Full Length Research Paper

Influence of pre-oxidation with potassium permanganate on the efficiency of iron and manganese removal from surface water by coagulation-flocculation using aluminium sulphate: Case of the Okpara dam in the Republic of Benin

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Okpara dam water contained significant amounts of iron and manganese that could create problems for consumers. This study perfected a deferrization and demanganization technique preceded or followed by oxidation with potassium permanganate. The maximum iron and manganese contents varied from 30 to 50 and 1.5 to 4.5 mg/L respectively. Under the best conditions, simple coagulation-flocculation was allowed to obtain iron removal yields of 18 to 75%. Manganese was eliminated between 8 and 24%. Pre-oxidation with 2.5 mg/L potassium permanganate allowed attaining of about 99% elimination of iron and about 72% of manganese at a pH of 6.5. The treatment plant composed of a clarification stage followed by oxidation with potassium permanganate at pH 8.5 allowed a complete elimination of iron and manganese.

Key words: Surface water, water clarification, chemical coagulant, potassium permanganate, deferrization, demanganization.

INTRODUCTION

Iron, and to a lesser degree manganese, are some of the most abundant elements in the earth's crust. They are found in waters emanating from soil leaching and industrial pollution. These elements pose no danger to human health or to the environment. But they cause esthetic and organoleptic inconveniences. Iron and manganese gives water colour that can stain linen and sanitary appliances. Iron and manganese, when not eliminated, could be progressively oxidized in the distribution network giving water colour, taste, smell, turbidity and favouring the development of microorganisms with serious consequences for users. In surface or ground waters, one finds iron and manganese in different chemical forms (dissolved, precipitated, free or complexed) in variable concentrations (Myint and Barry, 1999; Omoregie et al., 2002; Muwanga and Barifaidjo, 2006). They may be present in concentrations of the order of 2 to 5 mg/L of iron and 0.5 to 2 mg/L of manganese (Ellis et al., 2000; Roccaro et al., 2007) or that are markedly higher and capable of attaining 20 mg/L iron and 5 mg/L manganese (Berbenni et al., 2000).

The methods of iron and manganese removal from water consist of transforming the dissolved forms (Fe²⁺ and Mn²⁺) by oxidation, into precipitates (Fe(OH)₃ and MnO₂) followed by filtration. Oxidation can be carried out using powerful chemical oxidants like oxygen, chlorine, chlorine dioxide, ozone or potassium permanganate (El Araby et al., 2009; Katsoyiannisa et al., 2008) or biologically (Katsoyiannisa et al., 2008; Qin et al., 2009; Tekerlekopoulou et al., 2008; Tekerlekopoulou and Vayenas, 2007; Burgera et al., 2008). Predominantly,

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physical processes like clarification and adsorption on precipitates or activated carbon are evoked (Aziza and Smith, 1992; Llofd et al., 1983; Okoniewsk et al., 2007). Simple membrane techniques often preceded by oxidation are also cited among the methods of water deferrization and demanganization (Ellis et al., 2000; Teng et al., 2001; Choo et al., 2005). The plant treatment of Okpara water dam which consisted of a preoxidation phase with chlorine, a clarification phase with aluminium sulphate and a disinfection phase with chlorine did not allow a sufficient removal of iron and manganese contained in the water.

The objective of this work was to study a process for the optimum removal of iron and manganese contained in the dam water and to propose a scaled up process with procedures for dealing with the water treatment problem. The coagulant used was aluminium sulphate and the oxidizing agent, potassium permanganate.

MATERIALS AND METHODS

Study area

The dam is built at Parakou on a tributary of the Ouémé River called Okpara, in the North-East of Benin 450 km from Cotonou. The watershed of this dam is situated in the eastern part of the town and extends to the districts of Tchaourou, Pèrèrè, Nikki, N'dali, then to part of the south-east of Bembereke district. Parakou is situated at latitude $9^{\circ}21'$ north and longitude $2^{\circ}36'$ east. The dam supplies the potable water treatment plant of the town.

