# **Extractability of nickel and its concentration in cultivated plants in Ni rich ultramafic soils of New Caledonia**

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# **Abstract**

The presence of higher-than-normal quantities of nickel is one of the most general features of ultramafic soils and is often suspected as the reason for their infertility. This study on the bioavailability of Ni in ultramafic soils derived from peridotites in New Caledonia showed important variations depending on the position of the soil in the landscape. In piedmont and non hydromorphic colluvio-alluvial soils, Ni was poorly absorbed by cultivated plants. In contrast, crops species grown in the plain soils, especially those found in the colluvio-alluvial and plain soils subject to temporary reducing conditions, possessed very high and even toxic Ni concentrations. Extraction of Ni by DTPA 5 mM was an effective method of estimating Ni bioavailability in these soils. The regression equation developed with only DTPA-extractable Ni explained 88% of the variability in tomato Ni concentration. Extractable Ni might originate from the association of Ni with primary alterable minerals, organic matter and goethite.

# **Introduction**

Soils derived from ultramafic rocks (ultramafic soils) have been the focus of intensive studies, especially concerning the possible causes of their infertility. Proctor and Woodell (1975) pointed out that identification of the main factor conditioning plant growth in each case was difficult, owing to the great variability of these soils.

Nickel is known to have toxic effects on biological systems, especially on plants (Foy et al., 1978), and its abundance in ultramafic soils is often cited in the literature as an important factor (Robertson, 1992; Soane and Saunder, 1959). The total Ni concentration of ultramafic soils is often greater than 3000  $\mu$ g g<sup>-1</sup> (Baker et al., 1992; Brooks, 1987) whereas the majority of soils contain less than 500  $\mu$ g g<sup>-1</sup> Ni with a mean between 20 and 40  $\mu$ g g<sup>-1</sup> (Uren, 1992).

Many plants found on ultramafic soils accumulate and tolerate high Ni concentrations (Jaffré et al., 1976; Reeves, 1992; Vergnano Gambi et al., 1982) due to physiological adaptations (Gabbrielli et al., 1990; Lee et al., 1978), which suggests that Ni might be relative-

ly bioavailable in these soils. Crops plants grown on these soils could then exhibit a reduction of growth and yield, because they are in general very sensitive to Ni (Davis and Beckett, 1978; Frank et al., 1982; Soane and Saunder, 1959).

Ultramafic soils of New Caledonia possess amongst the highest concentrations of Ni in the world (between 0.5 and 2%). Moreover, the extensive area occupied by these soils (about 1/3 of the country, or 500,000 ha) and the development of agriculture in this region present an interesting model for testing Ni phytotoxicity.

The aim of this study is therefore: (1) to determine whether the problems encountered in crop development on these soils are related to excessive Ni uptake, (2) to test the efficiency of a common single chemical extractant in evaluating the bioavailability of Ni in these soils.

# **Materials and methods**

## *Soils*

Sixty surface soils (0-20 cm) were collected in southern New Caledonia in the La Coulee and La Lembi

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*Table 2.* Chemical and physical properties of the ultramafic soils used in the greenhouse experiment

Soil	Horizon	Particle size distribution $(\%)$			pН		Exchangeable cations $\pmod{kg^{-1}}$ C - N <b>CEC</b>					Total elements $(\%)$				
	(cm)	Sand Silt		Clay		$(H_2O)$ (KCl) $(\%)$		$(\%)$ Ca		$Mg$ K		Na	$(\text{cmol kg}^{-1})$ Si Fe Ni			
Piedmont 0-20		30.8		45.0 24.1	5.1	5.7	1.83	0.09				0.15 0.21 0.13 0.13 2.5			1.0 25.8 0.76	
Piedmont	40-60	35.7	473	170	4.7	6.1		0.42 0.03		$0.02 \cdot 0.01$		0.03 0.03 1.9		0.7	25.9 0.70	
Plain	$0-20$	29.1		49.6 21.3	6.6	6.2		2.10 0.15	1.21			4.48 0.45 0.22	121	57	16.9 0.85	
Plain	$40-60$	20.9	-614	- 17.6	6.6	6.2					145 0.11 0.56 4.53 0.16 0.13		-110	6.4		17.8 0.96



1 : Piedmont.

