

DISSOLUTION OF FERRIHYDRITE BY DISSOLVED ORGANIC COMPOUNDS DERIVED FROM SOME CROP RESIDUES

Saleh, M.E. and A. A. Abdel Salam

Department of Soil and Water Science, College of Agriculture, University of Alexandria, El-Shatby, Alexandria, Egypt.

ABSTRACT

The solubility of iron oxides and hydroxides is too low to cover the plant requirements especially in calcareous soils. Various attempts were carried out to overcome Fe deficiency in these soils. In the present study, the dissolved organic compounds (DOC) extracted from gramineous (rice, wheat, and corn) and legumeneous (peanut, faba bean, and clover) crop residues using water, 0.01M CaCl₂, or 0.01M CaSO₄ were used as DOC extractants to dissolve the laboratory-synthetic ferrihydrite-Fe. The results of infrared analysis for DOC showed a similarity in all residues with respect to the functional groups but different in their concentrations. The resulted DOC had a wide range of pH (7.0-5.1 in water, 7.1-5.3 in CaCl₂, and 6.9-5.2 in CaSO₄ solutions). Ferrihydrite showed a high affinity toward DOC; and different quantities of DOC were adsorbed on its surface. The DOC extracted by CaSO₄ was adsorbed in greater quantities than water- and CaCl₂-extracted ones. On the other hand, Fe dissolution by DOC indicated that the high ferrihydrite-Fe dissolution was observed with water-DOC of faba bean (1660.75mg Fe g⁻¹ mineral), clover (336.45), rice (245.14), corn (85.49), and peanut (18.13) then the CaCl₂-DOC of faba bean (533.18), clover 17.26), and corn (12.70). Ferrihydrite also showed a high affinity to adsorb the soluble Fe from wheat extracts and CaSO₄-DOC extracted from all residues. This result confirms that Fe was adsorbed in soluble complexes with DOC.

The results indicate that effectiveness of DOC to dissolve ferrihydrite-Fe is dependant, firstly, on the type of crop residue, the ionic strength of residue extracting media and the abundant ionic species. Secondly, it depends on the characteristics of DOC such as pH, type and concentration of the functional groups, reducing capacity, and the degree of lability (low or high molecular weight-DOC).

The results concluded that legume crop residues and corn stalks are considered efficient sources, as organic amendments, for ferrihydrite solubilization and maintaining high levels of soluble Fe in solutions.

INTRODUCTION

The chlorosis symptoms due to deficient Fe nutrition has long been known to cause yield reduction and even certain crop failure in calcareous soils. Under these conditions several attempts have been conducted from various naturally occurred microorganisms or from the grown crops to remedy the Fe situation and to promote its uptake by plants (Saleh *et al.*, 1999; Saleh, 1999; Marschner *et al.*, 1997; Bar-Ness *et al.*, 1991; Wang *et al.*, 1993; Wittenberg *et al.*, 1996; Fischer, 1988).

Iron oxides are the common source of Fe for plant nutrition. Since this Fe has to be supplied via solution, the solubility and the dissolution rate of Fe oxides are essential for the Fe supply (Schwertmann, 1991). The solubility products (K_{sp}) describing the effect of pH on Fe (III) ion concentration in solution are available for the well known Fe oxides occurring in soils such as goethite (FeOOH) and ferrihydrite (Fe₆(O₄H₃)₃; Chukhrov *et*

al., 1973) or $\text{Fe}_2\text{O}_3 + 2\text{FeOOH}$ (Russel, 1979). Thermodynamic calculations indicate that goethite and ferrihydrite represent the lowest and highest soluble forms, respectively (Lindsay, 1991). The K_{sp} of ferrihydrite is ranging from 10^{-37} to $10^{-39.4}$ (Schwertmann, 1977). The other factors rather than pH can affect the solubility of Fe in calcareous soils include the aeration status or the level of redox potential (Saleh, 1989) and the presence of substantial amounts of reactive calcium and magnesium carbonates.

One of the man-made attempts to increase the solubility and availability of Fe for plants grown in calcareous soils is the application of organic fertilizers (Bar-Ness and Chen, 1991, a and b). Dissolved organic compounds resulted from various organic amendments decomposition may significantly influence the mobility and complexation of heavy metals and micronutrients such as Pb, Cu, and Zn in soils (Kuiters and Mulder, 1989; and McBride *et al.*, 1997). Oden *et al.* (1993) found that the Cu mobility in a porous medium was highly affected by the soluble organic compounds extracted from six different sources. With respect to DOC characterization, Han and Thompson (1999) reported that the complexation of DOC extracted from anaerobically digested sewage biosolids with Cu decreased significantly with increasing molecular weight; the hydrophilic fraction showed a greater Cu-binding capacity than did the hydrophobic fraction.