Methods of analysis and testing

Raw water samples were taken from the surface (0.2 m) or at depths of from 2.0 to 4.5 m in 25 L plastic containers on the day of testing. The electrical conductivity and pH of the samples were measured using an LF 340-A/SET conductimeter, and a WTW pH 340/ion SET pH-meter, respectively. Iron and manganese were measured using the orthophenanthroline at 510 nm and the acid medium potassium periodate at 525 nm methods respectively. A DR 4000U spectrophotometer model 48100 was used to measure absorbance. Alkalinity was obtained by volumetric titration. The solutions were prepared from bidistilled water. Orthophenantroline (> 99%) and potassium periodate (> 99%) were purchased respectively from Acros Organics and Pancreac Quimica SA. The methods of analysis were based on AFNOR standards (Association de Normalisation). Oxidation with Francaise potassium permanganate was done in beakers containing the water to be treated and into which variable amounts of the stock solution (1% KMnO₄) prepared from crystals of 99% KMnO₄ (from Merck) were introduced.

Coagulation–flocculation tests were carried out according to the jar test protocol using an Orchidis 6-post miniflocculator equipped with a time switch and a tachometer. The volumes of water in the beakers were fixed at 1 L each. The different coagulation-flocculation phases consisted of a rapid agitation phase at a speed of 150 rpm for 3 min and a slow agitation phase at a speed of 25 rpm for 15 min. The samples were then allowed to settle for at least 30 min and the settled water filtered using a 0.22 μ m pore size Whatman filter paper. The addition of reagents like the coagulant made of aluminium sulphate (Al₂(SO4)₃.18H₂O) and lime for pH correction took place during the rapid agitation phase. Aluminium

sulphate (17 to 18% as Al_2O_3) and lime (> 92%) were purchased respectively from Société Tunisienne des Produits Alumineux, STPA and Balthazard et Cotte. The raw water tested did not undergo any pretreatment. The tests took place within 24 h of sampling.

EXPERIMENTAL RESULTS

Iron and manganese contents of the dam water

The raw water was characterized by low total salinity (60 to 120 μ scm⁻¹) and low alkalinity (30 to 50 mg/L CaCO₃). The pH varied from 6.7 to 5.6. The seasons of the year had a notable effect on the characteristics of the water. We monitored variations in iron and manganese concentrations in time and space. Water samples were taken from the surface at about 0.2 m depth and from a depth of 4.5 m. The results showed low variation in iron and manganese contents at the water surface in all seasons. On the other hand, the concentrations of these metals in depth were practically zero between the months of June and October. One noted an increase in these concentrations from the month of November, reaching a peak between May and June. These periods correspond to the beginning of the big rainy season and the small rainy season. The maximum values for iron varied between 30 and 50 mg/L and manganese 1.5 and 4.5 ma/L.

Figure 1 shows the evolution of dissolved iron and manganese concentrations according to depth; one observed generally that the concentrations of these elements varied little for depths below 2 m. From the 3 m depth, the concentrations increased rapidly, reaching the maximum value at the bottom of the dam. The study showed moreover that, the variation in oxygen concentration was reversed compared to those of iron and manganese. Iron and manganese concentrations were more significant in the zones of low oxygen content. In the superficial more aerated part of the dam, dissolved oxygen content was low or practically zero.

Coagulation-flocculation

Tests were carried out for doses of aluminium sulphate between 10 and 60 mg/L in the water. The residual concentrations of iron and manganese were measured on decanted and filtered water. The results without pH adjustment in Figure 2 shows that, clarification allowed eliminating part of the iron and manganese. The residual concentration of iron was in the order of 1.5 to 2.0 mg/L for an initial concentration of 9.1 mg/L and a coagulant dose between 50 and 60 mg/L. For the same doses of coagulant, the manganese concentration was brought down from 2.9 to about 2.3 mg/L. The same experiments were carried out at pH 6.5. The results (Figure 3) show

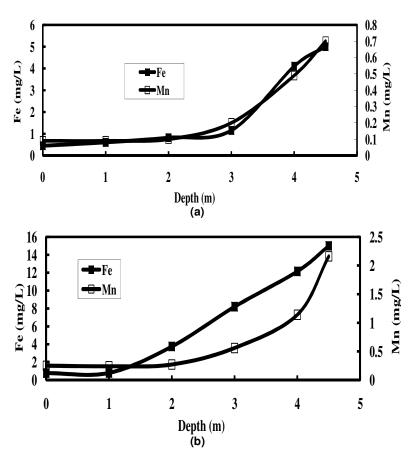


Figure 1. Evolution of iron and manganese concentrations as a function of depth in July (a) and June 2006 (b).

