

Studies of Nutrients Speciation in the Solid Substrate Polystyrene Nutrients Anchored Material Using pH-Metric Method

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ABSTRACT

Studies of relationship between pH variation and the nutrients distribution in the substrate/water mixture provides information about the concentration of the nutrients anchored in the substrate and the efficiency in which they are availed and controlled for plant relative growth. The technique for determining this relationship was based on measurement of changes in peak current (i_p) by DPASV method, following different concentration of HNO_3 added to a fixed amount of solid substrate polystyrene nutrients anchored material (M^+). The total nutrients in the substrate residual (ML_{n-1}) after several stripping experiments is determined by AAS method, the difference between the initial total nutrients concentration before stripping (ML_n) and ($M^+ + ML_{n-1}$), gives the concentration of the nutrients in the transition state. The method is advantageous in that the relationship can be measured under conditions where nutrients in the solution controlled directly the current. The results were then modeled so as to make an estimation of the availability of plants nutrients anchored in the substrate. The results shows that at pH 5.5, $5.02 \times 10^{-1} \mu\text{mol l}^{-1}$ of the nutrients had solubilized, $3.98 \times 10^{-1} \mu\text{mol l}^{-1}$ was complexed and $15.23 \times 10^{-1} \mu\text{mol l}^{-1}$ was in the transition state. This balance is enough to support plant growth without chemical burn.

Keywords : Distribution, Nutrients, Substrate, Stripping, Current, Species

I. INTRODUCTION

It has been discovered that growth of plants in soil may be improved by supplying a plurality of nutrients to soil by the use of synthetic water-insoluble ion exchange resins [3]. All of the nutrients absorbed by plants are of inorganic nature and they must be supplied by the addition of salts or a mixture of salts and inorganic materials, known generally as fertilizers. It has been discovered that additions of nutrients have

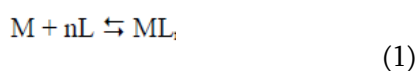
disadvantages, for instant, addition of excess salt concentration results in “fertilizer or chemical burn” or “salt injury to the plants [1; 3].

The total ionic concentration of a nutrient solution determines the growth, development and production of plants [20; 21]. The total amount of ions of dissolved salts in the nutrient solution exerts a force called osmotic pressure (OP), which is a colligative property

of the nutrient solutions and it is dependent on the number of dissolved solutes ^[11].

The stability of complexes can be employed in two ways, the thermodynamic stability and kinetic stability. Thermodynamic stability relates to the bond energy, stability constant and redox potential. On the other hand, kinetic stability is related to the rate of the reaction, formation of intermediate complexes, mechanism of reaction and activation for the process ^[14]. The thermodynamic stability of a species measures the extent to which the species will form or transform into other species in different conditions as the system reaches the equilibrium ^[13].

Let M be the nutrients and L be the substrate then the complexation process follows Equation 1:



If the value of [M], [L] and [ML_n] are known then the value of K, stability constant of the complex ML_n can be computed using Equation 2:

$$K = \frac{[ML_n]}{[M][L]^n} \quad (2)$$

II. METHODS AND MATERIAL

2.1 Introduction

Electroanalytical methods were used to determine the total and the labile fractions of metal ions in the substrate and also to study several metal-ligand (substrate) systems. Labile metal fraction is defined as the fraction that is easily available [19]. Since lability is an operationally defined concept in that it depends on the analytical method used, in this study, it was the fraction that can dissociate fast enough at the surface of the electrode to give a signal ^[12].

2.2 Determination of total zinc, iron and magnesium

The substrates were dried in the oven at 110.0 °C. They were allowed to cool in a desiccator after which 1.0 g of each was accurately weighed into a conical flask. 0.01M nitric acid (25.0 ml) was added and the mixture placed on a hot plate, then boiled for ten

minutes. It was left to cool and filtered through a whattman No. 41 filter paper into a 100 volumetric flask. The resulting solution was made to the mark with distilled water. These samples were then analyzed for the three metal ions. This was done by dispersing it in a support electrolyte, 10.0 ml, already in the polarographic cell, followed by purging the mixture with nitrogen, and an initial voltammogram of sample acquired. This was followed by volumes of 0.01 M ferrous ions (5.0 µl) were spiked in the same cell (standard addition method) without changing the machine settings and the resulting voltammograms acquired.