- 2 : Colluvio-alluvial **soil.**
- 3 : Colluvio-alluvial soil with temporary **reducing conditions.**
- 4 : Plain soil with temporary **reducing conditions.**
- 5 : Plain (alluvial terrace).

#### *Figure 1, Position in the landscape of the different type of ultramafic* soils studied.

valleys (166 $\degree$ 36' E-22 $\degree$ 14'S) that are the major agricultural areas of the region. They are highly weathered oxisols (Acrorthox) derived from peridotite. Samples were classified in five different soil types (Figure 1) previously described on the basis of differences in their chemistry and physical properties (Becquer et al., 1995; Bourdon and Becquer, 1992). Soils were air dried and sieved through a 2 mm sieve. Their characteristics are given in Table 1.

DTPA, (diethylenetriaminepentaacetic acid) was used as the extractant method for studying the bioavailability of Ni (DTPA-Ni): 5  $g$  of dry soil<2 mm was mixed in a solution of 25 mL DTPA 5 mM + CaCl<sub>2</sub> 10 mM at a pH of 5.3 for a period of 1 hour (Becquer et al., 1995; after Lindsay and Norvell, 1978). After centrifuging and filtration of solids, Ni was obtained by atomic absorption spectrophotometry AAS (Varian AA300).

# *Maize experiment*

A greenhouse experiment with maize *(Zea mays* L. cv. GH 5010) was conducted using a randomized block design with four types of oxisols (piedmont and non hydromorphic plain soils, sampled at depths of 0-20 cm and 40-60 cm) and ten repetitions of each. Soils characteristics are given in Table 2. They were sieved through a 6 mm sieve and homogenised before the filling of the pots (5.1 kg of plain soil per pot; 6.2 kg of piedmont soil per pot). They received an addition of nutrients previously determined to alleviate any deficiencies (mg kg<sup>-1</sup> of soil): 140 N, 1000 P, 69 K, 37 Ca, 23 Mg, 30 S, 0.7 B, 1.1 Zn, 1.6 Cu, 0.2 Mo. After 2 days of germination at 27 °C on cotton moistened with distilled water, the most vigorous maize seedlings were transferred to the pots (one plant by pot). Plants were harvested after 28 days of growth. Water was continually supplied to the pots via the capillary action of fibreglass mesh which was fed by an external water source. The temperature in the greenhouse varied from 24 to 38 °C, the relative humidity from 90 to 50% (day/night), and the photoperiod was 13 hours.

# *Plant sampling and analysis*

Plant material (except maize) was sampled in agricultural land from adult crop plants during their fruiting period. Mature non senescent leaves were sampled for chemical analysis. All the soils described above and the plant material were collected in association: the soil was taken just beside the plant.

At harvest, plants were rinsed with demineralised water, dried at 105 °C for 24 h and weighed. For analysis, samples of dried and ground plant material were ashed and digested with concentrated HC1. After filtration, the residue was dried, weighed and treated with hydrofluoric acid to eliminate silica. This element was determined by weight loss of the residue. Cation concentrations obtained after filtration were determined in diluted solutions containing  $1\%$  HCl,  $0.2\%$  H<sub>3</sub>BO<sub>3</sub> and  $0.5\%$  La<sub>2</sub>O<sub>3</sub> by flame emission spectroscopy (K) or AAS (Ca, Mg, Fe, Mn, Ni). Phosphorus was determined colorimetrically at 420 nm according to the vanadomolybdate method (Fiske and Subbarow,

1925). Nitrogen was determined using the Kjeldahl method.

#### *Statistical analysis*

Results obtained from the greenhouse experiment were examined by analysis of variance. The significant differences between means were analysed by Student t test at the 5% confidence limit. Regression equations were performed on all data using step by step regression available on Statview version 4.02, with significance defined at the 1% confidence level.

# **Results**

The geochemical nature of the different soils is relatively homogeneous (Tables 1 and 2). However, the colluvio-alluvial and plain soils possess the highest concentrations of Ni, and exceptionally high concentrations of Si and Mg. Levels of these elements are lower in the piedmont soils, but there is a higher concentration of Fe which corresponds to greater alteration.