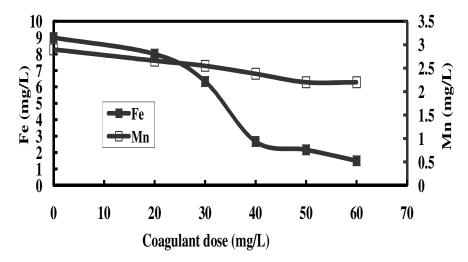


Figure 2. Removal of iron and manganese by coagulation without pH adjustment.

that at this pH, the elimination yields for iron and manganese improved significantly. Using between 40 and 60 mg/L of coagulant, the residual concentrations of

iron and manganese were lower than 0.5 and 2.0 mg/L respectively (initial concentrations of 9.1 mg/L of iron and 2.9 mg/L of manganese).

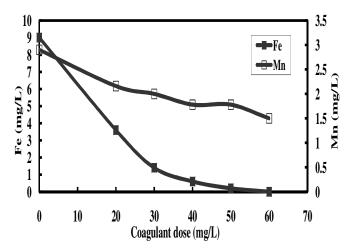


Figure 3. Removal of iron and manganese by coagulation at pH 6.5.

Table 1. Removal	yields	(%)	of	iron	and	manganese	by
coagulation (20 to	o 40	mg/L	of	coa	igular	nt) without	рΗ
adjustment and at pl	┨6.5.						

Treatment	% Fe removal	% Mn removal
Coagulation without pH adjustment	10 - 73	2 - 24
Coagulation pH = 6.5	18 - 75	8 - 20

Table 1 shows orders of magnitude of the percentages of iron and manganese removed without pH adjustment or pH 6.5 for several values of initial concentrations of iron and manganese in raw water sampled at different depths of the dam. The results showed that, for initial iron concentrations between 8 and 35 mg/L and for a coagulant dose from 20 to 40 mg/L, iron removal yields were between 10 and 73% during treatment without pH adjustment. At pH 6.5, the yields could reach 75%. Manganese was eliminated with a maximum yield of 24% (initial concentrations of manganese varied from 0.9 to 4.1 mg/L).

Influence of pre-oxidation

The potassium permanganate concentrations applied in pre-oxidation varied between 1 and 6 mg/L. The coagulant dose was fixed at 40 mg/L. The tests were carried out without pH adjustment and at pH 6.5. Figures 4 and 5 show that removal yield increased with potassium permanganate dose. In particular, for a KMnO₄ concentration of 4 mg/L and an initial iron concentration of 8.7 mg/L, the final concentration was in the order of 2.2 mg/L without pH adjustment and 2.0 at pH 6.5. Under these conditions, the residual manganese concentrations

Table 2. Removal yields (%) of iron and manganese, using 40
mg/L of coagulant without pH adjustment, pH 6.5 and a KMnO ₄
dose of 2.5 mg/L.

Fe (mg/L)	Mn (mg/L)		out pH stment	pH = 6.5		
(iiig/L)	(iiig/L)	% Fe % Mn		% Fe	% Mn	
8.0	1.1	98	66	99	72	
17.0	0.9	74	51	95	61	
24.0	2.0	76	28	85	35	
30.0	3.5	75	26	80	29	

were 0.4 and 0.3 mg/L without pH adjustment and pH 6.5 respectively (initial concentration of 1.8 mg/L).

