

Research Articles

X-RAY FLUORESCENCE SPECTRA OF ROOT (WILT) AFFECTED COCONUT PALM

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ABSTRACT

Trace element composition of soil, root and leaf samples collected from healthy, and root (wilt) diseased coconut palms were determined employing energy dispersive X-ray fluorescence technique. Non-nutrient trace elements such as Ti, Cr, Ni, Pb, Br, Rb, Sr and Zr were detected in soil samples. Sandy soil in the Kayangulam area is rich in Ti. Strontium content of laterite soils of Pilicode is less as compared to similar soils in the diseased tract. Root samples contained all these elements except Cr and Zr in detectable concentrations. Nickel content of roots from diseased palms was higher than that of roots from healthy palms. Low concentration of Sr in the leaves of diseased palms and relatively higher concentration of this element in the roots indicate that the translocation of Sr in diseased palms is hindered. Titanium, Cr, Pb and Zr were not detectable in coconut leaves.

INTRODUCTION

Most of the nutritional studies so far conducted in relation to the root (wilt) disease of coconut palm (*Cocos nucifera* L.) have been aimed at getting information on the role of well established plant nutrient elements in the incidence of this disease (Pillai, et al., (1981). However, none of these nutrients has been directly implicated in the incidence of the disease. The study reported herein examines mainly the non-nutrient elemental composition of soil, leaf and roots of coconut palms in relation to root (wilt) disease.

MATERIALS AND METHODS

Sampling procedure

Soil, leaf and root samples were collected from 25-45 years old 50 palms from locations representing diseased and disease-free areas of Kerala state (Table I).

Soil samples were collected from 0-30 cm depth at a distance of about 50 cm from the base of each palm. The samples were air-dried and sieved through 2 mm mesh prior to analysis. Leaf and root samples were also collected from the same palms.

Table I. *Particulars of soil, leaf and root samples collected for the investigation*

Group number	Location	Soil type	Number of palms sampled*	Condition of the palms
<i>Diseased area</i>				
1 a	Kayangulam	Sandy	5	Diseased
b	Kayangulam	Sandy	5	Apparently healthy
2 a	Kayangulam	Sandy	5	Diseased
b	Kayangulam	Sandy	5	Apparently healthy
3 a	Kumarakom	Clayey (marshy)	5	Diseased
b	Kumarakom	Clayey (marshy)	5	Apparently healthy
4 a	Kottayam	Laterite	5	Diseased
b	Kottayam	Laterite	5	Apparently healthy
<i>Disease-free area</i>				
5	Mannuthy	Laterite	5	Healthy
6	Pilicode	Laterite	5	Healthy

* Denotes number of samples each for root, leaf and soil.

Note: Diseased palms showed severe foliar yellowing, necrosis, flaccidity and ribbing of the laminae. Apparently healthy palms were healthy palms growing side by side with diseased palms in the same garden with practically no visual foliar symptoms of the root (wilt) disease.

Leaf samples were collected from the 14th lamina (Chapman, 1964). The leaflets were wiped with distilled water-soaked cloth, dried at 70-75°C in an oven and then ground in a mill with stainless steel blades.

Root samples were collected by exposing the soil near the base of the palm and chopping vertically at the rooting region of the bole. No distinction was made of the type and length of the roots sampled. The collected roots were first washed in running tap water to remove the adhering soil particles, then with 0.1 N HCl and finally with distilled water. The HCl treatment was given to get the surfaces of the roots especially those collected from laterite area, free of Fe

oxides (and other oxides) as far as possible. The samples were subsequently oven dried and powdered as done for leaf.

Spectral and total elemental analysis

The trace element analysis of the soil, leaf and root samples was carried out by employing energy dispersive X-ray fluorescence technique (EDXRF) making use of an indigenously built Si (Li) X-ray spectrometer (Lal and Kapoor, 1972) and a low power X-ray for the excitation of fluorescent X-rays (Kataria, Govil and Lal, 1980) at the Nuclear Physics Division of the Bhabha Atomic Research Centre, Trombay. The spectrometer has a resolution of about 200 eV for 5.9 KeV Mn K α X-rays and a

good performance upto an input count rate of about 20,000 cps (Lal, Bhatia and Kataria, 1979). The X-ray tube is of a transmission type with a Mo target and a thin Mo exit window.

