NICKEL ADSORPTION BY SOILS IN RELATION TO pH, ORGANIC MATTER, AND IRON OXIDES

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ABSTRACT: There is little information on nickel adsorption by Brazilian soils. The objective of this experiment was to determine the effect of pH, organic matter, and iron oxides on nickel adsorption by three soils: a clayey Anionic "Rhodic" Acrudox, a sandy clay loam Anionic "Xanthic" Acrudox, and a clayey Rhodic Hapludalf. Soil samples were collected from the 0-0.2 m layer and treated to eliminate organic matter and iron oxides. The nickel adsorption was evaluated in the original samples and in those treated to remove organic matter and to remove both, organic matter and iron oxides, using 2 g soil + 20 mL of 0.01 mol L^{-1} $CaCl_2$ solution containing 5 mg L^{-1} Ni, pH varying from 3.5 to 7.5. The nickel adsorption decreased with the elimination of organic matter. For the samples without organic matter and iron oxides, adsorption decreased only in the Anionic "Rhodic" Acrudox. The pH was the main factor involved in nickel adsorption variation, and for soil samples without organic matter and iron oxides, the maximum adsorption occurred at higher pH values.

Key words: Ni adsorption, tropical soils

ADSORÇÃO DE NÍQUEL EM SOLOS EM FUNÇÃO DE pH, MATÉRIA ORGÂNICA E ÓXIDOS DE FERRO

RESUMO: Há poucas informações disponíveis na literatura quanto à adsorção de níquel em solos do Brasil. O objetivo deste trabalho foi determinar a influência do pH, da matéria orgânica, e dos óxidos de ferro na adsorção de níquel em amostras da camada superficial (0 a 0,20 m) de um Latossolo Vermelho acriférrico típico (LVwf), textura argilosa, um Latossolo Amarelo ácrico típico (LAw), textura argilo-arenosa e um Nitossolo Vermelho eutroférrico (NVef), textura muito argilosa. Foram utilizadas amostras de solo natural, de solo sem matéria orgânica (MO), e de solo sem matéria orgânica e sem óxidos de ferro, para fazer envelopes de adsorção (2,0 g de solo + 20 mL de solução contendo 5 mg L^{-1} de Ni em $CaCl_2$ 0,01 mol L^{-1}, variando o pH de 3,5 a 7,5). A adsorção de níquel diminuiu com a eliminação da MO; a eliminação de MO e de óxidos de Fe só provocou diminuição na média de adsorção no LVwf; o pH foi o principal fator de variação na adsorção de níquel e, com a eliminação da MO e dos óxidos de Fe, os picos de adsorção foram atingidos a valores mais elevados de pH.

Palavras-chave: adsorção de Ni, solos tropicais

INTRODUCTION

Nickel (Ni) input in the environment is mainly derived from phosphate fertilizers, pesticides and biosolid sources. The Ni ionic species usually found in soil solution is $Ni(H_2O)_6^{2+}$, whose activity decreases with increasing pH, and also with the interaction with organic and inorganic compounds, forming complexes. In the solid soil fraction Ni may be found in organic and inorganic compounds and, also in this case, the pH is the main factor controlling the adsorption reactions of this metal to colloids (Uren, 1992). For Brazilian soils, the pH is usually considered the most important factor in Ni adsorption (Gomes et al., 2001). For two Ultisols from the State of Rio Grande do Sul, Brazil, Pombo et al. (1989) observed increasing Ni adsorption when pH varied from 4.0 to 6.0.

Ionic retention of heavy metal by soil colloids is an important process for the preservation of environmental quality. Knowledge on the adsorption mechanisms and their quantification are fundamental for the comprehension of polluter-metal movement in contaminated soils, providing possible alternatives for a preventive environment control.

Kaolinite, Fe and Al oxides, are important tropical soil secundary minerals, responsible for the low mobility and bioavailability of heavy metals. The metal ions can be eletrostatically adsorbed to such materials, or specifically, through covalent or partly covalent bindings to oxygen atoms from the mineral structure (Araújo et al., 2002). For soils as well as for kaolinite or Fe, Al and Mn oxides, the relative sequence of adsorption is: Cu > Zn > Ni. Goethite and hematite are among the inorganic colloids associated to higher Ni quantities (Singh & Gilkes, 1992).