Dissolution of soil iron minerals by DOC derived from organic amendments has very low attention and a little evidence exists for the widespread complexation of Fe by organic matter in soils although the two have some degree of association with one another. For example, appreciable quantities of Fe are invariably extracted with humic substances (Cheshire *et al.*, 1981), but this may be the result of co-extraction rather than chemical association (Goodman, 1988). The iron complexing ability to dissolved organic substances in the soils arises primarily from their carboxyl, phenolic hydroxyl and carbonyl groups, but other functional groups present in smaller amounts may have the ability to form very strong complexes under certain circumstances (Hayes and Swift, 1978; Stevenson, 1982).

In this paper the dissolution capacity of different crop residues-DOC extracted by water or calcium salts during the reaction with freshly precipitates of ferrihydrite was demonstrated. The objectives also extended to characterize the extracted DOC, and to show their importance to bring soluble Fe into solution.

MATERIALS AND METHODS

Characterization of Plant Residues

Six types of plant residues: rice and wheat straw, corn, peanut, and faba bean stalks, and clover hay were collected from different fields of calcareous soils. The samples of plant residues were air dried, crushed, passed through 2-mm screen, and stored in well-sealed plastic cans at room temperature. Total carbon, nitrogen, and iron were determined in the residue materials. On the other hand, the DOC, water-soluble organic compounds were collected after 24 h of incubation of one gram residue with 100 ml of distilled water in 250-ml Erlenmeyer polyethylene flasks. The mixtures were transferred into 100-ml polyethylene centrifuge tubes for centrifugation for 5

min at 6000 rpm then filtered on Whatmann paper no. 42. Electrical conductivity and pH of the filtrates were immediately measured using Radiometer CDM-83 conductivity meter and Orion Ion Analyzer 901, respectively. Concentrations of DOC were determined according to EPA (1982). Table (1) presents some properties of the studied plant residues. Portions of the plant residue extracts were frozen for 48 h then were lyophilized for 24 h. The solids were thoroughly mixed with potassium bromide for infrared testes.

Table (1): some properties of tested plant residues.

Properties	Plant residues					
	Rice	Wheat	Corn	Peanut	Faba bean	Clover
Total carbon, %	67.30	65.90	72.36	63.30	73.12	71.00
Total nitrogen, %	1.41	0.87	1.49	2.40	4.35	4.35
C/N ratio						
Total iron, %	47.80	76.07	48.72	26.40	16.82	16.33
	0.03	0.06	0.05	0.10	0.10	0.03
DOC, mg g ⁻¹						
PH	24.69	17.38	48.31	42.48	47.15	78.24
EC, dS m ⁻¹	7.00	7.10	5.30	6.70	5.10	5.10
	1.96	1.11	0.69	1.19	0.68	0.72

According to the results of DOC (Table 1), the clover hay had the highest concentration of DOC. To equalize the amounts of DOC released from each residue which will be reacted with ferrihydrite, proper volume from each extract was withdrawn and diluted to fixed volume

Preparation of Ferrihydrite

Ferrihydrite was prepared by dissolving 40 g Fe(NO₃)₃.9H₂O in 500 mL distilled water and adding 330 mL of 1M KOH to bring the pH to 7-8. The last 20 mL was added dropwise with constant checking of the pH. The system was stirred vigorously, centrifuged and then dialyzed rapidly until free from electrolytes; the produced solid was freeze-dried and stored. The resulted iron hydroxide was dark brown-red and agreed with that described by Schwertmann and Cornel (1991). There are various methods of ferrihydrite characterization such as color, X-Ray diffraction, electron microscopy, surface area, infrared spectroscopy and thermoanalysis (thermogravimetry and differential thermal analysis). The methods used in the present study to confirm the ferrihydrite formation were color, X-Ray diffraction and infrared spectral analyses. The results of X-Ray and IR were described previously (Saleh *et al.*, 1999).

