

**Research Article** 

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# Iron Removal by Atmospheric Air Oxydation and Adsorption on Iron Oxyhydroxide Tartar Deposited in Dan Daji -Illela Water Distribution Pipes (Tahoua-Niger Region)

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# Abstract

Groundwater in certain regions of Niger, notably the Illéla department in Tahoua region, is rich in iron. The use of these iron-rich waters could have consequences such as degradation of organoleptic quality (taste, odour, flavour), and damage to water distribution and storage equipment. As a results, the elimination of iron in the form of  $Fe^{2}$ + remains a major challenge. To this end, we have set ourselves the objective of applying a simple, effective and less costly treatment method for iron removal from Dan Daji drilling water. For these reasons, we have chosen to associate oxidation by simple agitation in atmospheric air with adsorption on iron oxyhydroxide tartar recovered from Dan Daji water distribution pipes. Experiments have shown that agitation in air makes the basic medium very conducive to iron oxidation and adsorption. IR and SEM analysis of the collected solid showed it to be amorphous. The results obtained proved that the adsorbent capacity of recovered pipe tartar is an excellent iron adsorbent. The rate of iron elimination is around 71.30% after 2min of agitation, for a 0.1g seed of iron oxyhydroxides. From 0.25g the removal rate is 100% after 2 minutes of reaction.

# Keywords: Elimination, oxidation-adsorption, iron, water, Dan Dadji.

#### 1. Introduction

Iron is the fourth element in the earth's crust, the second most abundant metal in the earth [1, 2, 3], and is mainly present in groundwater, but also in surface water. The presence of iron in natural sources is attributable to the decomposition of rocks and minerals, acid mine drainage water, landfill leachate, sewage effluent and discharges from iron-processing industries [4, 5]. Iron overload in the human body can lead to primary hemochromatosis (poor regulation of iron absorption by the intestine) and even hepatic carcinogenesis (risk of liver cancer). These disorders



generally occur when the concentration of iron in water exceeds 10 mg/l [5, 6, 7]. At lower concentrations, the problems associated with iron in drinking water are aesthetic. Indeed, the presence of this element leads to degradation of water quality, corrosion and clogging of water storage and piping systems [4, 8, 9, 10]. In addition, iron precipitation favors the growth of bacteria, which can cause reddish discoloration [11, 12]. The treatment of iron in water is the subject of numerous studies [13, 14, 15, 16, 17, 18]. Several treatment methods have been developed, the most common of which are chemical oxidation using oxidants of varying strengths (chlorine, potassium permanganate, oxygen and ozone), biological processes involving micro-organisms, catalytic processes based on adsorption and oxidation on the surface of a specific material, and membrane processes [2, 7, 13, 14]. These treatment methods are not without their limitations, both in terms of the use of certain materials and the control of certain physico-chemical parameters, and above all in terms of the accessibility of the necessary materials and chemicals. As part of a study to determine the optimum conditions for massive Fe<sup>2+</sup> removal. We have combined oxidation by simple agitation in atmospheric air and adsorption on iron oxyhydroxide tartar deposited in the water distribution pipes of Dan Daji -Illéla. The choice of combining these two methods is based not only on cost /efficiency, accessibility and ease of manipulation, but also on the fact that the mechanism of iron hydroxide (Fe(OH)<sub>3</sub>) formation could be attributable to two chemical processes, namely the oxidation-precipitation of iron during water transport and the adsorption of iron by the iron oxide that sediments in the form of lime.

# 2. Materials And Methods

### Presentation of Dan Daji water pipe tartar



Figure 1: Photo of iron tartar in the Dan Daji -Illéla water distribution pipes.

#### **Preliminary Tartar Treatment**

Tartar samples dug from the water pipe were crushed, then ground using a jaw crusher and sieved using a 200  $\mu$ m sieve. This step eliminates a number of impurities and reduces the sample to millimetric fragments.

#### **Tartar Characterization**

The solid-state analysis methods used in this study are Scanning Electron Microscopy (SEM) and Infrared Spectroscopy (IR). Analysis was carried out before and after each seeding. SEM was obtained using a JEOL J.S.M. 5350 LV. Infrared spectra were taken using a Perkin-Elmer FTIR 13600 spectrometer between (4000 and 200 nm). The method used is that of potassium bromide tablets. The tablet is prepared by mixing 2 mg of tartar with 400 mg of KBr.