Organic matter may affect negatively or positively the metal availability as a result of the formation of metal quelates or complexes. McBride (1989) suggested the following sequence of metal affinity with the organic matter: Cu > Ni > Pb > Co > Ca > Zn > Mn > Mg. As pH increases, only Cu adsorption by organic colloids is inner-sphere (especific adsorption) (Bloom & McBride, 1979), and thus, most cations stay moderately bound by these colloids, when compared to the inorganic forms (Uren, 1992). Although the relevance of organic matter and Fe oxides for the metal adsorption are widely known (Sauvé et al., 2000), studies about the behavior of such elements are scarce, especially in acric soils, which are high Fe- and Al-oxide weathered soils, and represent about 170.000 ha of the northern State of São Paulo (Alleoni & Camargo, 1995; Alleoni, 2000).

The soil is characterized as acric when the effective cation exchange capacity (ECEC) is equal to or lower than 15 $mmol_c\ kg^{-1}$ of clay and the pH (in 1 mol L^{-1} KCl) is higher than or equal to 5.0; or ΔpH (pH KCl - pH H_2O) is positive or null (Embrapa, 1999); or else, ΔpH is higher than or equal to 0.1, in the 125 cm depth layer (FAO, 1989). The objective of this research was to determine the effects of pH, organic matter and Fe oxides on nickel adsorption by three prevalent weathered soils.

MATERIAL AND METHODS

Superficial (0-0.2 m) and subsuperficial soil samples were taken from three soils: a clayey Anionic "Rhodic" Acrudox (from Ribeirão Preto - 21º10'39''S, 47º48'37''W), a sandy clay loam Anionic "Xanthic" Acrudox (from Guaíra - 20º19'06''S, 48º18'38''W) and a clayey Rhodic Hapludalf (from Ribeirão Preto), all in SP, Brazil. The soils were chosen for their contrasting textures, aiming to characterize the metal adsorption in different clay contents. The Rhodic Hapludalf is a less weathered soil, adequate for comparison with the two other contrasting, highly weathered Acrudox soils. Samples were submitted to chemical, physical and mineralogical analyses (Tables 1 and 2), according to procedures described in Camargo et al. (1986): organic carbon was oxidized with potassium dichromate; pH values were obtained in 0.01 mol L^{-1} $CaCl_2$ solution and in 1:2.5 soil:solution ratio suspensions; oxide concentrations were obtained after sulphuric acid digestion; iron was reduced with dithionite and complexed by sodium citrate; iron and aluminum, not well crystallized, were solubilized with oxalic acid and ammonium oxalate; and the clay concentration was determined by the pipette method. The zero point of salt effect (ZPSE) was determined by titration with H^+ and OH^- in three KCl concentrations (Alleoni & Camargo, 1994a). The net electrical charge was estimated from the ZPSE plotting (Alleoni & Camargo, 1994b). The soil specific surface was determined in the three original soil samples, using the ethylene glycol monoethyl ether (EGME) method, described in Camargo et al. (1986).

Organic matter removal from soil samples was made according to Lavkulich & Wiens (1970), as follows: 10.00 g of air dried fine soil were transferred to 100 mL centrifuge tubes; 20 mL of a pH 9.5 NaOCl solution containing 60.00 g L^{-1} of Cl were added to each tube. Tubes were then heated to 90-95ºC in a water-bath for 15 minutes and centrifuged at 2,500 rpm for 10 minutes, and the supernatant was discarded. During heating, all samples were stirred each 5 minute, with a glass stick. All procedures were repeated thrice, until complete organic matter removal, evidenced by the supernatant color change from yellow to uncolored (Moore & Reynolds Jr., 1989). After this, the soil samples were rinsed eight times with a 0.0025 mol L^{-1} calcium nitrate solution to eliminate the NaOCl excess, stirring and resuspending the sediment each time, following centrifugation and supernatant discarding. For each soil sample, around 450 g of soil without organic matter (OM-free soil sample) were obtained.