Individual samples were thoroughly ground again in agate mortar and pestle and mixed homogeneously with fine cellulose powder. For leaf and root samples, a 1:1 sample to cellulose (w/w) ratio was adopted while soil samples were mixed with 90% cellulose by weight. The mixture was then pressed to form a pellet of typically 40 mg/cm² thickness and subjected to spectral and total elemental analysis.

The X-ray fluorescence spectra were recorded on a 4-K multichannel analyser and the complete analysis of the spectra was done using the on-line data processor coupled to the analyser.

For quantitative elemental analysis, calibration curves were prepared from the analysis of pure element standards-cellulose based pellets. The fluorescent peak area in the spectrum corresponding to each element was then converted to its concentration in ppm.

As the samples were diluted by cellulose and were not too thick, the matrix enhancement effects due to multiple scattering of exciting primary beam and the fluorescent X-rays were assumed to be negligible. However, correction for the matrix absorption of the exciting and fluorescent X-rays was found to be necessary for all samples. The absorption correction (A) was obtained from the following equation (Kataria et al., 1977):

$$A = \frac{1 - \exp[-(b_0 \operatorname{cosec} a_1 + b_1 \operatorname{cosec} a_2)m]}{(b_0 \operatorname{cosec} a_1 + b_1 \operatorname{cosec} a_2)m}$$

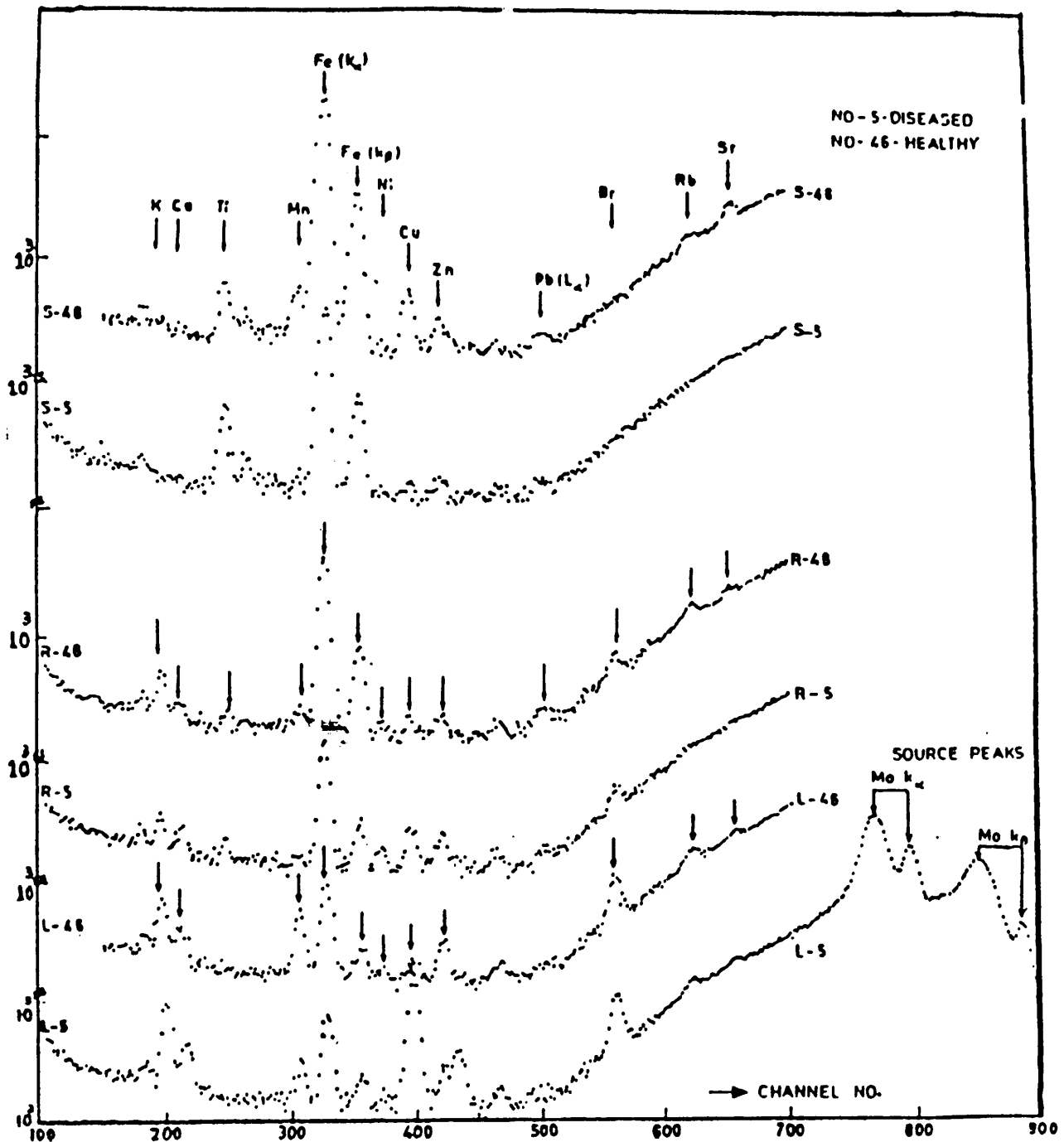
Where b_0 and b_1 are the total mass absorption coefficients for the primary beam and the fluorescent X-radiations respectively, a_1 and a_2 are the respective angles with the perpendicular to the sample and m is the mass thickness of the sample.

