Synthesis, Characterization and Application of 2-Line and 6-Line Ferrihydrite to Pb(II) Removal from Aqueous Solution

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KEY WORDS: Ferrihydrite, characterization, Point of zero charge.

ABSTRACT: Studies were carried out on the synthesis and characterization of 2-line and 6-line ferrihydrite. The XRD diffraction patterns for 2-line ferrihydrite exhibits two main peaks, a first-order peaks in the region 32.80° and 67.69° with an intensities 100% and 80% respectively. The 6-line ferrihydrite gave a characteristics five essential diffraction pattern with first order peaks in the regions 7.4, 30.0, 31.1, 33.8 and 62.8° correspondingly to 60, 60, 100, 100 and 50% intensities respectively. The XRF results of 2-line ferrihydrite and 6-line ferrihydrite showed Fe as the only major element. The percentage Fe was 95.20% and 93.42% for 2-line and 6-line ferrihydrite respectively. The Point of Zero Charge (pzc) of the 2-line and 6-line ferrihydrite was found to be 7.96 and 6.20 respectively. The 2-line ferrihydrite sorption capacity for Pb(II) was 127mg/g while it was 82mg/g for 6-line ferrihydrite. Adsorption was influenced by pH with maximum adsorption at pH 4 where 87.65% Pb(II) were adsorbed, after which sorption steadily decreased with increasing pH. The experimental results of Pb(II) adsorption were analyzed using Langmuir, Freundlich, and Temkin isotherms. Only Langmuir isotherm fitted the measured sorption data well. The constants obtained from the Langmuir model are 0.017, 1.72 and 0.768 for b, Q_m and the regression coefficient R^2 respectively; while the 6-line ferrihydrite are 0.02, 1.99 and 0.911 for b, Q_m and the regression coefficient R^2 respectively.© JASEM

There are sixteen known synthetic or naturally occurring iron oxides, hydroxides or oxyhydroxides [Mohapatra and Anand (2010).]. Ferrihydrite is one of the eight commonly occurring iron ores in the earth crust (Schwertmann and Cornell, 1991; Zhao, et al., 1994), groundwater aquifers, lake sediments, and hot - cold-spring deposits (Navrotsky 2001). Ferrihydrite is the only member with poorly ordered, none crystalline (Rancourt and Meunier, 2008) and nanoparticulate (<10 nm) in nature, which makes it thermodynamically unstable (Schwertmann, 1999.). It is a hydrous oxide with short range structural order that give broad range X-ray diffraction (XRD) peaks. Up to five and seven diffraction peaks have been observed for natural and synthetic 6-line ferrihydrite respectively [Schwertmann (1988); Parfitt et al., (1992)] and as low as two diffraction peaks for poor-orderly member sometimes referred to as ‘protoferrihydrite’ or 2-line ferrihydrite (Childs et al., (1982). Compared to most minerals, both 2-line and 6-line ferrihydrite show very broad diffraction line [4]. The broadness of the diffraction patterns makes it difficult to obtain accurate structural information. Several formulae have been proposed which can be reduced to FeOOH (Zhao et al., 1994)