For effective removal of iron oxides, 250 g of OM-free soil from each sample were separated and submitted to procedure according Jackson (1978), described in Camargo et al. (1986), as follows: 10.00 g of air-dried, fine, OM-free soil sample were transferred to 100 mL centrifuge tubes; 40 mL of a 0.3 mol L^{-1} sodium citrate + 1 mol L^{-1} sodium bicarbonate solution were added to each tube, which were heated to 75ºC in a water-bath. Following this, 1.00 g sodium dithionite was added to each tube, stirring for one minute. After 15 minutes, samples were removed from the water bath and 10 mL of a saturated NaCl solution plus 10 mL of aceton were added to each tube. Tubes were stirred and taken to the centrifuge for 10 minutes at 5.000 rpm, after what the supernatant was discarded. The entire procedure was repeated twice, when the sediment presented a gray color, evidencing the removal of iron oxides. Samples were rinsed once with deionized water and thrice with 0.0025 molL^{-1} calcium nitrate solution to eliminate salt excess. For each soil sample, around 200 g of soil without organic matter and iron oxides (OM+FeO-free soil samples) were obtained.

The Ni adsorption was determined in original samples (OS), OM-free samples and OM+FeOx-free samples. Adsorption envelopes were prepared for a pH range between 3.5 and 7.5. Three replications of the Ni adsorption envelopes were made. Each envelope consisted of the addition of 20 mL of a $NiCl_2$ solution (5 mg L^{-1} Ni) in support electrolyte, to 2 g oven dried (at 60ºC) fine soil samples, for a wide range of pH values, which were shaken for 24 hours. The support electrolyte chosen was a 0.01 mol L^{-1} $CaCl_2$ solution, because Ca^{2+} is the predominant cation in the exchange complex of the soils of the State of São Paulo. Immediately after the 24 hours shaking, Ni was determined in the equilibrium solution by atomic absorption spectrophotometry. The quantity of adsorbed Ni was calculated by subtracting the Ni determined in the equilibrium solution from the Ni added to the soil sample.

The results were submitted to analyses of variance of a completely randomized design, arranged in a 3x3x9 factorial scheme, with three replications, where the factors were three soil types, three soil treatments (OS, OM-free and OM+FeOx-free soil samples) and nine pH conditions. The triple interaction was decomposed to study the two-by-two combined effects. Comparisons of means among soils and soil treatments were made using Tukey test ($P = 0.05$).

RESULTS AND DISCUSSION

Nickel adsorption was different among the three original soil samples, in the following decreasing sequence: Rhodic Hapludalf > “Xanthic” Acrudox > “Rhodic” Acrudox (Table 3). After organic matter and iron oxides removal, the Rhodic Hapludalf presented the highest Ni adsorption capacity; however, no difference was observed between the two Acrudox soils. This higher-average soil Ni adsorption obtained for the Rhodic Hapludalf was attributed to the higher amount of variable and permanent charges present in its superficial horizon (Table 1). The permanent electrical charge, close to 30 $mmol_c$ kg^{-1} (Table 2), probably indicate the presence of 2:1 minerals, not detected by the X-ray analysis. The X-ray diffraction method for mineralogical analyses may not be accurate enough to detect small quantities of minerals with permanent charges (Fontes & Sposito, 1995). The higher Ni adsorption observed by the Rhodic Hapludalf as compared to the Acrudox points out that the metal retention capacity depends on chemical and mineralogical soil characteristics and, therefore, have to be considered when chosing areas for residue discarding or application, in order to minimize soil contamination (Silveira et al., 2002).

The Ni adsorption found for the original soil samples were different from the ones obtained from the OM-free soil samples (Table 3 and Figures 1a, 1b and 1c), evidencing a relevant effect of the organic matter on Ni adsorption. For the OM-free soil samples, the average adsorption values were 21.0, 11.0 and 28.0% lower than those obtained for the original soil samples (Table 3), of the “Xanthic” Acrudox, “Rhodic” Acrudox, and Rhodic Hapludalf, respectively, and these differences increased as pH decreased. For pH above 6.0-6.5, the effect of organic matter on Ni adsorption was not significant (Figures 1a, 1b and 1c), probably because great part of the negative, dependent pH charges would have the H^+ already dissociated. The organic matter provides sites for cation exchange, but its strong affinity to metals is due to the presence of specific binders or groups that form

Table 1 - Chemical and mineralogical attributes of the three soils.