For leaf and root samples, it was assumed that the cellulose formed the whole matrix as these samples are mainly composed of organic substances with traces of metals. Hence, for these two types of samples, absorption correction in cellulose alone was computed. Preliminary tests also confirmed the validity of this assumption as the test results agreed with the standard values with 10% deviations. In the case of soil samples, however, it was found necessary to compute the absorption correction in a matrix of 90% cellulose and 10% SiO₂ plus Fe content of the sample.

RESULTS AND DISCUSSION

Typical X-ray fluorescence spectra of soil, leaf and root samples from palms growing in the diseased tract (Kayangulam) and disease-free area (Pilicode) are presented in Fig. 1. The presence of the fluorescent peak of an element in the spectrum depends on its presence and concentration in the sample. The minimum detection limits for various elements are furnished in Fig. 2. Thus for soil samples, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Br, Rb, Sr and Zr (atomic numbers in the range of 19-40) could be detected and quantitated based on the fluorescent K X-rays and

FIG. 1. X-RAY FLUORESCENCE SPECTRA OF SOILS, ROOTS AND LEAVES OF HEALTHY AND ROOT (WILT) AFFECTED PALMS



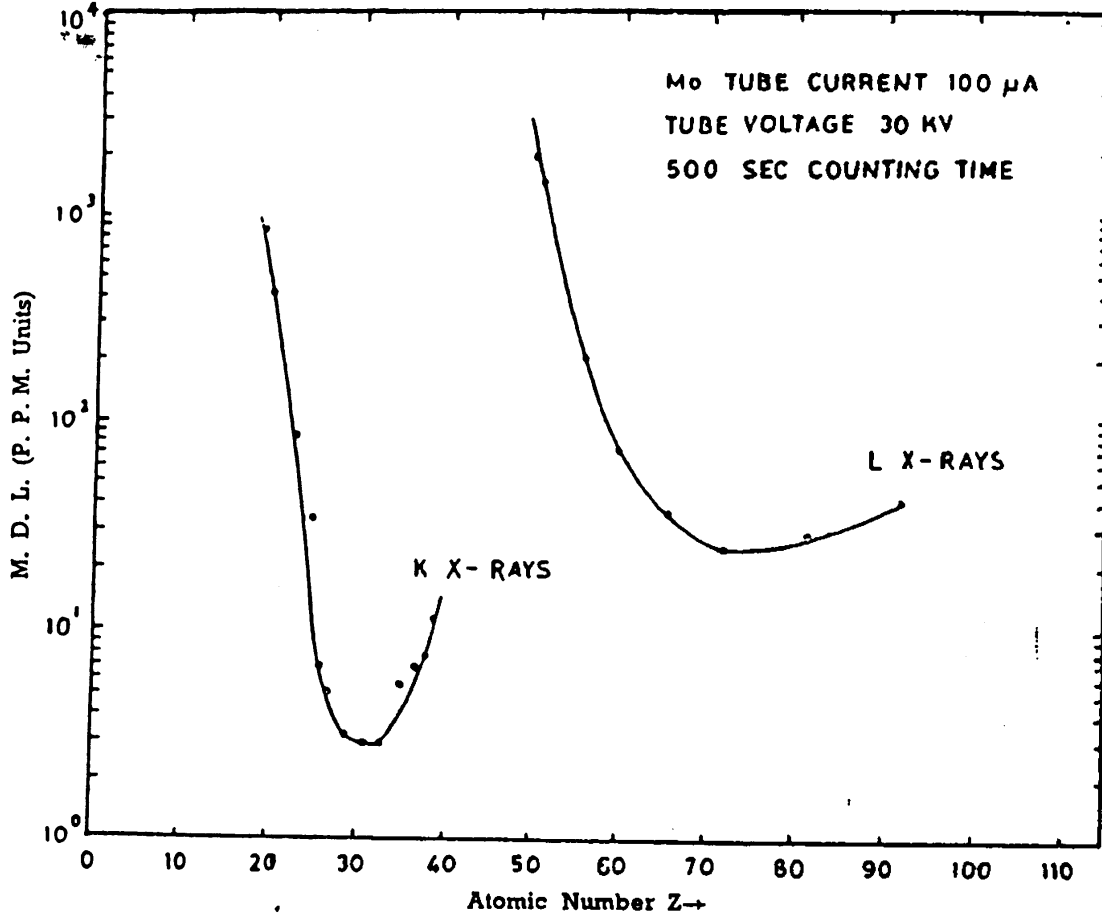
Note : S - Soil, R - Root, L - Leaf

Pb ($Z=82$) based on L X-rays. Quantitative analyses of K and Ca were not carried out as there is a great deal of information already available on these elements with reference to root (wilt) disease (Pillai et al., 1981). Therefore, the data are furnished only for trace metals (Table II). It may also be