In recent years, attention has been drawn to nanomaterials for its unique properties ranging from size, crystalinity, electronic and magnetic (Liu et al., 2006). Ferrihydrite had been shown to possess ultra-high surface area of about 200 m²/g (Cornell, R.M. and Schwertmann (1996) and sometimes classified as clay or active carbon materials which are known as good adsorbents for both organic and inorganic adsorbates (Cornell and Schwertmann, (1996); Matrajt and Blanot (2004). With its high surface area per volume, ferrihydrite is a very reactive mineral. It can interact, either by surface adsorption or by co-precipitation, with a number of environmentally important chemical species, including arsenic, heavy metals like lead or mercury, phosphate, as well as many organic molecules. Due to its unique physical and chemical properties, Ferrihydrite has found applications in medicine, catalysis, electronic device and environmental remediation (Ranjit et al., (2005); Xu and Bharttacharyya (2005); Mohapatra and Anand (2010). The most widely investigated application of ferrihydrite is its adsorbent property which is due to large surface area and ease of coordination of adsorbates to the unsaturated surface sites. In many soil and sedimentary systems, ferrihydrite conversion to goethite controls goethite occurrence.
and distribution (Cornell and Schwertmann 2003). Indeed, ferrihydrite is a metastable mineral and it is known to be a precursor of more crystalline minerals like hematite and goethite [5]. Ferrihydrite is one of the most important sorbents in soil, sediments, and water bodies and is frequently used for waste water treatment. Although the mineral is metastable and converts into more stable iron hydroxides such as goethite and hematite, it is constantly re-created by microbial mediated ‘redox’ cycles. Furthermore, its conversion is slow at temperatures below 20 °C and further retarded by adsorbed organic and inorganic species. Its sorption capacity is due to highly reactive surface sites and a surface area ranging from 200 to 800 m$^2$/g depending on the method of determination (Schwertmann and Murad (1983)

Experimental: The 2-line and 6-line ferrihydrite samples were synthesized using the outlined methods 1 and 2 respectively.

Method 1: A 2 L of deionised water was preheated to 75 °C in an oven. 20 g of unhydrolysed crystals of Fe (NO$_3$)$_3$·9H$_2$O was added rapidly to this hot water and the resulting solution was stirred for about 3 min. The solution was then returned to the oven at the same temperature for 10-12 min, after which it was cooled rapidly by plugging into ice water for 10 min and transferred into a dialysis bag. The dialysis was done changing the water several times each day until a constant value for conductivity close to that of de-ionized water was obtained. The suspension was filtered using vacuum pump and the product kept in a desiccator. The dried form was then used for characterization and other applications (Schwertmann and Cornell, 1991).

Method 2: 40 g of unhydrolysed crystals of Fe (NO$_3$)$_3$·9H$_2$O was dissolved in 500 ml deionized water and 330 ml 1 M KOH was added to bring the pH to 7-8. Then, another 20 ml 1 M KOH was added drop-wise with constant checking of the pH with vigorous stirring followed by centrifuging. The dialysis was done by changing the water several times each day until a constant value for conductivity close to that of de-ionized water was obtained. The suspension was filtered using vacuum pump and the product kept a desiccator to dry. The dried form was then used for characterization and other applications (Schwertmann and Cornell, 1991).

The pH was determined potentiometrically in deionised water at a sample-water ratio of 1:10 (REF). The point of zero charge (pzc) was determined by both mass titration and potentiometric methods (Noh and Schwarz, 1989). This involve measurement of pH of the Ferrihydrite (0.5-2.0 g) suspension in 25 ml of different ionic strength (10$^{-3}$ – 10$^{-1}$ M). The pH values at the start of experiment and after 24 h were recorded. The pH of the mixture was then adjusted to pH 5-6 with 0.01 M HNO$_3$, equilibrated for 24 h and pH measured. The pH of these mixtures was then re-adjusted to pH 10-11 with 0.01 M KOH, equilibrated for 24 h and the pH measured. The acid and base adjustments were made in order to determine the influence of pH on the pzc (Noh and Schwarz, 1989).

The ferrihydrite samples were analyzed to determine their constituents and qualities using X-Ray florescence (XRF) model Axios of Analytical type with a 2.4kWatt Rh X-ray Tube. XRD analysis of the samples were obtained on a Phylip analytical X-ray model PW 1800 diffractometer type equipped with Cu-target X-ray tube operated at 55 mA and 40kV (λ=1.5406 nm). The scanning analysis was accomplished on a Leo 1430 VP Scanning Electron Microscope.