Soil[1]	Profile depth	pH H_2O	ΔpH	C	H+Al	Al	SB[2]	CEC_t[3]	CR[4]	ZPSE[5]	Kt[6]	Gb[7]
	m			g kg^{-1}	---------	mmolc kg^{-1}	---------		mmolc kg^{-1} clay		--- g kg^{-1} ---	
1	0.0-0.2	5.7	-0.9	26	59	2	106	165	131	3.7	249	59
	0.7-0.9	6.0	-0.7	7	27	0	54	81	77	3.6	263	80
2	0.0-0.2	6.4	-0.9	20	30	0	63	93	105	3.6	172	375
	1.0-1.4	5.3	+0.3	5	14	0	8	22	13	5.7	216	431
3	0.0-0.2	4.6	-0.5	16	36	6	20	56	38	3.4	248	200
	1.0-1.3	5.1	+0.6	6	11	0	9	20	7	6.1	196	189

[1] 1=clayey Rhodic Hapludalf; 2=clayey Anionic “Rhodic” Acrudox; 3=sandy clay loam Anionic “Xanthic” Acrudox; [2] SB = sum of bases; [3] CEC_t= CTC total; [4] CR= cation retention; [5] ZPSE= zero point of salt effect;; [6] Kt= kaolinite; [7] Gb= gibbsite.

Table 2 - Iron and aluminum concentrations, ki index, clay concentration, specific surface and negative charge density of the soil samples.

Soil[1]	Prof depth	Fe_t[2]	Fe_d[3]	Fe_o[4]	Al_t[5]	Al_d[6]	Al_o[7]	ki[8]	Clay	ES[9]	Negative charge density	
											Variable (σ_H)	Permanent (σ_O)
	m	--------------------------			g kg^{-1}			--------------------------		m^2 g^{-1}	---------- $mmol_c$	kg^{-1} ----------
1	0-0.2	270	150	12	167	23	8	1.89	820	134	63.1	30.2
2	0-0.2	320	164	13	195	26	10	0.93	600	65	39.9	8.2
3	0-0.2	66	39	2	91	22	6	0.98	350	65	32.3	8.4

[1] 1=clayey Rhodic Hapludalf; 2=clayey Anionic “Rhodic” Acrudox; 3=sandy clay loam Anionic “Xanthic” Acrudox; [2] Fe_t = total iron; [3] Fe_d = iron extracted with dithionite, citrate and sodium bicarbonate solutions; [4] Fe_o = iron extracted with ammonium oxalate; [5] Al_t = total aluminum; [6] Al_d = aluminum extracted with dithionite, citrate and sodium bicarbonate solutions; [7] Al_o= aluminum extracted with ammonium oxalate; [8] ki index= (% SiO_2/60)/(Al_2O_3/102); [9] ES = specific surface of original sample.

metal complexes or chelates. Nickel, as well as other heavy metals, form complexes with several soil organic constituents. Nevertheless, these complex formations occur in great part with the fulvic and humic acids, present in the organic matter in large quantities, the insoluble combinations being associated with the humic acid and the soluble ones, with the fulvic acid (Stevenson, 1991). Several researchers have already observed these associations of organic matter with metal chelate formations in many soil types, Acrudox included (Camargo et al., 1989; Alleoni, 2000; Mellis & Casagrande, 2001).

The “Rhodic” Acrudox presented higher Fe_t, Fe_d, Al_t and Al_d concentrations when compared to the two other soils, and higher and similar amorphous Fe-oxide concentration when compared to “Xanthic” Acrudox and Rhodic Hapludalf, respectively (Table 2). Although the hydrated Fe oxides and hydroxides, that appear amorphous under X-ray diffraction, present higher adsorption capacity (Okazaki et al., 1986) and also higher amount of positive charges (Yu, 1997) than the crystalline ones, they are not responsible for the higher Ni adsorption capacity of “Rhodic” Acrudox, despite the fact that this latter soil presents higher Fe_o concentration. On the other hand, the “Xanthic” Acrudox and Rhodic Hapludalf presented higher quantity of kaolinite than “Rhodic” Acrudox, which might have compensated lower Ni ad-

Table 3 - Nickel adsorbed by soil samples from the 0-0.2 m layer depth, before and after the organic matter removal (OM-free soil) and organic matter + Fe-oxide removal (OM + FeOx-free soil).