# *Greenhouse experiment*

The maize grown in the test soils exhibited significant differences in growth (Table 3). Maize showed a weaker root and shoot development in plain soils (0-20 cm and especially 40-60 cm: respectively 15% and 39% reduction in shoot dry weight) than in piedmont soils. No particular symptoms were observed during maize development.

Mineral analysis of maize shoots revealed significant differences for many elements between plants grown on the four soils (Table 3). However, variations in growth showed the stronger correlation with shoot Ni concentrations (r significant at 1%, after Figure 2).

Results of extraction of Ni by DTPA showed similar differences between soils (Table 5): plain soils released much more Ni than piedmont soils, but no significant differences were observed between horizons. Figure 3 shows that the Ni extracted by DTPA correlated relatively well with Ni concentrations in maize shoots.

#### *Sampling from agricultural land*

Sampling and analysis were carried out on the soils and the associated crops that are grown in the region. They were classified in terms of their position in the land-



*Figure 2.* Correlation between shoot dry weight and shoot Ni concentration, and between root dry weight and shoot Ni concentration



*Figure 3.* Correlation between Ni concentration in maize shoots and Ni extracted by DTPA. Data from the greenhouse experiment. **\*\*** Significant at 1%.

scape (Figure 1) according to Becquer et al. (1995), and Bourdon and Becquer (1992). Important differences were noted in relation to their position: plain and colluvio-alluvial soils with temporary reducing conditions in particular were distinguishable by the highest levels of leaf Ni concentrations in crop plants

Soil		Yield (mg plt <sup>-1</sup> )	Si	N	P	K	Ca	Мg	Fe	Mn	Ni
	Root Shoot			(%)				$(\mu$ g g <sup>-1</sup> $dw$ )			
Piedmont	35.1a	2.0a	1.5a	2.8a	0.22a	1.5a	0.54a	0.46a	121a	115a	4.8a
$0-20$ cm	$\pm 3.9$	$\pm 0.4$	$\pm 0.1$	$\pm 0.3$	$\pm 0.02$	$\pm 0.2$	±0.05	±0.03	±32	±9	$\pm 0.6$
Piedmont	33.0a	2.6a	0.7 <sub>b</sub>	3.0a	0.23a	1.4a	0.68a	0.29 <sub>b</sub>	103a	73 <sub>b</sub>	2.9 <sub>b</sub>
$40-60$ cm	$\pm 3.0$	$\pm 0.6$	$\pm 0.1$	$\pm 0.4$	$\pm 0.01$	$\pm 0.3$	$\pm 0.08$	$\pm 0.02$	±26	±9	$\pm 0.3$
Plain	29.8 <sub>b</sub>	1.4 <sub>b</sub>	2.0 <sub>c</sub>	3.1a	0.38 <sub>b</sub>	2.5 <sub>b</sub>	0.24 <sub>b</sub>	0.64c	134a	56c	6.6c
$0-20$ cm	$\pm 3.1$	$\pm 0.3$	$\pm 0.1$	$\pm 0.3$	$+0.05$	$\pm 0.3$	$+0.02$	$\pm 0.04$	$\pm 60$	±7	±1.6
Plain	21.5c	1.1 <sub>b</sub>	2.1c	3.4a	0.37 <sub>b</sub>	2.5 <sub>b</sub>	0.25 <sub>b</sub>	0.82d	102a	75 <sub>b</sub>	12.5d
$40-60$ cm	$\pm 2.3$	$\pm 0.3$	$\pm 0.1$	$\pm 0.2$	$\pm 0.03$	$\pm 0.3$	$\pm 0.03$	$\pm 0.07$	±50	$\pm 6$	$\pm 1.7$

*Table* 3. Dry matter yield and mineral composition of shoots of maize grown on ultramafic soils used in the greenhouse experiment. Values are means  $\pm$ SE (n=10)

Different letters in a column indicate significant difference at the 0.05 confidence level.

