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Contribution to the Study of the Formation of Iron Oxyhydroxide Tartar Deposited in the Water Distribution Pipes of Dan Daji -Illela (Tahoua-Niger Area): Effect of HCO_3^- Ions

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ABSTRACT

One of the main problems associated due of using Dan Daji water was the massive precipitation of limescale. Transporting the water was a vital operation. The water arrived under pressure and, given the sudden drop in pressure as it left the plant, the dissolved carbon dioxide it contained escaped into the atmosphere, came into contact with the oxygen in the air and caused tartar precipitation. The main objective of this work was to study the influence of bicarbonate ions on the formation of ferruginous tartar deposited in Dan Daji water distribution pipes. To do this, we experimentally examined the effect of the concentration of bicarbonate ions on the precipitation of Fe^{2+} by the method of oxidation in air and degassing of CO_2 under agitation. Changed in Fe(II) concentration were monitored using UV-visible molecular absorption spectrophotometry at a wavelength of 510 nm. The results showed that no iron precipitation was observed in the absence of bicarbonate ions. Fe^{2+} precipitation ratios of 57% and 85% were observed for bicarbonate ion concentrations of 244 mg/L and 488 mg/L respectively. The high scaling behavior of Dan Daji water was due to its high iron and bicarbonate ion concentration. The addition of bicarbonate ions lead to massive precipitation of iron oxyhydroxyde, $Fe(OH)_3$.

1. INTRODUCTION

Niger drunk water supply and sanitation service was among the poorest in the world, due to the country's socio-economic conditions. Niger had only one water treatment plant to drink, located in the capital (Niamey). However, numerous drillings have been carried out in rural areas by water sector projects [1]. Today, some of these drillings are menaced by iron contamination. The inevitable provision of iron-charged water generates a very complicated problem in the distribution canalization, which manifested itself essentially in the formation of tartar (Figure-a). The presence of red iron tartar in water distribution pipes generated red water with a disagreeable taste (Figure I- b). The accumulation of this tartar with time results in the complete obstruction of the water distribution system, or finally the total stoppage of water (Figure I-c). This water shortage obliged the population of this locality to use traditional water sources (wells, ponds and marshes) contaminated by bacteria. This absence of hygiene can result the risk of bacterial diseases such as typhoid fever, bacillary dysentery and cholera, name but the most important. A study was necessary to understand the mechanism by which this type of tartar forms. It was with these ideas that the present study was initiated. The principal causes of iron deposits in natural water could be related to the presence of the following elements: cations (Ca^{2+}, Na^{+}, K^{+}, Mg^{2+}, Fe^{2+}, etc....), anions (Cl^{-}, SO_4^{2-}, CO_3^{2-}, HCO_3^{-}, NO_3^{-}, etc), organic matter (humic acids), dissolved gases (CO_2, O_2, and H_2S)[2]. Among these different dissolved elements, a certain number were susceptible to react with each other or to favor certain reactions, in particular the precipitation of iron oxyhydroxides. This was why we had fixed as an objective to study the influence of bicarbonate ions on the formation of iron oxyhydroxides.