#### **Description of the Experimental Method**

All precipitation reactions were carried out at ambient temperature. In a beaker containing 1L of Dan Dadji's water. The initial pH was set at 7.325, the pH of Dan Daji water in its natural environment by stirring in CO<sub>2</sub>. Tartar quantities corresponding to 0g; and 0.1g; 0.25g and 0.5g were used. Tartar enrichment was carried out at time t = 0 min before starting the timer and stirrer for 60 minutes, which is the cut-off time for all reactions.

Mechanism of Iron Oxyhydroxide Formation



The formation of iron hydroxide by aeration involves introducing atmospheric oxygen into the solution by stirring. Contact of the solution with  $CO_2$  poor air enables the dissolved  $CO_2$  to be degassed, resulting in alkalinization of the medium. This increase in pH favors the oxidation of Fe<sup>2</sup>+ to Fe<sup>3</sup>+ and the precipitation of Fe (OH)<sub>3</sub>. The global precipitation reaction is written as follows:

$$\operatorname{Fe}^{2+}_{(\operatorname{aq})} + 2\operatorname{HCO}_{3}_{(\operatorname{aq})} + \frac{1}{4}\operatorname{O}_{2}_{(g)} + \frac{1}{2}\operatorname{H}_{2}\operatorname{O}_{(l)} \leftrightarrow \operatorname{Fe}(\operatorname{OH})_{3(s)} + 2\operatorname{CO}_{2} \uparrow (\operatorname{R}_{1})$$

It should be remembered that the iron hydroxide formed according to Reaction  $R_1$  is a metastable phase which evolves towards a more thermodynamically stable iron oxyhydroxide according to the following reaction [19]:

$$Fe(OH)_3 \rightarrow FeOOH + H_2O$$
 (R<sub>2</sub>)

### Calculation of iron elimination rate

The iron removal rate,  $\tau$ (Fe), is calculated from the following equation:

$$\tau_{(Fe)} = \frac{\left[\operatorname{Fe}\right]_{0} - \left[\operatorname{Fe}\right]_{t}}{\left[\operatorname{Fe}\right]_{0}} \times 100$$

With:

[Fe]0 = The initial ionic iron concentration in mg/L [Fe]t = Total iron concentration at time t in mg/L

#### 3. Results And Discussion

#### IR spectra of tartars recovered before and after enrichment

The figures below show the IR spectra before and after each enrichment.







Figure 4 : IR spectra of tartar after enrichment of 0. 25g



**Figure 3 :** IR spectra of tartar after enrichment of 0.1g



**Figure 5** : IR spectra of tartar after enrichment of 0. 5g

IR spectra of the solids recovered before (figure 2) and after (figures 3, 4 and 5) seeding show virtually identical bands characterizing a mixture of iron oxyhydroxides. Bands at 640, 1400, 1640 and 3000 cm<sup>-1</sup> characterize the presence of goethite ( $\alpha$ -FeOOH). Bands at 750 and 1020 cm<sup>-1</sup> indicate the presence of lepidocrocite ( $\gamma$ -FeOOH). The band at 465 cm<sup>-1</sup> characterizes the presence of ferrihydrite Fe5HO<sub>8</sub>.4H<sub>2</sub>O. This confirms that the red coloration of the various tartars recovered is associated with iron oxyhydroxide. These findings corroborate those of Wided. M, 2017 who showed that the solids recovered from the surface of drippers and at the pumping station at Dar Chichou



(Menzel Temim-Tunisia), are mainly precipitates of iron oxides or oxyhydroxides. In addition Wided. M, 2017 also asserted that the red coloration of the various tartars recovered is associated with Iron Oxyhydroxide [20].

#### SEM of recovered tartar before and after enrichment.

The following figures show SEM photos before and after each enrichment.



Figure 6 : SEM of tartar before enrichment



Figure 7 : SEM of tartar after enrichment of 0, 1g



Figure 8 : SEM of tartar after enrichment of 0, 25g



Figure 9 : SEM of tartar after enrichment of 0, 5g

The SEM photos shown in the figures above confirm that the tartar recovered from the Dan Daji site (figure 6) and the solids collected after the enrichment experiments (figures 7, 8 and 9), are all amorphous and similar solids. These results show that adsorption of iron oxyhydroxides on limescale is cyclic. As a result, the adsorbents can be reused for future applications. This affirmation has been supported by other authors such as Fernand K, Lanciné GD, Touchard AJ, (2008) and Gani Hichem Mohamed Cherif; Khelifi Iheb, (2021) [6, 19].