*Table 4.* Ni concentrations in the leaves ( $\mu$ g g<sup>-1</sup> dw) of some plants cultivated on the ultramafic soils studied. Values are means  $\pm$  SE (n)

	1. Piedmont	2. Colluvio- alluvial soil	3. Colluvio- alluvial soil with reducing conditions	4. Plain soil with reducing conditions	5. Plain soil
Banana tree				$53.5 \pm 11.5(2)$	$22.1 \pm 11.0(7)$
Carrot				$59.1 \pm 6.0(2)$	$41.0 \pm 10.1(3)$
Chinese cabbage					$48.5 \pm 10.5(2)$
Courgette		$11.6 \pm 1.2(3)$			$55.5 \pm 1.5(2)$
Eggplant		$19.4 \pm 4.6(4)$			94.0(1)
Lemon tree		$44.0 \pm 2.0(2)$	$82.1 \pm 16.3(2)$		
Mango tree	$25.0 \pm 8.6(4)$	$23.5 \pm 6.5(2)$			
Pawpaw tree		$14.5 \pm 1.5(2)$			
Radish					85.0(1)
Tomato		$13.9 \pm 4.8(14)$			$47.5 \pm 4.7(5)$

*Table 5.* Concentrations in DTPA-extractable Ni (Ni-DTPA) in the ultramafic soils used in the greenhouse experiment or sampled from the field. Values are means  $\pm$  SE (n)



grown on these types of soil, with Ni concentrations often exceeding 50  $\mu$ g g<sup>-1</sup> dw (Table 4). Despite the high levels of fertilization applied by the farmers, crop yields on these soils were much lower than their production potential (about 1.5 to 3 fold lower). Even in the absence of precise information on yields, it appears that there were no important differences between the five types of soil. Only the lemon trees (for the same age and same species *Citrus aurantifotia)* grown in colluvio-alluvial soils with reducing conditions showed chlorotic leaves and significantly lower growth and production than in colluvio-alluvial soils. Except for lemon trees, no particular symptoms were observed in the field.

The same differences as for leaf Ni concentrations between the type of soils were found at the level of DTPA-extractable Ni (Table 5).

In addition, there is a significant correlation between Ni concentrations extracted by DTPA (Ni-DTPA) from the different soils and Ni concentrations in the crop plants examined (Figure 4a). The significance of this correlation is even greater if only tomato plants or eggplants are selected  $(r = 0.95$  and 0.99; Figures 4 b,c).

Regression equations for the prediction of Ni concentration in tomato leaves could be developed (Table 7). Only equations with all variables significant at 0.01 probability are presented. DTPA-extractable Ni alone accounted for 88% of the variability in tomato Ni concentration.  $\mathbb{R}^2$  values were not significantly improved by the inclusion of other soil characteristics in the equation.

## **Discussion**

The flora of ultramafic soils has been the focus of many studies due to its originality (Brooks, 1987; Lee et al., 1977; Reeves, 1992). In contrast, there is still very little known on the effect of Ni within these soils on plant development (Baker et al., 1992; Robertson, 1992; Uren, 1992).

This study shows the existence of very important variations in Ni bioavailability in the ultramafic soils examined which relate to the position of the soil in the landscape and the soil horizon.

Results of the greenhouse experiment (Table 3) showed that Ni concentrations in maize shoots were all significantly different between the four soils (piedmont and plain soils, for the horizons 0-20 and 40-60 cm). This was also noticed in the growth on the test

plants. The high Ni concentrations recorded for maize grown in the plain soils - especially the horizon 40-60 cm - are probably connected with their poor growth, because they were similar or superior to the concentrations considered toxic in the literature (see Table 6). The strong correlation between the dry weight of roots and shoots and Ni concentrations in maize shoots stronger than all other elements - reinforce further the hypothesis (Figure 2). However, based on the mineral composition of the maize shoots (Table 3), it appears that four factors might intervene: (1) a low Ca concentration in maize grown in the plain soils. But Ca concentrations of 0.24% are not considered as deficient for maize (Jones, 1967); (2) an excess of Mg, which is often reported for ultrarnafic soils (Proctor et al., 1981); (3) a strongly imbalanced Mg/Ca ratio; (4) an excess of Ni. It is also possible that combined excess concentrations of Ni and Mg led to a decrease in the growth of maize.

