



# COMPARING SEDIMENTATION AND DISSOLVED AIR FLOTATION (DAF) FOR ORGANIC IRON COMPOUNDS REMOVAL FOCUSING COAGULATION/FLOCCULATION PROCESSES

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## Abstract

This paper presents results comparing iron organic complexes, color, turbidity and manganese removal efficiencies of two techniques: oxidation/coagulation/sedimentation and oxidation/coagulation/dissolved air flotation (DAF). The focus was the suitable adjustment of the coagulation processes rather than the oxidation process. In this way, lower dosages of oxidant and coagulant could be added increasing the removal efficiency and saving chemicals. The results showed that DAF is an attractive alternative to promote the removal of organic iron compounds (97% of removal efficiency, residual of 0.17 mg/L), color (98% of removal efficiency, residual <2 CU) and turbidity (95% of removal efficiency, residual of 0.7 NTU) by using low dosages of chemicals (15 mg/L of ferric chloride; 3.1 mgFe/L) in a lower pH value (6.3) than the commonly recommended one (>7.5). The manganese removal was better (>84% of removal efficiency, residual of 0.04 mg/L) in a high pH value (8.0) when sedimentation was used as clarification technique (dosage of ferric chloride of 45 mg/L; 9.3 mgFe/L in a pH value of 8.0) but a reasonable efficiency (52% of removal efficiency, residual of 0.12 mgMn/L near to Brazilian drinking water standard 0.10 mgMn/L) was achieved when DAF was applied with the dosage of ferric chloride of 15 mg/L (3.1 mgFe/L) in a pH value of 6.3. The solid/liquid separation technique can be a planner's decision but the matter is to focus in coagulation process rather than oxidation. For DAF and sedimentation THM concentrations were always between 4 and 6.5 µg/L.

*Key words:* dissolved air flotation, organic iron compounds, drinking water treatment.

## Introduction

It's consensus that waters containing iron forms cause problems in different stages in water treatment systems. The occurrence of taste and color in treated water as well as encrustation in pump lines, growth of specific

microorganisms and staining sanitary devices are commonly found troubles in the iron presence.

As widely related in several works, the humic substances responsible for the color of natural water easily associate with metals and oxides forming usually colored

complexes. Due to its high stability when associated with NOM, the iron and manganese removal becomes more difficult than the removal of the free forms of these metals. As observed by Knoche *et al.* (1991), the organic complexes are very hard to be removed by oxidation and subsequent precipitation of  $\text{Fe}(\text{OH})_3$ . An efficient removal of these organic complexes of iron must involve processes capable of removing organic matter.

Most of the time, waters containing natural organic matter (NOM), as humic substances, require pH in the range of 5.5 and 7.0 to be coagulated with coagulant dosages more economic than those required when coagulating in higher pH range (7.0 to 9.0). On the other hand, iron and manganese oxidation/precipitation usually requires pH value in a high pH range to be effective (favoring the kinetics of the oxidation process). Thus, it is common in Brazil to find water treatment plants working with high coagulant dosage in a high pH range conjointly with the chlorine oxidation step as a strategy to promote both metals and NOM removal by using chlorine/coagulation/sedimentation/filtration steps. Consequently, in these cases, the coagulation process is not the focus but the oxidation is. So, some points must be highlighted. Firstly, the ferric chloride is the most used coagulant due to its large working pH range comparing to other coagulants. The other point is a consequence of the first one: the ferric chloride dosage required must be pretty high so that adequate conditions of coagulation/flocculation and clarification by sedimentation can be reached. As results shorter filters runs, higher sludge production and operation costs are commonly related. Furthermore, it is common to observe problems related to THM formation in these plants when using chlorine as oxidant.

Dissolved air flotation (DAF) is a confirmed alternative to promote NOM removal by using lower coagulant dosages and working with a higher loading rate than the sedimentation units does. Several works report results of DAF when treating colored waters in lab studies, pilot plants or facilities (Zabel, 1985; Edzwald, 1991a; Reali & Campos, 1995; Reali & Marchetto, 2001; among others). Reali and Marchetto (2001) present some results of a pilot unit of DAF called high-rate unit. In this article the authors presented that efficiencies around 90% of color removal could be reached even when a high loading rate value ( $19 \text{ mh}^{-1}$ ) and low air recirculation ( $2.1 \text{ g of air m}^{-3}$ ) was applied.

This paper aims iron organic forms removal by focusing coagulation process rather than oxidation seeking both DAF and sedimentation clarification steps. For this purpose, sedimentation and dissolved air flotation (DAF) in laboratory units using synthetic water were investigated.

The oxidation step was also investigated as a single process in order to evaluate its contribution for the metals precipitation.

## Methods

### *Synthetic water*

Synthetic water was used to carefully control experimental conditions and guarantee the same metals concentration during all the investigation period.