The peak currents obtained from the voltammograms and Equation 3 were used to calculate the concentration of the three metal ions in the substrate:

$$\frac{\Delta i}{0.05916} = \log \left\{ \frac{(V_x/V_T)C_x + (V_s/V_T)C_s}{C_x} \right\} = \log \left(\frac{V_x}{V_T} - \frac{V_s C_s}{V_T C_x} \right) \quad (3)$$

2.3 Flame atomic absorption spectroscopy method

A Shimadzu AA-680 atomic absorption spectrometer (AAS) which was equipped with single element hollow cathode lamp at 15.0, 30.0 and 20.0 mA for zinc, iron and magnesium respectively and 10.0 cm of burner air/acetylene burner head was used for the determination of iron, zinc and magnesium metals in the substrate. The wave length/resonance line was set at 580.2 nm with the spectral band width at 0.20 nm and the ratio of air-acetylene at 3.2 nm. The total nutrients released as a function of metal concentration was determined by mixing 1.0 g of finely ground modified polystyrene nutrients' anchored material with 10.0 mL of HCl solution (concentrations 10–250 mg L⁻¹) buffered at the optimum pH of 5.5. The mixtures were agitated for 30 min, filtered and the concentrations of metal ions in the filtrate were determined.

2.4 Determination of labile metal ion content by DPASV

A sample, 1.0 g, was dispersed in a support electrolyte, 10.0 ml of 1 M KNO₃, already in the polarographic cell, followed by purging the mixture with nitrogen, and an initial voltammogram of sample acquired. Cyclic voltammograms (CV) were then obtained at a scan rate of 50 mV s⁻¹ under the diffusion-limited conditions starting from a range of -1.6 V to 2.0 V. Starting with the blank followed by volumes of 0.01 M nitric acid (5.0 µl) were spiked in the same cell (standard addition method) without changing the machine settings and the resulting voltammograms acquired.

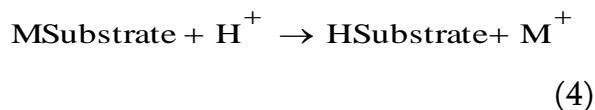
2.5 Potentiometric determination of stability constants

A pH titration study was carried out to measure stability constant of some selected metal-ligands complexes to investigate the effect of ternary complexes on the solubilization of Zn (II), Fe (II) and Mg (II) systems. According to Onindo [15; 16], mono glycinate complex held copper in solution in physiologically important pH range. His procedure was adopted to study the ligand system on iron, zinc and magnesium complexes.

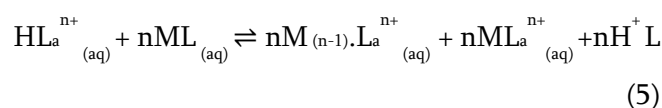
For the three ligands system studied, iron, zinc and magnesium, formation constants were calculated from potentiometric titration curves of metal ions in both the presence and absence of the HL_n ligand at 25.0 °C (298 K) and ionic strength maintained with 1.0 M KNO₃. Changes in pH were monitored using a glass microelectrode on a molspin pH meter. The devised apparatus to carry out Potentiometric titration consisted of a computer, micro syringe, titration vessel, molspin pH meter and the Polarographic analyzer Epsilon model E2 serial number 1177.

De-protonation process (Equation 4) is responsible in availing the nutrients for plant growth and this was done by regulating the pH releasing the ions from the

resin. The nutrients were made available for plant uptake by regulating the pH of the soilless media which resulted in the protonation of the substrate material [9]:



Modeling studies were carried out to investigate the effect of simple or ternary complexes found in the environmental sample on the release of the nutrients for plant uptake. This helped to simulate the natural system and predict the distribution of nutrient species at different environments. The expected reaction is as shown in Equation 5 [9]:



From the equation, the stability constant values of these various possible complexes of the various nutrients were determined. Our study used potentiometry by monitoring the pH of the resulting solution to predict the distribution of nutrient species at different environments.

