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Use of a Cation Exchange Resin for Determination of Plant Available Micro Nutrients in Upland Soils of Sri Lanka

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ABSTRACT. The efficiency of the cation exchange resin (CER), Duolite 255-NH₄⁺, in uptake of micro nutrient cations such as Fe^{3+} , Mn^{2+} , Cu^{2+} and Zn^{3+} and the possibility of recovering them into 1M NH₄Cl solution by a shaking method was initially investigated. Standard solutions of the respective cations were used to evaluate the efficacy of the method. The results showed that Fe^{3+} , Mn^{2+} and Zn^{2+} uptake by CER from the solution was almost 100% and that of Cu^{2+} was higher than 96%. However, the rates of recovery of cations from the resin to 1M NH₄Cl solution were 42, 42, 57 and 58 % for Fe^{3+} , Cu^{2+} , Mn^{2+} and Zn^{2+} , respectively. Due to low recovery %, there were difficulties in analysing of those cations in the 1M NH₄Cl effluent particularly when the initial quantity of cations in the resins is low.

The possibility of the use of the cation exchange resins (CER) for determination of plant available micro nutrients in upland soils of Sri Lanka was investigated using twenty two soils, distributed across the agro-ecological regions of WL_3 , IL_1 , IL_3 , IM_3 , DL_1 and DL_3 . The quantities of each cationic micro nutrient of the soils were determined using Duolite 255-NH₄⁺, and compared with DTPA extractable Fe^{3+} , Cu^{2+} , Mn^{2+} and Zn^{2+} , $EDTA/NH_4HCO_3$ extractable Zn^{2+} and 0.01M HCl extractable Zn^{2+} . The quantities of Fe^{3+} and Cu^{2+} extracted by CER (CER-Fe³⁺ and CER-Cu²⁺) from the soils were extremely low and could not be detected by effluent solution analysis. Considerable quantities of Mn^{2+} and Zn^{2+} could be extracted from soil by using CER. The CER-Mn²⁺ did not correlate with DTPA-Mn²⁺. Therefore, interpretation of CER-Mn²⁺ require further studies on soil plant relationships of Mn. Nevertheless, CER-Zn²⁺ correlated significantly with EDTA/ NH_4HCO_3-Zn²⁺ (r=0.55; p < 0.01) and 0.01 M HCl -Zn²⁺ (r=0.75; p<0.001) indicating that CER can be used for determination of plant available Zn in

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upland soils of Sri Lanka. The method has to be further improved by changing the soil:resin ratio and modifying CER elution technique to increase the efficiency of recovery.

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INTRODUCTION

One of the major purposes of testing soils for nutrients is to determine the quantities required by plants to produce optimum crop yield. Soil tests also provide indices pertaining to plant nutrient availabilities. Biological or chemical extraction procedures are been mainly used for determination of available nutrients in soil. However, chemical methods are generally considered as cheaper and quicker. Among numerous chemical methods suggested for available plant nutrient extractions in soils, use of multi element extractants are becoming more attractive. A simultaneous determination of a range of nutrients is more economical in routine analysis for advisory purposes.

Cation exchange resins (CER) have been successfully used as multi element extractants in determination of cationic soil nutrients in many countries (Van Raij *et al.*, 1986; Wimaladasa and Sinclair, 1988; Somasiri, 1991). Hence, the prime objective of this study was to evaluate the applicability of a CER for the determination of availability of micro nutrient cations in some upland soils of Sri Lanka.

MATERIALS AND METHODS

Evaluation of uptake efficiency of iron, copper, manganese and zinc by the cation exchange resin from solutions

Six standard solutions with different concentrations of Fe^{3+} , Cu^{2+} , Mn²⁺ and Zn²⁺ were prepared (Table 1) using chloride salts of each element. The CER (Duolite 255–NH₄⁺) was used for the experiments. Three analytical determinations on those solutions were made viz. (i) quantities (mg) of Fe³⁺, Cu²⁺, Zn²⁺ and Mn²⁺ in initial standard solutions before treating with CER, (ii) quantities (mg) of the residual elements of (i) after shaking with the CER for 16 h (residual solution), and (iii) quantities (mg) of Fe³⁺, Cu²⁺, Zn²⁺ and Mn²⁺ in mg in 50 ml of NH₄Cl solution, after shaking with CER for 30 minutes. The amount of Fe³⁺, Cu²⁺, Zn²⁺ and Mn²⁺ extracted by the resin was calculated as the difference between (i) and (ii) above [*i.e.* (i)-(ii)].