# Study of the influence of enrichment quantity

Figures 10-A and 10-B show, respectively, the variation curves for iron concentration and pH as a function of time at different enrichment quantities.



*Figure 10*: Changes in  $Fe^{2+}$  concentration and pH as a function of time at different enrichment quantities.

Fig. 10-A shows that in the absence of iron oxyhydroxide enrichments, the iron(II) concentration remains virtually constant throughout the experiment. On the other hand, a drop in iron content is observed after around 2 minutes of reaction when higher quantities of iron oxyhydroxides are introduced. Analysis of the curves in Figure 10-A shows that Fe(II) removal is greater when the amount of iron oxyhydroxide enrichment is also greater. In fact, with 0.1g iron oxyhydroxide enrichment, 71.30% of iron was eliminated in less than 2 minutes. Iron elimination is complete with 0.25g enrichment. These results allow us to confirm that enrichment with a quantity of iron oxyhydroxides leads to massive elimination of iron from the water. These results are in agreement with those obtained from many other studies. Charles, (2011) showed that ferric hydroxide is a highly effective adsorbent for metal cations in aqueous solution, particularly ferrous ions [3]. In 1981, B. Mark M. Benjamen and James O. Leckie studied the competitive adsorption of Cd, Cu, Pb and Zn on amorphous iron oxyhydroxide (Fe<sub>2</sub>O<sub>3</sub>. H<sub>2</sub>O) in systems containing pairs of metal adsorbates. They have clearly demonstrated the performance of Oxyhydroxide on the adsorption of these metals [21]. Miller and Orbock Miller (2007) stated that the oxygen atom of Fe(OH)3 plays the role of a LEWIS base towards a transition metal, which constitutes a LEWIS acid, explaining the immobilization of metals by ferric hydroxides through adsorption [22]. Examination of Figure 10-B shows that, in the absence of oxyhydroxide enrichment, the evolution of pH remains practically constant throughout the experiment. However, a pH drop from 7.325 to 6.420 is observed before the pH starts to rise again. The drop in pH may be linked to the conversion of  $Fe_{3+}$  to  $Fe(OH)_{2+}$  (reaction R3), which takes place in the acidic medium (pH below 6). This phenomenon has been reported by other authors [23,24].

$$\operatorname{Fe}^{3+} + 2\operatorname{H}_2 O \longrightarrow \operatorname{Fe}(OH)_2^+ + 2\operatorname{H}^+(R_3)$$

The rise in pH during the experiments could be attributable to agitation in the atmospheric air. This agitation brings the solution into contact with atmospheric oxygen, degassing dissolved CO2 and rendering the medium alkaline. This alkanization of the medium inhibits dissolution of the solid (iron oxyhydroxide) introduced, confering it a high adsorption capacity. This explains the massive elimination of iron. This allows us to corroborate that adsorption on iron oxyhydroxides takes place at alkaline pH. Figure 10-B shows the curves with almost identical and coincident curves. This figure also shows that when 0.1g, 0.25g and 0.5g iron oxyhydroxides are enrichments, the pH increases over time. Indeed, the pH rises from 7.325 to 7.530 after around 2 minutes of the experiment. The pH continues to rise, reaching a value of pH = 8 after 40 minutes. But increasing the amount of iron oxyhydroxide has little effect on the pH trend. This accords well with the findings of Wided (2017), who showed that increasing Fe(III) hydroxide concentration has no effect on pH above 10mg/L Fe(III) [20].

#### 4. Conclusion

The objective of our study is to remove iron from Dan Daji drilling water by simple air agitation and adsorption on the scale recovered from the water distribution pipes. Our results showed that oxidation by simple agitation takes place at alkaline pH. SEM analysis showed that both the scale recovered from water distribution pipes and the tartar collected after the enrichment experiments were amorphous. A study of the adsorption capacity of the solid recovered from the pipes revealed that it is an excellent iron adsorbent.

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