



MINISTÉRIO DA AGRICULTURA — MA
Empresa Brasileira de Pesquisa Agropecuária — EMBRAPA
Centro de Pesquisa Agropecuária do Trópico Úmido — CPATU

1º Simpósio
do Trópico Úmido

1st Symposium
on the Humid Tropics

1er Simposio
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ANAIS
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Volume I

CLIMA e SOLO

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BELEM - PARÁ - BRASIL

1986



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Belém, Pará, 12 a 17 de Novembro de 1984

Belém, November 12 through 17, 1984

Belém, 12 a 17 de novembre de 1984

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EMBRAPA-CPATU. Documentos, 36

Exemplares desta publicação podem ser solicitados à
EMBRAPA-CPATU

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Caixa Postal, 48

66000 Belém, PA - Brasil

Tiragem: 1.000 exemplares

Observação

Os trabalhos publicados nestes anais não foram revisados pelo Comitê de Publicações do CPATU como normalmente se procede para as publicações regulares. Assim sendo, todos os conceitos e opiniões emitidos são de inteira responsabilidade dos autores.

Simpósio do Trópico Úmido, I, Belém, 1984.

Anais. Belém, EMBRAPA-CPATU, 1986.

6v. (EMBRAPA-CPATU. Documentos, 36)

I. Agricultura — Congresso — Trópico. I. Empresa Brasileira de Pesquisa Agropecuária. Centro de Pesquisa Agropecuária do Trópico Úmido, Belém, PA, II. Título. III. Série.

CDD: 630.601

SOIL SAMPLING FOR ESTIMATION OF MICRONUTRIENT AVAILABILITY AND PHYSICO-CHEMICAL PROPERTIES OF A REPRESENTATIVE SOIL TYPE OF MARAJÓ ISLAND, PARÁ

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e José Ferreira Teixeira Neto³

ABSTRACT: A study was carried out to determine the number of core samples required to make a composite sample for a given area to estimate micronutrient availability and physico-chemical properties of a representative soil type, viz. Ground Water Laterite (having sandy surface and clayey subsoil, poor drainage and periodic flooding and level slope) of Marajó Island. Fifty two core samples (0-20 cm) were taken at random from 1 ha area under native pasture at the rate of 13 samples per block of 0.25 ha. The C.V. for the available Zn, Cu, Mn and Fe extracted by 0.005M DTPA (pH 7.3) varied from 40.5 to 51.8% with the sequence of the nutrients for it being: Cu > Mn > Fe > Zn. A confidence limit $\pm 30\%$ of the mean value of different soil characteristics was used to determine the number of core samples required to make a composite sample. In the case of micronutrients it was found that 7, 9, 11 and 13 core samples were needed to prepare a composite sample to estimate the availability of Zn, Fe, Mn and Cu respectively. Estimation of soil pH (H₂O) required only one representative soil core sample. To estimate macronutrient fertility, while total N and available P (Mehlich 1) required 3 core samples, organic carbon required 5 to make a composite sample. In the case of available K extracted by Mehlich 1, C.V. was found to be 123.8%, the highest for any soil characteristic and thus required 69 core samples for making a composite sample. For determination of effective C.E.C., exchangeable Ca²⁺, Mg²⁺, Na⁺, H⁺ and Al³⁺ could be analysed from a composite sample made respectively from 13, 12, 4, 3 and 2 core samples. Soil texture could be defined from the analysis of soil separates of a composite sample prepared from a minimum of 6 core samples as in the case of silt, C.V. (37.6%) was much higher than that for sand or clay. In general, it was concluded that for the soil studied under the given field conditions, a good estimation of available micronutrients, macronutrients and other their physico-chemical properties required a composite sample prepared from at least 15 core samples per hectare. The only exception to this was found to be available K.

Index terms: Soil fertility evaluation, micro-and macro-nutrients, availability, soil properties, soil variability and unit of sampling.