Determination of available Fe, Zn, Cu and Mn in soils

Soil samples were collected from twenty two soil series distributed across six agro-ecological regions of Sri Lanka viz., WL₃, IL₁, IL₃, IM₃, DL₁ and DL₃. Some important soil properties of each series are given in the Table 2. Soils were air-dried, passed through a 2 mm sieve and treated with four extractants. The content of micro nutrients were determined in those extracts.

Table 1. Composition of the four micro nutrient standard solutions.

		Micro nutrient concentration (ppm)				
Fe	Zn	Cu	Mn			
16.0	16.0	16.0	16.0			
8.0	8.0	8.0	8.0			
1.0	1.0	1.0	1.0			
0.5	0.5	0.5	0.5			
0.2	0.2	0.2	0.2			
0.1	0.1	0.1	0.1			
	8.0 1.0 0.5 0.2	8.0 8.0 1.0 1.0 0.5 0.5 0.2 0.2	8.0 8.0 8.0 1.0 1.0 1.0 0.5 0.5 0.5 0.2 0.2 0.2			

Determination of micro nutrients in soils

Cation exchange resin (CER) extraction

Five grams of each soil were taken into a 250 ml bottle. Thereafter, 100 ml of distilled water and a mesh bag containing 2.88 g of the cation exchange resin (Duolite $255-NH_4^+$) was introduced into the bottle and contents were shaken for 16 h in a reciprocal shaker. The resin bags were then recovered and washed with distilled water to make them free of soil residues. To determine available micro nutrients adsorbed to the resin, the resin bag was shaken in 50 ml of NH₄Cl solution for 30 min. The solution was analysed for micro nutrients as described by Somasiri and Edwards (1992).

Soil series	pH 1:1	O.M. %		Texture		
	soil:H _: O		cmol, kg ⁻¹	Sand %	Silt %	Clay %
Boralu (BOR)	4.89	1.34	17.33	54.45	10.06	35.49
Gambura (GAM)	6.19	0.39	9.06	66.36	4.05	29.59
Warakapola (WAR)	4.93	1.47	16.56	64.49	13.08	22.43
Kuliyapitiya (KUL)	4.47	1.06	14.08	77.45	3.05	19.50
Melsiripura (MEL)	6.18	1.12	2.27	49.34	11.23	39.43
Aluthwewa (ALU)	6.29	1.10	7.75	71.62	6.01	22.37
Wariyapola (WAY)	5.99	0.93	9.43	45.32	8.05	46.63
Maho (MAH)	6.31	0.18	6.93	66.32	4.05	29.63
Kurunegala (KUR)	6.81	0.91	10.44	75.18	6.38	18.44
Ranorawa (RAN)	5.74	0.99	3.19	78.38	3.05	18.57
Kiriwana (KIR)	5.21	0.82	14.46	67.58	6.03	26.39
Andigama (AND)	5.48	0.99	2.24	63.16	9.32	27.52
Mavillu (MAV)	6.78	0.72	2.84	78.59	2.92	18.49
Weliketiya (WEL)	6.26	0.37	4.40	95.62	1.79	2.59
Welipelessa (WEP)	5.55	0.56	1.74	88.55	3.98	7.47
Ambekalle (AMB)	5.36	0.19	5.30	84.62	2.01	13.37
Rajakadaluwa (RAJ)	5.33	0.12	5.82	81.59	3.02	15.39
Palugaswewa (PGA)	5.46	0.29	2.02	88.51	3.95	7.54
Ratupasa (RAT)	5.76	0.27	2.69	93.95	3.80	2.25
Wilttawa (WIL)	5.85	0.17	1.46	81.62	2.01	16.37
Madampe (MAD)	5.43	0.80	1.56	88.63	4.56	6.81
Pallama (PAL)	5.46	0.29	2.02	88.51	3.95	7.54

Table 2.Some important chemical and physical properties of the
experimental soils used and the abbreviations.

