Research Article
Investigation of Chemical Fraction of Nickel in Maize and Soil Using the Cold Extraction Technique

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Abstract: An investigation of chemical fraction of Nickel in maize and soil samples was conducted. The objective of the study was to determine the concentration of Nickel in maize and soil samples by Flame Atomic Absorption Spectrometry (FAAS) using Cold Extraction Technique. The results indicated that the soil samples collected from various locations contain varying amounts of Nickel and were distributed between residual, oxide, Fe and Mn oxide and carbonate/organically bound phases. The result of the study also showed that in some of the sampling locations, the Nickel concentration is above the tolerance limit value of 50 mg/kg and the ANOVA (p = 0.000<0.05) indicated a significant difference in the nickel concentrations across the various maize crops. Similarly, the ANOVA (p = 0.000<0.05) also showed a significant difference in the nickel concentrations across the various maize grown soils.

Keywords: AAS, cold extraction, maize, nickel, soil

INTRODUCTION

Nickel and nickel compounds have many industrial and commercial uses and the progress of industrialization has led to increased emission of pollutants into ecosystems. Although Nickel is vital for the function of many organisms, concentrations in some areas from both anthropogenic release and naturally varying levels may be toxic to living organisms (Diaggomanolin *et al.*, 2004). Inhalation exposure in occupational settings is a primary route for Nickel induced toxicity and may cause toxic effects in the respiratory tract and immune system (Bennett, 1982). The general exposure to Nickel mainly concerned oral intake, primarily through waste and food, as a contaminant in drinking water or as both a constituent and contaminant of food (Haber *et al.*, 2000).

Nickel is generally distributed uniformly through the soil profile but typically accumulates the surface from disposition by industrial and agricultural activities. Nickel may present a major problem in land near towns, in industrial areas or even in agricultural land receiving waste such as sewage sludge. Nickel can exist in soils in several forms such as inorganic crystalline minerals or precipitates, complexes and absorbed on organic cat ions surface or on inorganic cat ions exchange surfaces, water-soluble and free-ion or chalated metal complexes in soil solution (Bennett, 1982).

The total heavy metal content in soils provide a convenient means of expressing a measure of pollution, numerous reports have highlighted that such measures are deficient in predicting toxicity of metal pollutants (Yusuf, 2006). Heavy metals may be distributed among many components of the soil or sediment and may be associated with them in different ways (Harrison *et al.*, 1981; Chlopecka *et al.*, 1996; Kabala and Singh, 2001; Khairah *et al.*, 2009). The nature of the association is referred to as speciation. The general approach for the soil speciation studies has been to separate the soil using different chemical reagents or solvents fractions and by analyzing each fraction to determine the amount of element combined or associated with each soil fraction or phase (Yaman *et al.*, 2000).

Therefore, the identification of the chemical form or phases of Nickel in soil is necessary for estimating its biological availability, physico-chemical reactivity and transport in the environment and into the food chain (Yaman and Yusuf, 2002).

In this research, Nickel concentrations in maize and soil samples were determined by Flame Atomic Absorption Spectrometry (FAAS) using Cold Extraction Technique. The soil samples were extracted using chemical reagents such as the mixture of HNO_3/H_2O_2, oxalic acid, Na_2EDTA, citric acid and acetic acid. The relation between the maize Ni contents and the soil extracts was investigated.

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MATERIALS AND METHODS

The soil samples were dissolved by using the chemical reagents such as mixture of HNO_3/H_2O_2, $(COOH)_2$, Na_2EDTA, $C_6H_8O_7$ and CH_3COOH. The relation between the maize contents and the Ni contents of soil extracts was also investigated. In the digestion and extraction procedures, concentrated HNO_3, H_2O_2, 1.0 M $(COOH)_2$, 0.05 MNa_2EDTA, 1.0 M CH_3COOH and Citric acid $C_6H_8O_7$ were used. Stock solution of Ni (1000 mg/L) was prepared by dissolving $Ni (NO_3)_2$ in 1.0 mol/L nitric acid.