Soil sample	pH	Ni adsorbed					
		"Xanthic" Acrudox		"Rhodic" Acrudox		Rhodic Hapludalf	
		mg kg^{-1}	%	mg kg^{-1}	%	mg kg^{-1}	%
Original Soil	3.4	17.77	35	7.77	16	29.46	59
	4.1	25.42	50	15.42	31	35.12	70
	4.6	30.89	62	17.89	36	44.50	89
	5.2	36.74	74	31.02	62	47.25	94
	5.5	39.22	78	37.86	76	47.97	96
	6.1	46.61	93	45.24	90	48.70	97
	6.4	48.52	97	49.62	99	49.35	99
	7.0	49.90	100	49.89	100	49.67	99
	7.5	49.90	100	49.89	100	49.67	99
Means[1]		38.33 Ba	75	33.84 Ca	68	44.43 Aa	89
OM-free Soil	3.4	5.23	10	5.23	10	4.51	9
	4.1	7.39	15	7.39	15	9.15	18
	4.6	13.87	28	13.87	28	19.60	34
	5.2	26.11	52	26.29	52	32.17	64
	5.5	29.71	59	30.07	60	39.60	79
	6.1	40.16	80	39.80	79	41.96	84
	6.4	49.52	99	49.52	99	47.00	94
	7.0	49.52	99	49.52	99	49.52	99
	7.5	49.52	99	49.51	99	49.52	99
Means		30.11 Bb	60	30.19 Bb	60	32.03 Ab	64
OM + FeOx-free soil	3.4	4.68	9	4.68	9	19.67	39
	4.1	8.01	16	8.01	16	22.34	45
	4.6	14.34	28	14.34	28	23.67	47
	5.2	22.00	44	22.00	44	24.67	49
	5.5	31.00	62	31.00	62	31.66	63
	6.1	38.66	77	38.66	77	30.00	60
	6.4	46.65	93	46.65	93	36.66	73
	7.0	48.65	97	48.65	97	43.03	86
	7.5	49.65	99	49.65	99	45.37	91
Means		29.29 Bb	58	29.29 Bc	58	31.88 Ab	64

[1] Means followed by the same letters, small letters in the columns and capital letters in the lines, do not differ by Tukey test at 5 %.

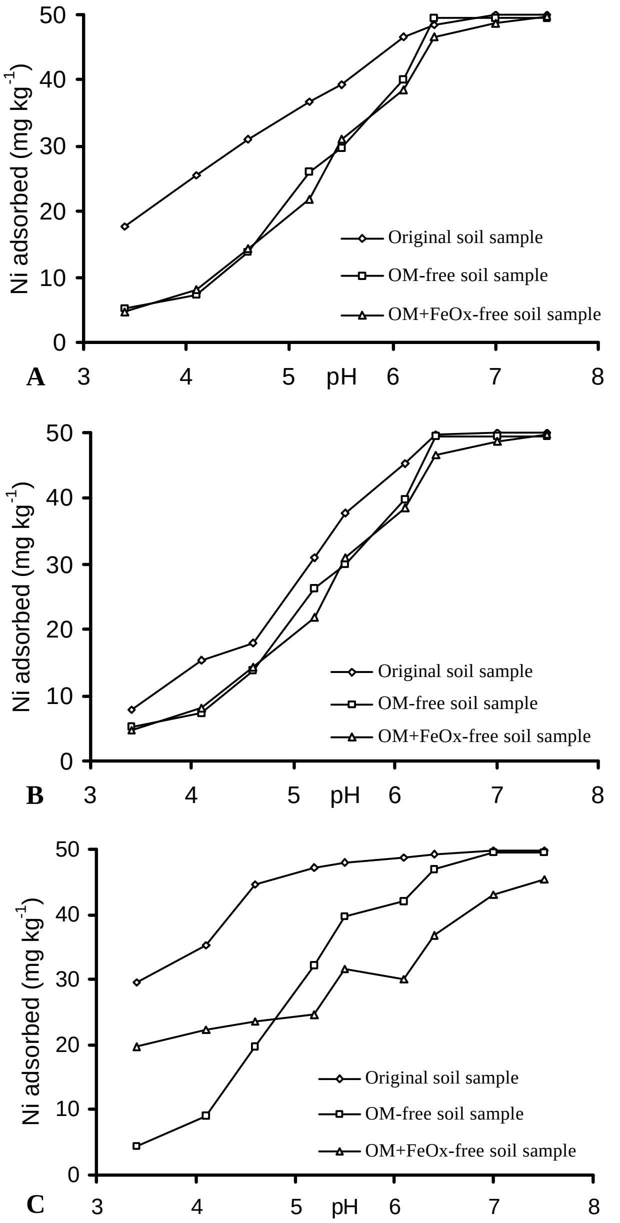

Figure 1 - Ni-adsorption envelope for the Anionic "Xanthic" Acrudox (A); Anionic "Rhodic" Acrudox (B) ; and Rhodic Hapludalf (C).

sorption resulting from the Fe-oxide removal, because heavy metals might be electrostatically adsorbed on the kaolinite surface (Spark et al., 1995).