Exploiting the information on the formation constants of the various nutrient systems, this study developed an effective, efficient, affordable and reusable method for the growth of crops in a soilless media.

III. RESULTS AND DISCUSSIONS

3.1 Effect of pH variation on concentration of the species in the substrate

The peak currents from the voltammograms in pH titration studies and the Nernstian equation were used to calculate the concentration of the metal ions in the substrate, AAS was used to determine the complexed metal in residual substrate after stripping.

The difference between the initial metal in the substrate and both the solubilized metal ion and metal complexed in the residual substrate gives the metal in the transition state and the results were tabulated and presented in Table 1

Table 1. concentration of the various species in the substrate

pH	Concentration of the species($\times 10^{-1} \mu\text{mol l}^{-1}$)		
	M ⁺	ML _{n-1} (Res. Subst.)	M(tran.state)
1	12.30	1.52	1.23
2	10.71	2.51	1.91
3	9.43	3.12	3.34
4	6.40	3.92	5.17
5	5.32	4.18	14.4
6	3.62	11.26	4.63
7	2.93	10.56	2.75
8	1.11	12.44	1.77

It was observed that the concentration of metal ions increased with decrease in pH up to pH 4.0 where a plateau on concentration was realized with 90% of the complexed metal having been de-protonated. This is due to the decrease in the competition for the binding sites on the substrate anchored material preventing the release of the metal ions [6].

Based on the data in Table 1, it can be observed that when the metal ions concentration is relatively low in high pH values, most of metal ions are bound to form complexes with the amino groups in the substrate moiety. Because of the strong binding power of the substrate to capture metal ions, its metal ions uptake dramatically increases at this stage. And because the amino groups were occupied quickly by metal ions and cannot interact with water molecules by H-bond, the substrate is water insoluble. In the sorption process, metal ions exchange with hydrogen ions to directly interact with the functional amino groups in the substrate polymeric structure. Therefore, the more the functional amino groups are occupied to

bind the nutrients, less water will be taken up by the substrate.

The study has confirmed that when the metal ions concentration is relatively low, most of metal ions are bound to form complexes with the amine groups in the substrate moiety [23]. This is because of the availability of active sites on the substrate for mineral attachment. Due to this strong binding power of the substrate to capture metal ions, metal ions release and thus availability of labile metal ions for plant uptake dramatically decreases.

The complexation phenomenon inhibits any interaction of the substrate with water molecules through H-bonding. Therefore, the more the number of functional groups complexed with nutrients, less water will be taken up by the substrate [23]. The choice of iron (II) was made due to its uniqueness to biological transition metal ions in that it has a high charge-to-radius ratio and as a hard acid forms high-affinity complexes with ligands [10]. Protonation of metal complexed substrate material was done to investigate the release of metal ions with reference to pH.

Many metal ions cannot exist in solution above pH 5 due to precipitation. But they exist in natural systems above this pH due to complexation with ligands from decayed organic matter [16]. Copper forms complexes predominantly with ligands containing nitrogen, oxygen and sulphur donor groups. At pH values below 5, copper (II) salts readily dissolve in water to form aqua complex $[\text{Cu}(\text{H}_2\text{O})]^{+}$. The water molecule can easily be replaced by another stronger ligand to form a different complex [8]. In order to model substrates speciation, three metal-ligands were considered for the study namely iron, zinc and magnesium with the substrate.

In this study chemical speciation is defined as the distribution of an individual metal element between

the bound and the ionized species or groups of species in the substrate and it reflects the chemical complexity of substrate media. The substrate is macromolecular and contains many sites of different types.

The interaction of metal M and ligand L can be expressed in the following mass balancing Equations 7 and 8:



From the equations it's clear that there exists a relation between the non-complexed metal and all forms of metal in solution [4; 14].