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O.M. : Organic matter, CEC : Cation exchange capacity

Diethylene triamine penta acetic acid (DTPA) extraction

Twenty five grams of each soil were shaken with 50 ml of DTPA solution in a 250 ml polyethylene bottle for 2 h using a mechanical shaker. The extracts were filtered through Whatman No. 42 filter paper and Zn, Cu, Fe, and Mn in the filtrate were determined using a Shimadzu AA-670 atomic absorption spectrophotometer (Soltanpour and Schwab, 1977).

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Ammonium bicarbonate $(NH_4HCO_3)/ethylene$ diamine tetra acetic acid (EDTA) extractable Zn and Cu

Ten grams of each soil were shaken with 20 ml ammonium bicarbonate/EDTA extraction solution in a polyethylene container for 30 min at room temperature and filtered through Whatman No. 42 filter paper. Concentrations of Cu and Zn in the filtrate were determined by Shimadzu AA-670 atomic absorption spectrophotometer (Best *et al.*, 1985).

0.01 M HCl extractable Zn

Twenty grams of each soil were shaken with 50 ml of 0.01 M HCl solution for 5 min in a 100 ml glass bottle and filtered through Whatman No. 42 filter paper. The concentration of Zn was determined by Shimadzu AA-670 atomic absorption spectrophotometer.

RESULTS AND DISCUSSION

Efficiency of uptake of Fe3+, Cu2+, Mn2+, and Zn2+ by CER

The residual Fe³⁺, Zn²⁺ and Mn²⁺ in standard solutions after extracting with CER were extremely low and thus could not be determined by the atomic absorption spectrophotometer with a C_2H_2/O_2 flame. This implies that those three elements were efficiently extracted by the CER. Although residual Cu present in standard solutions after resin extraction was considerable, percentage of Cu sorbed by CER was always greater than 96% (Table 3) even in case of low (0.1 ppm-0.2 ppm) Cu concentrations in initial standard solution. The results suggest that Cu adsorption by the CER is efficient.

Recovery of resin sorbed micro nutrients

The quantities of micro nutrient cations recovered from CER by 50 ml of 1 M NH₄Cl solution are shown in Table 4. The results show that although the resin could extract almost the total quantity of Fe³⁺, Mn^{2+} , Cu^{2+} and Zn^{2+} from the standard solutions, it was unable to elute them completely from the resin using a 1 M NH₄Cl effluent solution.

Solution No.	Initial quantity of Cu (mg)	Residual quantity of Cu (mg)	% uptake
1	1.60	0.0062	99.61
2	0.80	0.0016	99.80
3	0.10	0.0011	98.90
4	0.05	0.0009	[.] 98.20
5	0.02	0.0008	96.00
6	0.01	0.0003	97.00

Table 3.Comparison of quantities of Cu in standards, adsorbed to
resin and in residual solution subsequent to CER treatment.

In case of high concentrations of Mn^{2+} , Zn^{2+} , Fe^{3+} and Cu^{2+} in standard solutions, the recovery of Mn^{2+} and Zn^{2+} from CER was greater than that of Fe³⁺ and Cu²⁺. When the concentration of each element in initial standard solution was about 0.5 mg l⁻¹ (standard 4), the extraction into resin followed by recovery into 1 M NH₄Cl solution was low to such an extent that the concentration of those elements in the effluent solution was very close to the detection limit by the atomic absorption spectrophotometer. For uptake studies, only 100 ml of each standard solution was used and therefore, 0.05 mg of Cu, Mn, Zn and Fe were present in the standard 4. The results indicate that if the amount of each element on the resins falls below 0.05 mg, shaking of resin bags in 50 ml 1 M NH₄Cl for 30 min is not satisfactory to recover the resin sorbed Fe³⁺, Zn²⁺, Cu²⁺ and Mn²⁺.

