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COMPARISON OF CONVENTIONAL AND INNOVATIVE SOIL PHOSPHORUS TESTS IN ESTIMATING PLANT-AVAILABLE PHOSPHORUS

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Introduction

Soil phosphorus tests are fundamental analytical tools for assessing the P nutrient status of the soil. Normally, the plants can utilise only a small percentage of the total soil P, such a quantity being strictly correlated to the labile soil P (portion of P relatively loosely bound onto soil colloids). Thus, an ideal extractant for available soil P should dissolve or desorb some reproducible and consistent proportion of the labile P.

Conventional soil test methods generally use a wide number of chemical reagents (or a mixture of reagents as well) that may extract not only forms of P available for plant use but also some stable and nonlabile soil P. Furthermore, these solutions may fail to extract plant-available P if they are used on soil types for which they are inappropriate (Menon *et al*., 1989; Sharpley, 1991).

In the regions of North America, the current tendency is to encourage the use of mild reactants for the simultaneous extraction of both major elements and micronutrients, in order to perform simple, low-cost, and time saving routine procedures. Among these "multiple-element" or "universal soil" extractants, the most popular is the last reactant developed by the late Dr. A. Mehlich designed as M3 (Mehlich, 1984).

Nowadays, M3 is widely adopted in U.S.A. and Canada. Conversely, most of the soil testing laboratories of European countries still employ the Olsen reagent (Olsen *et al*., 1954) as official soil test (since decades), whilst the universal soil extractants have been only recently investigated (Buondonno *et al*., 1992).

Procedures developed to overcome the limitations of the extracting solutions include the use of anion exchange resin and iron oxide-impregnated filter paper. Originally, resins were used as beads freely mixed in the soil-water suspension (Amer *et al.*, 1955). Later, Sibbesen (1977) simplified the procedure by placing the resin beads in nylon netting bags which were immersed in the soil suspension.

This procedure allows for an easier separation of the resin from soil, but fine root material and soil particles could be trapped in the bag and interfere in further analyses. A new technique has been therefore developed in which anion exchange resin in membrane form (AERM) is used (Saggar *et al*., 1990). This way, the exchanger can be easily separated and allows for its use in routine soil testing.

Recently, a soil test for P has been proposed using iron oxide-impregnated filter paper (Pi) strips. The use of Pi has been found to be an effective approach to soil testing for plant available P (Menon *et al*., 1988; Menon *et al*., 1990). In the Pi procedure the ions are removed from the soil by adsorption onto the iron (hydro)oxide surfaces of the reactive strip.

Unlike the extracting solutions used in the conventional soil P test, the resin and the Pi strip do not react with the soil but act as a sink, which sorbs and retains the P entering the soil solution. In this way, low concentrations of P are maintained in the extraction solution, thereby preventing solution P from increasing to levels, which restrict further P release from the soil. The resin and Pi tests have been found to be less sensitive to soil type than are the others P tests, and they can be used in acid, alkaline and calcareous soils (Menon *et al*., 1989; Quian *et al*., 1992).

The Olsen method is the most used soil P test in Italy. It has been calibrated on a wide range of soils and different crops and it is known to accurately estimate P fertiliser requirement of soil. On the other hand, little or none information is available in our country about the suitability of the more innovative P sink methods in evaluating plant available P.

The objectives of this study were i) to compare soil P test levels determined by two conventional methods (Olsen and M3) and two P-sink procedures (the Pi and AERM tests), and ii) to assess the ability of the Pi and AERM extraction procedures to predict plant available P. The research was carried out on five widely ranging Italian soils added with five rates of P, using maize as test crop.

Materials and Methods

The surface horizons of five soils (Fontana Nuova, Capobarile, Val Furella, Sterparo, Colle Castello) were collected from cultivated areas in the Latium region. Some selected physical and chemical properties of the soils studied (air dried and 2-mm sieved soils) are reported in Table 1, together with the relative classification (FAO-UNESCO, 1975).