Adsorption Experiment: A 0.5 g of ferrihydrite (2-line or 6-line ferrihydrite as the case may be) was weighed into 100 mL reactor flask and 25 cm$^2$ of working standard solutions (250, 500, 750, 850, 1000 ppm) of Pb(II) prepared by serial dilution of the standard solution, was added to the adsorbent in the beaker. The solutions were mechanically agitated for 7 hr and then filtered separately into plastic vials. The filtrates were analyzed using Atomic Absorption Spectroscopy (AAS) to determine the quantity of lead remaining in the solution. The quantity sorbed was then evaluated using the following equation (Megat et al., 2007).

\[
q_e = \frac{C_i - C_f}{M} \times V \tag{1}
\]

\[
q_e = \text{amount sorbed at equilibrium (mg/g)}
\]

\[
C_i = \text{initial concentration of Pb(II) solution (mg/L)}
\]

\[
C_f = \text{final concentration of Pb(II) solution (mg/L)}
\]

\[
V = \text{volume of Pb(II) solution used (mL)}
\]

\[
M = \text{Mass of ferrihydrite used (g)}
\]

The quantity sorbed was plotted against the initial concentration to determine the equilibrium concentration which is the concentration with the highest sorption capacity of ferrihydrite for Pb(II).
This equilibrium concentration was used subsequently for the sorption kinetics.

**Effect of Reaction Time** The effect of reaction time was investigated using 25ml of 850 mg/L (or 750 mg/L) Pb(II) solutions and 0.5g of synthesized samples of 2-line ferrihydrite (or 6-line ferrihydrite respectively) in 100 mL reactor flask. The flaks were mechanically shaken and the mixtures were allowed to equilibrate at different time intervals, 10, 20, 30, 45, 60, 90 and 360 min. The flaks were removed at designed intervals and content filtered. The Pb(II) in the filtrates were analyzed using AAS and the amount sorbed were calculated from equation (1).

**Effect of pH:** 25 mL of 850 mg/L (or 750 mg/L) Pb(II) solution was added to 0.5 g of synthesized samples of 2-line ferrihydrite (or 6-line ferrihydrite respectively) in 100 mL reactor flask and the initial pH was noted. The pH of the resulting solution was then varied between pH 3 and 7 using 0.1 M HNO₃ and/ or 0.1 M KOH solution. The mixture was equilibrated for 7 hr and then filtered after the equilibration time. The filtrate was analyzed for Pb(II) using AAS and the quantity adsorbed calculated from equation 1.

**Fitness of Pb(II) Adsorption Data into Langmuir, Freundlich, and Temkin Equations:** The equilibrium concentrations data obtained in the contact experiment were subjected to the Langmuir, Freundlich, and Temkin adsorption isotherms to find the equation that best fits the data. Data were fed into equations 2-4 separately and constants were calculated.

Langmuir:
\[
\frac{Q}{Q_e} = \frac{1}{aQ_m} + \frac{C_e}{Q_m} \tag{2}
\]

Freundlich:
\[
\log Q_e = \log K_f + \frac{1}{n} \log C_e \tag{3}
\]

Temkin:
\[
Q_e = B \ln A + B \ln C_e \tag{4}
\]

where \(Q_e\) is the quantity of sorbed at equilibrium (mg/g), \(C_e\) is the equilibrium concentration of adsorbate (mg/L). The Langmuir adsorption isotherm constants, \(b\) and \(Q_m\), were determined from the intercept and slope of the plot of \(C_e/Q_e\) against \(C_e\) (Krishnan and Anirudhan 2003, Hema and Arivoli, 2007). The Freundlich adsorption isotherm was determined by plotting \(\log Q_e\) against \(\log C_e\).

The slope and intercept obtained from the graph were used to calculate the Freundlich constants, \(n\) and \(K_f\). In the case of Temkin, the quantity sorbed \(Q_e\) was plotted against \(\ln C_e\) and the constants \(B\) and \(A\) were determined from the slope and intercept respectively.