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Std.*	Fe/Zn/Mn uptake (mg)	Fe (mg) recovery	Zn (mg) recovery	Mn (mg) recovery	Cu (mg) uptake	Cu (mg) recovery
1	1.60	0.56	0.91	0.91	1.590	0.680
2	0.80	0.31	0.49	0.45	0.790	0.280
3	0.10	0.03	0.05	0.04	0.090	0.004
4	0.05	0.02	0.01	0.02	0.040	0.010
5	0.02	0.01	0.01	0.01	0.020	-
6	0.01	0.01	0.01	-	0.009	-

Table 4. Quantities of Fe, Zn, Mn and Cu sorbed by CER and recovered by NH₄Cl.

* Standard solution

The relationship between quantities of the elements on the CER and eluted from the resin was linear for all four micro nutrients (Figure 1). For the four elements, correlation coefficients were highly significant and therefore, the amounts eluted from the resin should be proportional to the amounts adsorbed. The gradient of the graphs (Table 5) can be used to calculate the actual quantity of element that was in the resin provided that the amount eluted is known.

Somasiri and Edwards (1992) showed that the quantities of P, Ca, Mg and K eluted from mixed cation and anion exchange resins (CER+AER) by 50 ml of 1 M NH₄Cl was proportional to the amounts of those elements in the resin prior to elution. They also suggested the need of correction factors to calculate the actual quantities of each element sorbed by resins, when (CER+AER) were used for soil testing.

However, the per cent recovery for Cu and Fe was low (42%). The per cent recovery for Mn and Zn was also less than 58%. The results indicate that when the quantity of element in the resin was low, the concentration of that element in the elute was also low. Therefore, the quantity of the element cannot be analysed by atomic absorption spectrophotometer with C_2H_2/O_2 flame. However, the use of more concentrated NH₄Cl solution to enhance the efficiency of elution is also not appropriate. High salt concentration in the effluent may cause problems in flame analysis techniques such as clogging of salts in aspirators and lowering the burner temperatures. Interference of background elements present in the NH₄Cl solution also could become a limitation when used in high concentrations.

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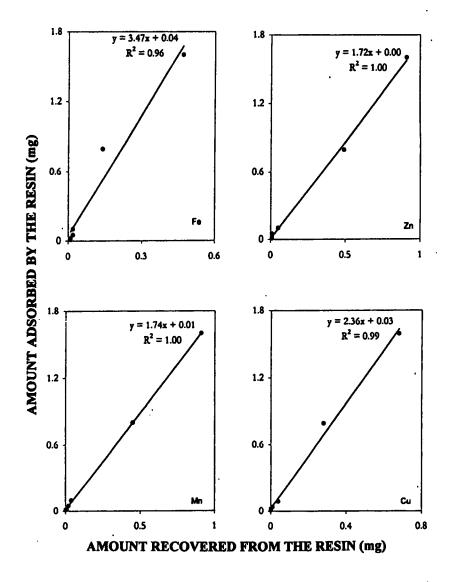


Figure 1. Relationship between amount of micro nutrients adsorbed and recovered by CER.

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_	Element	Recovery (%) by
		CER*
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	Mn	57.0
	Cu	42.0
	Fe	42.0
	Zn	58.0

Table 5. Percentage of recovery of micro nutrients from resins.

* CER : cation exchange resin

Extraction of Fe and Cu in soils by CER

The Fe and Cu in CER extracts from soils could not be detected by the atomic absorption spectrophotometer since their concentration in the elute was below the detection limit of the instrument. However, the Fe content in DTPA extraction of 22 soils ranged from 4.49 to 72.49 mg kg⁻¹. The highest Fe content was observed for Aluthwewa series and the lowest for Pallama series (Table 6). The DTPA solution could extract the chelating forms of the Fe from soils (Lindsay and Norvell, 1969). Olson (1965) reported that in most well aerated soils that were neutral, slightly acidic, or alkaline, very low exchangeable Fe was present. The failure of extracting an appreciable quantity of Fe from soil by ion exchange resins could therefore, be attributed to its low availability in Fe³⁻ form.