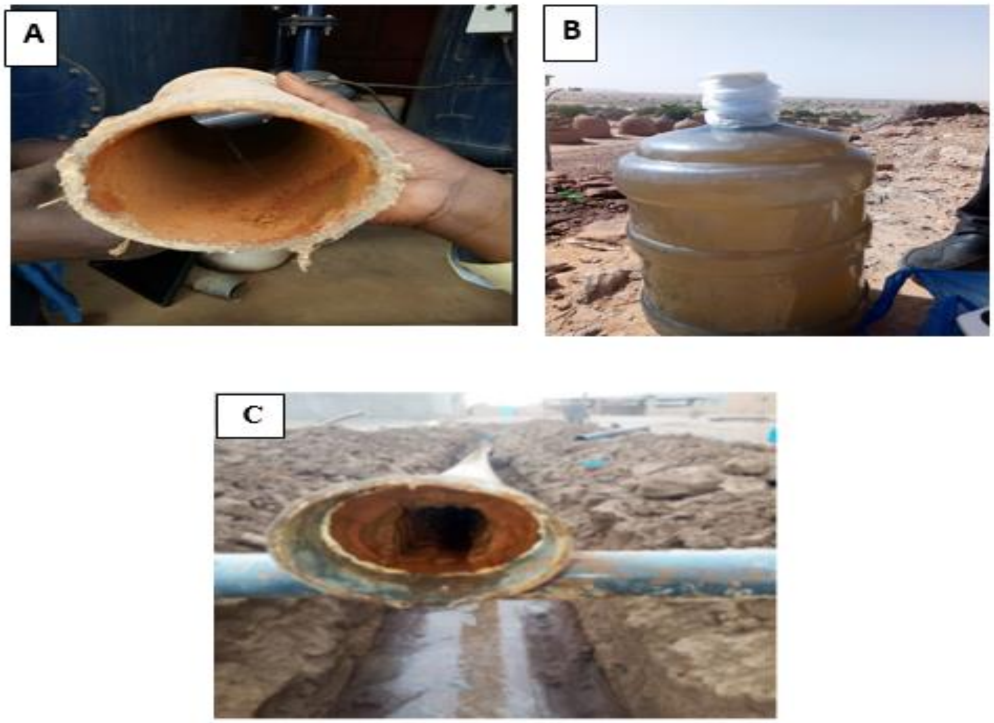

Figure I: Some photos illustrating the consequences of the presence of iron tartar in the Dan Daji -Illela water distribution pipes.

2. MATERIALS AND METHODS

2.1. Water sampling at Dan Daji

The water samples were taken in specially prepared bottles after running in a vacuum for about three minutes. The water samples were immediately stored in a cooler containing ice.

2.2. Physicochemical analysis of Dan Daji water

Measurements of physical parameters such as pH and temperature were taken immediately at the sampling site, using a VWR pH enomenal pH1100H pH meter. Conductivity and the major chemical elements, namely iron (Fe^{2+}), sodium (Na^+), chloride (Cl^-), sulfate (SO_4^{2-}), fluoride (F^-), bicarbonate (HCO_3^-), nitrate (NO_3^-), nitrite (NO_2^-), magnesium (Mg^{2+}), potassium (K^+) and manganese (Mn^{2+}) were analyzed less than 72 hours after sampling, in the laboratory of the geology department of the Abdou Moumouni University in Niamey. These analyses were carried out with a spectrophotometer, used the standard methods described by Rodier et al, 2009 [3].

2.3 Study of the influence of bicarbonate ions on the natural water of Dandaji

The experiments were carried out in a 1000mL beaker placed on a magnetic stirrer. At each step, the pH of the water in the beaker was brought back to pH =7.35 (the pH of the water in its natural environment) by bubbling in CO_2. The stirrer and stopwatch were then started at the same time for 60 minutes. Stirring introduces atmospheric oxygen into the water in the beaker. Contact of the water with the atmospheric air causes the release of dissolved CO_2, which alkalinises the medium and increases the pH. This increase in pH promotes the oxidation of Fe^{2+} to Fe^{3+} and the precipitation of Fe^{3+} to $Fe(OH)_3(s)$ according to the following reactions:

$$4\ Fe^{2+} + O_2 + 2H_2O \rightarrow 4\ Fe^{3+} + 4OH^- \ (R_1)$$

$$4\ Fe^{3+} + 4OH^- + 8H_2O \rightarrow 4\ Fe(OH)_3 + 8H^+ \ (R_2)$$

Four (4) bicarbonate concentrations were studied: $[HCO_3^-]$ = 37.5mg/L, $[HCO_3^-]$ = 122mg/L, $[HCO_3^-]$ = 244mg/L and $[HCO_3^-]$ = 488 mg/L. Of these concentrations, only 37.5mg/L was the concentration of bicarbonate in the natural environment. The bicarbonate concentrations 122mg/L, 244mg/L and 488mg/L were obtained by adding 0.116g, 0.284g and 0.620g of sodium bicarbonate ($NaHCO_3$) respectively to natural water already containing 37.5mg of bicarbonate. After every 10 minutes, a 1ml sample of the water in the beaker was taken and placed in a 50 volumetric flask to determine the residual Fe^{2+} concentration. The 1ml sample was taken using a syringe fitted with a 0.45µm membrane microfilter (Figure II). A pH value was recorded for each sample to monitor changes in pH during the reaction.