pointed out here that no attempt was made to determine the available fraction of these elements in soil.

Kayangulam sandy soil is rich in Ti as compared to laterite and Kumarakom (marshy area) clayey soils. On an average, the Ti content of sandy

FIG. 2. MINIMUM DETECTION LIMITS (M. D. L.) OF ELEMENTS IN RELATION TO THEIR ATOMIC NUMBER



soils is of the order of 2.4 per cent while it is less than 1 per cent in laterite and Kumarakom soils. Almost a reverse trend was observed in the case of Cr. Laterite and Kumarakom soils contained comparatively more Cr than sandy soils. Not much difference was seen in the total Mn content of the soils. However, laterite soil at Mannuthy had the highest value for Mn (above 500 ppm) as against a value of less than 100 ppm for other soils. The Fe content of laterite soil was in the range of 3-5.8 per cent while it was around 2 per cent in Kumarakom soil and less

than 1.2 per cent in sandy soils of Kayangulam area. Nickel was detected in a few soils notably in laterite soils. Similarly, Br was detected only in a limited number of soils. Not much variations were observed in the concentrations of Cu, Zn and Pb among soils while Rb and Sr contents were relatively more in laterite and Kumarakom soils. On the contrary, Zr content of sandy soils was higher than that of laterite and Kumarakom soils. Apart from these differences due to soil type, no clear-cut pattern was evident in the distribution of these elements in

Table II. Elemental composition (ppm) of coconut soil basins in the diseased and disease-free regions of Kerala