The results showed moreover that, with larger doses (5 to 6 mg/L) of KMnO₄, one could completely remove iron and manganese from water. Table 2 shows the removal yields for a dose of 40 mg/L of coagulant and a preoxidation dose of 2.5 mg/L KMnO₄. At pH 6.5, the yields varied from 80 to 99% for iron concentration from 8 to 30 mg/L. For manganese, the highest yields (60 to 72%) were obtained when the initial concentration was between 0.9 and 1.1 mg/L. For larger (4 mg/L of KMnO₄) oxidant doses and for raw water concentrations of from 8 to 17 mg/L of iron and from 0.9 to 1.1 mg/L of manganese, the removal yields were greater than 99% for iron and between 94 and 96% for manganese.

Clarification and oxidation at pH = 8.5

To improve removal yields of the elements (manganese in particular) we implemented the following treatment scheme, based on optimal coagulation-flocculation conditions:

1. coagulation at pH 6.5 with 40 mg/L of aluminium sulphate

2. oxidation of coagulated and decanted water with potassium permanganate at pH 8.5

3. filtration and measurement of iron and manganese concentrations.

The results show a clear improvement in removal yields (Figures 6 and 7). Manganese, even at high concentration (3.5 mg/L) was completely removed from the water with oxidant doses of between 3 and 4 mg/L. The sequence/ clarification then oxidation and filtration (two filters)/ was more efficient than the sequence/ oxidation and clarification (only one filter). Table 3 shows the evolution of removal yields. One observed that manganese was removed at between 65 and 95% for concentrations varying between 0.9 and 3.5 mg/L. Iron was completely removed from the water for concentrations

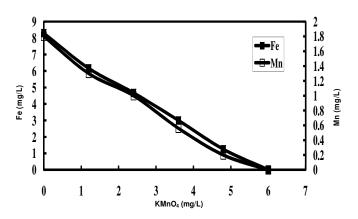


Figure 4. Influence of pre-oxidation on iron and manganese removal by coagulation without pH adjustment.

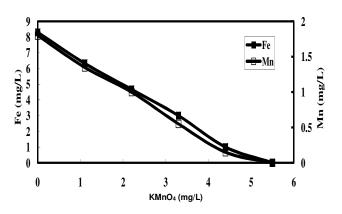


Figure 5. Influence of pre-oxidation on iron and manganese removal by coagulation at pH 6.5

of potassium permanganate of about 2.5 mg/L; higher concentrations than 2.5 mg/L (3 to 4 mg/L) are required to completely eliminate manganese.

DISCUSSION

Mechanisms of iron and manganese removal

Without pH adjustment or at pH 6.5 we determined iron and manganese removal yields by coagulation. The influence of pH was not systematically studied because several studies show that the range of pH, favourable for coagulation with aluminium sulphate lies between 5.0 and 7.0 (Bawa et al., 1998a; Van Benschoten and Edzwald, 1990; Gregor et al., 1997; Zhong et al., 2010). The study showed that coagulation allowed the removal of a significant proportion of iron and manganese. The eliminated fractions took different forms (hydroxide, oxide) sometimes associated with solid particles like clays. We observed that iron removal declined steadily as

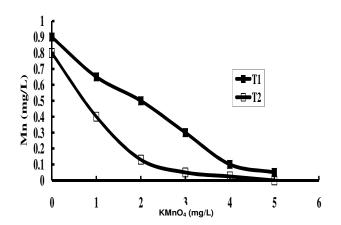


Figure 6. Removal of manganese with KMnO₄. Initial water contents: 0.9 mg Mn/L, 17 mg Fe/L (TI : oxidation by KMnO₄ of coagulated water with 40 mg/L at pH 6.5 ; T2 : coagulated water with 40 mg/L at pH 6.5, decanted, filtered and oxidized with KMnO4 at pH 6.5 and filtered).