Preparation of samples: The study covered seven agricultural sites in Kaduna, Nigeria (Fig. 1). The sites are: Nasarawa (NS), Sabon Tasha (ST), Unguwar Mu'azu (UM), Tudun Wada (TW), Kakuri (KK), Mando (MD) and Kabala west (KB). The samples were collected during the harvest season (Oct-Nov., 2008, 2009 and 2010). The soil samples were collected from different areas enumerated at the depth of 10 cm below the surface (Yaman *et al.*, 2005). The maize samples were collected at each of the locations. Kachia, a town situated about 130 km away from Kaduna, was taken as control. The sample was washed with water and was allowed to dry on filter papers. Both samples were dried at 85°C. All the analyses were carried out in the analytical laboratory of the Department of Applied Science, College of Science and Technology, Kaduna Polytechnic, Kaduna-Nigeria.
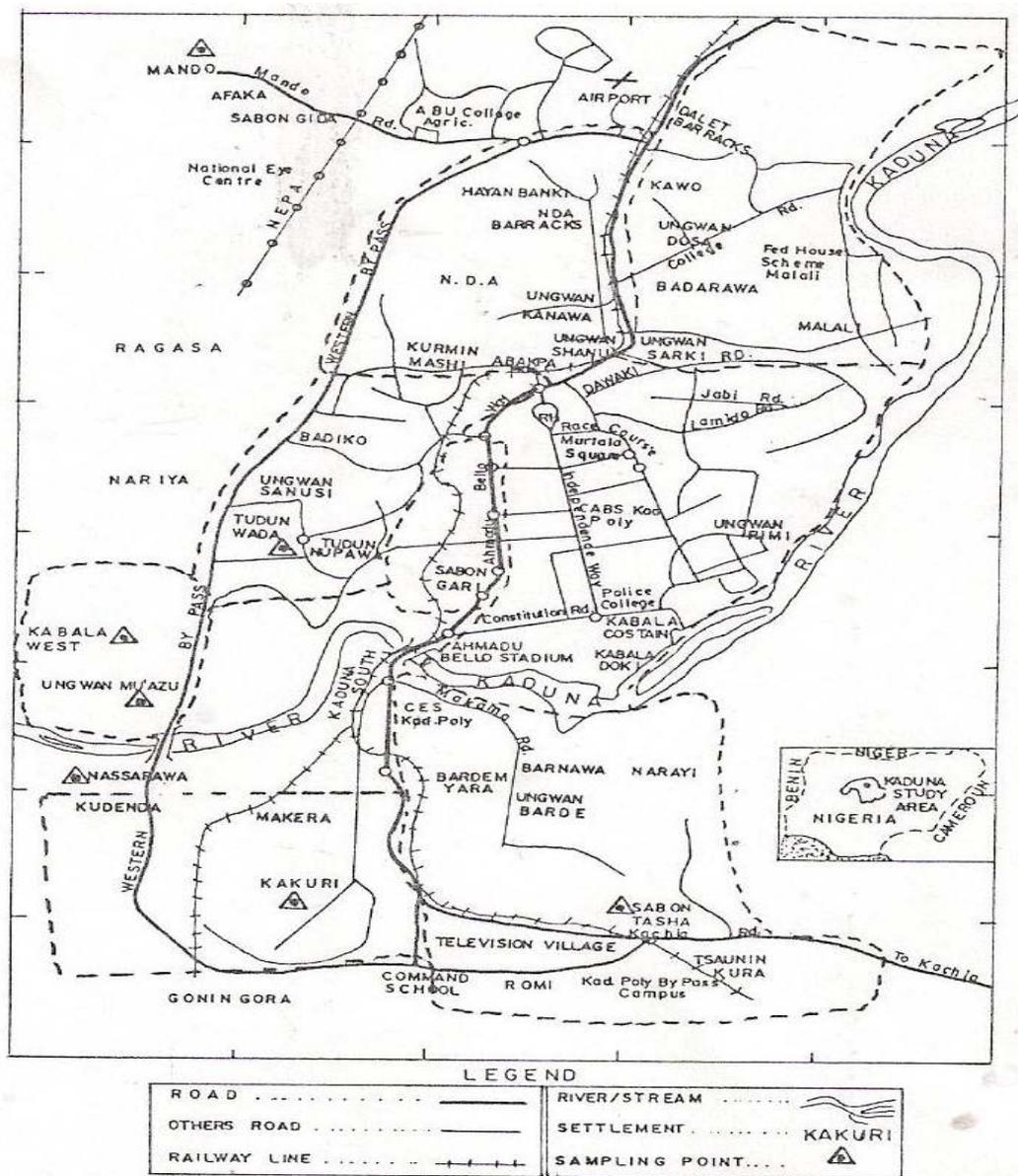
Fig. 1: Map of Kaduna metropolis

