



Full Length Research Paper

Determining the suitability of multipurpose extractants for soil under traditional smallholder food crop production systems

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Abstract

Eight extractants: Bray's P-1 (0.03N NH_4F + 0.025N HCl), Bray's P-1 + 0.01M EDTA, Bray's P-1 + 0.005M DPTA, Bray's P-1 + 0.01M EDTA, AHDF (0.1N $\text{NH}_4\text{F.HF}$), AHDF + 0.005M DPTA, Modified Olsen's 0.5M NaHCO_3 + 0.01M EDTA, Modified Olsen's 0.5M NaHCO_3 + 0.005M DPTA and 1.0N NH_4OAc (pH 7.0) were evaluated for predicting the availability of plant nutrients in thirty surface (0-15cm) soil samples collected from smallholder farmers' plots in Ekiti State, Nigeria. The minus-one nutrient technique was used for maize grown in the greenhouse for 28 days and regression analyses carried out between amounts of nutrients determined with the extractants and uptake of respective nutrients. Correlation coefficients show that Bray's P-1 and AHDF, with or without chelates, and modified 0.5M NaHCO_3 can be used to assess available P and exchangeable K in the soils but not exchangeable Mg. The correlation coefficients of Bray's P-1 + DPTA and 0.5M NaHCO_3 + 0.01MEDTA with P, K, Mg, Cu, Zn and Mn uptake were significant and so meet one of the criteria for selecting them multipurpose extractants. The need to remove the dark colour from extracts would make the alkaline 0.5M NaHCO_3 extractant less suitable for a rapid routine analytical procedure.

Keywords: Extractant, suitability, maize, nutrient uptake, correlation coefficients, Ekiti State.

INTRODUCTION

The desire to increase the reliability of soil tests necessitates an update of methods and instrumentation. The development of new procedures, refinement and standardization of existing ones ensure increased accuracy and precision of tests, and reproducibility of results needed in the rapid analysis of soil samples. The development of a multipurpose (multi-nutrient) extractant is one of such advances needed to cut down cost, save time and increase the efficiency in routine analysis of large numbers of soil samples (Jones, 1990; Westerman, 1990). This is compatible with the increased automation and sophisticated instrumentation needed for rapid soil tests that have characterized the operations in some laboratories since the 1980s.

Salt solutions buffered to specific pH values have been developed, with which to determine several nutrients in the same extract. Morgan's reagent (0.25N acetic acid, HOAc-0.25N sodium acetate, NaOAc. pH 4.8), introduced in 1941 and adapted to acid soils with

$<10 \text{ cmol.kg}^{-1}$ CEC, was used to determine most nutrients elements in the soil and so referred to as a "universal soil extracting solution" (Jones, 2001). Extraction reagents that contain exchange cations of sufficient strength would remove most, if not all, of the cations from the colloidal complex. Thus, neutral normal ammonium acetate (1N NH_4OAc , pH 7.0) that uses ammonium ion (NH_4^+) as the exchange cation has been the most commonly adopted method of analysis given its effectiveness in wetting soil, ability to replace exchangeable cations (for determination of total exchangeable bases) and suitability for use in flame emission photometry (Anon, 1994a). The multi-nutrient role of ammonium acetate in determination of micronutrients is fortuitous, since the extracting solution alone or with inclusion of a chelate (EDTA) would be acidified (buffered) to attain different pH values for some of the micronutrients (Jones, 2001). The DPTA soil test developed for extractable Cu, Fe, Zn and Mn (Lindsay

and Norvell, 1978) has been widely adopted (Johnson, 1992; Liang and Karamanos, 1993). Addition of 1M NH_4HCO_3 to the DPTA widened its scope to include the determination of some macronutrients (Soultanpour and Schwab, 1977).