Dissolution Experiment

Ten mL of three replicates of plant residue extracts were diluted to 35 mL by distilled water and transferred into 100-mL polyethylene centrifuge tubes containing 20658.4 ppm ferrihydrite-Fe. The suspensions were shaken for two hours using twist shaker. The tubes were centrifuged for 20 minutes at 11,000 rpm then filtered on Whatmann paper no. 42. In filtrates, pH and electrical conductivity were measured immediately after filtration and the concentrations of solubilized Fe in residue extracts were determined before and after the reaction with ferrihydrite using the Perkin Elmer 373 single beam atomic absorption spectrophotometer. On the other hand, the concentrations of DOC were determined after the reaction between the residue extracts and ferrihydrite.

RESULTS AND DISCUSSION

Infrared of the dried water extracts for the six studied plant residues were shown in Fig. 1. The IR spectra of DOC extracted from gramineous and legumeneous residues were similar in many absorbance bands. Bands at the following wave numbers were observed: 3300 cm^{-1} (stretching of OH), 1700 cm^{-1} (stretching of C=O in COOH groups), 1390-1400 cm^{-1} (stretching of C-O in phenolic OH, OH deformation, C-H deformation in CH_2 and CH_3 groups, and antisymmetric stretching of COO^- groups), and 950 - 1170 cm^{-1} (stretching in polysaccharides or similar compounds). Bar-Ness and Chen (1991) reported that the IR spectrum of Fe-enriched water-soluble fraction of various organic materials (farmyard, poultry manure, and peat) indicated that most of the ligands in the complex formed are polysaccharides or polysaccharide-like compounds.

The results in Table (2) show that the DOC has a wide range of pH regardless of the type of extracting solution. The extracting solutions did not significantly affect the pH values of DOC of corn stalks (5.3, 5.3, and 5.2 in water, CaCl_2 and CaSO_4 solutions, respectively). While the pH values of faba bean and clover were highly affected. Although the thermodynamic calculations indicate that ferrihydrite (and the other Fe hydroxides) dissolution is pH dependent (Lindsay, 1979), the relationship between pH and dissolved ferrihydrite-Fe (the difference between Fe values in columns 6 and 2 in Table 2) revealed no correlation ($r = -0.41$, $n = 16$) between pH decrease and Fe solubilization.

After the reaction with ferrihydrite, the pH values increased in most solutions with ranges between 0.4 and 1.0 in water, 0.2 and 1.1 in CaCl_2 and 0.5 and 1.3 in CaSO_4 solutions. The least increase in pH was observed in the rice, wheat and peanut residue solutions while the highest values were noticed for corn, faba bean and clover solutions (Table 2). However, the dissolved ferrihydrite-Fe did not correlated with the increment values of pH (the difference between columns 5 and 2). Therefore it is anticipated that soluble organics of the tested plant residues have other Fe solubilization forces, rather than pH, work in a wide range of pH. In an attempt to evaluate the reducing capacities of two fulvic acids at different pH values by varying the Fe to fulvic acid ratio over a wide range of values, Goodman (1988) found that at very low Fe(III) : acid ratios, some reduction could be seen with pH values as high as 5.

Concentrations of DOC before and after the reaction with ferrihydrite are listed in Table (2). The lowest concentrations of DOC were released from wheat and rice straws where the highest amounts were released from faba bean, clover and corn (in CaSO_4 extract). The results showed the ratios between DOC and hydroxide greatly differed from 2.11 : 1 to 0.081 : 1. After the reaction with ferrihydrite, the remaining amounts of DOC in solution were varied widely and were not related to the initial concentrations or extracting solutions. The dissolved Fe was not affected by the initial concentrations of DOC (Table 2).

Fig1

Table (2): Values of pH, DOC and solubilized Fe before and after the reaction of plant residue extracts with ferrihydrite