Table 1. Selected properties of the soils

Soil	FAO-UNESCO classification	pH (H_2O)	Silt %	Clay %	Corg %	$CaCO_3$ %	CDB-Fe_2O_3 %	Olsen-P mg/kg	P-SI
Fontana Nuova	Regosol	6.6	48	8	0.75	0.0	0.46	6.5	5.5
Capobarile	Fluvisol	7.4	46	18	2.30	16.2	0.89	11.3	8.7
Val Furella	Regosol	7.1	33	11	3.46	0.0	0.52	7.6	12.3
Sterparo	Regosol	7.8	50	32	1.26	2.5	1.42	4.8	17.7
Colle Castello	Luvisol	6.6	45	20	2.62	0.0	1.32	9.1	22.7

Soil pH was determined with a glass electrode in a 2.5:1 water to soil (v/w) suspension. Particle size analysis was performed by sedimentation procedure. Organic carbon (Corg) was determined by the wet oxidation procedure (Raveh and Avnimelech, 1972).

Total carbonate content ($CaCO_3$) was measured by a Dietrich-Fruhlig calcimeter. Free iron oxide was extracted with citrate-dithionite-bicarbonate solution (Mehra and Jackson, 1960) and Fe in the extracts (CDB-Fe_2O_3) determined by atomic adsorption spectrometry.

The phosphorus sorption index of soils (P-SI), a measure of soil P retention, was calculated by the quotient X/logC, were X (mg/100 g) is the amount of P sorbed from 1 g of soil after shaking for 24 hours with 20 ml of a 0.02M KCl solution containing 75 mg P L^{-1} (as KH_2PO_4), and C is the solution P concentration expressed as moles L^{-1} (Bache and Williams, 1971). The quotient was highly correlated with P sorption maxima calculated from a Langmuir sorption plot for a wide range of soils (Mozaffari and Sims, 1994).

Soil-P equilibration

For laboratory studies, 100-g samples of each soil were treated with 0, 25, 50, 75, and 100 mg P kg^{-1}. After addition of P as $Ca(H_2PO_4)_2 \cdot H_2O$ solution to the surface of the soil, the sample was mixed and incubated at field capacity at 24°C for 28 d. After the incubation, the samples were allowed to air dry.

Phosphorus extractability of incubated soils

Anion exchange resin membrane-P (AERM-P): one anion exchange resin membrane preliminarily converted to HCO_3^- form (Saggar *et al.*, 1990) and cut into strips measuring 62.5 mm x 25 mm, and 1 g of soil were shaken with 30 mL of deionized water for 16 h. The strip was then removed and washed with water to eliminate adhering soil particles and fine roots. The phosphate extracted from the soil by resin was analysed after shaking the membrane for 30 min with 20 mL 0.5M NaCl solution.

Paper-strip P (Pi-P) was determined by using the procedure described by Menon *et al.* (1989) as follows: iron oxide-impregnated strips were prepared by immersing filter paper circles (15 cm diameter, Whatman 541 or equivalent) in a 10% $FeCl_3.6H_2O$ solution for 1 h, air dried, immersed in 2.7M NH_4OH solution to convert $FeCl_3$ to Fe-oxide, and then air dried. Immersion in NH_4OH was carried out as rapidly as possible (about 1 min) to avoid uneven oxide deposition on the paper (Guo *et al.*, 1996).

The paper was then cut into 2x10 cm strips, and immediately used or stored. To extract P, 1 g of soil was shaken for 16 h with 40 mL 0.01M $CaCl_2$ solution together with a strip. The strip was removed, rinsed for adhering soil particles, and air-dried. Phosphorus retained on the strip was then dissolved for 1 h in 40 mL 0.1M H_2SO_4 and measured.

Mehlich3 extractable P (M3-P) was determined by shaking soil with the reagent solution (a mixture of 0.2M CH_3COOH, 0.25M NH_4NO_3, 0.015M NH_4F, 0.013M HNO_3, and 0.001M EDTA) for 5 min, at a soil to solution ratio of 1:10 (w/v) (Mehlich, 1984).

Olsen extractable P (Ols-P) was determined by shaking soil with a 0.5M $NaHCO_3$ solution (pH=8.5) for 30 min at a soil to solution ratio of 1:20 (w/v), (Olsen *et al.*, 1954).