Table 1: Results of Ni concentrations in maize and soil samples at Kabala

| | | | | Cold extraction | | | |
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
KB_1	5.16	5.850±2.90	5.850±2.9	17.56±2.9	44.88±1.7	46.83±2.9	11.71±5.9
KB_2	5.46	36.10±17.8	14.63±2.9	5.850±2.9	8.780±2.9	17.56±2.9	8.780±2.9
KB_3	5.36	58.54±29.3	17.56±2.9	5.850±2.9	11.71±5.9	20.49±2.9	8.780±5.9
KB_4	5.26	5.850±2.90	8.780±2.9	20.49±2.9	43.90±5.9	46.83±5.9	11.71±2.9
KB_5	5.66	29.27±2.90	17.56±2.9	8.780±5.9	11.71±2.9	20.49±5.9	11.71±2.9
KB_6	6.12	35.12±2.90	20.49±5.9	11.71±5.9	14.63±5.9	23.41±5.9	14.63±2.9
KB_7	6.12	32.20±2.90	20.49±5.9	11.71±2.9	11.71±2.9	23.41±5.9	11.71±2.9
KB_8	6.12	38.05±2.90	22.44±6.1	11.71±2.9	17.56±2.9	23.41±5.9	17.56±5.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 2: Results of Ni concentrations in maize and soil samples at Nasarawa

| | | | | Cold extraction | | | |
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2(2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
NS_1	5.49	11.71±5.9	11.71±2.9	8.780±2.9	81.95±2.9	30.24±4.5	23.41±2.9
NS_2	6.12	29.22±2.5	11.71±2.9	20.49±2.9	11.71±5.9	20.49±2.9	14.63±5.9
NS_3	5.33	43.90±2.9	14.63±5.9	23.41±5.9	8.780±5.9	20.49±5.9	17.56±5.9
NS_4	5.92	14.63±5.9	11.71±2.9	11.71±2.9	84.88±5.9	32.20±2.9	26.34±5.9
NS_5	6.12	32.20±2.9	14.63±2.9	23.41±5.9	14.63±2.9	23.41±2.9	17.56±2.9
NS_6	6.45	38.05±2.9	17.56±2.9	26.34±2.9	17.56±2.9	26.34±5.9	20.49±2.9
NS_7	6.45	11.71±2.9	11.71±5.9	14.63±2.9	17.56±2.9	26.34±5.9	23.41±5.9
NS_8	6.45	32.20±2.9	14.63±2.9	26.34±5.9	23.41±5.9	20.49±5.9	17.56±5.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 3: Results of Ni concentrations in maize and soil samples at Mando