Residue	Before Ferrihydrite			After Ferrihydrite		
	pH	DOC(mg g ⁻¹ oxide)	Fe (ug g ⁻¹ oxide)	pH	DOC (mg g ⁻¹ oxide)	Fe (ug g ⁻¹ oxide)
<u>Water extract</u>	7.0	162.370	519.17	7.5	93.045	764.31
Rice	7.1	114.298	207.27	7.5	66.162	85.50
Wheat	5.3	317.845	344.59	6.2	246.622	430.08
Corn	6.7	279.450	82.91	7.4	191.782	101.04
Peanut	5.1	428.031	62.18	6.1	315.700	1722.93
Faba bean	5.9	514.750	59.59	6.8	356.998	386.04
Clover						
<u>CaCl₂ extract</u>	7.1	112.084	38.08	7.0	58.319	45.34
Rice	7.0	81.976	39.89	6.8	67.870	nd*
Wheat	5.3	195.704	26.29	6.3	176.728	38.99
Corn	6.8	133.906	26.29	7.1	128.782	29.01
Peanut	5.4	485.465	10.88	6.5	416.139	544.06
Faba bean	5.5	319.932	25.39	6.2	241.878	42.65
Clover						
<u>CaSO₄ extract</u>	6.8	367.941	54.01	7.3	259.020	39.96
Rice	6.3	133.084	48.85	6.9	55.726	nd*
Wheat	5.2	2108.466	45.89	6.5	186.026	42.93
Corn	6.9	321.324	37.01	7.4	152.186	25.91
Peanut	6.0	606.831	28.13	7.1	288.495	40.71
Faba bean	6.4	367.245	31.83	7.3	264.776	16.28
Clover						

* not detected

Adsorbed DOC on ferrihydrite is shown in Fig. 2. The results demonstrate that the

DOC of faba bean and clover extracted by CaSO₄ was adsorbed in higher amounts than water and CaCl₂ extracted ones. Generally, for all residues, the CaSO₄ extracted DOC was highly adsorbed by ferrihydrite surfaces especially the corn stalks-DOC. The relationship between the dissolved Fe and the amounts of adsorbed DOC failed to interpret the concentrations of soluble Fe (non significant correlation, see Table 2).

One of the important observations that the soluble Fe in initial DOC of wheat straw extracted by water and rice, wheat, corn, peanut and clover residues extracted by CaSO₄ solution was adsorbed on ferrihydrite after the reaction with DOC (Fig. 3). It can be investigated that the adsorbed Fe was in the form of Fe-organocomplexes. On the other hand, the DOC of faba bean residue appeared a strong force to dissolve ferrihydrite-Fe in all extracting solutions (dissolved Fe: 1660.75, 533.18, and 12.58 ug g⁻¹ oxide for water, CaCl₂, and CaSO₄ extracts, respectively). The results of ferrihydrite dissolution indicated that not only the concentration, functional groups, or pH of the produced DOC are strongly responsible to Fe solubilization, but also the reducing force (represented by redox potential, for example) may have the major responsibility. The redox strength and the functional groups of DOC may together be responsible on the reductive dissolution of ferrihydrite-Fe and maintain a high concentration of Fe in soil solutions against the precipitation reactions with the reactive solid phases in calcareous soils such as CaCO₃ and oxides. Stone and Morgan (1987) showed that changes in

Fig2+3

oxidation state of Fe (reduction of Fe³⁺ to Fe²⁺ and vice versa) have a dramatic impact on the solubility of Fe in all soils. The reductive dissolution of ferrihydrite can take place in high oxidation conditions (high values of redox potential) of soil by organic compounds. In other words, the DOC in this study are classified as labile organic fractions, which have the high reductive dissolution for Fe comparing with the nonlabile organics.

The wide variation in Fe dissolution according to extracting solution can be interpreted by the occurrence of calcium ions in the dissolution medium, which may eliminate the dissolution rate, and subsequently the soluble Fe released to solution. Also we can speculate that, in the presence of ions in soil solution such as Ca²⁺, a part of DOC dissolution force is inhibited by the complexing formation with Ca²⁺.

In conclusion, it is important to use the plant residues as organic amendments for calcareous soil for the purpose of solubilizing oxide- and/or hydroxide-Fe and bringing high concentrations to soil solution in available forms. In the salt-affected calcareous soils, faba bean stalks-DOC gave a good results for Fe solubilization under high concentrations of Ca salts.

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ذوبان الفيريهدريت بواسطة المركبات العضوية الذائبة المستخلصة من بعض المخلفات النباتية

ماهر السيد صالح وعبد السلام عباس عبد السلام
قسم علوم الأراضى والمياه - كلية الزراعة بالشاطبي - جامعة الإسكندرية