**Adsorption Kinetics:** Useful information could be derived from adsorption kinetic models when they are applied to the removal of toxic metals from the environment by an adsorbent because it provides guide on pollution flux. Identifying the rate determining step is crucial in kinetic studies. In order to determine the controlling mechanism of adsorption process of Pb(II), the pseudo-first order and pseudo-second order rate equations were used (Ho and McKay, 1998, Ho and McKay, 1999, Abdus-Salam and Itiola, 2012). Sorption data obtained for 2-line and 6line ferrihydrite were subjected to both pseudo-first and pseudo-second order models in order to establish the model that best fits the data.

**Pseudo-first order model:** The equation for this reaction is
\[
\frac{dq_t}{dt} = k (q_e - q_t) \tag{5}
\]

where \(q_t\) is the quantity of solute adsorbed at equilibrium per unit mass of adsorbent (mg/g), \(q_e\) is the amount of solute adsorbed at any given time \(t\), (mg/g) and \(k\) is the rate constant of first order sorption (min⁻¹). In order to integrate eq. 5, the boundary conditions were set within the range \(t = 0\) to \(t = t\) and \(q_t = 0\) to \(q_t = q_e\) and simplifying the result of integration, Eq. (5) becomes
\[
\log (q_e - q_t) = \log q_e - \frac{k}{2.303} t \tag{6}
\]

The plot of \(\log (q_e - q_t)\) versus \(t\) gives the slope and intercept from which \(k\) and \(q_e\) were evaluated.

**Pseudo-second order model:** The data used for pseudo-first order model was independently substituted into the pseudo-second order model equation:
\[
\frac{dq_t}{dt} = k \frac{q_e}{q_t} \tag{7}
\]

If this equation is integrated setting the boundary conditions between \(t = 0\) to \(t = t\) and \(q_t = 0\) to \(q_t = q_e\), then Eq. (7) was simplified to yield equation
\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{8}
\]

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where \( k_2 \) is the rate constant of second order of sorption (g/mg min). Evaluation of the curve obtained from the plot of \( t/q \) versus \( t \) gave values of \( k_2 \) and \( q_e \).

**RESULTS AND DISCUSSION**

The physical properties of 2-line an 6-line ferrihydrite are summarized in Table 1. The synthesized 2-line ferrihydrite came out with colour brown while the 6-line was discovered to be dark brown in colour. The colours are within the variable colours (brown-dark brown) depending on the conditions of synthesis. The pH of the water-sample mixture of 2-line ferrihydrite was slightly above neutral (7.8) while that of 6-line was a little below neutral (6.5).

**Spectroscopic Studies:** The XRD patterns was consistent with 2-line ferrihydrite where diffraction pattern exhibits two main peaks, a first-order peaks in the region 32.8\(^0\) and 67.69\(^0\) 20 with an intensities 100 and 80% respectively. The symmetry of this sample is hexagonal structure and a monoclinic system. The 6-line ferrihydrite gave a characteristics five essential diffraction pattern with first order peaks in the regions 7.4, 30.0, 31.1, 33.8 and 62.8\(^0\) 20 corresponding to 60, 60, 100, 100 and 50% intensities respectively. The other data obtained for the 6-line ferrihydrite were cubic shape and a monoclinic system. In all cases, the diffraction peaks were broad which are attributed to nanoparticle sizes rather than structural distortion of the poor orderly Ferrihydrite (Parfitt et al., 1992).

The XRF results of 2-line ferrihydrite and 6-line ferrihydrite showed Fe as the only major element in the synthesized samples of ferrihydrite as seen indicated in Table 2 and Table 3 respectively. The spectroscopic data obtained for 2-line and 6-line ferrihydrite showed that the sample synthesized were actually ferrihydrite. The percentage Fe was 95.20% and 93.42% for 2-line and 6-line ferrihydrite respectively. XRF is ideal for rapid and accurate whole bulk elemental analysis in rock/soil samples or synthesized crystals or powdered samples. Apart from the major elements present other elements were reported at trace levels. The sum of these minor or ultra-trace elements constitute about 4.807% and 6.685% by mass of 2-line and 6-line ferrihydrite respectively. The source of these elements are attributed to the degree of purity of Fe(NO\(_3\))\(_3\)·9H\(_2\)O used. The assay constituents of the starting salt indicated some of these elements. The results of the SEM phase-morphological analysis of synthesized 2-line and 6-line ferrhydrite are shown Figs. 1 and 2 respectively.