| | | | | Cold extraction | | | |
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
MD_1	4.15	8.780±2.9	23.41±2.9	17.56±2.9	93.66±2.9	23.41±2.9	14.63±2.9
MD_2	4.45	32.20±2.9	8.780±2.9	8.780±2.9	14.63±5.9	11.71±5.9	26.34±5.9
MD_3	4.56	35.12±2.4	11.71±2.4	8.780±2.4	17.56±2.4	14.63±4.8	29.27±8.7
MD_4	4.35	14.63±2.9	29.27±2.9	23.41±2.9	99.51±5.9	29.27±2.9	20.49±5.9
MD_5	4.26	35.12±2.9	35.12±2.9	11.71±2.9	17.56±2.9	14.63±5.9	29.27±2.9
MD_6	4.75	38.05±2.9	17.56±2.9	14.63±2.9	20.49±5.9	17.56±2.9	29.27±2.9
MD_7	4.75	35.12±2.9	17.56±2.9	20.49±5.9	17.56±5.9	23.41±2.9	20.49±5.9
MD_8	4.75	32.20±2.9	8.780±2.9	8.780±2.9	17.56±2.9	14.63±2.9	29.27±2.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 4: Results of Ni concentrations in maize and soil samples at Kakuri

| | | | | Cold extraction | | | |
Sample site	pH	Metal conc in Maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
KK_1	5.08	5.850±2.9	5.850±2.9	20.49±2.9	96.590±2.9	52.68±2.9	5.850±2.9
KK_2	4.98	28.30±2.8	5.850±2.9	46.83±5.9	46.830±5.9	46.83±5.9	58.54±29.3
KK_3	5.14	35.12±2.9	8.780±2.9	46.83±5.9	49.760±2.9	49.76±5.9	64.39±2.9
KK_4	5.14	11.71±2.9	11.71±2.9	26.34±2.9	102.44±2.9	58.54±29.3	11.71±2.9
KK_5	4.34	32.20±2.9	32.20±2.9	46.83±2.9	51.710±6.1	49.76±5.9	61.46±2.9
KK_6	5.15	35.12±2.9	11.71±2.9	49.76±2.9	52.680±5.9	52.68±2.9	64.39±2.9
KK_7	4.50	32.20±2.9	11.71±2.9	49.76±2.9	49.760±5.9	52.68±2.9	61.46±2.9
KK_8	4.50	35.12±2.9	11.71±5.9	52.68±5.9	52.680±2.9	52.68±5.9	61.46±2.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 5: Results of Ni concentrations in maize and soil samples at T/Wada

| | | | | Cold extraction | | | |
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
TW_1	5.07	8.780±2.9	9.760±3.4	7.7100±3.5	79.020±2.9	29.270±2.9	14.63±2.9
TW_2	5.12	66.33±6.0	73.00±0.9	102.44±2.9	102.44±2.9	110.93±3.4	93.66±5.9
TW_3	5.25	70.24±2.9	76.10±2.9	105.37±2.9	105.37±5.9	111.22±2.9	96.59±5.9
TW_4	5.27	14.63±2.9	14.63±5.9	52.680±2.9	17.560±2.9	84.880±5.9	20.49±2.9
TW_5	5.17	70.24±2.9	70.24±2.9	105.37±2.9	105.37±5.9	114.15±2.9	96.59±5.9
TW_6	5.49	73.17±2.9	79.02±2.9	108.29±5.9	108.29±2.9	117.07±2.9	99.51±5.9
TW_7	5.49	73.17±5.9	76.10±2.9	105.37±2.9	108.29±2.9	114.15±2.9	99.51±2.9
TW_8	5.49	67.32±2.9	73.17±5.9	106.34±3.4	108.29±2.9	112.20±	99.51±5.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 6: Results of Ni concentrations in maize and soil samples at T/Wada

			Cold extraction				
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
ST_1	5.14	8.780±0.0	9.7600±2.9	7.710±2.9	79.02±2.9	29.270±3.5	14.63±2.9
ST_2	5.20	90.88±5.4	99.510±2.9	87.80±2.9	96.59±5.9	105.37±2.9	96.59±2.9
ST_3	5.25	96.59±2.9	99.510±5.9	87.80±2.9	96.59±2.9	108.29±2.9	99.51±2.9
ST_4	5.82	8.780±2.9	29.270±2.9	23.41±5.9	93.66±2.9	20.490±5.9	26.34±2.9
ST_5	5.25	96.59±5.9	102.44±2.9	90.73±2.9	99.51±2.9	108.29±2.9	99.51±2.9
ST_6	6.10	99.51±2.9	105.37±5.9	93.66±2.9	102.44±2.9	111.22±5.9	105.85±8.3
ST_7	6.10	100.49±4.5	105.37±2.9	93.66±2.9	99.51±2.9	111.22±5.9	99.510±2.9
ST_8	6.10	96.59±5.9	102.44±2.9	90.73±2.9	96.59±5.9	105.37±2.9	101.46±8.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 7: Results of Ni concentrations in maize and soil samples at U/Muazu