3.2 A comparison of hydrogen ion uptake and iron ion release

The concentration of hydrogen and iron ions were monitored with respect to the metal in the solution and the results presented in Figure 1:

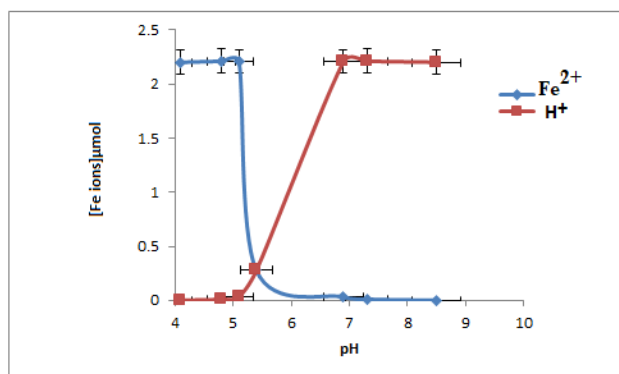


Figure 1. A comparison of hydrogen ion uptake and iron ion release as a function of equilibrium concentration of iron in the solution

As can be seen from Figure 2, the hydrogen ion uptake and iron released can serve as a function of equilibrium concentration of iron ions in the solution. At low concentrations, the molar quantity of hydrogen ion uptake is more than two times that of iron ion release. This is because in addition to exchange between iron and hydrogen ions, some hydrogen ions in the substrate dissociate and enter

the solution phase [24; 23]. However, at relatively high iron concentrations in the solution, the iron ions will complex with the amide groups, resulting in a hydrogen ion uptake to iron release ratio of slightly lower than [2; 10].

Based on the above analysis, it can be seen that at sorption equilibrium, all iron ions and hydrogen ions will reach equilibrium between the solution phase and the substrate phase. The sorption uptake of hydrogen ions influences the equilibrium of iron ions and water between the solution phase and substrate phase significantly.

3.3 Modeling

Modeling is simulating the natural system and predicting the distribution of chemical species. As such, modeling uses a chemical analysis of a system to calculate the distribution of the species in that system. It has found important areas of application in the speciation of chemical components likely to be present under a given situation [5]. The species are identified and chemical behavior predicted. The second application of modeling is in the prediction of environmental behavior for systems, which need not be considered over a very long timescale or are too complex for direct experimental approach as it enables building of a realistic picture.

In chemical modeling, formation constants, which are prerequisite in modeling, are generated in laboratory conditions and their use usually reflects the environment with a great deal of accuracy [5]. Stability constant values are a good measure of the stability of the complex species; it describes the competition of metal ions with protons for binding. A higher value of the stability constant shows the possibility of complex formation.

3.4 Stability constants

For the three ligands system studied, iron, zinc and magnesium, protonation constant values were

calculated from the pH titration curves and the constants are reported in Table 2:

Table 1. Stability constant values for deprotonation of iron, zinc and magnesium

Metal-ligand	P	β_p	$\log \beta_p$
Fe-ligand	2.19	1.78×10^{13}	13.25
Zn-ligand	2.12	2.13×10^{16}	16.33
Mg-ligand	2.12	9.55×10^{12}	12.98

The values obtained in this study by potentiometric analysis for the metal-ligand complexes compared very well with literature values [7; 13]. The reactivity of metallic centers in chemistry rests in their Lewis acid or redox-active characters. Metal centers that are strong Lewis acids, activate coordinated ligands for reactivity, a nitrogen atom coordinated to a Zn (II) center becomes a potent nucleophile for non-metals bonding to the substrate. In terms of redox activity, a wide variety of transition metals with variable oxidation states are found incorporated as catalytic cofactors to carry out oxidation/reduction reactions. Units like, iron-sulphate clusters, and blue copper nitrate donate electrons to other salts that require redox reactions for their release, while other redox salts catalyze multi-electron oxidation/reduction reactions directly on a substrate.

From this study, increasing the Lewis basicity of the donor enhances metal-ligand bonding. And this fact can be used to regulate metal-ligand affinity for nutrient release, increasing the basicity of a substrate increases its pKa. And since the metal ions compete with protons for ligand binding in aqueous solution, such adjustment decreases the effective metal binding at a desired pH.

3.5 Species distribution

Figures 2-4 represents the species distribution as calculated using the stability constant values obtained in this work for the various single ligand complexes of iron, zinc and magnesium at T= 298 K and ISAB= 1.0

M KNO₃. The diagram in Figure 2 represents the deprotonation of iron.