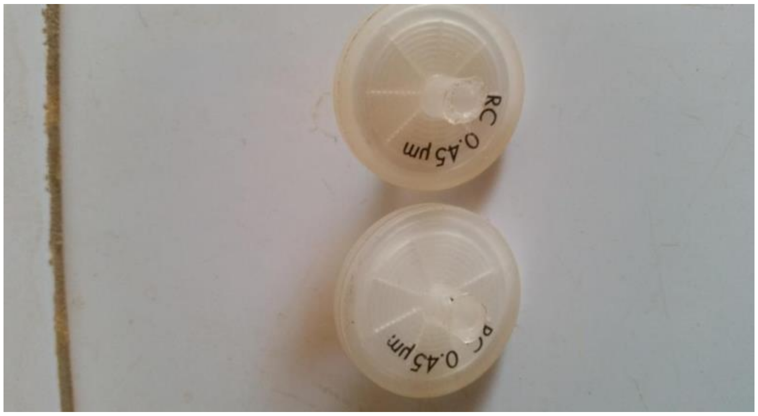

Figure II: Photo of 0.45μm microfilter

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2.4. Dosage of residual Fe^{2+} by UV-visible molecular adsorption spectrophotometer

To 1mL of solution taken, we successively added :

1ml hydroxylamine hydrochloride: This reactant reduces all dissolved Fe^{3+} iron to Fe^{2+} iron and prevents the precipitation of iron hydroxides. The reduction of Fe^{3+} iron ions by hydroxylamine chlorohydrate was given by the following reaction [5]:

$$4\,Fe^{3+} + 2\,NH_2OH \rightarrow 4\,Fe^{2+} + N_2O + 4\,H^+ + H_2O \quad (R_3)$$

2ml ammonium acetate: This reactant is used to keep the environment at a pH of between 3.5 and 5.5 (this pH range was very favorable for the color stability of the iron-orthophenanthroline complex).

2ml orthophenanthroline: This reactant forms a red-orange complex with the ferrous ion (Fe^{2+}), the colour intensity of which obeys Béer-Lambert's law. The complex forms instantly after the reactant added and becomes stable over a long period. It was formed by 3 orthophenanthroline molecules and a Fe^{2+} ion, as shown in the following reaction equation [6]:

$$Fe^{2+} + 3\text{ O-phen} \rightarrow [Fe\text{ (O-phen)}_3]^{2+} \text{ (R4)}$$

After introduced these reactants, the mixture was made up to 50mL with distilled water. The optical density was read using a Zuzi Spectrophotometer Model 4101 at a wavelength of 510 nm.

2.5. Détermination of the precipitation ratio

The iron hydroxide precipitation ratio, τp(Fe), is calculated from the following equation:

$$\tau_{p(Fe)} = \frac{[Fe]_0 - [Fe]_t}{[Fe]_0} \times 100$$

With:

$[Fe]_0$ = The initial ionic iron concentration in mg/L

$[Fe]_t$ = The total concentration of iron at time t in mg/L

2.6. Solid phase characterization techniques

The analytical methods used in this study were X-ray diffraction and scanning electron microscopy. Powder diffractograms of the precipitated solids were produced using a PW 1050/37 diffractometer using a K**α** wavelength anticathode (λ = 1.5418 and 1.5405). The method used was the potassium bromide pastille method. The pastille was prepared by mixing 2 mg of the product with 400 mg of KBr. Scanning Electron Microscopy (SEM) was obtained using a JEOL J.S.M. 5350 LV.

