumulative dry weight of shootings over the experimental period and total dry matter production (including roots) were taken as yields of the crop.

Anion Exchange Resin Method

Anion exchange resins (Amberlite IRA-400, Cl form, mesh size 14-52, exchange capacity 1.4 meq ml⁻¹ wet resin) were initially backwashed with distilled water in a column to remove impurities and light weight particles. Four ml of washed anion exchange resins were scooped into a mesh bag (11 x 3.5 cm) made out of polypropylene gauze (52 mesh size). The open end of each mesh bag was folded over three times in 0.5 cm wide strips and fastened with plastic clips. Five grams of soils used for the pot experiment (prior to treatment application) were weighed into a 250 ml polypropylene bottle containing 100 ml distilled water. One anion exchange resin bag was included into each bottle. The contents were shaken in an end-over-end shaker for 16 hours.

The resin bags were recovered from the soil suspension and washed with distilled water to remove soil residues from the resin. In order to recover phosphorus, each resin bag was put into polypropylene bottles containing 50ml 1 M NH₄Cl (pH = 2) and shaken in an end-over-end shaker for 30 minutes. The phosphorus in the solution was estimated by the molybdenum blue method (Murphy and Riley, 1962). The whole procedure was repeated but employing shaking times of 1h, 2h, 4h and 8h respectively.

2.5% Acetic Acid Extraction

Soils equivalent to 2.5 grams were shaken with 100 ml of 2.5% (v/v) acetic acid solution for 2 h. The mixture was filtered through Whatman No 42 filter paper (Anon, 1985) and the filtrate was analysed for phosphorus.

Olsen's Bicarbonate Extraction

Five grams of soil were shaken with 100 ml of 0.5 M sodium bicarbonate solution (pH 8.5) for 30 minutes. The mixture was filtered through Whatman No 42 filter paper and polyacrylamide was added as a decolourizing agent. The pH of the filtrate was adjusted to 5 using para-nitrophenol indicator and the filtrate was analysed for phosphorus (Olsen *et.al.*, 1954).

Bray and Kurtz I Extraction

Three grams of soil were shaken with 20 ml of extraction solution (0.03N NH_4F and 0.025N HCl) for one minute. The mixture was filtered through Whatman No 42 filter paper and the filtrate was analysed for phosphorus (Bray & Kurtz, 1945, Olsen and Sommers, 1982).

Bray and Kurtz II Extraction

Same procedure as above was adopted except that the concentration of extracting solution was 0.03 N NH_4F and 0.1 N HCl (Bray & Kurtz, 1945).

RESULTS AND DISCUSSION

The quantity of soil P extracted during the 16h shaking period was variable depending on the soil type. P values of different soils ranged from 0.3 to 75.8 mg/kg. It was found that the extractable P increased with the increase of shaking time from 1h to 16h as shown in Fig. 1. For all the soils except Melsiripura series there was a sharp increase in resin extractable P up to 4h of extraction and from 4h to 16h extraction there was hardly any increase (Fig 1). The phosphorus level of Melsiripura series was considerably high compared to the other soils. Melsiripura series also showed a curvilinear relationship between the resin extractable P and shaking period indicating a sharp increase during first 4h, a relatively lower rate of increase from 4h to 8h and a levelling off gradually to a constant at 16h shaking period. The rate of increase was very low beyond 16h (Fig. 1).

The curvilinear relationship between the resin P and the shaking period obtained in the present study was much similar to that obtained by Amer et. al. 1955, Barrow and Shaw (1979) and Sibbesen (1978). Amer et. al. (1955) explained that the curvilinear relationship was due to occurrence of three simultaneous reactions, i.e., two rapid reactions completed in first 2 of 3 hours and the other reaction which was a slow one which continued upto 72 hours. Cooke (1966) showed that the relationship between the resin extractable P and square root of the shaking time can be expressed by a straight line for shorter extraction times less than 2h. Aura (1978a) also obtained a similar relationship. The linearity of the plots of resin P against $(\text{time})^{1/2}$ could be due to the diffusion of phosphate from porous soil medium. Previous studies by Sibbesen (1978), Barrow and Shaw (1979) and Bache and Ireland (1980) showed that

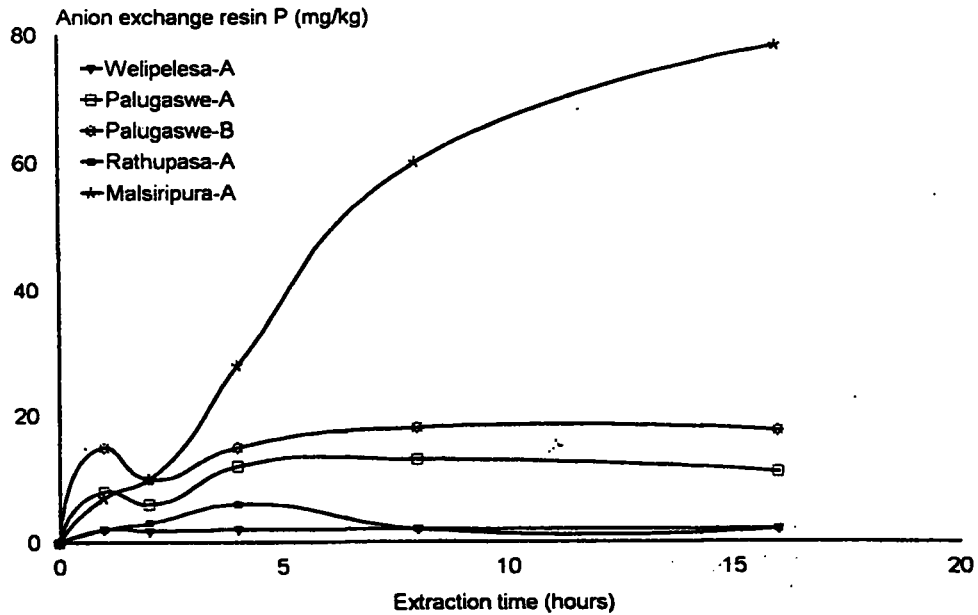


Fig. 1 The relationship between anion exchange resin P and the extraction time

the resin P increased up to a extraction time of 90 hours but in all cases there was a sharp increase in resin P within first 4h. The present study also showed the same trend (Fig. 1).