			Cold extraction				
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
UM_1	4.01	5.850±2.90	14.63±2.9	23.41±2.9	99.510±2.9	17.56±2.9	5.850±2.9
UM_2	4.10	99.49±3.00	90.73±2.9	8.780±5.9	87.80±29.3	76.10±5.9	73.17±5.9
UM_3	4.22	107.32±4.5	93.66±2.9	90.73±2.9	76.100±2.9	87.80±5.9	76.10±2.9
UM_4	5.82	17.560±2.9	26.34±5.9	35.12±2.9	111.22±5.9	29.27±2.9	17.56±2.9
UM_5	4.11	102.44±2.9	93.66±2.9	58.54±2.9	90.730±2.9	79.02±2.9	76.10±2.9
UM_6	4.54	105.37±2.9	93.66±2.9	81.95±5.9	93.660±2.9	81.95±2.9	79.02±2.9
UM_7	4.54	105.37±2.9	93.66±2.9	81.95±2.9	90.730±2.9	81.95±2.9	79.02±2.9
UM_8	4.54	99.510±2.9	93.66±2.9	81.95±5.9	90.730±2.9	79.02±5.9	76.10±2.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Table 8: Results of Ni concentrations in maize and soil samples at Kachia

			Cold extraction				
Sample site	pH	Metal conc in maize sample HNO_3/H_2O_2 (2+1)	Metal conc in soil sample HNO_3/H_2O_2 (2+1)	EDTA 0.05 M	Oxalic acid 1.0 M	Citric acid 1.0 M	Acetic acid 1.0 M
KC_1	6.16	14.63±2.9	73.17±5.9	2.930±0.0	5.850±2.9	8.780±2.9	5.850±2.9
KC_2	6.24	17.56±5.9	5.850±0.0	11.71±5.9	5.850±2.9	11.71±2.9	8.780±2.9
KC_3	6.15	17.56±2.9	5.850±2.9	5.850±2.9	8.780±5.9	5.850±0.0	17.56±2.9
KC_4	6.08	17.56±2.9	2.930±0.0	23.41±3.4	23.41±2.9	23.41±2.9	29.27±2.9
KC_5	6.07	49.76±2.9	38.05±2.9	49.76±5.9	52.68±5.9	49.76±5.9	46.83±2.9
KC_6	6.14	46.83±2.9	49.76±2.9	43.90±2.9	49.76±5.9	52.68±2.9	49.76±5.9
KC_7	6.01	49.76±5.4	43.90±5.9	43.90±2.9	38.05±2.9	35.12±2.9	38.05±2.9

Results of mean values (mg/kg) ±S.D. (n = 3)