3. RESULTS

3.1 The results of the physico-chemical analyses of the Dan Daji water are presented in Table 1.

Table 1: Physico-chemical parameters of water in the village of Dan Daji

Physico-chemical parameters	Units	Physico-chemical parameter values	Guideline values WHO, 2011
pH	-	7,35	6,5- 8,5
CE	µS/cm	212	250
T	°C	26,5	25
SO_4^{2-}	mg/L	36	250
F^-	mg/L	**0,03**	1, 5
HCO_3^-	mg/L	37,5	ND
NO_3^-	mg/L	1,32	50
NO_2^-	mg/L	0,0099	3
Cl^-	mg/L	1	200
Fe^{2+}	mg/L	**1,69**	**0,3**
Ca^{2+}	mg/L	12	ND
Na^+	mg/L	7,49	200
Mg^{2+}	mg/L	4,35	50
K^+	mg/L	15,93	12
Mn^{2+}	mg/L	1,2	0,4

3.2 Study of the influence of bicarbonate ions on iron precipitation

Figures III-a and III-b showed the variation graphs for iron concentration and pH as a function of time, respectively.

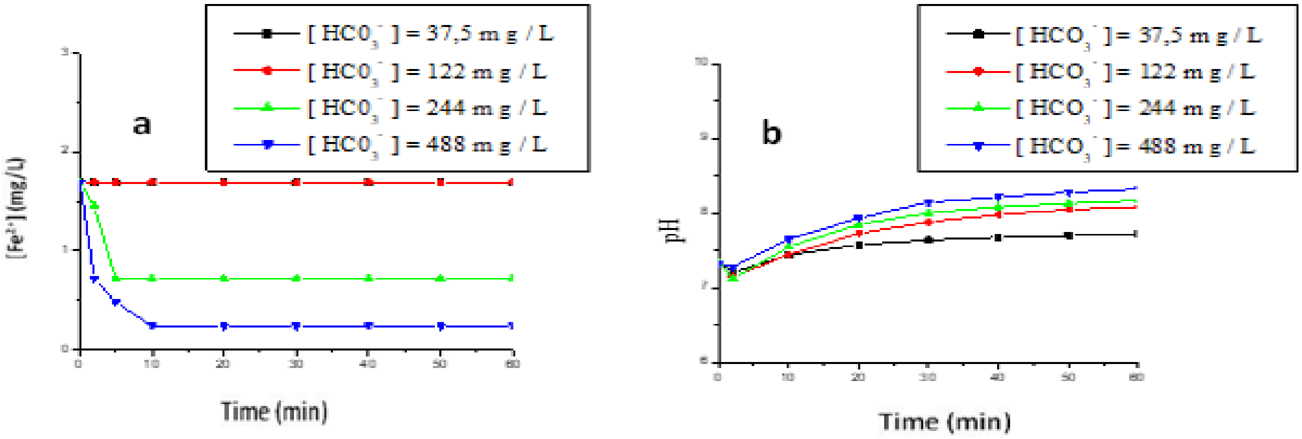

Figure III: Evolution of dissolved iron content (a) and pH (b) during precipitation in the bicarbonate environment at different [HCO_3^-] values.

Figure III-a showed that iron Fe^{2+} precipitation in the carbonated environment under agitation was instantaneous and massive as the bicarbonate concentration increased. For [HCO_3^-] = 37.5 mg/L, precipitation was very low or even non-existent, and the iron concentration [Fe^{2+}] = 1.69 mg/L remained constant throughout the operation. For [HCO_3^-] = 244 mg/L, precipitation is instantaneous and after one minute of precipitation, the iron concentration dropped to [Fe^{2+}] = 1.45 mg/L. After 10 minutes of precipitation, the iron concentration dropped to 0.72 mg/L and remained constant throughout the operation. For [HCO_3^-] = 488 mg/L, precipitation was instantaneous, after one minute of precipitation, the iron concentration dropped to [Fe^{2+}] = 0.72 mg/L and after 20 minutes of precipitation, the iron concentration dropped to 0.24 mg/L and remained constant until the end of the experiment. The precipitation rates corresponding to the drop in iron content at the different bicarbonate concentrations are recorded in Table 2. Figure III-b showed a drop in pH after just 2 minutes with all bicarbonate concentrations, followed by a gradual rise in pH.