تعتبر درجة ذوبان أكاسيد وهيدروكسيدات الحديد فى الأرض منخفضة جدا لدرجة أن الحديد الذائب منها لايفى بحاجة النباتات النامية خاصة فى الأراضى الجيرية. وكان هناك محاولات عديدة فى دراسات سابقة للتغلب على أعراض نقص الحديد فى هذه الأراضى. فى الدراسة الحالية استخدمت مواد عضوية الذائبة المستخلصة من قش الأرز وتبن القمح والفول البلدى وحطب الذرة وعرش الفول السودانى والبرسيم كمركبات ذات أثر فى ذوبان معدن الفيريهدريت. تم إستخلاص المواد العضوية الذائبة بالماء أو بمحلول ٠,٠١ جزئى من كل من كلوريد الكالسيوم وكبريتات الكالسيوم. وتم تقدير خواص تلك المواد. كما تم تخليق معدن الفيريهدريت معمليا وتم التعرف عليه من خلال كل من اللون والأشعة السينية والأشعة تحت الحمراء. بعد ذلك أجريت عملية الذوبان بتفاعل المواد العضوية الذائبة مع الفيريهدريت لمدة ساعتين وتم تقدير الحديد الذائب وكذا المتبقى من المواد العضوية الذائبة.

أوضحت نتائج الأشعة تحت الحمراء على المواد العضوية الذائبة فى الماء تشابه المخلفات نوعا ما فى نوعية المجاميع الفعالة بها بينما اختلفت تركيبات المواد العضوية المستخلصة من كل مخلف نباتى حسب نوع المحصول ونوع محلول الاستخلاص. كذلك تميزت محاليل المواد العضوية المستخلصة بتفاوت فى درجة حموضتها (pH ١,٥-٧,١ فى الماء، ٥,٣-٧,١ فى كلوريد الكالسيوم، ٥,٢-٦,٩ فى كبريتات الكالسيوم). أبدى معدن الفيريهدريت قدرة واضحة على إدمصاص المركبات العضوية الذائبة المستخلصة من جميع المخلفات المختبرة مع وجود تفاوت كبير فى الكميات المدمصة على سطح المعدن. أقصى إدمصاص للمواد العضوية على سطح المعدن كان لمستخلص حطب الذرة بكبريتات الكالسيوم (١٩٢٢,٤٤ ملليجرام/جرام معدن) وأقل إدمصاص كان لمستخلص عرش الفول السودانى بكلوريد الكالسيوم (٥,١٢٤ ملليجرام/جرام معدن). وبصفة عامة كانت المواد العضوية المستخلصة بكبريتات الكالسيوم أكثر إدمصاصا من غيرها على سطح المعدن. من ناحية أخرى تفاوتت قدرة المواد العضوية الذائبة فى ذوبان الفيريهدريت وانطلاق الحديد منه حيث كان أعلى ذوبان لصالح المواد المستخلصة بالماء وهى على الترتيب: تبن الفول (١٦٦٠,٧٥)، عرش البرسيم (٣٢٦,٤٥)، قش الأرز (٢٤٥,١٤)، حطب الذرة (٨٥,٤٩)، وعرش الفول السودانى (١٨,١٣) ملليجرام/كجم فيريهدريت). جاء بعد ذلك فى الترتيب المواد المستخلصة بكلوريد الكالسيوم. بالنسبة للمواد العضوية المستخلصة بكبريتات الكالسيوم، فقد أبدى الفيريهدريت قدرة عالية على سحب (إدمصاص) الحديد الذائب المستخلص من المخلفات نفسها ولم يحدث ذوبان للفيريهدريت إلا بمستخلص الفول (١٢,٥٨ ملليجراما حديد/كجم معدن) مما يعتقد معه أنه حدث إدمصاص للحديد فى صورة معقدات عضوية. توضح نتائج هذه الدراسة تفاوت فعالية المواد العضوية الذائبة والمستخلصة من المخلفات النباتية المذكورة فى ذوبان الحديد من الأكاسيد والهيدروكسيدات. وأن هذه الفعالية مرتبطة بنوع المخلفات النباتية والقوة الأيونية لمحلول الاستخلاص من ناحية وخواص المواد العضوية المستخلصة (ال pH، المجاميع الفعالة السائدة، تركيز المواد العضوية المستخلصة، السعة الإختزالية لهذه المواد وكذلك الوزن الجزيئى للمواد المستخلصة) من ناحية أخرى.

وتخلص الدراسة الى أن مخلفات المحاصيل البقولية وحطب الذرة تملك قدرة عالية (عندما تضاف للأرض) على ذوبان الحديد من معدن الفيريهدريت (المتوفر فى الأرض) وتوفير مستويات مرتفعة من الحديد الذائب فى بيئة الذوبان.