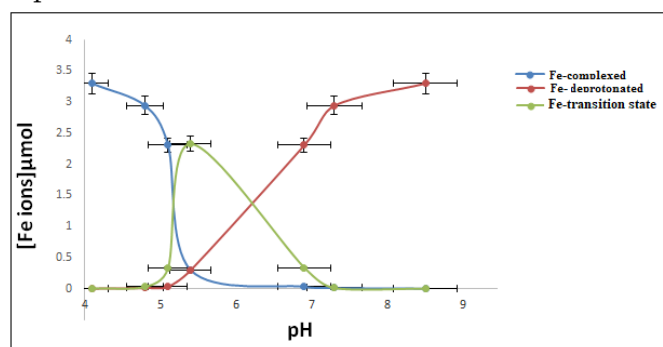


Figure 2. Speciation diagram for deprotonation of iron

From Figure 2, it was observed that substrate exists as diprotic ligand, ML₂, up to a pH of 6.9 after which it gains one proton due to the high concentration of H⁺ ions and this will result in its decomplexation with any available Lewis base like a hydrogen ion. At pH 5.5 almost all the iron metal was in solution form and at pH 7.4 most of the metal was complexed.

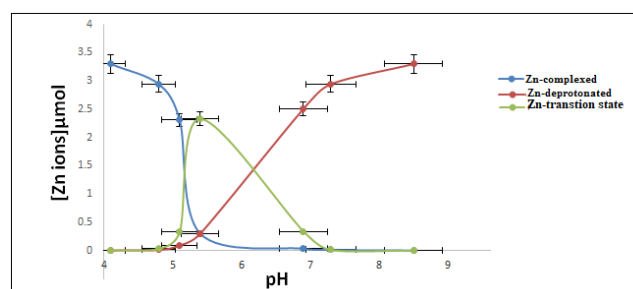


Figure 3 Speciation diagram for deprotonation of zinc
Zinc, as shown in Figure 3, starts to deprotonate at pH 6.5, which is lower than the iron because zinc is more reactive than iron and may de-complex faster with any available Lewis base like a hydrogen ion. At pH 4.8 almost all the zinc metal was in solution form and at pH 7.0 most of metal was complexed.

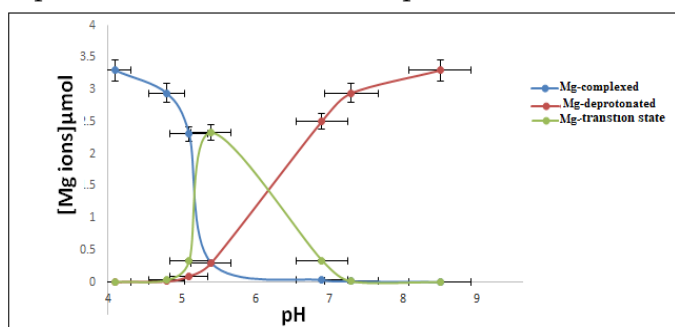


Figure 4. Speciation diagram for deprotonation of magnesium

In the case of magnesium represented in Figure 4, the ligand exists as ML_2 up to a pH of 5.7, which is lower than iron and zinc. This is because it's more reactive, hence it is a better leaving group than iron and zinc and therefore a better deprotonation. At pH 4.1 almost all the magnesium metal was in solution form and at pH 7.0 most of metal was complexed.

The analysis of these diagrams shows that at low pH value, most of the metal ions are present as free ions. This indicates that little complexation occurs in an acidic medium. Increasing the pH of the solution during the titration increases the concentration of the ligand, and this tends to decrease the mole fraction of the free metal ion, while that of ML_2 species develops at moderately acidic media [2].

According to the above observations, a dilute acid can directly influence the pH of the solution, stability constant and the state of amino groups, and eventually affects the coordination number of functional groups in the macromolecular ligand and the amount of metal ion binding. The average coordination number of substrate-metal ion complexes depends on the pH (1-6) of the aqueous solution and the molar ratio of amino groups to the metal ions. It can vary from 0 to more than two [23]. For divalent metal ions (iron, zinc and magnesium), at a pH of 4, when the equilibrium concentration of metal ions increases from 0 to $1 \mu\text{m}$, the average coordinate number decreases. This result shows that with an increase in equilibrium concentration of metal ions in the solution and pH, the complexes with one or two ligands are predominant.