Greenhouse experiment

Three kg of the five soil samples were used for greenhouse experiment and prepared with a procedure similar to that used for the laboratory studies. Each sample was placed in plastic bags and treated with 0, 25, 50, 75, and 100 mg P kg^{-1} applied as $Ca(H_2PO_4)_2 \cdot H_2O$ solution to the soil surface. To ensure an equilibrated plant nutrition, supplemental micro (Mg, B, Mo, Cu, Mn, Fe, and Zn) and macro elements (N and K) were added using reagent grade chemicals.

The soils were then mixed and incubated at field capacity at 24°C for four weeks. The pots were arranged in a randomised block design with three replications. One corn (*Zea mays* L., hybrid Dekalb 743) plant was allowed to grow in each pot for four weeks in the greenhouse, with the soil moisture content being maintained at 70% of field capacity.

Table 2. Amount of soil extractable P$^{)}$.as assessed by the Olsen (Ols-P), Mehlich 3 (M3-P), iron oxide-coated paper strip (Pi-P) and anion exchange resin membrane strip (AERM-P) procedures, in function of P added. All data are expressed as mg P kg^{-1} soil.

Soil	Added P	Ols-P	M3-P	Pi-P	AERM-P
Fontana Nuova	0	7.0	15.8	11.5	9.8
	25	16.8	31.5	21.4	22.0
	50	30.2	48.8	38.3	42.6
	75	38.5	66.1	47.5	51.8
	100	52.0	85.2	65.0	68.6
Capobarile	0	10.2	6.6	13.5	3.5
	25	16.0	20.6	29.1	14.0
	50	29.0	41.2	42.8	29.2
	75	42.3	53.0	51.6	44.8
	100	53.2	73.5	68.0	62.3
Val Furella	0	8.1	47.0	5.0	17.5
	25	17.5	62.0	13.5	31.0
	50	24.0	83.0	24.8	48.5
	75	37.5	93.2	38.0	60.2
	100	48.2	112.0	52.3	73.2
Sterparo	0	4.2	30.2	8.5	7.9
	25	11.0	48.2	21.0	20.2
	50	18.6	60.1	28.8	35.2
	75	30.3	74.8	41.2	48.6
	100	42.2	90.0	52.3	57.0
Colle Castello	0	10.0	13.8	12.0	13.5
	25	17.8	25.0	23.2	25.6
	50	25.2	36.2	30.1	38.7
	75	33.0	47.0	41.5	50.5
	100	42.6	55.2	53.0	64.0
Mean		33.4	52.8	26.6	37.6

Total (root and shoot) dry-matter production (DM) were determined after drying the fresh samples in an oven at 75°C for 48 h. Plant tissue (ground to pass through a 2.0-mm-opening screen) was dry ashed at 500 °C, and treated with diluted (1:1) HCl. The P uptake of the plants was calculated by multiplying the P concentration in the tissue and the DM yield per pot. Relative shoot dry-matter and P uptake values were calculated by dividing the actual yields or P uptake by the maximum shoot dry-matter and P uptake values obtained in the corresponding soil.

The concentration of P in soil and plant digestion extracts was measured by the ammonium molybdate method with ascorbic acid as the reducing agent (Murphy and Riley, 1962). All determinations were made in duplicate and results given as means. Data processing and statistical analyses were performed with the aid of the Excel Package Software (Excel, 1977).

Results and Discussion

The soils used in this study (three Regosols, one Fluvisol, and one Luvisol) presented a quite wide range in their properties. They were sand to loam in texture with pH ranging from 6.6 to 7.8 (Table 1). Soil P sorption index ranged from 5.5 to 22.7, indicating a low-medium phosphate retention capacity of the soils (Indiati and Loj, 1998).

The amounts of P extracted according to the different P test methods are reported in Table 2 for soils added and not with P. Generally, the original soils (not treated samples) showed an extractable P content below the sufficiency level with the lowest variation among the soils shown by the Olsen test (4.2 to 10.2 mg P kg^{-1} soil) and the highest variation by the M3 test (6.6 to 47 mg P kg^{-1} soil).