AMOSTRAGEM DE SOLOS PARA ESTIMAR A DISPONIBILIDADE DE MICRONUTRIENTES E AS CARACTERÍSTICAS FÍSICO-QUÍMICAS DE UM SOLO REPRESENTATIVO DA ILHA DO MARAJÓ, PARÁ

RESUMO: Foi conduzido um estudo para determinar o número necessário de amostras simples para obter-se uma amostra composta por unidade de área, visando estimar a disponibilidade

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de micronutrientes e as propriedades físico-químicas de um solo representativo identificado como Literita Hidromórfica, arenosa/argilosa, fase campo higrófilo, relevo plano, ocorrente na ilha do Marajó. Cinquenta e duas amostras simples (0-20 cm) foram coletadas ao acaso, de uma área de 1 ha sob pastagem nativa, distribuídas em treze amostras por bloco de 0,25 ha. O CV para Zn, Cu, Mn e Fe extraídos por DTPA 0.005M (pH 7,3) variou entre 40,5 e 51,8% o qual obedeceu a seguinte seqüência: $Cu > Mn > Fe > Zn$. Foi estabelecido o limite de confiança de $\pm 30\%$ do valor médio das diferentes características de solo para se determinar o número necessário de amostras simples para constituir uma amostra composta. No caso de micronutrientes achou-se que sete, nove, onze e treze amostras simples foram suficientes para a coleta de uma amostra composta visando estimar a disponibilidade de Zn, Fe, Mn e Cu, respectivamente. A estimativa do $pH(H_2O)$ do solo requereu apenas uma amostra simples. Para o nitrogênio total e o P disponível (Mehlich 1) foram necessárias três amostras simples, enquanto que para o carbono orgânico, cinco amostras. No caso do K disponível extraído por Mehlich 1, o coeficiente de variação foi de 123,8%, o maior de todos para as características do solo, havendo necessidade de 69 amostras simples para uma amostra composta. A CTC efetiva pode ser determinada em amostras compostas constituídas de treze, doze, quatro, três e duas amostras simples, respectivamente para Ca^{2+} , Mg^{2+} , Na^+ , H^+ e Al^{3+} . É possível determinar a textura do solo em uma amostra composta constituída com o mínimo de seis amostras simples, como no caso de limo que apresentou C.V. (37,6%) muito maior que os obtidos para a areia e a argila. Em geral, concluiu-se que nas condições de campo do solo estudado para se obter uma boa estimativa da disponibilidade de micronutrientes, macronutrientes e de outras propriedades físico-químicas, há necessidade, por hectare, de uma amostra composta constituída de pelo menos quinze amostras simples. A única exceção encontrada foi para o potássio assimilável.

Termos para indexação: Avaliação de fertilidade de solo, disponibilidade de micro e macro nutrientes, propriedades do solo, variabilidade de solo e unidade de amostragem.

INTRODUCTION

Proper soil sampling is requisite of pragmatic evaluation on soil fertility in order to facilitate more efficient use of soil amendments and fertilizers. Soil as a heterogeneous body naturally presents a spatial variability in its physical and chemical composition and properties. Besides, interacting factors like topography, climate, vegetation, cultural and management practices among others are also determinants of the dynamics of any soil system. The soil sampling has to be thus area and soil-plant system specific, and there is continual need to define some of its important parameters such as depth of sampling, unit of sampling and number of core samples required to prepare a composite sample representative of the unit area.

The need to establish a more adequate system and techniques of collecting samples for soil fertility evaluation has been constantly emphasized by a number of Brazilian workers (Catani et al. 1954, Barreto

et al. 1974, Alvarez & Carraro, 1976, and Teixeira et al. 1984). However, these and other studies have been limited to evaluation of the status of macronutrients and soil properties and not much seems to have been done for the evaluation of micronutrient fertility of the soils. This study was thus undertaken to determine an appropriate sampling procedure for estimation of micronutrient and macronutrient fertility status apart from factors that affect their availability in a soil type predominantly representative in Marajó Island, state of Pará.

