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## Determination of the Zero Point Charge (PZC) and the Isoelectric Point (IEP) of the Iron Hydroxide

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### ABSTRACT

Throughout this work, we have studied the surface chemistry of iron ferric hydroxide  $Fe(OH)_3$  by the potentiometric method, allowing the monitoring of the pH evolution of colloidal suspensions as a function of contact time,  $pH=f(Tct)$ .

Indeed, two techniques were chosen, namely, the variable mass potentiometric titration (PMT) and variable time potentiometric titration (PTT). This study shows the values of the two parameters  $PZC=7.45$  and  $IEP=7.65$

**Keywords:** Iron hydroxide, potentiometric, adsorbent, PZC, IEP

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### INTRODUCTION

Pollutants introduced in the environment and particularly in water, are of an organic nature, mainly from industrial waste water [1,2] used liquid of domestic [3], agricultural [4-6] or hospital origin, and inorganic such as copper, zinc and cobalt. However, other elements, such as mercury, lead, cadmium and chromium are toxic and therefore undesirable.

The environmental awareness requires reducing the impact of these liquid discharges on the quality of the water, the amount of suspended solids and their heavy metal content.

For this purpose, numerous methods are employed, such as precipitation [7], ion exchange [8], solvent extraction [9,10] and adsorption [11,12]. Among these methods, adsorption is one of the most used processes to reduce the content of trace elements from different sources of pollution [13,14]. This process is carried out on various conventional and non-conventional adsorbents, such as silica, zeolites, coal and iron hydroxide [15]. Iron hydroxide recognized by its specific surface area of order  $100\text{ m}^2/\text{g}$  [16] and its high adsorption capacity of traces metals [17].

The physicochemical properties of these materials, in this case the iron hydroxide, as well as their surface chemistry are examined by the potentiometric titration method, namely the variable mass potentiometric titration (PMT) and variable time potentiometric titration (PTT) [18,19].

Also, the control of the surface interaction of this hydroxide with the organic and inorganic pollutants to be able to optimize its use in the water depollution imposes a surface study of this adsorbent, which consists of determining its physicochemical parameters of surface, to namely, the point of zero charge (PZC) and the isoelectric point (IEP) [20]. Hence the object of this study.

### MATERIAL AND METHODS

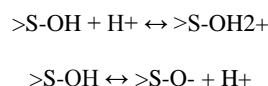
#### Preparation of solutions

All the chemicals used are of analytical quality and have not undergone any prior treatment. The colloidal suspensions of iron hydroxide are prepared by rapid addition of NaOH (0.05 M) to acid solutions of iron (III). The solutions used correspond to 20 g, 40 g and 60 g iron (III) purliners per liter of solutions.

The surface reactivity measured by the ion exchange or the adsorption of organic or inorganic species by iron hydroxide depends, in addition to the PZC and IEP parameters, on the hydration time or contact time.

To determine the value of the point of zero charge (PZC) and that of the isoelectric point (IEP), we used the conventional potentiometric technique. It allows following the evolution of the pH of the colloidal suspensions as a function of the contact time. These solutions are obtained by the dissolution of different masses of iron chloride ( $m=0.5$  g, 1 g and 1.5 g), ie 20 g/L, 40 g/L and 60 g/L of iron (III) in 25 ml of NaOH (0.05 M). The contact time varied between 15 minutes and 72 hours for different initial pH (pHi between 3 and 11).

The adjustment of pHi was done by adding NaOH (0.05 M) or HCl (0.05 M). The reactions protonation-deprotonation involved in acid-base behavior of the substrate are performed by the following reactions:

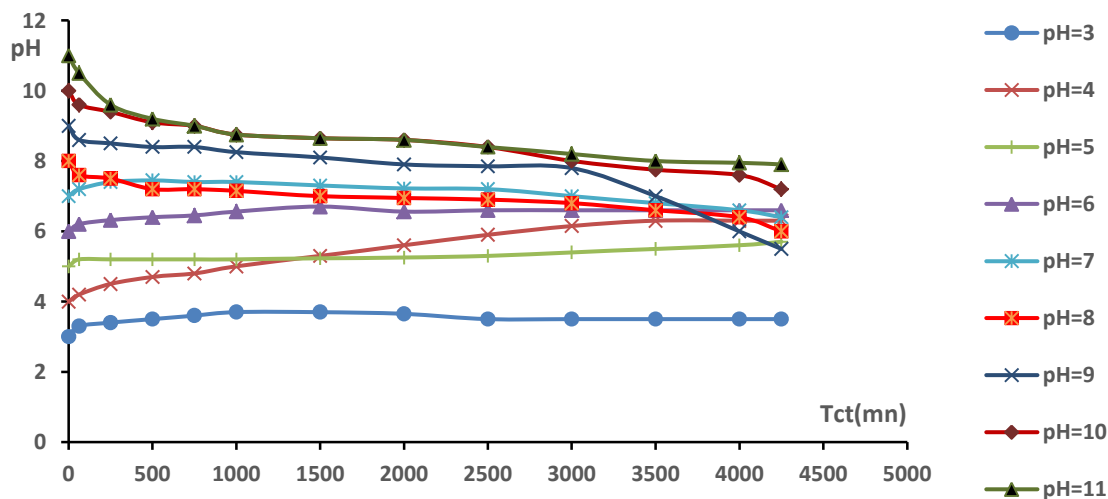


The determination of the constants of acidity of a material can be realized by modeling potentiometric titration curves of this adsorbent.