Table 2: Influence of [HCO_3^-] on precipitation pH (pHp), precipitation rate τp(Fe)

Concentration en mg/L	tp (min)	pHp	$\tau_{p(Fe)}$ en%
[HCO_3^-] = 37,5	10	7,48	-
[HCO_3^-] = 122	10	7,48	-
[HCO_3^-] = 244	10	7, 52	57%
[HCO_3^-] = 488	10	7,70	80%

Table 2 shows an increase in pH at all concentrations during the precipitation tests. In addition, from [HCO_3^-] = 244 mg/L, the precipitation pH increases progressively with the bicarbonate concentration.

Bicarbonate influences the precipitation rate (τp(Fe)). Indeed, for bicarbonate concentrations equal to 37.5 mg/L and 122 mg/l precipitation did not observe. The τp(Fe) was practically zero despite the change in pH from 7.35 to 7.48. However, for bicarbonate concentrations equal to 244 mg/l and 488 mg/l, precipitation was observed. The τp(Fe) is 57% and 80%, respectively.

3.3. X-ray diffraction of deposit tartar

The red deposit extracted from the Dan Daji water distribution installations was analyzed by XRD. The results obtained are shown in Figure IV below:

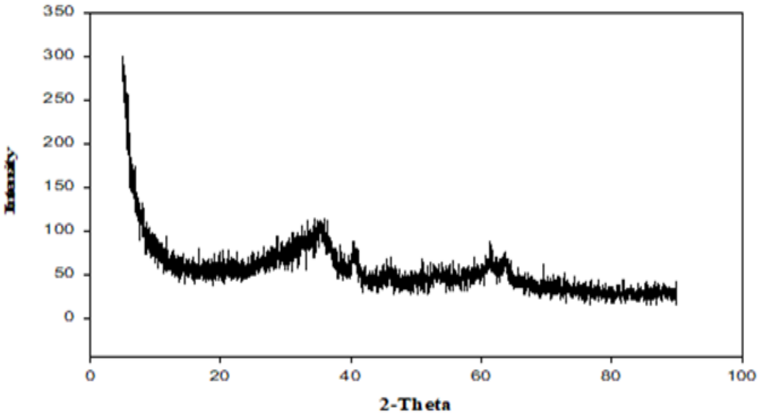

Figure IV: X-ray diffractograms of the solid recovered from the Dan Daji -Illela water distribution pipe.

3.4. SEM of deposit tartar

To confirm the exact nature of the precipitate, we carried out additional analyses using Scanning Electron Microscopy (SEM) (Figure V-2).

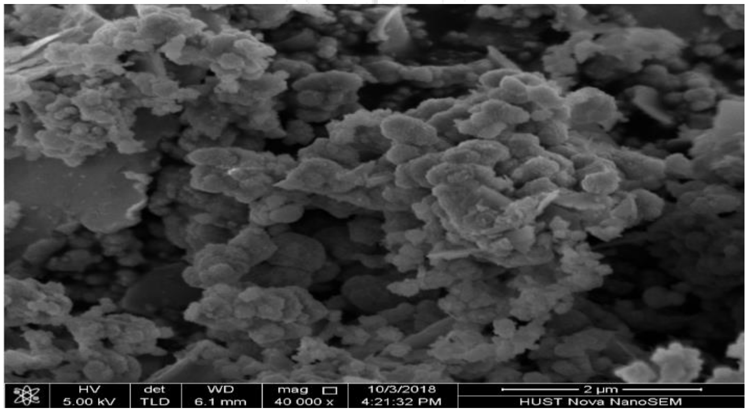

Figure V: SEM of tartar in the water distribution pipe from the Dan Daji Illéla drilling.