The tendency has been to develop an available P extractant and then increase the number of nutrients that can be determined with it. The double acid (DA, 0.05N HCl –0.02N H_2SO_4) also known as North Carolina or Mehlich No. 1 extractant, the Mehlich No. 2 (0.2N NH_4Cl –0.2N HOAc –0.015N NH_4F –0.012N HCl) and Mehlich No. 3 (0.02N HOAc –0.25N NH_4NO_3 –0.015N NH_4F –0.01M HNO_3 –0.001MEDTA) were developed for available P and used to determine exchangeable K, Ca and Mg, and available Mn and Zn (Mehlich, 1984). The adoption of Mehlich No 3 for P in the United States is because of the multi-nutrient capability and correlation with other extraction methods (Anon, 1994b). Bray's P-1 and Olsen's 0.5M NaHCO_3 have potentials for use as exchangeable K extractants in soils of Nigeria (Sobulo, 2000). The efficiency of double F⁻ to extract Al-bound P, the most abundant and available inorganic P fraction in acid soils enabled acidic AHDF (0.1N NH_4F .HF) extractant to give similar or better performance than Bray's P-1 for available P in soils of South-Western Nigeria (Ayodele and Agboola, 1981; Agboola and Ayodele, 1985). It was, thus, reasoned that the addition of a chelate to the acidic P extractants would widen the range of nutrients that can be determined from the extracts. This was accomplished in the modified 0.5M NaHCO_3 , developed by adding 0.01M EDTA, to become an extractant for P, K, Cu, Fe, Zn, Mn and possibly boron (B) and sulphur (S) in acid to alkaline soils (Hunter, 1974). This study was carried out to compare the efficiency of chemical solutions for determination of macro- and micronutrients in the soils of Ekiti State, Nigeria with a view to selecting a suitable multipurpose extractant for the soil testing programme.

MATERIALS AND METHODS

Soils used for the study

Surface (0-15cm) soil samples were randomly collected from thirty (30) farmers' fields cultivated to major food crops (maize, yam, cassava, cowpea, upland rice, cocoyam, plantain, leaf and fruit vegetables etc) in the main agricultural lands of Ekiti State, Nigeria. Ekiti State is located between long. 4°51' to 5°45' E and lat 7°45' to 8°05' N and has a land mass of approximately 5,804.6km², spread over 16 Local Government Areas. The samples were bulked to make a composite sample for each site, air-dried and sieved (< 2.0mm); and analyzed for pH (in distilled water), particle size distribution, organic carbon, total N, exchangeable cations and total exchange acidity (IITA, 1979).

Evaluation of extractants

The extractants developed using chemical reagents are as follows:

Bray's P-1 (0.03N NH_4F + 0.025N HCl); Bray's P-1 + 0.01M EDTA;

Bray's P-1 + 0.005M DPTA; AHDF (0.1N NH_4F .HF, pH 4.1-4.3)

AHDF + 0.005M DPTA; Modified Olsen's 0.5M NaHCO_3 + 0.01M EDTA, pH 8.5)

Modified Olsen's 0.5M NaHCO_3 + 0.005M DPTA; and 1.0N NH_4OAc (pH 7.0)

The extraction time was 15 minutes stirring of a 1:10 soil to solution ratio, on a Multiple Stirrer (Custom Laboratories) and the mixture filtered. P in the extracts was determined by colorimetry, K by flame emission while Mg, Zn, Cu and Mn were estimated by atomic absorption spectrophotometer (IITA, 1979).

Greenhouse study

Four hundred millilitres (ml) of soils were measured into half-litre (500 ml) plastic cups, for the respective nutrients. The minus-one nutrient technique in which nutrient solutions prepared with reagent grade chemicals had the nutrient being evaluated missing was used. The nutrients were added at 100 mg N as NH_4NO_3 , 20 mg P as orthophosphoric acid, 80 mg K as KCl , 20 mg Mg as $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 10 mg Zn as ZnCl_2 , 7.5 mg Fe as $\text{FeSO}_4 \cdot 5\text{H}_2\text{O}$ and 2.0 mg Cu as $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ per litre. The respective solutions were added to each set of soils, watered to field capacity and allowed to equilibrate for 24 hours. Maize (TZPB variety) seeds were planted, and the seedlings thinned to five per cup after emergence. The plants were watered daily with a solution containing 1.5 g NH_4NO_3 per 5 litres of water. After 28 days' growth, the maize tops were cut at soil surface, rinsed in water and dried at 60°C for 48 hours, weighed and milled. One-half gram (0.5 g) samples were digested with a ternary mixture of concentrated nitric, sulphuric and perchloric acid (25:5:5 v/v) on a hot plate until white fumes evolved. The digest was diluted to 100 ml in volumetric flasks. P in the digest was determined by the vanado-molybdate yellow method, K by flame photometry Mg and micronutrients by atomic absorption spectrophotometry (IITA, 1979). Nutrient uptake was calculated as the product of nutrient concentration (%) and dry matter yield.