## RESULTS AND DISCUSSION

### Study of variation $pH=f(Tct)$

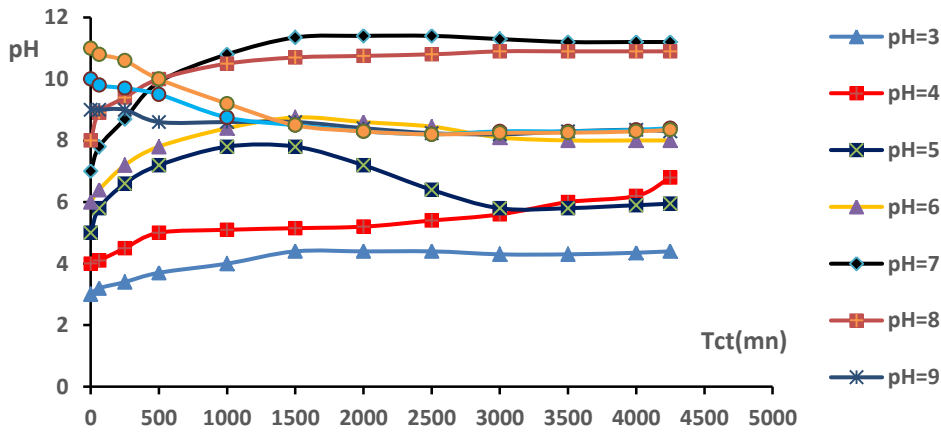
We performed at different initial pH (pHi), monitoring the pH evolution of colloidal iron suspensions of 20 g/L, 40 g/L and 60 g/L as a function of contact time (Tct). The volumes of the examined solutions are 25 ml. The results obtained are illustrated in (Figures 1-3).



**Figure 1:** Variation in  $pH=f(Tct)$  of a suspension of iron hydroxide 20g/L at  $pH=(3, 4, 5, 6, 7, 8, 9, 10, 11)$

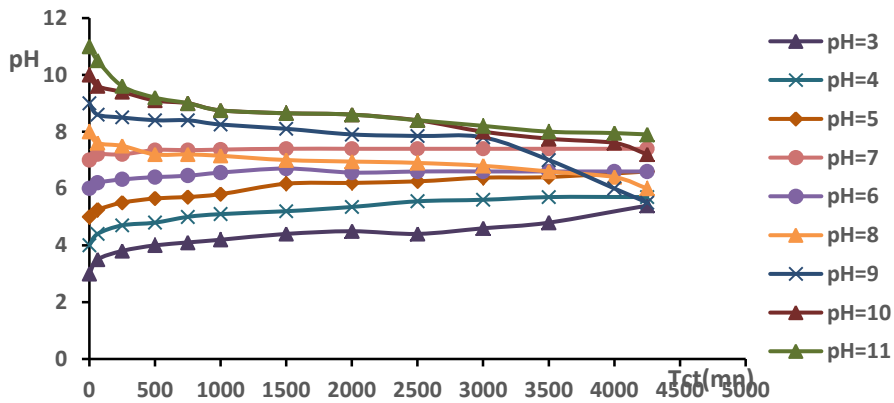
Analysis of the Figure 1, shows that the pH of all the solutions varies as a function of the contact time. Indeed, for the pHi range between 2 and 7, the pH increases slightly to stabilize after 2 to 3 hours, while for pHi greater than 7 it decreases significantly. This change is generally due to the exchange of the  $H^+$  and  $OH^-$  ions with the active functional sites  $>S-OH$  present on the surface of the adsorbent.

Study of the surface chemistry of iron hydroxide



**Figure 2:** Variation in  $pH=f(Tct)$  of a suspension of iron hydroxide 40g/L, at  $pHi=(3, 4, 5, 6, 7, 8, 9, 10, 11)$

It follows from the examination of the Figure 2, that the pH also changes with the contact time so that it increases notably for the interval  $2 < pHi < 8$  and decreases beyond this interval. This change is generally due to the exchange of the  $H^+$  and  $OH^-$  ions with the active functional sites  $>S-OH$  present on the surface of the adsorbent.