Cultivated plants grown in the field (Table 4) also showed very strong variations in foliar Ni concentration depending on the position of the soil in the landscape. Moreover, leaf Ni concentrations were often very high. In general, crop plants contain less than 5  $\mu$ g g<sup>-1</sup> dw Ni in their shoots (Hutchinson and Whitby, 1974; Vanselow, 1966). Nearly all the crops sampled from the plain soils, and especially the colluvio-alluvial and plain formations subject to temporary reducing conditions, possessed Ni concentrations greater than 40  $\mu$ g g<sup>-1</sup> dw (Table 4) which are normally considered toxic for most cultivated plants (Table 6). For instance, Ni concentrations found in eggplants and lemon trees cultivated in the plain and colluvio-alluvial soils subject to temporary reducing conditions were probably toxic (compare Tables 4 and 6). This could be connected with the chlorotic leaves and the low growth and production of lemon trees in colluvio-alluvial soils with reducing conditions.

Despite the lack of information in the literature about upper critical levels of Ni for crops, it is likely that Ni plays a significant role in growth of plants on ultramafic soils. However, Ni toxicity is probably limited to certain types of oxisols particular to specific areas in the landscape: plain soils, and especially colluvio-alluvial and plain soils subject to temporary reducing conditions (Figure 1).

The very strong variability in the bioavailability of Ni in relation to the position of the soil in the landscape reinforces the need to establish a method of chemically evaluating Ni bioavailability in these soils. We therefore tested the efficiency of DTPA that is corn-



*Figure 4.* **Correlations between DTPA-extractable Ni and shoot Ni concentrations in all crop plants (a), in tomato (b), or in eggplant (c) sampled from the field on ultramafic soils. \*\* Significant at** 1%.

**monly used (Haq et al., 1980; Lindsay and Norvell, 1978; Sauerbeck and Hein, 1991). For the soils used in the greenhouse experiment, the correlation between Ni concentrations in maize shoots and Ni-DTPA was significant at 1% (r=0.74; Figure 3). However, this correlation is dangerous since figure 3 show two different data populations, corresponding to two types of soil very different: plain soils with high Ni-DTPA and piedmont soils with low Ni-DTPA. The difference in Ni uptake by maize between the surface (0-20 cm) and sub-surface (40-60 cm) horizons of plain soils was not**  **found with the DTPA extraction. The reasons for that are not clear. Nevertheless, a satisfactory correlation was obtained between Ni concentrations in all crop plants sampled in the field and Ni-DTPA (r=0.61; Figure 4a). The correlations were particularly strong for tomato plants (r=0.94) and eggplants (r=0.99; Figures 4b,c).** 

**A very satisfactory result was obtained from the regression equations, whereby Ni extraction by DTPA explained by itself 88% of the variation in Ni concentration in tomato leaves (Table 7). The multiple cor-** 

Plant	Stage	Ni concentration $(\mu g g^{-1} dw)^a$	<b>Effects</b>	Reference
Alfalfa	83 days	44 (S)	Reduced growth	Halstead et al. (1969)
<b>Barley</b>	control at 5 L	12(S)	Upper critical level	Davis and Beckett (1978)
Bean	75 days	47 $(L)$	$-60\%$ seeds weight	Piccini and Malavolta (1992)
<b>Citrus</b>	٠	40 $(L)$	Toxic	Vanselow (1966)
Eggplant	4 months	24(L)	$-16\%$ plant dw	Salim et al. (1988)
Maize	18 days	8.4(S)	$-6\%$ shoots dw	<b>Wallace (1989)</b>
	75 cm at $9th$ L.	12(S)	Upper critical level	L'Huillier(1994)
Oat	adult	42 $(L)$	Reduced growth	Hunter and Vergnano (1952)
Soya	18 days	13(L)	$-22\%$ shoots dw	<b>Wallace (1989)</b>
Wheat	23 days	25(L)	-10% leaves dw	Taylor (1988)

*Table 6.* Toxic Ni concentrations in different crop plants

~Organ analysed: L: Leaves, S: Shoots.