The amounts of extracted P increased in the order: Pi-P<Ols-P<AERM-P<M3-P. Taking the Olsen test as the reference method, this sequence can be expressed by calculating the ratio between the mean values of the P extracted by each procedure and that extracted by the Olsen test multiplied by 100: 80<100<113<158. Extractable P by the Olsen, M3, Pi and AERM methods in all soils under study were correlated among themselves. Linear regression models were fitted to the data as given in Table 3.

Table 3. Regression equation and coefficients of determination (R^2) for relationships among soil tests (all values are significant at $p<0.001$).

Soil P test	Regression model	R^2
M3 *vs.* Pi	Y = 0.98 x + 20.2	0.411
Ols *vs.* Pi	Y = 0.80 x – 0.2	0.939
Ols *vs.* M3	Y = 0.40 x + 5.3	0.550
AERM *vs.*Pi	Y = 1.00 x + 4.3	0.752
AERM *vs.* M3	Y = 0.66 x + 3.0	0.755
AERM *vs.* Ols	Y = 1.30 x + 3.0	0.880

All of the equations are very closely significant ($p<0.001$), and the highest coefficient of determination resulted for the relationship between Olsen-P and Pi-P ($R^2=0.939$) The determination of conversion equations is especially important when studying new soil tests, as it allows the use of relations between soil nutrient contents and crop response evaluated by the usual methods.

In addition, knowledge of the parameters of the regression conversion equations permits i) the comparison of P data, which are obtained using the different extractants, and ii) the determination of critical P levels for one extractant when using another.

Following application of P, the content of soil extractable P linearly increased with added P, for all soils and P tests (R^2=0.98-0.99, p<0.001). The fractional amount of added P remaining in extractable P form is commonly named P availability index (F). It is calculated for each soil and soil test by the slope of the regression equation of the relationship between soil extractable P and added P. In this experiment, the mean F index values were 0.40 (Ols), 0.47 (Pi), 0.53 (AERM), and 0.60 (M3) (Table 4).

Table 4. Values of the P availability index (F) for the soils used.

Soil	F-Ols	F-M3	F-strip	F-resin	Mean
Fontana Nuova	0.45	0.69	0.53	0.59	0.57
Capobarile	0.45	0.66	0.53	0.59	0.56
Val Furella	0.40	0.64	0.48	0.56	0.52
Sterparo	0.38	0.58	0.43	0.51	0.48
Colle Castello	0.32	0.42	0.40	0.42	0.39
Mean	0.40	0.60	0.47	0.53	

These values indicated that the Olsen test was the least efficient method in extracting added P from the soils. On average, for all soil P test methods studied, the F index increased in the soils in the order: Colle Castello <Sterparo <Val Furella <Capobarile <Fontana Nuova. Generally, soils with the lowest P sorption capacity showed the highest F value, and a very close correlation was found for the relationship between soil P-SI and F index (Figure 1).

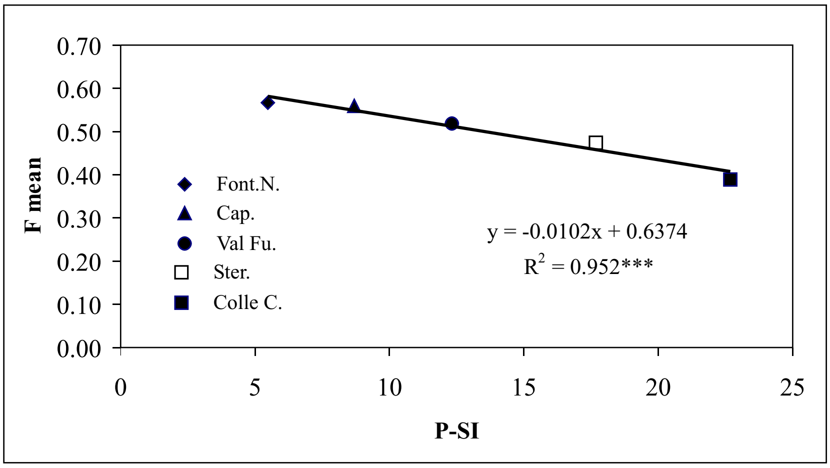

Figure 1. Relationship between P sorption index (P-SI) and P availability index, for the soils (Font.N. = Fontana Nuova; Cap. = Capobarile; Val. Fu. = Val Furella; Ster. = Sterparo; Colle C. = Colle Castello). F mean represents mean F index value relative to all soil P tests studied.