**Figure 3:** Variation in  $pH=f(Tct)$  of a suspension of iron hydroxide 20g/L at  $pHi=(3, 4, 5, 6, 7, 8, 9, 10, 11)$

The same conclusions drawn from the analysis of Figures 1 and 2, in terms of pH variations, are elucidated in Figure 3. This implies that the same phenomenon occurs regardless of the sorbent content.

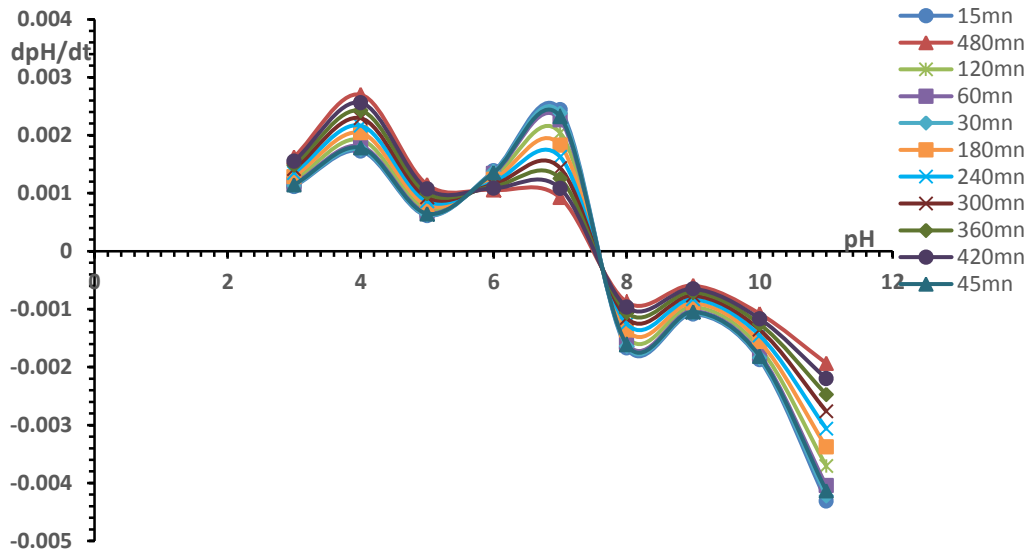
In all cases, the results indicate a change in pH during the first 200 minutes, to stabilize after 500 minutes. This change is generally due to the exchange of the  $H^+$  and  $OH^-$  ions with the active functional sites  $>S-OH$  present on the surface of the adsorbent.

Indeed, when  $pHi$  is between 6.5 and 8, the variation of acidity of the suspension becomes less significant. This result is due to effect of the surface charge, which contributes to the stability of the pH, when it is nearby of zero. So that, we can estimate at first that the values of PZC and IEP of the studied adsorbent are between 6,5 and 8. These results are comparable to the bibliographical data [17]. However, these results seem insufficient and need more and more precision. In this objective the method for mesasuring  $pH=f(tct)$  must be coupled with another technique in order to achieve accurate values of PZC and IEP. The method used for this purpose, is the derivative of the function  $pH=f(Tct)$ .

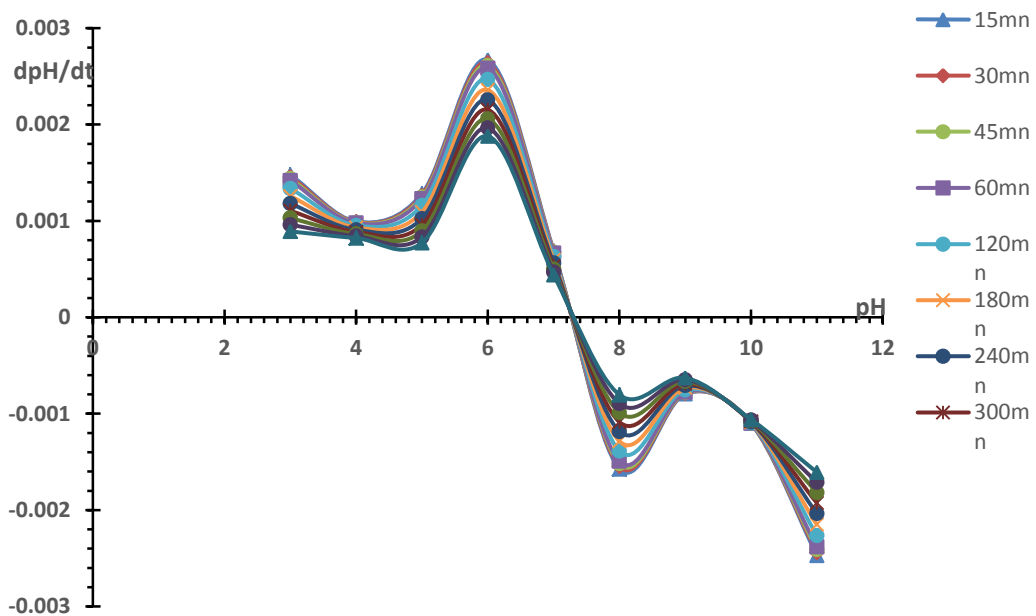
The parameters, PZC and IEP can be obtained only from the derivative of the fonction  $pH=f(Tct)$ . This is why the titration curves are smoothed by polynomial equations.