4. DISCUSSION

4.1. Physico-chemical analyses

A study of the physico-chemical parameters of Dan Daji-Illela's water showed that, on in whole, the values conformed to the guide values recommended by the WHO (WHO, 2011) [7] for human drunk water. However, an iron content of 1.69 mg/L was recorded, well above the WHO standard. This value of iron concentration in the present study was in accord with those found by Haoua et al. (2014) in certain localities in the Tahoua area in Niger [8]. The high iron content may be related to the nature of the aquifer in this locality, which was of the Terminal continental type (CT1) (Maxime Monfort, 1997) [9]. The lithology of CT1 was characterized by ferruginous oolitic formations. These results were similar to other works on groundwater such as Greigert et al, 1966, Haoua A et al, 2014, Rabilou et al, 2018[3, 10, 11] who reported that the continental terminal (CT1) aquifers were highly loaded with iron. One of the most important elements in Dan Dadji's water was potassium, with a concentration of 15.93 mg/L above the WHO guide value of 12 mg/L. Potassium was present in water in the form of potassium ions (K^+). Potassium is essential for plant, animal and human nutrition [12]. At the last, potassium ions play an essential role in many vital cell functions, such as metabolism, growth, repair and volume regulation, as well as in the electrical properties of cells. Harmful effects linked to higher than-normal concentrations of K^+ in the plasma (hyperkalaemia) may occur in certain segments of the population when these individuals consume drunk water from water softeners that use potassium chloride. The people most at risk were the people suffered from renal disease or other conditions (heart disease, coronary artery disease, hypertension, diabetes) and who took medicaments that inhibit the normal potassium-depended functions of the body. Harmful effects can also be produced when plasma K^+ concentrations were lower (hypokalaemia) than normal. Hyperkalaemia and hypokalaemia were all produced by perturbations in transcellular homeostasis or renal regulation of K^+ excretion [13]. K^+ ions are introduced into the nappe by the mechanisms of alteration of minerals containing potassium, such as orthoclase [11]. This water had a pH of 7.35. This pH showed that the water studied did not aggressive. In another aspect, this pH indicated that the water came from the terminal continental nappe (CT1). This characterized by a pH that was generally neutral to slightly basic, according to several authors such as Maxime, 1997, Haoua et al, 2014, Yaou k, 2020[9, 8, 14]. The alkalinity of the pH of this water could be due to the presence of bicarbonate ions, which were the most important parameter in this water with a concentration of 37.5mg/L. These results were in accord with those obtained by Natchia et al. (2013) and Ahoussi et al. (2013) [15,16] who worked respectively on the waters of the alterite nappe and the source waters of the mountainous west in Côte d'Ivoire. These results were also similar to previous studies by Sandao (2013) and Rabilou et al, 2018 [11,17] which showed that the very acidic pH in the Hamadian Continental and

Terminal nappes (ferruginous sandstones) was essentially due to the geological process involving the reduction of bicarbonate ions and the liberation of hydrogen ions with the precipitation of siderite. Bicarbonates were produced by the dissolution of carbonates in water containing CO_2 carbon dioxide.

4.2 Influence of bicarbonate ions on iron precipitation

During transport, which was a crucial operation, the water arrived under pressure. As a result of the sudden drop in pressure at the water exit, the dissolved carbon dioxide it contained escaped into the atmosphere, came into contact with the oxygen in the air, leading to the oxidation of Fe^{2+} into Fe^{3+} and the precipitation of tartar. If a deposit was to be produced from this water, it would be made up of the less soluble salt, which in most cases was $Fe(OH)_3$. The precipitation-oxidation mechanism, which illustrates all the possible reactions, proceeds as follows:

The reactions of oxidation of Fe^{2+} by oxygen and precipitation of ($Fe(OH)_3$) are given by the combination of R_1 and R_2:

$$4\ Fe^{2+} + O_2 + 10H_2O \rightarrow 4\ Fe\ (OH)_3 + 8H^+ \quad (R_5)$$

The H^+ protons liberated during reaction R_5 acidify the environment, rendering $Fe(OH)_3$ unstable. In the presence of bicarbonate, these protons react with HCO_3^- ions, according to the following reaction:

$$H^+ + HCO_3^- \leftrightarrow CO_2 + 2H_2O \quad (R_6)$$

This reaction gave the environment a stable alkaline pH that was very favourable to the precipitation of iron hydroxides ($Fe(OH)_3$). The oxidation-precipitation mechanism of $Fe(OH)_3$ can be summarised by the following global reaction:

$$Fe^{2+}(aq) + 2HCO_3^-(aq) + 1/4O_2(g) + 1/2H_2O(l) \rightleftarrows Fe(OH)_3(s) + 2CO_2\uparrow \quad (R_7)$$

The loss of CO_2 through degas and/or heat causes the reaction to move in the direction of $Fe(OH)_3$ precipitation [6].

The study of the influence of bicarbonate ions was carried out on four (4) bicarbonate concentrations 37.5mg/L, 122mg/L, 244mg/L and 488mg/L, in to explain the effect of bicarbonate concentration on iron precipitation ratio τp(Fe and precipitation pH. Our experiments showed that τp(Fe and pHp increase with bicarbonate concentration. These results can be explained by the speciation of Fe(II) in aqueous solution. A study of the speciation of Fe(II) in a bicarbonate environment showed that several Fe(II) complexes could be formed: $FeCO_3$, $Fe(OH)_3$, $Fe(CO_3)_2^{2-}$, $FeHCO_3$, $Fe(OH)^+$, $Fe(CO_3)OH^-$, $Fe(HCO_3)^+$ [18; 19; 20]. The formation of these complexes results in a very high rate of precipitation of Fe(II) at certain concentrations of $NaHCO_3$ [21]. Among these complexes, if a precipitate is to be formed from these waters, it would consist of the least soluble salt, which in most cases was $Fe(OH)_3$ [2, 6].

4.3 X-ray diffraction of deposited tartar

The results showed that the tartar recovered was amorphous, as it is characterized by low-intensity peaks (Figure IV). This explained why the solid formed in the Dan Daji water pipes was an iron oxyhydroxide, ferrihydrite ($Fe(OH)_3$), which was generally a solid without a specific form. This result was in good accord with that of John M. Z et al, 2011[22].

4. 4. SEM of deposited tartar

To confirm the exact nature of the amorphous precipitate, we carried out complementary analyses using Scanning Electron Microscopy (SEM). The SEM analysis (Figure V) approves that the tartar formed in the Dan Daji water distribution pipes effectively corresponded to a solid with no defined form. This allowed us to confirmed that the red tartar deposited in the Dandaji water distribution pipes was no doubt amorphous iron oxyhydroxide, ferrihydrite ($Fe(OH)_3$).

5. CONCLUSION

The study of the precipitation of iron oxyhydroxides in the carbonate environment was carried out on samples of natural water from Dan Daji. The physicochemical analysis showed that the water was loaded with iron, with a concentration of 1.69 mg/L, exceeding the limit set by the WHO. The precipitation reactions took place in four different carbonate media, with bicarbonate concentrations of 37.5 mg/L, 122 mg/L, 244 mg/L and 488 mg/L. Our studies showed that iron precipitation in the bicarbonate environment resulted in iron precipitation at bicarbonate concentrations of 244 mg/L and 488 mg/L, with precipitation ratios of 57% and 80% respectively. These results showed that the evolution of pH increased at all bicarbonate concentrations. In another aspect, this study showed that the pH trend increased more at higher bicarbonate concentrations. The XRD showed that the tartar recovered is characterized by low-intensity peaks. Further analysis using Scanning Electron Microscopy (SEM) confirmed that the amorphous tartar formed in the Dan Daji water distribution pipes indeed correspond to amorphous iron oxyhydroxide, ferrihydrite ($Fe(OH)_3$).

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