Copper determined by DTPA and EDTA/NH₄HCO₃ methods ranged from 0.046 to 2.45 mg kg⁻¹ and 0.19 to 6.12 mg kg⁻¹ soil, respectively. The lowest quantities of Cu determined by DTPA and EDTA/NH₄HCO₃ were for Rajakadaluwa and Weliketiya series, respectively and the highest values were for Aluthwewa and Boralu series, respectively (Table 6). According to Fiskell (1965), even in soils that received large amounts of Cu, the amounts of exchangeable Cu was quite low. This is mainly due to the complex form of Cu present in soils as shown by Cheng and Bray (1953). The results of the standard solution work of the present study (Table 3) showed that resin uptake of Cu should be at least 0.05 mg for an appreciable recovery by eluting with 1 M NH₄Cl (50 ml) for analysis. The DTPA and EDTA/NH₄HCO₃ extractions revealed that the available quantities of Cu in 5 g soils were much less than 0.05 mg. Therefore, the resin method used in this experiment was not sensitive enough to determine available Cu in soils.

In contrast to Fe and Cu, Zn and Mn were detectable in the resin elute of 50 ml of 1 M NH₄Cl (Tables 7 and 8). The quantity of DTPA extractable Zn was high for Gambura, Madampe and Andigama soil series when compared to others. However, such a trend was not observed in EDTA/ NH₄HCO₃. The CER extractable Zn was the lowest for Rajakadaluwa series and the highest for Wilaththawa series (Table 7). The DTPA extractable Mn was the highest for Warakapola series and the lowest for Rajakadaluwa series.

The correlations between DTPA and CER methods were very poor for both Zn and Mn. The correlations between EDTA/NH₄HCO₃ and CER (r=0.55; p<0.01) as well as that between 0.01 M HCl and CER (r=0.75; p<0.001) were highly significant (Table 9).

The results indicated that CER method could be used for determination of Zn instead of EDTA/NH₄HCO₃ or 0.01 M HCl. Nevertheless, the extractable quantity of Zn by CER is less than by that of EDTA/NH₄HCO₃ as well as 0.01 M HCl. The DTPA extractable Mn was also higher than resin extractable Mn.

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The EDTA/NH₄HCO₃ method may extract complexed, chelated and adsorbed forms of micro nutrients (Cu and Zn) which are considered as the most important sources of micronutrient forms for plant growth (Viets, 1962). Trieweiler and Lindsay (1969) also found that a solution of 0.01 M EDTA/ 1 M NH₄HCO₃ was an excellent extractant to determine plant available zinc in calcarious soils. The CER method showed significant correlations with standard EDTA/1M NH₄HCO₃ and 0.01 M HCl methods only for Zn. The results suggest that the application of the CER method for available Zn determination in soils is successful.

The overall results of the experiment showed that the quantities of Fe and Cu extractable from soils by the CER method was not sufficient for precise determination by the atomic absorption spectrophotometer. The quantity of Mn extracted by the CER did not correlate with DTPA extractable Mn. Only the quantity of Zn extractable by CER significantly correlated with

a 11	Cu (mg kg ⁻¹ so	oil)	Fe (mg kg ⁻¹ soil)
Soil Series*	EDTA/NH, HCO,	DTPA	DTPA
BOR	6.12	0.34	4.48
ALU	4.72	2.45	72.49
MEL	4.34	1.86	32.64
MAH	4.02	1.54	24.80
KUR	3.61	2.16	28.89
WAY	2.64	1.64	32.64
KIR	1.75	76.00	18.17
AND	1.65	0.61	25.83
KUL	1.65	0.62	28.65
RAN	1.64	0.66	25.50
RAT	1.30	0.05	17.52
GAM	0.96	0.34	26.97
WAR	0.89	0.15	21.16
MAD	0.86	0.09	60.09
WEP	0.73	0.24	26.05
AMB	0.65	0.19	46.30
PAL	0.53	0.11	4.48
WIL	0.53	0.24	21.48
MAV	0.45	0.18	5.85
PGA	0.33	0.19	16.45
RAJ	0.23	0.05	6.44
WEL	0.19	0.45	6.38

Table 6. Extracted amounts of Cu and Fe by DTPA and EDTA/NH4HCO₃ methods.