The Point of Zero Charge (pzc) of the 2-line and 6-line ferrihydrite by potentiometric method were found to be 7.96 and 6.20 respectively which are within (5.77 and 7.97) values reported for ferrihydrite in literature (Rhoton and Bigham, 2005, Brinza et al., 2008). Comparative values obtained for the same samples by mass titration were between 6.1-6.5 range for the different ionic strengths (0.1, 0.01, 0.001 M) but after acid adjustment, the pzc increased proportionately to the range 6.4-6.9. After alkaline adjustment, the pzc was observed at pH 8.2. The variable pzc obtained from mass titration is often affected by the nature of contaminants such as basic and acidic elements in the sample (Preocanin and Kallay, 1998). The potentiometric method may therefore be preferred because it is free from the interference of acidic/basic contaminants. At pH below this pzc value, the acidic water donates more protons than hydroxide groups and so the termite hill soil will have a positive surface charge characteristic and will therefore electrostatically repel cations and attract anions to its surface. Conversely, above pzc, the surface charge characteristic will be negative (Abdus-Salam and Adekola, 2005). The presence of ionic species or complexing agents in the reaction medium may change this adsorption pattern by conferring a net negative surface charge at pH below pzc value. This explains the adsorption of metal or hydrated metal ions onto surfaces at pH lower than its pzc value.

**Sorption Capacity of Ferrihydrite** Figs. 3 represents the plot of amount of Pb(II) adsorbed against equilibrium concentrations for 2-line and 6-line FHY. In each case, a two steps process was involved corresponding to a fast followed by a slow adsorption. As the initial concentration increases the amount adsorbed increases proportionally until a plateau was reached, where there was no corresponding rise in the amount adsorbed. The plateau was reached when the favourable sites with lower adsorption energies had been completely filled leaving the unfavourable sites more difficult to access. This is an indication of surface saturation or a monolayer adsorption. As the available sites get saturated, adsorption becomes slower due partly to common ion repulsion effect or differential in the adsorption energy of the available binding sites (Rhoton and Bigham 2005). The second rise may be due to multi-layer adsorption on the ferrihydrite.

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The 2-line ferrihydrite sorption capacity for lead was 127mg/g while it was 82mg/g for 6-line ferrihydrite.

**Adsorption Isotherms:** The adsorption data obtained from the adsorption of Pb(II) experiment were tested for fitness of data against three common adsorption equations, Langmuir, Freundlich and Temkin adsorption isotherms, respectively. Fig. 6 is a Langmuir adsorption isotherm for the sorption of Pb(II) by 2-line ferrihydrite. The adsorption isotherm data as analyzed from Fig. 6 are 0.017, 1.72 and 0.768 for b, Qₘ and the regression coefficient R² respectively. The Langmuir adsorption constants for 6-line ferrihydrite as obtained from Fig. 7 are 0.02, 1.99 and 0.911 for b, Qₘ and the regression coefficient R² respectively. The observed b value (b <<1) shows that the Langmuir isotherm fitted the adsorption data for Pb(II) fairly well. The favourability of this adsorption process was subjected to the equation of separation factor Rᵣ (Mohanty et al., 2006) given as:

$$Rᵣ = \frac{1}{1 + bCᵢ}$$ (9)

where b = Langmuir equilibrium constant (K), Cᵢ = Initial concentration

For a favourable adsorption, 0 < Rᵣ < 1, while for an unfavourable adsorption, Rᵣ > 1 and when Rᵣ = 0, adsorption is linear and irreversible. The Rᵣ values obtained for 2-line and 6-line 0.073 and 0.063 respectively indicate that the adsorption process was favourable.