Group number*	Ti	Cr	Mn	Fe	Ni	Cu	Zn	Pb	Br	Rb	Sr	Zr
1 a	13950 (67.0)	90 (14.3)	80 (5.9)	8970 (29.7)	ND	22 (3.3)	27 (4.7)	95 (8.9)	ND	ND	ND	120 (7.7)
b	15255 (68.7)	160 (14.7)	45 (6.7)	8850 (31.9)	ND	20 (3.2)	25 (3.2)	18 (6.3)	ND	11 (3.2)	ND	190 (9.2)
2 a	39795 (126.0)	340 (18.7)	32 (6.7)	12060 (62.3)	10 (3.8)	38 (3.8)	50 (4.5)	108 (8.2)	ND	10 (4.5)	15 (5.9)	273 (10.5)
b	28630 (67.0)	130 (12.8)	25 (5.4)	8060 (27.4)	ND	25 (3.4)	25 (2.6)	60 (6.3)	ND	9 (4.1)	15 (3.3)	310 (9.8)
3 a	5175 (11.6)	320 (10.9)	45 (7.7)	21140 (60.6)	ND	16 (2.6)	25 (2.3)	50 (7.4)	15 (4.5)	48 (3.2)	190 (5.5)	85 (4.5)
b	5210 (33.0)	455 (12.4)	55 (5.4)	22720 (91.8)	ND	30 (3.2)	33 (2.6)	73 (5.8)	ND	52 (4.7)	193 (5.6)	87 (6.7)
4 a	7030 (39.3)	525 (13.9)	55 (7.7)	44340 (85.6)	10 (4.5)	35 (5.0)	60 (3.8)	50 (7.1)	10 (4.5)	27 (5.4)	75 (4.5)	80 (8.2)
b	8380 (40.9)	550 (17.2)	75 (8.4)	55160 (84.6)	ND	28 (2.8)	45 (4.2)	130 (8.7)	ND	12 (4.6)	58 (4.2)	97 (6.0)
5	5430 (21.2)	180 (11.6)	580 (13.4)	30865 (48.4)	17 (2.4)	65 (7.7)	40 (3.9)	95 (7.7)	ND	36 (5.3)	220 (5.5)	142 (6.9)
6	9445 (32.9)	440 (19.1)	60 (7.7)	58230 (38.0)	5 (3.2)	35 (4.2)	19 (2.0)	110 (6.7)	17 (2.4)	18 (3.2)	16 (4.9)	153 (10.6)

* Please see Table I for details

Note : a. Diseased palm. b. Apparently healthy palm

Group numbers 5 and 6 correspond to healthy palms in the disease-free area.

Values are means of 5 analyses

Figures in parentheses denote S. D. of the mean.

ND = Not detectable

relation to disease incidence. The elemental composition of Kumarakom soil is quite similar to laterite. In the present study, Mannuthy is considered as a disease-free area although recent reports show the presence of localised pockets of diseased palms in the nearby areas (Anonymous, 1981). In view of the striking dissimilarities of the physical and chemical characteristics of the soils studied, comparison of their chemical composition in relation to disease incidence may be made within the same soil type between the diseased and disease-free areas. The sandy soils of Kayangulam area are derived mainly from the coastal alluvial deposits of the Quilon-Alleppey tract which is rich in minerals like ilmenite, monosite, zircon etc. Soils of similar nature are however absent in the disease-free areas (northern parts) of Kerala. Such an approach would, therefore, restrict the comparison to only laterite soils which are widely distributed in the mid upland regions of the state. Thus comparison of laterite of soils of the two regions shows lowest content of Sr and Zn in the soils of Pilicode area.

High concentrations of Ti and Fe were noticed in the roots compared to other elements (Table III). As the soils are rich in these two elements, it is possible that the presence of their respective oxides as coating on the root surface would lead to over-estimation of their concentration in the sample. Perhaps, this would explain, in part, the high content of Ti and Fe in roots. Chromium and Zr were not detected in the root samples. The concentration of Mn was higher in roots of the palms growing in sandy soil than that in

laterite soil. Nickel and Cu contents of the diseased roots were found to be comparatively higher than healthy roots. The healthy roots on an average contained less than 5 ppm Ni. Not much differences were observed in the concentrations of Zn, Pb and Br in the roots of the palms. Strontium was present in the roots of all palms at concentration of about 10 ppm. A notable exception was, however, the Pilicode samples in which Sr was not detected. Rubidium was detected only in a few samples.

In general, the concentrations of the metals in the leaf were less than in roots. Titanium, Cr and Pb were not detectable in the leaves of the palms. Nickel was detected only in two cases (Table IV). No regular pattern was discernible in the foliar levels of Fe, Cu, Zn, Br and Rb in relation to the disease. However, it was found that foliar Mn levels were lower in apparently healthy palms than in the diseased palms. The leaf samples collected from Kayangulam (location 1) showed abnormally high concentrations of Cu, probably due to the Cu fungicide spray on these palms. The leaf samples collected from Kayangulam (location 1) showed abnormally high concentrations of Cu, probably due to the Cu fungicide spray on these palms. Strontium content of the leaves of the diseased palms was found to be generally low compared to that of healthy or apparently healthy palm. On the contrary, roots of the diseased palms accumulated more Sr. It was also found that foliar Sr level was negatively correlated with Sr content of roots ($r = -0.63$) indicating that the translocation of Sr from the roots to top is hindered in the diseased palms.