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* See Table 2 for the soil series; EDTA- ethylene diamine tetra acetic acid; DTPA- diethylene triamine penta acetic acid

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		Extractab	le Zn (mg kg ⁻¹)	
Soil Series*	DTPA	EDTA	0.01 M HCl	CER
WEL	0.07	0.21	0.07	0.77
RAN	0.13	0.19	0.16	1. 67
WEK	0.33	0.60	0.36	1.11
MEL	0.43	0.81	0.43	0.56
WAR	0.61	1.20	0.81	0.25
MAV	0.62	0.71	0.47	0.75
KUL	0.63	1.04	0.59	0.03
KIR	0.64	0.63	0.40	0.30
KUR	0.74	1.25	0.32	0.18
AMB	0.99	2.29	1.01	0.29
RAJ	1.13	1.25	0.88	0.99
MAH	2.92	2.91	1.60	1.48
PAL	3.67	5.92	2.66	1.54
PAU	5.92	1.34	0.93	0.52
WAR	6.05	3.04	4.95	1.36
RAT	6.42	0.46	0.38	0.49
ALU	6.38	5.77	5.12	2.83
WIL	6.48	0.05	0.01	0.53
BOR	7.06	0.07	7.40	2.25
GAM	16.49	0.29	0.12	0.53
MAD	26.09	0.04	0.03	0.74
AND	46.20	0.22	0.08	0.46

Table 7.	Amounts of Zn extracted by different methods from each
	soil series.

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* See Table 2 for the soil series; EDTA – ethylene diamine tetra acetic acid; DTPA- diethylene triamine penta acetic acid.

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	Extractable M	In (mg kg ⁻¹)
Soil Series*	DTPA	CER
RAJ	0.38	0.37
MAD	0.87	0.65
BOR	1.42	0.65
RAN	1.68	0.46
RAT	2.25	0.29
AND	2.55	0.87
GAM	3.10	0.96
WEK	4.37	1.93
WIL	10.53	6.23
PAL	10.80	3.35
MAV	10.98	2.21
PAL	14.69	1.19
KUL	16.05	3.99
ALU	20.85	2.71
WEL	30.98	4.63
WAR	32.93	1.09
KIR	38.54	0.84
MAH	45.68	0.95
MEL	71.85	1.18
KUR	74.03	1.07
AMB	92.49	3.40
WAR	104.54	1.14

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Table 8.Amounts of Mn extracted by 0.005 M DTPA method and
CER method from each soil.

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•See Table 2 for the soil series; DTPA - diethylene triamine penta acetic acid; CER - cation exchange resin

		Co	rrelation coefficients		
Resin Method	DI	РА	EDTA/NH,HCO3	0.01 M HCI	
	Mn	Zn	Zn	Zn	
CER	0.01	0.03	0.55**	0.75***	

Table 9.Correlation coefficients between CER and other methods
for Zn and Mn.

*** p < 0.001 ** p < 0.01; DTPA - diethylene triamine penta acetic acid; EDTA - ethylene diamine tetra acetic acid; CER - cation exchange resin

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 $EDTA/NH_4HCO_3$ extractable Zn and 0.01 M HCl extractable Zn. These observations suggest that the resin technique should be further improved to increase the extractable quantity of Fe and Cu, thus the application of CER for determination of those elements could be further evaluated.

CONCLUSIONS

The CER method used for determination of exchangeable bases in soil has very limited application in determination of available soil micro nutrients mainly due to the availability of these nutrients in minute quantities in soils. Cation exchange resins (CER) efficiently adsorb cationic micro nutrients. However, quantitative determination of the amounts adsorbed is difficult, particularly when the quantity involved is very small. A nondestructive and efficient elution technique for determination of micro nutrient cations by using a CER should be developed to overcome such difficulties.

Moreover, to increase the quantity of cationic micro nutrients that could be adsorbed by CER from resin, soil:resin ratio has to be increased. A higher soil:resin ratio would enhance the extractable quantity of each element. Nevertheless, determination of Zn by using CER is successful to the extent that the quantities correlate with the results of conventional solution extractions. The usefulness of extractable Mn by CER should be further investigated by studying soil plant relationships.

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