RESULTS AND DISCUSSION

The means and ranges of the properties of soils used for the study are shown in Table 1. The soils were sandy loams to sands; this coarse texture is typical of soils in the sub-humid forest and derived savannah zones formed on basement complex rocks (Murdoch et al.,

Table 1. Means and ranges of properties in the soils used for the study

Property	Mean	Range
pH (in distilled water)	6.5	4.9-8.3
Sand, %	67.0	46.0-85.0
Silt, %	19.0	4.0-28.0
Clay, %	14.0	1.0-38.0
Textural Class*	SL	S – CL
Organic matter, %	3.4	1.3-6.4
Total N, %	0.14	0.06-0.25
Effective CEC cmol.kg ⁻¹ soil	8.4	2.6-30.3
Base saturation, %	95.0	87.0-99.0

* SL = Sandy loam; S = Sand; CL = Clay loam

1976). The low clay content is related to intensive weathering of the parent materials that contain little weatherable minerals, such that soil mineralogy is dominated by low-activity, non-swelling 1:1 lattice clays, mainly kaolinite, quartz and oxides (Juo, 1986; Ogunkunle, 2009).

In evaluating the fertility status of the soils, comparison was made with the criteria and fertility classes established for the soils in Nigeria (Agboola and Ayodele, 1985; FMANR, 1990; FDALR, 2004; Anon, 2006). The mean soil pH (6.5) was near neutral while the least value (pH 4.9) is at the lower limit of strong acidity. The mean organic matter content (3.4%) falls within the high fertility class probably because the soils were collected from smallholder food crop production farms which had been cleared from bush fallows of varying periods- the main soil management practice needed to build up organic matter and nutrients (Lal, 1999). Effective CEC varied between 2.6 to 30.3 cmol.kg⁻¹ soils, 95% of which was made up of basic cations suggesting that liming would not be a condition to enhance the utilization of nutrients whose availability is influenced by soil pH. Besides, the low intensive slash-and-burn bush fallow practices would mean addition of various amounts of ash (Araki, 1993) such that acidity may not pose problems for the traditional food crops being grown.

The means and ranges of nutrients determined with various extractants are shown in Table 2. Bray's P-1 + EDTA extracted the highest amount of available P followed by Bray's P-1, AHDF, AHDF + EDTA and Bray's P-1 + DPTA which did not differ significantly ($P=0.05$) while 0.5M NaHCO₃ + DPTA extracted the least. The extractants clearly reflected the variation of available P in the soils and gave mean values that exceeded or were similar to critical levels of 8.5 - 10 mg.kg⁻¹ soil established with conventional procedures for the soils in south-western Nigeria (Ayodele and Agboola, 1981). 0.5M NaHCO₃ extracted the highest amounts of exchangeable K and Mg while the least values at 0.41 and 1.14 cmol.kg⁻¹ soil, respectively with Bray's P-1 + DPTA suggest that all extractants were efficient in assessing K and Mg status when compared to the performance of the

conventional neutral 1.0N NH₄OAc. Based on 0.20 cmol.kg⁻¹ soil critical level (FMANR, 1990; FDALR, 2004; Anon, 2006), these soils were sufficient in exchangeable K. The basement complex rock parent materials are rich in K minerals (Esu, 2010) and so would produce soils with ample supplies of exchangeable K, while the ash from slash-and-burn bush clearing practices would increase surface layer soluble K. The K extracted is largely the water-soluble form and that displaced from the exchange sites as water and weak salt solutions would extract ions in the soil that are in equilibrium with that on the exchange complex (Jones, 2001). These solutions would extract the water-soluble K, especially in these coarse-textured soils, and also contain the cations that can displace K such that extractant performance would be a function of the ionic components. The highest means from 0.5M NaHCO₃ relate to the efficiency of Na⁺ in displacing K⁺ while H⁺ and NH₄⁺ appear equal, as indicated by similar values from Bray's P-1, AHDF and NH₄OAc. Based on 0.30-0.40 cmol.kg⁻¹ soil critical level established for soils in south-western Nigeria (FMANR, 1990), Mg was adequate in the soils.