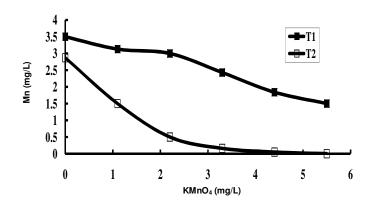


Figure 7. Removal of manganese with KMnO₄. Initial water contents: 3.5 mg Mn/L, 30 mg Fe/L (TI : oxidation by KMnO₄ of coagulated water with 40 mg/L at pH 6.5; T2 : coagulated water with 40 mg/L at pH 6.5, decanted, filtered and oxidized with KMnO₄ at pH 6.5 and filtered).

the dose of coagulant increased and the elimination of manganese tended to stabilize from 30 to 40 mg/L of coagulant applied. Higher doses of up to 60 mg/L did not improve manganese elimination, neither did it cause a restabilization of flocs. Knocke et al. (1992) obtain a 63% total iron removal during the coagulation of raw river water using aluminium sulphate (coagulant dose of from 40 to 50 mg/L, pH between 6.3 and 6.5, initial iron concentration from 0.6 to 2.4 mg/L) and Montiel and Welte, (1990) obtained a manganese elimination yield of 25 to 35% (initial concentration of 0.5 to 1.0 mg/L) during coagulation using ferric chloride in the presence of alginate at pH between 7.6 and 7.8.

Many mechanisms of particle elimination by coagulation such as complexation between the hydrolyzed soluble forms of metals and organic or colloidal

Fe (mg/L)	Mn (mg/L)	% Fe	% Mn
8.0	1.1	> 99	97
12.2	1.1	> 99	95
17.0	0.9	> 99	75
24.0	2.0	> 99	79
30.0	3.5	> 99	69

Table 3. Removal yields (%) of iron and manganese for 40 mg/L of coagulant at pH 6.5 and a $KMnO_4$ dose of 2.5 mg/L at pH 8.5.

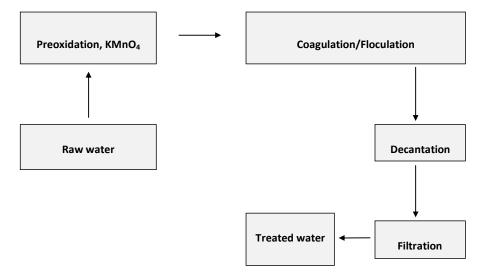


Figure 8. Structure of the treatment plant (plant a).

matter, double layer reduction, adsorption on flocs of metallic hydroxides, charge neutralization, particle trapping in a mesh and co- precipitation are generally cited in coagulation-flocculation studies. The conditions of treatment, particularly the pH, may favour some approaches. Thus, Jian-Jun et al. (2006) and Zhong et al. (2010) showed that, between pH 4.5 and 5.2, the contribution of the charge neutralization mechanism is more important.

At pH greater than 5.5, the contribution of coagulation by neutralization declined and the mechanism of coagulation by adsorption combined with trapping and co-precipitation dominated. It is therefore probable that in this study, the mechanism of particle neutralization was dominant during coagulation without pH adjustment (the pH decreased rapidly after coagulant addition from 5.4 to 4.5). The mechanism by adsorption was dominant at pH 6.5 (the pH having varied between 6.5 and 6.0). It was recently shown that at pH between 4.5 and 9.0, aluminium in the form of sulphate in water hydrolyzes forming 8 products of hydrolysis (AI^{3+} , $AI(OH)^{2+}$, $AI(OH)_{2}^{+}$, $AI(OH)_{3}$, $AI(OH)_{4}^{-}$, $AI_{2}(OH)_{2}^{4+}$, $AI_{3}(OH)_{4}^{-5+}$ and AIO_{4}^{-2} $AI_{12}(OH)_{24}(H_{20})^{7+}$ is the dominant type of the total

aluminium added to water and the cation containing 13 atoms of aluminium $(AIO_4 AI_{12}(OH)_{24}(H_20)^{\prime+}_{12})$ is most important for the neutralization and destabilization of colloids which are generally negatively charged (Xiao et al., 2008). Franceshi et al. (2002) also showed that between pH 6 and 8, the mechanism of clay elimination, responsible for water turbidity, is adsorption and neutralization. With regard to the coagulation-flocculation carried out at pH 6.5, pre-oxidation allowed the increase of removal yield from 50 to 95%. Iron was practically eliminated. Manganese was still present in high residual concentrations. lt was shown that potassium permanganate was a good chemical reagent for the oxidation of manganese in water. But pH is a limiting factor in this treatment. It is necessary to operate at a pH greater than 7.0 (Doré, 1989; EPA, 1999; Roccaro et al., 2007).