MATERIALS AND METHODS

Soil Samples

Soil samples for the study were collected in August 1984, before the onset of dry period, from Santo André Ranch, Anabijú River, Muaná County of Marajó Island, state of Pará. The soil of the area has been classified as Ground Water Laterite, characterized by sandy surface and clayey sub-

soil, poor drainage and periodic flooding and level slope.

The area had vegetation cover of native pasture predominantly composed of grasses and about 5% of forage legumes.

The unit area of sampling was 1 ha and for the convenience of sampling it was divided into four blocks of 0.25 ha each. In all, 52 surface soil samples (0-20 cm) were collected by selecting at random 13 core sites per block. To avoid contamination soil samples collected by a wooden tool were packed in polyethylene bags.

The samples were then air dried, ground with the help of wooden roller and board, passed through 1 mm sieve and finally stored in polyethylene lined cardboard boxes.

Analyses of soils

The physico-chemical properties of the soils were analysed as per method described by Guimarães et al. (1970). Organic carbon was determined by the method of Walkley & Black (1934). Available zinc, copper, manganese and iron were extracted by 0.005M DTPA (pH 7.3) (Lindsay & Norvell 1978). The micronutrients in the extract were analysed by atomic absorption spectrophotometry.

Statistical Analyses

The data were analysed statistically by the standard methods to determine the mean, standard deviation and C.V. of each of the available macronutrients and physico-chemical properties of the soil. Simple correlation coefficients were also determined between available micronutrients and soil physico-chemical properties.

The number of core samples (n) required to make a composite sample for determination of available micronutrients and macronutrients and soil physico-chemical properties was calculated by the following formula cited by Snedecor & Cochran (1967):

$$n = \frac{4 \sigma^2}{L^2}$$

Where:

n = number of core samples

σ^2 = variance of the population mean

4 = value of $(t)^2$ at the confidence level of 95%, assuming normal distribution.

L^2 = limit of confidence interval about mean

In this study, L value for any soil parameter studied was calculated as $\pm 30\%$ of its mean value. This criteria was adopted from a similar study conducted by Teixeira et al. (1984) on soil fertility evaluation of two Amazonian eco-systems.

RESULTS

The results of available micronutrients extracted by 0.005M DTPA (pH 7.3) are given in Table 1. As per the critical limits of 0.8 ppm zinc, 0.2 ppm copper, 1 ppm manganese and 8 ppm iron extracted by 0.005M DTPA (pH 7.3) (Lindsay & Norvell 1978) and the mean values of each of the available micronutrient in the soil, zinc, copper and manganese were found to be deficient whereas iron was sufficient. In such a comparison was made on the basis of individual determinations then 9.6% core samples for copper and 7.6% core samples for manganese could be classified as sufficient.

The C.V. was found to vary from 40.5 to 51.8% with the sequence of the micronutrients for it being $Cu > Mn > Fe > Zn$.

The number of core samples required to constitute a composite sample determined for confidence limit of $\pm 30\%$ of mean value of each of the micronutrient studied with 95% probability was found to be 13, 11, 9 and 7 for copper, manganese, iron and zinc respectively.

TABLE 1. Minimum number of core samples (n) required to make composite sample for confidence limit of $\pm 30\%$ of mean value (\bar{x}) and other statistical parameters of available zinc, copper, iron and manganese in the soil.