Wet ashing of maize: Five grams of oven dried crushed and sieved maize sample was weighed into an evaporating dish and ashed at 480°C for 4 h. Ten cm^3 of a mixture of nitric acid-hydrogen peroxide (2+1) was added to the ashed sample and dried with occasional shaking on a hot plate and cooled. Four cm^3 of 1.5 mol/dm^3 nitric acid was then added and centrifuged. The digest was diluted to 60 cm^3 of water and filtered. This was analyzed for Ni using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Digestion of soils: Soil pH was measured (1:5, w/v) using microprocessor pH METER, model pH 210. 10 cm^3 of mixture of nitric acid-hydrogen peroxide (2+1) was added to 5 g soil sample and dried with occasional shaking on a hot plate and cooled. Four cm^3 of 1.5 mol/dm^3 nitric acid was added and centrifuged and diluted to 60 cm^3 with water and filtered. The clear digest was analyzed for Ni using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Extraction of soil: Soil extracts were obtained by shaking separately, 5 g of soil samples with 10 cm^3 of 0.05 mol/dm^3 Na_2 EDTA (for carbonate and organically bound phases), 1.0 mol/dm^3 oxalic acid (for oxide phases), 1.0 mol/dm^3 acetic acid (for carbonate phases) and 1.0 mol/dm^3 citric acid (Fe and Mn oxide phase) at room temperature and centrifuged after stirring for 5 min. This procedure is referred to as cold extraction (Yaman *et al.*, 2005). The digest was diluted to 60 cm^3 with water and analyzed for Ni using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Ni content in maize and soil: The Ni content for the samples from the eight different locations in Kaduna is as shown in Table 1 to 8. The maize samples from KB, NS, MD, UM, KC had higher Ni concentrations than the corresponding soil samples. Maize sample obtained from site UM_3 had the highest Ni concentration (107 mg/kg) compared to all the sites. This could be attributed to agricultural activities, heavy traffic and

other anthropogenic activities within the locations. This is in agreement with the results of other investigators (Hickey and Kittrick, 1984; Kashem and Singh, 1998; Aydinalp and Marinova, 2003; Khairah *et al.*, 2004; Chamon *et al.*, 2005, 2009; Kashem *et al.*, 2007; Ana-Irina *et al.*, 2008; Pei-Fang *et al.*, 2008).

The soil samples from TW and ST had higher Ni concentrations than the corresponding maize samples. Similar results were reported by several authors (Chamon *et al.*, 2009; Krishna *et al.*, 2010).

The lower Ni values obtained for some soil sites compared to others may be due to the impact of anthropogenic sources of pollution in the various locations (Yaman and Bakirdere, 2002; Yusuf, 2007). The Ni concentration in the soil is least in site KC_4 and highest in sampling sites ST_6 and ST_7. In some of these sampling sites, the Ni concentration is above the tolerance limit value of 50 mg/kg (Baralkiewicz and Siepak, 1999).

The highest Ni concentration at the sampling sites is not only a problem to plant nutrition and food chain but may constitute to a direct health hazard. The highest concentration of total Ni in maize and extractable Ni could be attributed to agricultural processes, heavy traffic and other anthropogenic activities within the locations (Kashem and Singh, 1998; Chamon *et al.*, 2009).

The ANOVA (p = 0.000<0.05) indicated a significant difference in the Nickel concentrations across the various maize crops. The differences in Nickel concentrations can further be analyzed by a post-hoc test using the Duncan Multiple range test where means of homogeneous subgroups are clearly displayed. Moreover, the mean plots that follow clearly depict the mean values of the Nickel concentrations across the various maize crops.

The Duncan multiple range tests showed that Kakuri, Nasarawa, among others, had the least Nickel concentration in maize crops. While Sabon Tasha and Ungwan Muazu had the highest Nickel concentration as depicted in Fig. 2.

Metal speciation: The Ni distribution in the soil samples collected from Kabala (KB) showed that the metal exists in the forms; residual, oxide, carbonate/organic and Fe-Mn oxide.