Silveira et al. (2002) have also observed decrease on the "Rhodic" Acrudox copper adsorption with the Fe oxide removal, and suggested that in the "Xanthic" Acrudox and Rhodic Hapludalf soils, the Fe oxides might be covering sites of negative charges, since part of these oxides are combined to the clay minerals in the soil. Thereby, after iron oxide removal, such sites would be available for Ni adsorption through electrostatic retention mechanisms. The Fe oxide removal can drastically reduce the soil positive charges, which could favor the soil-metal adsorption reactions (Yu, 1997). Under low pH conditions, the oxides may contribute to metal retention by specific mechanisms involving covalent bindings. According to the same author, the Fe-oxide removal by the sodium citrate-bicarbonate method, can also remove part of the active aluminum, thus altering the balance of superficial charges. Hence, when soil pH is lower than the zero point saline effect (ZPSE), the iron oxide removal may contribute to the Ni adsorption (Figure 1c). Ni adsorption was higher in the 3.5 to 4.0 pH range as compared to the adsorption values obtained for the same pH range in soil samples only without organic matter. This was due to the reduction in the metal repulsion by the positively charged surface, in consequence of the oxide removal (Silveira et al., 2002).

For pH close to 7.0, practically all added Ni was adsorbed, corroborating results obtained by several authors that also observed drastic increase in the metal adsorption when soil pH were raised above 7.0 (Harter, 1979; Barrow et al., 1989; McBride, 1989; Schulthess & Huang, 1990; Scheidegger et al., 1996; Mellis & Casagrande, 2001). Even for pH around 7.0 and 7.5, all Ni^{2+} tend to disappear from solution as a result of adsorption, because the Ni^{2+} pKa_1 is close to 9.6, which is far above the values of pKa_1 found for metals like Cu^{2+}, Pb^{2+}, Zn^{2+} (Rodela et al., 2002). Therefore, even for the higher pH values tested in this research, the possibility of finding Ni in the form $Ni(OH)_2^0$ was very low.

The Ni adsorption, for all soils and independently of the treatment, increased with increasing pH. However, changes in the maximal adsorption peaks occurred depending on the treatment utilized. That is, for the original samples, the maximal Ni adsorption peak occurred at pH 6.0, approximately. Meanwhile, for the MO-free and MO-FeOx-free samples, the peaks occurred at pH 6.5 and 7.0, respectively (Figures 1a, 1b and 1c). This deviation of the maximal Ni adsorption peak observed for higher pH values evidenced that Ni adsorption is affected not only by soil pH, but also by CEC, clay contents, organic matter and Fe oxides present in the soil and other factors, as also observed by Melo et al. (1997).

After Fe oxide removal, there was lower variation in the Rhodic Hapludalf -Ni adsorption in function of pH (Figure 1c). This soil presented practically 2-fold variable charges and 4-fold the permanent charges values found in the other soils (Table 2). The Rhodic Hapludalf permanent charges have the same magnitude of the two Acrudox variable charges. With pH variation, the significantly higher Rhodic Hapludalf permanent charges will remain steady; meanwhile the variable charges will decrease more significantly than for the two other soils. In this way, the permanent charges will work as a buffer, that is, will avoid the pH dependent Ni adsorption variation.

On the other hand, the more significant decrease in the "Rhodic" Acrudox and "Xanthic" Acrudox Ni adsorption might be due to the predominance of variable charges in these soils, 4 to 5-fold the permanent ones, as compared to the 2-fold difference for the Rhodic Hapludalf. Considering that a clear reduction in Ni adsorption occurred at pH below 4.5, it was inferred that below this pH a higher amount of variable positive charges would probably be produced, mainly for the "Rhodic" Acrudox and "Xanthic" Acrudox soils. Above pH 6.5 there would be an effect of the pool of all negative variable charges, which would induce a similar Ni adsorption by all the three soil types.

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