Available Micronutrient	Minimum	Maximum	\bar{x}	Standard Deviation	C.V. (%)	n
Zinc (ppm)	0.08	0.44	0.168	0.068	40.5	7
Copper (ppm)	0.02	0.29	0.118	0.061	51.8	13
Iron (ppm)	47.3	284.2	125.68	56.32	44.8	9
Manganese (ppm)	0.10	1.60	0.610	0.301	49.4	11

The soil properties are known to affect availability of micronutrients, therefore correlation coefficients among them were determined. Matrix of correlation given in Table 2 shows significant relationship of cop-

per with organic carbon ($r = 0.32^*$), clay ($r = 0.66^{**}$), effective C.E.C. ($r = 0.49^{**}$) and base saturation ($r = -0.36^{**}$) and of iron with clay ($r = 0.73^{**}$) and effective C.E.C. ($r = 0.36^{**}$).

TABLE 2. Matrix of correlation.

	1	2	3	4	5	6	7	8	9
1 Zn (ppm)	1	0.35*	0.20	0.32*	-0.24	0.05	0.06	-0.02	-0.03
2 Cu (ppm)		1	-0.13	0.61**	-0.13	0.32*	0.66**	0.49**	-0.36**
3 Mn (ppm)			1	0.06	-0.04	0.10	-0.11	0.27	-0.08
4 Fe (ppm)				1	0.02	0.05	0.73**	0.36**	-0.05
5 pH (H ₂ O)					1	0.14	0.02	0.06	0.36**
6 Org. C (%)						1	0.18	0.44**	-0.11
7 Clay (%)							1	0.59**	-0.21
8 Effective C.E.C. (meq/100g)								1	-0.32*
9 Base saturation (%)									1

* Significant at 0.05 level.

** Significant at 0.01 level.

Unmarked figures are not significant at 0.05 level.

In Table 3 are presented the results of the analyses of macronutrients, pH, organic carbon and the components of effective C.E.C. The soil reaction was found to be highly acidic (\bar{x} of pH < 4.). The C.V. of soil pH was found to be 9.3% which was the lowest among all the soil properties and thus required only one representative sample for its estimation. The macronutrient fertility of the soil may be considered poor as it was medium in organic carbon, low in total N, low in available P (Mehlich 1) and low in exchangeable K⁺. The C.V. for organic carbon, total N and available P was found to be varying between 24.7 and 32.4% and thus required 5.3 and 3 core samples respectively for constitution of a composite sample. The exchangeable K⁺

presented a C.V. of 123.8% which was the highest for any soil parameter. To estimate the exchangeable K⁺ in the soil thus would require a composite sample constituted from 69 core samples.

The results of the components of effective C.E.C. showed that soil was low in base saturation (\bar{x} =2.92%) and high in Al³⁺ saturation (\bar{X} =89.7%). The Ca²⁺ and Mg²⁺ presented C.V. of 53.5 and 51.5% respectively. On the other hand, the C.V. of Na⁺, H⁺ and Al³⁺ varied between 21 and 31%. Accordingly, the number of core samples required for Ca²⁺, Mg²⁺, Na⁺, H⁺ and Al³⁺ analyses was found to be 13, 12, 4, 3 and 2 respectively for constituting a composite sample.

TABLE 3. Minimum number of core samples (n) required to make composite sample for confidence limit of $\pm 30\%$ of mean value (\bar{x}) and other statistical parameters of chemical properties of the soil.

Soil Properties	Minimum	Maximum	\bar{x}	Standard Deviation	C.V. (%)	n
pH (H ₂ O)	3.20	5.60	3.942	0.368	9.3	1
Organic Carbon (%)	0.41	2.23	1.293	0.419	32.4	5
Nitrogen (%)	0.04	0.14	0.092	0.023	24.7	3
Available P (ppm)	1.00	2.88	1.730	0.463	26.8	3
K ⁺ (meq/100g)	0.02	0.44	0.062	0.077	123.8	69
Ca ⁺⁺ (meq/100g)	0.01	0.16	0.052	0.028	53.5	13
Mg ⁺⁺ (meq/100g)	0.02	0.19	0.046	0.024	51.5	12
Na ⁺ (meq/100g)	0.02	0.05	0.027	0.008	30.1	4
H ⁺ (meq/100g)	1.28	7.33	4.838	1.206	24.9	3
Al ⁺⁺⁺ (meq/100g)	0.71	2.31	1.631	0.343	21.0	2

The results of the analysis of soil separates are presented in Table 4. The C.V. was found to be maximum in the case of silt followed by clay and sand. The number of core samples required for making a composite sample was found to be 1, 6 and 3 for sand, silt and clay respectively.