In these conditions, potassium permanganate is more stable in water and reacts with its highest oxidation state. Roccaro et al. (2007) obtained a 95% removal yield for manganese in underground water, after oxidation with potassium permanganate at pH 8.5 and an initial manganese concentration of about 2 mg/L. Moreover, studies show that the oxidized forms of iron (Fe(OH)₃)

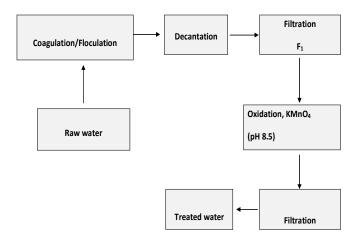


Figure 9. Structure of the treatment plant (plant b).

and manganese (MnO_2) , like aluminium hydroxide $(Al(OH)_3)$, are good adsorbents (Mazet et al., 1990; Berbenni et al., 2000). It is therefore probable that oxidized forms of iron and manganese, in playing an adsorptive role, might have contributed a lot to improving the efficiency of coagulation (coagulation by adsorption, trapping and co-precipitation).

Next. we treated the water. associating physicochemical treatments with oxidation. usina potassium permanganate (KMnO₄) at pH 8.5. The treatment plant included first, a clarification stage (coagulation with aluminium sulphate at pH 6.5, decantation and filtration) and second, an oxidation stage with potassium permanganate at pH 8.5 followed finally by filtration. This plant allowed a complete removal of iron and manganese for coagulant doses of 40 mg/L and potassium permanganate concentrations greater than 2.5 mg/L. The first module of the plant allowed clarifying water and reducing high molecular weight organic compounds like humic substances (Bawa et al., 1998b; Van Benschoten and Edzwald, 1990; Jian-Jun et al., 2006), the second module allowed, under the best conditions, the oxidation of iron and particularly manganese.

Proposed treatment plants

We tested treatment plants including, firstly, physicochemical (coagulation/flocculation/decantation/ filtration) and secondly, chemical oxidation procedure using potassium permanganate (Figures 8 and 9). With plant a), the results showed that it was possible to get a complete removal of iron and manganese, if their initial concentrations were low (< 8 mg/L iron and < 1 mg/L manganese). In the dam, the lowest iron and manganese concentrations were encountered in the superficial parts (depths < 2 m). We therefore concluded that when the

dam water was sampled at shallow depth, plant a) could allow attaining the objectives of the treatment.

Plant b) represented in Figure 9 included a clarification phase with coagulation at pH 6.5 and an oxidation phase with potassium permanganate (KMnO₄) at pH 8.5. Potassium permanganate oxidation was preceded by one filtration, followed by a second filtration stage. The first stage eliminated the residual flocs resulting from the physicochemical treatment and the second allowed to retain the metallic oxides and hydroxides of iron and manganese (mainly $Fe(OH)_3$ and MnO_2). The results showed that this plant could also be used but it was indispensable that the decantation and filtration phases after coagulation-flocculation be well executed, to ensure a complete removal of flocs, for if they escaped to the clarification module, they could reach the KMnO4 oxidation post at pH 8.5. At this pH, the residual flocs, rich in aluminium salts, are restabilized, water is recoloured and soluble aluminium increases in the treated water. Actually at pH 8.5, the dominant form of aluminium in the water is no longer the solid AI(OH)₃ but the very soluble type, the aluminate AIO²⁻, (Van Benshoten and Edzwald, 1990, Chow et al., 2009). In fact, the sampling and implementation conditions, if respected, one could supply water that conforms to the norms of portability with regard to iron particularly to manganese (< 0.4 mg/L) (WHO, 2006).

Conclusion

The water studied contained significant amounts of iron and manganese. For a better appreciation of its quality by consumers, it was necessary to propose a method of deferrization and demanganization. In this study, we tested two treatment plants including an oxidation stage with potassium permanganate. The first plant was adapted for waters with low iron and manganese contents (depths < 2 m) and the second was more suited for waters sampled in depth. In both cases the treated water conformed to the norms of potability relative to iron and manganese.

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