In order to compare ability of the different conventional and P-sink soil P test methods in predicting plant P availability, the amounts of extracted P were linearly related to relative dry matter yield and shoot P uptake (Table 5). However, use of quadratic equations generally improved all correlations as the data points of the individual soils could be defined by curves described by second-order polynomial functions (Table 6).

Table 5. Linear correlation analysis for the studied parameters.

Soil P tests	Rel. dry matter (%)	Rel. shoot P uptake (%)
Pi-P	0.856	0.744
M3-P	0.734	0.747
Ols-P	0.867	0.772
AERM-P	0.901	0.880

Table 6. Relationships between extracted phosphorus (x-mg P kg^{-1}) and relative dry-matter production (y) and plant P uptake (y_1) for soil P test.

Soil P test	Regression model	$R^{2(*)}$
Ols-P	$y=-0.053x^2+4.74x-6.13$	0.854
Pi-P	$y=-0.029x^2+3.46x-2.76$	0.804
AERM-P	$y=-0.025x^2+3.19x-3.43$	0.908
M3-P	$y=-0.011x^2+2.01x+2.26$	0.635
Ols-P	$y_1=-0.039x^2+3.89x-0.71$	0.650
Pi-P	$y_1=-0.023x^2+2.93x+1.29$	0.601
AERM-P	$y_1=-0.018x^2+2.77x-3.69$	0.819
M3-P	$y_1=-0.008x^2+1.80+0.33$	0.602

(*) All values are significant at 0.1% probability level

Generally, relative plant yield was better indicator of soil P availability than relative P uptake, for all the extractants. Any case, all soil P test methods were very closely correlated ($p<0.001$) to the biological parameters studied. Comparison among the extractants as to their ability to accurately reflect the plant P availability of the soils, showed that AERM method gave the best results, by accounting for 91 and 82 % of the variability in relative dry-matter yield and relative shoot P uptake, respectively (Figure 2).

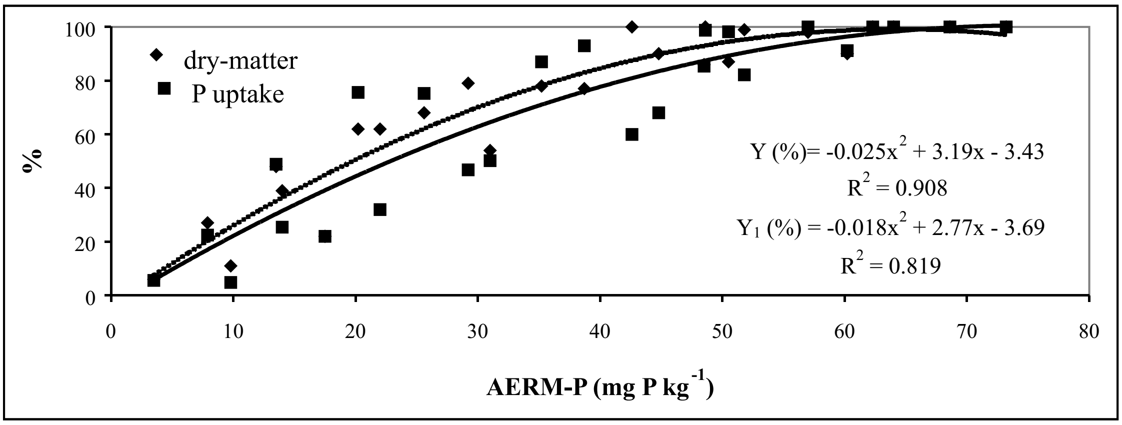

Figure 2. Correlation between resin membrane-P (AERM-P) test and relative dry matter yield (---) and shoot P uptake (—).

This agrees with findings from other studies by Schoneau and Huang (1991), and Fernandes *et al.*, 2000.

Conclusions

In this study, the AERM soil P test method resulted the best accurate in predicting plant P availability, followed by the Olsen, Pi, and M3 soil P extraction procedures. Further research is needed in order to test the P-sink methods in different soil types of the Mediterranean environment.