X-ray fluorescence spectra of root (wilt) affected coconut palm

Table III. Elemental composition (ppm) of coconut roots in the diseased and disease-free regions of Kerala

Group Number*	Ti	Mn	Fe	Ni	Cu	Zn	Pb	Br	Rb	Sr
1a	498 (20.2)	17 (4.1)	573 (10.7)	8 (1.7)	28 (4.7)	15 (2.2)	13 (3.9)	11 (2.2)	ND	10 (2.6)
b	534 (16.0)	66 (11.4)	380 (16.1)	5 (3.2)	25 (5.9)	10 (2.4)	15 (3.5)	13 (2.4)	ND	10 (3.3)
2a	3310 (64.1)	78 (12.5)	1030 (32.1)	13 (4.4)	20 (5.6)	39 (7.7)	74 (11.4)	20 (4.5)	16 (4.1)	26 (5.5)
b	602 (25.2)	ND	351 (14.9)	5 (2.2)	ND	7 (2.4)	ND	14 (2.0)	ND	12 (3.2)
3a	320 (17.7)	ND	870 (18.7)	5 (1.4)	10 (2.8)	9 (2.2)	8 (2.8)	21 (3.3)	ND	13 (2.6)
b	112 (8.5)	12 (3.7)	825 (18.8)	5 (1.0)	5 (2.0)	8 (1.7)	ND	22 (2.8)	ND	10 ^f (2.8)
4a	164 (9.9)	6 (2.8)	723 (15.6)	12 (4.0)	5 (2.0)	11 (1.4)	17 (4.0)	10 (2.4)	14 (2.4)	12 (2.4)
b	140 (10.0)	12 (3.9)	997 (16.4)	5 (1.7)	ND	8 (1.0)	10 (3.0)	16 (2.2)	11 (2.2)	7 (2.2)
5	260 (10.2)	20 (3.7)	1087 (21.4)	ND	ND	7 (1.0)	20 (4.7)	11 (2.2)	25 (2.8)	12 (1.7)
6	235 (6.3)	21 (3.7)	1495 (17.5)	ND	ND	11 (2.0)	11 (3.5)	16 (2.2)	ND	ND

* Please see Table I for details

Note: a = Diseased palm; b = Apparently healthy palm; Group number 5 and 6 correspond to healthy palms in the disease-free area

Values are means of 5 analyses

Figures in parantheses denote S. D. of the mean

ND = Not detectable

Table IV. *Foliar elemental composition (ppm) of coconut palms in the diseased and disease-free areas of Kerala*

Group number*	Mn	Fe	Ni	Cu	Zn	Br	Rb	Sr
1 a	120 (6.3)	155 (8.0)	ND	60 (8.4)	9 (2.8)	33 (3.2)	11 (2.2)	8 (2.2)
b	58 (6.7)	70 (6.9)	9 (4.1)	42 (7.1)	4 (2.0)	15 (2.8)	ND	15 (2.6)
2 a	90 (7.3)	123 (7.3)	ND	5 (1.4)	10 (2.2)	32 (2.4)	ND	8 (2.0)
b	95 (5.9)	130 (7.4)	ND	5 (1.0)	10 (1.4)	27 (2.4)	ND	12 (2.4)
3 a	85 (5.1)	112 (5.8)	ND	7 (1.7)	8 (1.4)	40 (3.2)	7 (2.2)	10 (2.2)
b	26 (6.0)	110 (5.0)	ND	6 (1.7)	10 (1.7)	37 (3.2)	10 (2.2)	16 (2.0)
4 a	172 (13.5)	102 (6.5)	ND	5 (1.4)	12 (1.7)	15 (2.2)	24 (3.2)	11 (3.7)
b	46 (6.3)	92 (4.2)	6 (2.6)	6 (1.4)	14 (2.2)	14 (2.0)	15 (2.6)	21 (2.2)
5	188 (6.9)	120 (5.4)	ND	5 (1.0)	16 (2.2)	33 (2.4)	18 (3.2)	16 (2.4)
6	248 (8.2)	96 (4.7)	ND	7 (1.7)	12 (2.0)	38 (2.8)	ND	16 (2.6)

* Please see Table I for details

Note: a — Diseased palm; b — Apparently healthy palm; Group number 5 and 6 correspond to healthy palms in the disease-free area

Values are means of 5 analyses

ND — Not detectable

Figures in parentheses denote S. D. of the mean

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