**Study of variation  $dpH/dTct=f(pHi)$**

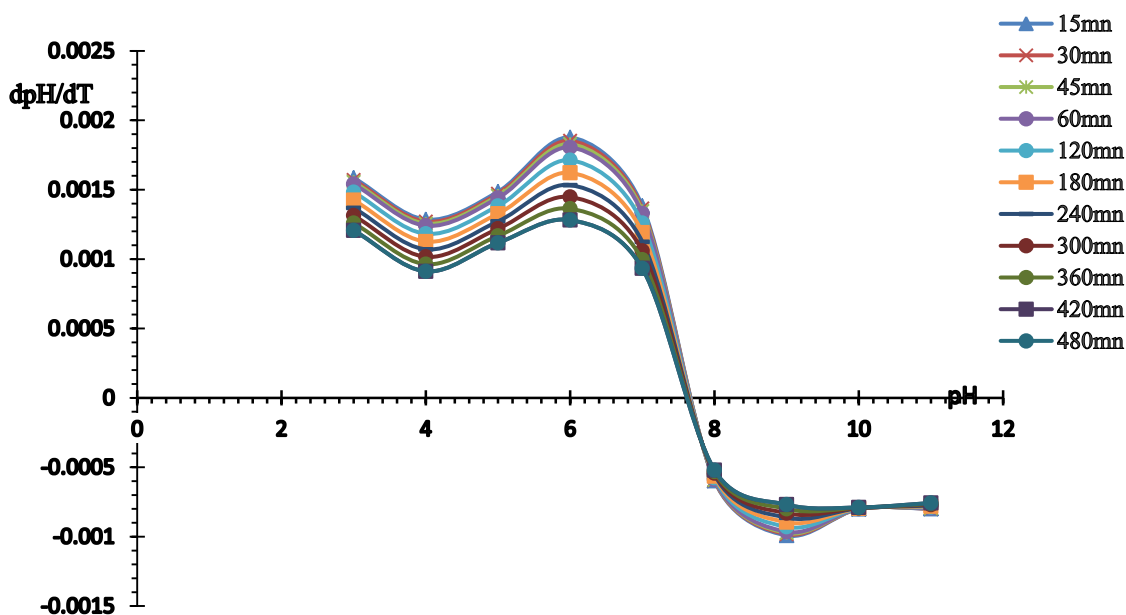
The Figures 4-6, present the variations  $dpH/dTct=f(pHi)$ , for a substrate suspension of 20 g/L, 40 g/L and 60 g/L.



**Figure 4:** Variations  $(dpH/dTct)=f(pHi)$  obtained for a substrate suspension of 20 g/L.



**Figure 5:** Variations  $(dpH/dTct)=f(pHi)$  obtained for a substrate suspension of 40 g/L.



**Figure 6:** Variations  $(dpH/dT) = f(pH_i)$  obtained for a substrate suspension of 60 g/L.

The PZC is identified as being the point of intersection of the curves with the  $pH_i$  axis corresponding to  $dpH/dT = 0$ , while the IEP point is the point of intersection of these curves with each other.

In-depth analysis of curves 4, 5 and 6 highlights the value of PZC and IEP:

\*For a substrate of 20 g/l (Figure 1). IEP=7.8 and  $7.4 < PZC < 7.7$ ; the difference between the PZC and IEP values is due to a slight adsorption of  $Cl^-$  chloride ions.

\*For a substrate of 40 g/l (Figure 5). IEP=PZC=7.4

\*For a substrate of 60 g/l (Figure 6). IEP=7.8 and  $7.4 < PZC < 7.5$ ; the difference between the PZC and IEP values is due to a slight adsorption of chloride ions  $Cl^-$ .

Taking into account the results obtained, the values of PZC and IEP are such that; PZC=7.45 and IEP=7.65. These values are in agreement with literature data [21].

## CONCLUSION

In this study, we have process to determine the zero charge point of iron hydroxide as adsorbent as of the metals in the state of traces. Therefore, the technical monitoring pH changes biosorbent of suspensions as a function of hydration time  $pH_i = f(Tct)$  indicates that the values of PZC and IEP are between 6.5 and 8. For more precision, the first order derivative curve  $pH_i = f(Tct)$  smoothed by polynomial equations allowed to calculate the PZC=7.45 and IEP=7.65.

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