AHDF extracted the highest amount of Zn, and the efficiency decreased with addition of a chelate, unlike the inclusion of EDTA and DPTA in 0.5M NaHCO₃, which gave same mean values. The significantly higher Mn when acid extractants were modified with chelates is shown by 411.4 and 330.4% increase in amount determined using Brays P-1 + EDTA and Bray's P-1 + DPTA -1 respectively. Mean Cu was high in the soils based on 0.7 mg.kg⁻¹ soil value for high fertility class. The highest amount was extracted by 0.5M NaHCO₃ + EDTA. The addition of EDTA increased the amount of Cu extracted with AHDF while DPTA gave higher value than EDTA added to the Bray's P-1 extractant. The 1.0N NH₄OAc was not comparable with these methods because of the different pH values required in the procedures for the determination of respective micronutrients.

The correlation coefficients of simple regression analyses between amounts of nutrients extracted and uptake by maize are presented in Table 3. The corre-

Table 2. Means* and ranges of nutrients determined with various extractants

Extractant	K	Mg	P	Cu	Mn	Zn
Bray's P-1	0.55bc (0.21-0.91)	1.52cd (0.26-3.20)	10.4b (2.5-43.7)	-	-	-
AHDF	0.54cd (0.21-0.81)	1.61cd (0.14-4.34)	10.9b (0.5-51.4)	0.9c (0.2-1.6)	75.3d (2.0-306.0)	8.5a (3.0-15.0)
Bray's P-1 + EDTA	0.51d (0.26-0.88)	1.74c (0.64-3.52)	13.4a (2.1-50.4)	0.9c (0.1-2.3)	385.1a (58.0-726.0)	7.4b (5.9-9.3)
AHDF + EDTA	0.52cd (0.12-0.91)	1.35de (0.62-2.94)	10.3bc (1.5-38.6)	1.7b (0.4-3.2)	213.4c (39.0-379.0)	2.8e (0.6-7.1)
NaHCO ₃ + DPTA	0.68a (0.26-1.04)	2.45b (0.84-4.32)	6.2d (2.1-21.6)	1.6b (0.7-3.0)	29.4d (6.6-67.2)	4.3d (2.8-7.9)
NaHCO ₃ + EDTA	0.58b (0.21-1.01)	3.15a (1.02-5.34)	8.2cd (0.5-40.6)	2.1a (0.3-4.3)	68.2d (2.0-149.0)	4.3d (2.9-6.7)
Bray's P-1 + DPTA	0.41e (0.16-0.78)	1.14e (0.66-2.10)	9.0bc (3.9-20.6)	1.4b (0.3-3.1)	324.1b (48.0-658.0)	5.9c (2.2-8.9)
NH ₄ OAc	0.55bc (0.20-1.06)	1.77c (0.83-3.50)	-	-	-	-

() ranges of soil test values

*Means for each nutrient followed by same alphabets do not differ significantly (P = 0.05)

Table 3. Correlation coefficients of relationships between extractable nutrients and uptake by maize grown in the greenhouse

Extractants	P	K	Mg	Mn	Cu	Zn
Bray's P-1	0.88*	0.93**	0.27	-	-	-
AHDF	0.89**	0.86**	0.20	0.81*	-0.20	0.13
AHDF+DPTA	0.87**	0.72**	0.31	0.01	0.20	0.05
Bray's P-1+DPTA	0.88**	0.78**	0.41*	0.60**	0.45*	0.55*
Bray's P-1+EDTA	0.87**	0.85**	0.31	0.01	0.36	0.22
NaHCO ₃ +EDTA	0.84**	0.82**	0.23	0.49*	0.44*	0.83**
NaHCO ₃ +DPTA	0.88**	0.51*	0.37	0.53*	-0.36	0.10
NH ₄ OAc	-	0.88**	0.34	-	-	-

*, ** denote significance at 5 and 1% respectively

lation coefficients between amounts extracted by the methods and P uptake were similar suggesting that all the extractants would adequately measure available P in the soils. All the exchangeable K extractants had significant correlation coefficients which are due to the fact that most available K is water-soluble and in light-textured soils, this solubilization would readily take place

and so explains the suitability of the extraction methods. Exchangeable Mg was poorly correlated with uptake, however, the Bray's P-1 + DPTA with $r = 0.41^*$ shows promise, being better than 0.5M NaHCO₃ + EDTA and conventional 1N NH₄OAc. The low correlations could be due to high values of exchangeable Mg in the soils compared to the established critical levels. This suggests