In cold extraction, the concentration of the metal bound to carbonate is lowest in KB_1, KB_2, KB_3, KB_4 and KB_5. Ni concentrations of cold CH_3COOH extracts of soils from these locations were lower than the other fractions. The Ni concentration bound to residual fraction is highest in KB_6, KB_7 and KB_8. The metal in these sites was residual species available for plant uptake. Similar results were reported by many authors (Sposito *et al.*, 1982; Hickey and Kittrick, 1984; Yaman and Bakirdere, 2002). The concentration of the metal bound to Fe-Mn oxide fraction is highest in all the sampling sites. Hence Ni is said to be Fe-Mn oxide
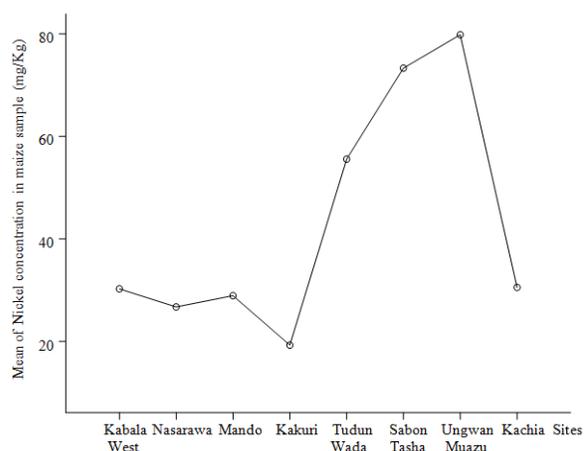
Fig. 2: Mean plot for nickel concentration in maize crops
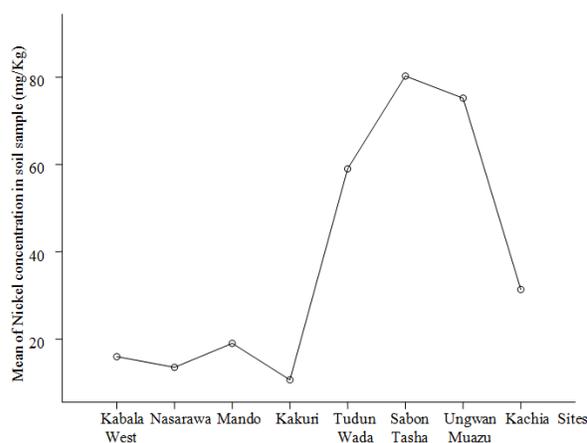
Fig. 3: Mean plot for nickel concentration in maize soil

species in these soils (Hickey and Kittrick, 1984; Yaman and Bakirdere, 2002).

The Nickel concentration in soil from Nasarawa (NS), showed that the metal also exist in the forms; residual, oxide, carbonate/organic and Fe-Mn oxide.

The nickel concentration bound to carbonate/ organic fraction is highest in NS_3 and NS_8. Hence, the Ni in these soils was organically bound or carbonates species (Yaman and Bakirdere, 2002). The concentration of the metal bound to oxide phase is highest in NS_1 and NS_4, while that bound to Fe-Mn oxide fraction is highest in NS_2, NS_5, NS_6 and NS_7. Similar result were reported by Sposito *et al.* (1982) and Hickey and Kittrick (1984).

In Tudun Wada (TW), the nickel concentration bound to Fe-Mn oxide fraction is highest in the sampling sites. Similar observations were made in the other sampling locations. The HNO_3/H_2O, EDTA, $(COOH)_2$, CH_3COOH and $C_6H_8O_6$ extractable Ni was considered as available (Lindsay and Norvell, 1978).

The pH of the soil samples from the various locations is acidic. This could increase the solubility and mobility of the metal in the soils (Kashem and Singh, 1998; Baranowski *et al*., 2002; Chamon *et al*., 2005).

The ANOVA (p = 0.000<0.05) indicated a significant difference in the nickel concentrations across the various maize grown soils. The real differences in nickel concentrations deduced by a post-hoc test using the Duncan Multiple range test where means of homogeneous subgroups are clearly displayed. Moreover, the mean plots clearly depict the mean values of the nickel concentrations across the various maize soils.

The Duncan multiple range tests showed that Kakuri, Nasarawa and Kabala West had the least Nickel concentration in maize grown soils while Tudun Wada, Ungwan Muazu and Sabon Tasha had the highest Nickel concentration as depicted in Fig. 3.

CONCLUSION

Total trace metal composition of soil is of little importance in determining its uptake by plants and consequently, in contaminating the food chain since the different forms have different mobility's, bioavailability and potential environmental contamination potential. The results on heavy metal speciation in the study indicated that the soil samples collected from various areas contain varying amounts of the metal. The metal was distributed between residual, oxide, Fe-Mn oxide and carbonate fractions. An increase of the metal concentration in some areas suggests that heavy use of agrochemical materials for planting activities could cause increase in the content of heavy metals in the soil.

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