DISCUSSION

The fertility evaluation of a representative soil type, viz. Ground Water Laterite, of Marajó island, Pará, showed that it was highly acidic in reaction, low in base saturation, high in Al³⁺ saturation and poor in macronutrient and micronutrient fertility. The only exception to this was available iron which was found to be sufficient in the soil.

The available micronutrient contents showed high C.V. values (40.5 to 51.8%). The variations in available copper and iron in the soil were found to be associated with organic carbon, clay, effective C.E.C. and base saturation and clay and effective C.E.C. respectively. The variations in zinc and manganese may be perhaps due to other factors such as variations in composition of parent material, differential influence of vegetation, relief etc.

It was interesting to note that at the confidence limit of $\pm 30\%$ of the mean value with 95% probability < 13 core sample were required to constitute a composite sample representing unit area of 1 ha for the analyses of available zinc, copper, manganese and iron.

The pH was the only soil parameter which presented the lowest C.V. and thus required only one representative core sample for its determination. This finding was in conformity with the findings of Catani (1954), Barreto et al. (1974), Alvarez & Carraro (1976) and Teixeira et al. (1984). On the other hand, the maximum C.V. was found in the case of K⁺ which required 69 core sample for its determination. Highest values of C.V. for K⁺ among the various soil parameters studied were also reported for a soil having 32% slope by Barreto et al. (1974), for two soils studied by Catani et al. (1954) and for a soil under pasture for 5 years whether on slope or level ground by Teixeira et al. (1984).

For other macronutrients such as N and P and organic carbon as an index of nitrogen availability, the C.V. was found to vary from 24.7 to 32.4% and the confidence limit used in the study did not require > 5 core samples for constituting a composite sample for their analyses. In respect of the components of effective C.E.C., barring K⁺, the C.V. varied between 21.0 and 53.5% with its value for Ca²⁺ and Mg²⁺ being > 50%. Such high values of C.V. for Ca²⁺ and Mg²⁺ have also been reported by Teixeira et al. (1984) for a soil under pasture for 5 years. Taking this fact into consideration, determinations of all the components of effective C.E.C. would require a composite sample constituted from a minimum of 13 core samples.

TABLE 4. Minimum number of core samples (n) required to make a composite sample for confidence limit of $\pm 30\%$ of mean value (\bar{x}) and other statistical parameters of the soil separates.

Soil Separate	Minimum	Maximum	\bar{x}	Standard Deviation	C.V. (%)	n
Sand (%)	14	79	63.6	11.2	17.6	1
Silt (%)	16	83	28.0	10.5	37.6	6
Clay (%)	3	14	8.4	2.2	26.2	3

Soil texture could be defined from the analysis of soil separates of a composite sample prepared from a minimum of 6 core samples as, in the case of silt, C.V. (37.6%) was much higher than that for sand or clay. The comparative high variability of silt contents may have been introduced partly due to its migration by periodic flooding and microrelief characteristics of the soil.

In general, it was concluded that for the soil studied under the given field condition, estimation of available micronutrients and macronutrients and other physico-chemical properties at a confidence interval of $\pm 30\%$ of mean value with 95% probability required 15 core samples to prepare a composite sample to be representative of the unit area of sampling, i.e. 1 ha, used in the study. The only exception to this was found to be exchangeable K^+ which requires separate study in detail not only on method of sampling but also on the methodology used in determination of its availability in the soil. Further, it is suggested that this type of research should be extended to different agro-ecological conditions so that more judicious evaluation of soil fertility was possible.

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