*Table 7.* Regression equations for the prediction of Ni concentrations in Tomato developed with DTPA-extractable Ni and other soil characteristics as independent variables  $(n=19)$ 

			Final equation				
Regression step $R^2$		Variable added	Coefficient	Standard error	F		
	0.88	Ni-DTPA (Constant: $12.3 \pm 1.5$ )	0.375	0.035	$113.2**$		

\*\* Significant at the 0.01 confidence level.

relation coefficient  $(R<sup>2</sup>)$  did not significantly improve when other soil characteristics were included into the regression equation. The data from Table 7 can be presented in equation form as follows:

Ni in Tomato ( $\mu$ g g<sup>-1</sup> dw) = 12.3 + 0.375 Ni-DTPA  $(\mu g g^{-1} \text{ soil}) (\text{R}^2 = 0.88, n = 19)$ 

DTPA is therefore a chemical extractant that by itself is a good indicator of Ni bioavailability and toxicity risks in ultramafic soils. Hughes and Noble (1991) are among the few who have tested chemical extractants in ultramafic soils. They showed that none of the ten single extractants they tested were capable of indicating Ni availability for the flora of ultramafic soils in the Eastern Transvaal. However, the correlations with the vegetation they studied probably lacked some precision and DTPA was not tested.

The regression equations for the prediction of Ni extractability by DTPA (Table 8) allowed us to highlight soil characteristics which may play a significant role in Ni extractability. The levels of CEC, pH(KC1), silica, iron and carbon in the soil explained 89% of the variation in Ni extractability by DTPA with significant probabilities at 1%. This suggests that Ni was more extractable - and probably more bioavailable - in soils with a high level of silica, iron and probably organic matter (for CEC and carbon). The negative coefficient for pH(KCI) suggests that Ni was more extractable in low pH soils. CEC in these soils mainly originates from organic matter, so this one might be a significant source of bioavailable Ni, especially in the surface horizons of these soils as already shown (Becquer et al., 1995). Silicates might constitute an important source of bioavailable Ni as they can originate from easily weatherable primary minerals (rich in Ni and Si), which can be transported by alluviation and colluviation in colluvio-alluvial and plain soils. Nickel has also been shown to occur in association with iron oxides in mafic soils (Schwertmann and Latham, 1986; Singh and Gilkes, 1992; Uren, 1992) which may represent another significant source of Ni for plants, in particular goethite which is more abundant in plain oxisols (Schwertmann and Latham, 1986). These results are in accordance with Jenne (1968) who showed the important effect of hydrous Fe oxides on Ni availability and emphasised the crucial role of redox potential and pH in determining the availability of these hydrous oxides.

In conclusion, our results show that Ni bioavailability in ultramafic soils is very variable. The levels are low in non hydromorphic colluvio-alluvial and piedmont soils, as previously observed by Angeione et al.

			Final equation				
Regression step	$R^2$	Variable added	Coefficient	Standard error	F		
	0.76	CEC	4.8	1.2	$16.0**$		
$\mathbf{2}$	0.81	pH(KCl)	$-36.2$	10.5	$11.8***$		
3	0.85	Si	31.1	5.6	$30.6***$		
4	0.87	Fe	11.6	2.9	$15.8***$		
	0.89	C	2.3	0.9	$5.6***$		
(Constant: $-126.2 \pm 106.4$ )							

*Table 8.* Regression equations for the prediction of DTPA-extractable Ni developed with soil characteristics as independent variables  $(n=60)$ 

\*\* Significant at the 0.01 confidence level.

(1993) on ultramafic soils. In contrast, Ni bioavailability is high in plain soils and especially in colluvioalluvial and plain soils subject to temporary reducing conditions. Crops cultivated on these soils probably suffer from an excess in Ni uptake. Extraction by DTPA gives a good estimation of Ni bioavailability in ultramafic soils.

The regulations concerning the maximum concentrations of Ni allowed in agricultural soils treated with sewage sludge (30 to 75 mg kg<sup>-1</sup> of dry soil in the EEC; McGrath, 1993) are not practically adaptable to ultramafic soils. It would probably be desirable to establish different limits based on the types of soil, or to take into account the bioavailability of Ni in the soil assessed by a chemical extractant.

Complementary studies are necessary to determine the applicability of DTPA for other soils and also to define the upper critical levels of Ni for many crop species. This would enable a greater understanding of the toxicity risks on ultramafic soils and soils which are in general rich in Ni.

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