Table 4. Correlation coefficients of the relationships between 0.5 NaHCO₃ + EDTA and other extractants

Extractants	P	K	Mg	Mn	Cu	Zn
Bray's P-1	0.96*	0.89**	0.81*	-	-	-
Bray's P-1+DPTA	0.96**	0.95**	0.49*	0.82*	0.89**	0.66**
Bray's P-1+EDTA	0.87**	0.85**	0.57*	-0.26	0.18	0.29
AHDF	0.94**	0.87**	0.23	0.76**	0.21*	-0.13
AHDF+DPTA	0.90**	0.68**	0.25	0.12	0.01	-0.50*
NaHCO ₃ +DPTA	0.96**	0.96**	0.71**	0.90**	0.80**	0.81**

*, ** denote significance at 5 and 1% respectively

the need to re-evaluate the indices of Mg availability. The Mg/K ratio was suggested as a better indicator of availability than exchangeable Mg and the critical ratio of 2.0 (FMANR, 1990) would correspond to 1.20-1.25 cmol.kg⁻¹ soil which is much higher than 0.30-0.40 cmol.kg⁻¹ soil established criteria for Mg. It was further observed that uptake can be suppressed by K even when Mg is adequate. Extractable Zn and Cu with Bray's P-1 + DPTA and 0.5M NaHCO₃ + EDTA correlated significantly with uptake while the order for Mn was AHDF > Bray's P-1 + DPTA > 0.5M NaHCO₃ extractants.

Bray's P-1 + DPTA and 0.5M NaHCO₃ + 0.01M EDTA show promise as multipurpose extractants. First, the performance for available P will be related to dissolution through ligand exchange of Al- and Fe-bound surface P for the OH⁻, HCO₃⁻, F⁻ and Cl⁻ (Tisdale et al., 1993). Since Al-P and Fe-P are the most abundant inorganic P forms in these soils (Ayodele, 1986), then these extractants will solubilize them and so give a proper estimation of available P status. Alkaline extractants selectively dissolve the Fe-P while Al-P makes the greatest contribution to available P determined with Bray's P-1 and 0.5M NaHCO₃ (Jones, 2000). From the correlation coefficients between the extractants, shown in Table 4, they probably extract the same proportionate amounts of the amorphous and colloidal P associated with the surfaces of Al, Fe-oxides rather than the crystalline Al-P and Fe-P, extractable with NH₄F and NaOH respectively (Brady and Weil, 1999).

Second, the high correlation coefficients between the K extractants are expected since they can solubilize the water-soluble K and displace proportionate amounts of K from the exchange complex. Third, the significant correlations with 0.5M NaHCO₃ + DPTA for Mg, Cu, Zn and Mn indicate that the same forms of these nutrients are probably extracted, in relation to the similarity in ionic composition.

Thus, by using Bray's P-1 + DPTA and 0.5M NaHCO₃ + EDTA as extractants, the soil available P, exchangeable K and Mg, available Cu, Mn and Zn can be determined and would predict their availability. However, the 0.5M NaHCO₃ method presents some problems for adaptation to rapid routine testing of soils. The modified extractant contains sodium EDTA and a flocculant and is buffered to pH 8.5 with 0.1N NaOH. Extracts are dark in

colour due to dissolved organic matter, in relation to the level in the soil, which tends to mask development of the molybdate blue colour whose intensity is read on spectrometers. Although the dark colour can be easily removed with activated charcoal or drops of concentrated sulphuric acid, it is doubtful if the additional expenses on the reagents and work are justified as the 0.5M NaHCO₃ had no demonstrable advantage over Bray's P-1 for the determination of available P in the soils. The next step will be to correlate the extractable amounts with responses to the respective nutrients in greenhouse and field studies, to determine critical levels for various crops. Thereafter, calibration of soil test values into low, medium and high fertility classes would be done to provide the basis for fertilizer response predictions and efficient fertilizer recommendations in the soils.

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