With respect to the coordination number and the state of the metal ions in a broader pH range from less than 3 to over 9, Yokoi [24] reported that there are two factors determining the status of metal ions in an aqueous solution. One is the pH of the solution and the other one is the ratio of the residual functional groups in substrate to metal ions concentration. If pH

is less than 3, the metal ions can exist as ordinary hydrated M^{2+} in aqueous M^{2+} -substrate solutions, and no metal complexes can be formed. In a pH range of 1-6, mononuclear complexes of metal ions with one (ML) or two (ML_2) amino groups from substrate can be formed in an aqueous solution.

The coordination of metal ions to amino groups in substrate is strain-free. At a pH below 4, ML_2 is the preferential complex. Furthermore, when the ratio of amino groups to metal ions is relatively low (≤ 10), a large amount of binuclear M^{2+} complexes can be formed at pH 7. When the pH raises further to 9, all mononuclear and binuclear complexes of M^{2+} finally disappear. It is worth noting that when the pH increases to 6, inorganic compound $M(OH)_2$ can be gradually formed.

However, because of a high local concentration of amino groups and a polyelectrolyte field effect, $M(OH)_2$ does not precipitate in the aqueous solution but remains in the solution due to hydrophilic interaction with substrate. Therefore, in the range of solution pH 7-9, metal binding capacity of the substrate increases with an increase in pH [24; 23] due to formation of mononuclear and binuclear metal (M^{2+}) ion complexes. It is a complicated process for metal ion to associate with such macromolecular ligands as aminated polystyrene. The apparent dissociation constant, K_a , is influenced by the counter ions as well as the flexibility of the polymer ligands [17; 18]. According to the counter ion condensation theory, the change of the apparent dissociation constant involves electrostatic interactions between the metal ions and the polymer and chemical binding of counter ions of different valence.

The total free energy for amino groups in the substrate binding metal ions can be considered to consist of electrostatic and entropic mixing contributions. The bound fraction of metal ions leads to a decrease in charge of the substrate by a fraction which reduces the electrostatic interaction between

amino groups and metal ions in the solution. With an increase in chemical binding, more negative value of the intrinsic free energy of binding is obtained resulting in a high binding capacity.

The anchoring was by complexation with a high stability and as such nutrients cannot be leached off the media as is the case in normal soils. This offered a solution for the utilization of a non-biodegradable material initially used as packing material and latter bound to waste. The resulting soilless media is a low-density material suitable for use even in space experiments. This is then an environmentally friendly and sustainable method for mitigating effects of littering the environment by this low-density material affecting the scenic beauty.

IV. CONCLUSION

From this study it can be observed that a dilute acid can directly influence the pH of the solution, stability constant and the state of amino groups, and eventually affects the coordination number of functional groups in the macromolecular ligand and the amount of metal ion binding. The average coordination number of substrate-metal ion complexes depends on the pH (1-6) of the aqueous solution and the molar ratio of amino groups to the metal ions. In a pH range of 1-6, mononuclear complexes of metal ions with one (ML) or two (ML₂) amino groups from substrate can be formed in an aqueous solution.

The coordination of metal ions to amino groups in substrate is strain-free. At a pH below 4, ML₂ is the preferential complex. Furthermore, when the ratio of amino groups to metal ions is relatively low (≤ 10), a large amount of binuclear M²⁺ complexes can be formed at pH 7. When the pH raises further to 9, all mononuclear and binuclear complexes of M²⁺ finally disappear. At pH 5.5, $5.02 \times 10^{-1} \mu\text{mol l}^{-1}$ of the nutrients had solubilized, $3.98 \times 10^{-1} \mu\text{mol l}^{-1}$ was complexed and $15.23 \times 10^{-1} \mu\text{mol l}^{-1}$ was in the transition state. This

balance is enough to support plant growth without chemical burn. The results confirms that modification of the polystyrene material has a potential application for and cleaning the environment and subsequent use the growth of plants as a source of nutrients.

V. ACKNOWLEDGMENTS

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