The correlations between relative yields of pueraria in each soil (Table 2) and the corresponding resin P values obtained for shaking period of 1h, 2h, 4h, 8h and 16h respectively were tested by the goodness of fit of data to Cate and Nelson statistical model (Cate and Nelson, 1971). The goodness of fit of soil/plant data to Cate and Nelson model was determined by the magnitude of R^2 values. For 2h shaking period, the R^2 values were low but for shaking periods of 4h and above, R^2 values were high (Table 2). Resin P by the shaking period of 4h explained 67.6% and 70% of the variation of relative yields of vegetative parts and total dry matter of pueraria respectively.

The goodness of fit of soil test data and corresponding plant indices to Cate and Nelson statistical model can be used to evaluate the suitability of soil P testing methods. Such a soil test can also be calibrated to get the critical soil P levels. The soil critical P levels deduced from the relationship between relative yield and resin P at different shaking periods (Table 2) showed that soil critical P levels corresponding to 4h, 8h and 16h shaking periods were nearly the same. But the soil critical P levels deduced by using resin P values for shaking period less than 4h were low. In all cases, the critical values obtained by total dry matter content and vegetative parts were similar. Although the extractable resin P has gradually increased with increasing shaking period, there has been no improvement in R^2 value and therefore there is no advantage in employing a long shaking time. It follows that the 4h extraction time for the anion

Anion Exchange Resin Method for Soil Phosphorus

exchange resin method is sufficient for estimating phosphorus in soils.

Table 2 - Goodness of fit of resin P and plant data to Cate and Nelson model and soil P critical levels.

Extraction Time	R ²		Critical soil P levels	
	RY based on total dry matter	RY based on weight of vegetative parts	Based on total dry matter	Based on weight of vegetative parts
AER - 1h	0.504***	0.638***	1.78	1.78
AER - 2h	0.301*	0.368***	0.89	0.89
AER - 4h	0.700**	0.676***	2.98	2.98
AER - 8h	0.611***	0.592***	2.69	2.38
AER - 16h	0.468***	0.616***	2.31	2.93

Significant level P < 0.001 *** P < 0.01 ** P < 0.05 *

The other conventional chemical soil P testing methods, viz. Bray and Kurtz I & II extractions and Olsen's extraction showed low R² values correspond to relative yields of Pueraria and only 50% of the variation of the relative yields was explained by those methods. Only 2.5% acetic acid extraction showed a high correlation explaining 70% of the variation of relative yield of Pueraria (Table

3). The linear regression between other conventional methods and anion resin method (modified to 4h shaking) was also very highly significant. The mutual correlations between each method were higher for resin method (4h shaking) than for the other methods (Table 4). It shows that the anion exchange resin method is superior to other conventional soil P testing methods for routine phosphorus analysis in agricultural soil samples.

Table 3 - Goodness of fit of soil P by different methods and plant data to Cate and Nelson model and soil P critical levels.

Soil P test method	R ²		Critical soil P levels	
	RY based on total dry matter	RY based on dry weight of vegetative parts	Based on total dry matter	Based on dry weight of vegetative parts
2.5% Acetic acid	0.700***	0.681***	6.86	6.40
Bray I	0.406***	0.556***	5.78	5.78
Bray II	0.495***	0.677***	4.84	4.84
Olsen	0.495***	0.677***	4.90	4.90

Significant level P < 0.001 *** P < 0.01 ** P < 0.05 *

The anion exchange resin extractable P vary depending on the relative ratio of soil, resin and solution, the shaking period and

the temperature (Bache and Ireland, 1980; Barrow and Shaw, 1979). Nevertheless, results of the present experiments show that resin extractable P during 4h extraction time can explain 70% the variation in the yield of a crop for a short period like 3 months. It appears that the fraction of soil P desorbed by resin during a shorter extraction time is responsible for the immediate growth of a plant. The situation may be different in the case of evaluation of residual phosphorus availability in soil by the resin method where long shaking periods should be employed. For most annual cash crops, the response to applied phosphorus fertilizer or the soil P availability over a short period such as 3-6 months is more important because of there short life span. In such circumstances, 4h extraction is more appropriate to evaluate soil P status than lengthy extraction times.

CONCLUSION

Anion exchange resin method can be used successfully for routine soil P analysis of agricultural advisory soil samples by modifying the procedure to employ only 4h extraction time instead of 16h as in the standard method. The modified method is much economical in saving both time and electricity. The soil P determined by the modified method highly correlated with relative yields of Pueraria and soil P determined by other conventional methods for a wide range of soil in the Intermediate zone-Low country region of Sri Lanka. The applicability of the method for the other agro-ecological regions should be further investigated.

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Anion Exchange Resin Method for Soil Phosphorus

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Wijebandara, D.M.D.I and L.L.W, Somasiri

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