The substitution of the adsorption data obtained for 2-line and 6-line ferrihydrite into Freundlich isotherm (eq. 3), gave very poor plots (Figs 8 and 9) with the regression coefficient R² = 0.4564 and 0.0138 respectively. The test of adsorption data on eq. 4 yielded poor graphs (Figs 10 and 11) with regression coefficient R² = 0.339 and 0.0316 for 2-line and 6-line ferrihydrite respectively. When the R² values are compared, the data fitted into Eq. 2 only but fitted poorly into eqs 3 and 4. Since Langmuir is physical and mono-layer adsorption process, the possibility of multi-layer and chemo-absorption may be ruled out because of the poor fitness into Eqs 3 and 4.

**Conclusion:** The spectroscopic data obtained for 2-line and 6-line ferrihydrite showed that the sample synthesized were actually ferrihydrite. The
percentage Fe was 95.20% and 93.42% for 2-line and 6-line ferrihydrite respectively. The XRD gave characteristic peaks for 2-line and 6-line ferrihydrite at 29.20° and 28.00° respectively. The morphology of 2-line was hexagonal-monoclinic structure while that of 6-line was cubic-monoclinic system.

Ferrihydrite is environmentally friendly; it can remove heavy metal from wastewater. It is therefore recommended that further research be done carried out on samples of Ferrihydrite synthesized to confirm the degree of purity and large scale environmental application in area of waste management.

The experimental results of Pb(II) adsorption were analyzed using Langmuir, Freundlich, and Temkin isotherms. Only Langmuir isotherm fitted the measured sorption data well. The constants obtained from the Langmuir model are 0.017, 1.72 and 0.768 for b, Qm and the regression coefficient R² respectively; while the 6-line ferrihydrite are 0.02, 1.99 and 0.911 for b, Qm and the regression coefficient R² respectively.

### Table 1 Physical properties of 2-line an 6-line ferrihydrite

<table>
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<tr>
<th>SN</th>
<th>PROPERTIES</th>
<th>2-LINE FERRIHYDRITE</th>
<th>6-LINE FERRIHYDRITE</th>
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<tbody>
<tr>
<td>1</td>
<td>Colour</td>
<td>Brown</td>
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<tr>
<td>2</td>
<td>Texture</td>
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<td>Fine</td>
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<tr>
<td>3</td>
<td>% Yield</td>
<td>60.90%</td>
<td>77.2%</td>
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<tr>
<td>4</td>
<td>% by weight of Fe</td>
<td>95.20%</td>
<td>93.42%</td>
</tr>
<tr>
<td>5</td>
<td>pH</td>
<td>7.96</td>
<td>6.20</td>
</tr>
<tr>
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### Table 2: XRF Result of 2-Line Ferrihydrite

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<th>Si</th>
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<th>Cl</th>
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<th>Zn</th>
<th>Ga</th>
<th>Rh</th>
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<td>300</td>
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<tr>
<td>Conc. (%)</td>
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<td>0.89</td>
<td>0.01</td>
<td>0.047</td>
<td>0.11</td>
<td>95.20</td>
<td>0.37</td>
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<td>0.28</td>
<td>0.30</td>
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### Table 3: XRF Result of 6-Line Ferrihydrite

<table>
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<tr>
<td>Conc. (%)</td>
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![Fig.1: SEM of 2-line ferrihydrite](image1)

![Fig.2: SEM of 6-line ferrihydrite](image2)

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Fig. 3: Determination of equilibrium concentration

Fig. 4: Influence of time on adsorption of Pb(II) on 2-line and 6-line Fenhydrite

Fig. 5: Effect of pH on adsorption of Pb(II) on 2-line and 6-line Fenhydrite

Fig. 6: Langmuir isotherm for adsorption of Pb(II) on 2-line Fenhydrite

Fig. 7: Langmuir isotherm for adsorption of Pb(II) on 6-line Fenhydrite

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