References

Amer, F., D.R. Bouldin, C.A. Black, and F.R. Duke. 1955. Characterisation of soil phosphorus by anion exchange resin adsorption and P^{32} equilibrium. Plant and Soil 151: 143-146.

Bache, B.W., and E.G. Williams. 1971. A phosphate sorption index for soils. J. Soil Sci. 22: 289-301.

Buondonno, A., E. Coppola, D. Felleca, and P. Violante. 1992. Comparing tests for soil fertility: 1. Conversion equations between Olsen and Mehlich 3 as phosphorus extractants for 120 soils of South Italy. Comm. Soil Sci. Plant Anal. 23: 699-716.

Excel. 1977. Excel Statistical Program. Microsoft Corp., Seattle, WA.

FAO-UNESCO. 1975. Carte mondial des sols. UNESCO, Rome.

Fernandes, M.L., F. Calouro, R. Indiati, and A.M. Barros. 2000. Evaluation of soil test methods for estimation of available phoshporus in some Portuguese soils: a greenhouse study. Comm. Soil Sci. Plant Anal. 31: 2535-2546.

Guo, F., R.S. Yost, and R.C. Jones. 1996. Evaluating iron-impregnated paper strips for assessing available soil phosphorus. Comm. Soil Sci. Plant Anal. 27: 2561-2590.

Indiati, R., and G. Loj. 1998. Use of alternative soil phosphorus test for improving environmental management of soil phosphate. Agr. Med. 128:298-306.

Mehlich, A. 1984. Mehlich 3 soil test extractant: A modification of Mehlich 2. Comm. Soil Sci. Plant Anal. 15: 1409-1416.

Mehra, O.P. and M.L. Jackson. 1960. Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate. Clays and Clay Miner. 7:317-327.

Menon, R.G., L.L. Hammond, H.A. Sissingh. 1988. Determination of plant available phosphorus by the iron hydroxide-impregnated filter paper (Pi) soil test. Soil Sc. Soc. Am. J. 53: 110-115.

Menon, R.G., S.H. Chien and L.L. Hammond. 1989. Comparison of the Bray1 and Pi test for evaluating plant-available phosphorus from soils treated with different partially acidulated phosphate rocks. Plant and Soil 114: 211-216.

Menon, R.G., S.H. Chien and L.L. Hammond. 1990. Development and evaluation of the Pi soil test for plant-available phosphorus. Comm. Soil Sci. Plant Anal. 21: 1131-1150.

Mozaffari, M. and J.T. Sims. 1994. Phosphorus availability and sorption in an Atlantic coastal plain watershed dominated by animal-based agriculture. Soil Sci. 157: 97-107.

Murphy, J. And J.P. Riley. 1962. A modified single solution method for the determination of phosphate in natural water. Anal. Chem. Acta 27: 31-36.

Olsen, S.R., C.V. Cole, F.S. Watanabe, and L.A. Dean. 1954. Estimation of available phosphorus in soils by extracting with sodium bicarbonate. USDA Circ. 939. U.S. Gov. Print. Office, Washington D.C.

Quian, P., J. Schoneau, W.Z. Huang. 1992. Use of ion exchange membranes in routine soil testing. Comm. Soil Sci. Plant Anal. 23: 1791-1804.

Raveh, A. and Y. Avnimelech. 1972. Potentiometric determination of soil organic matter. Soil Sci. Soc. Amer. Proc. 35: 967.

Saggar, S.M., J. Hedley, R.E. White. 1990. A simplified resin membrane tecnique for extracting phosphorus from soils. Fert. Res. 24: 173-180.

Schoneau, J.J., W.Z. Huang. 1991. Anion-exchange membrane, water and sodium bicarbonate extractions as soil test for phosphorus. Comm. Soil Sci. Plant Anal. 22: 465-492.

Sharpley, A.N. 1991. Soil phosphorus extracted by iron-aluminum-oxide-impregnated filter paper. Soil. Soc. Am. J. 55: 1038-1041.

Sibbesen, E. 1977. A simple ionexchange procedure for extracting plant-available elements from soil. Plant Soil 46:665-669.