The synthetic water was obtained by adding a commercial humic acid (5 mg/L), iron ( $\text{Fe}^{+3}$ ), manganese ( $\text{Mn}^{+2}$ ) and caolin (Kaolin) in a deep well water. This way the water presented color (150 CU), turbidity (15 NTU), iron ( $3,5 \text{ mgFe}^{+3}/\text{L}$ ) and manganese ( $0,25 \text{ Mn}^{+2}/\text{L}$ ). Table 1 shows results of synthetic raw water quality analyses.

All iron and manganese concentrations were obtained in digested and non-digested samples. The determinations of iron and manganese residual concentrations were performed by using an atomic absorption spectrum photometer following the procedure described in the Standard Methods 20<sup>th</sup> edition section 3111. The  $\text{HNO}_3/\text{HCl}$  chemical digestion process of the samples followed the normalized procedure of the Standard Methods 20<sup>th</sup> edition section 3030F.

### *Laboratory scale essays strategies*

First of all, previous essays were conducted to certify the amount of colloidal complexes present in different conditions. For this, samples with different pH values were filtered in 0.45 micron pore size. Later, oxidation essays applying chlorine in a wide pH range followed by filtration in 0.45 micron pore size were investigated (Figure 2a).

Dissolved air flotation equipment in lab scale called Flotatest was used for coagulation/oxidation/flotation essays (Figure 1). For the coagulation/oxidation/sedimentation essays identical columns to the ones of flotation were used.

For each technique, several dosages of coagulant in different pH values were studied. For flotation, the ferric chloride dosages applied ( $D_{\text{FeCl}_3}$ ) were of: 0; 5; 10; 15; 20; 25; 30; and 35 mg/L and pH values were around 5.8 and 9.3 (Figure 2b). Table 1 shows the DAF parameters adopted in these essays as recommended by Dombroski (1996), apud Reali & Campos (2002):

During the sedimentation essays, ferric chloride dosages ( $D_{\text{FeCl}_3}$ ) of 30 and 45 mg/L (pH in the range of 3.8 and 8.7) were applied. The rapid mix was carried out in a mean velocity gradient (Gmr) of  $800 \text{ s}^{-1}$  in a period of 20 seconds. For flocculation the mean velocity gradient (Gf) studied were of 40 and  $60 \text{ s}^{-1}$  and for each Gf value the flocculation times of 20 and 35 min were studied (Figure 2c). Table 1 presents a summary of the operating conditions used for experiments comparing DAF and sedimentation.

### Experimental Results and Discussion

#### Oxidation essays

Chemical oxidation essays showed that only the oxidation process was not enough to promote the precipitation of iron organic compounds. Although iron and manganese residuals presented improvement when oxidant dosage was

increased, it can be observed in Table 2 that iron kept a reasonable level ( $0.36 \text{ mgFe}^{+3}/\text{L}$ ) even for the highest dosage of oxidant ( $32 \text{ mg/L}$ ). On the other hand, manganese residuals were pretty low when oxidant dosage was higher than  $5.3 \text{ mg/L}$ . This is an indication that iron organic compounds are much more stable than the manganese ones.

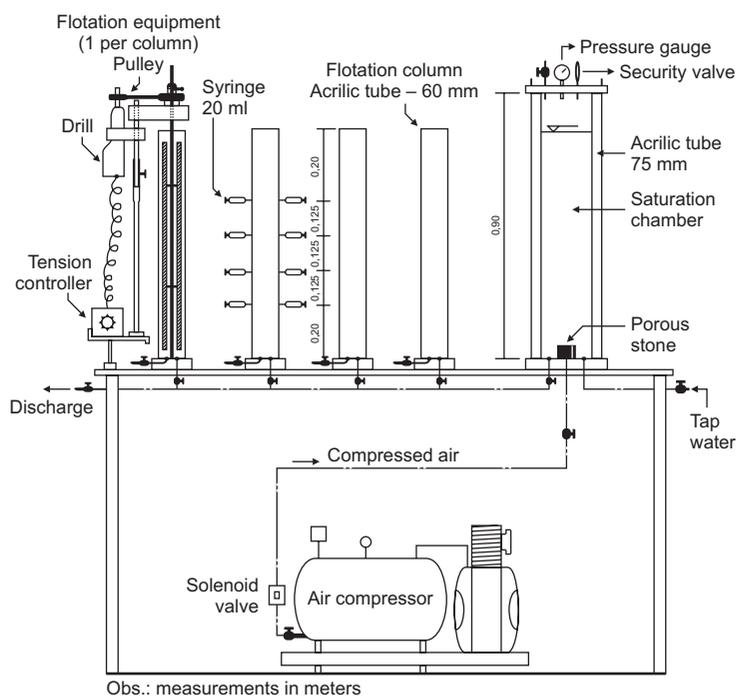


Figure 1 A scheme of the bath flotation unit used in the study (Flotatest) – Reali (1991).

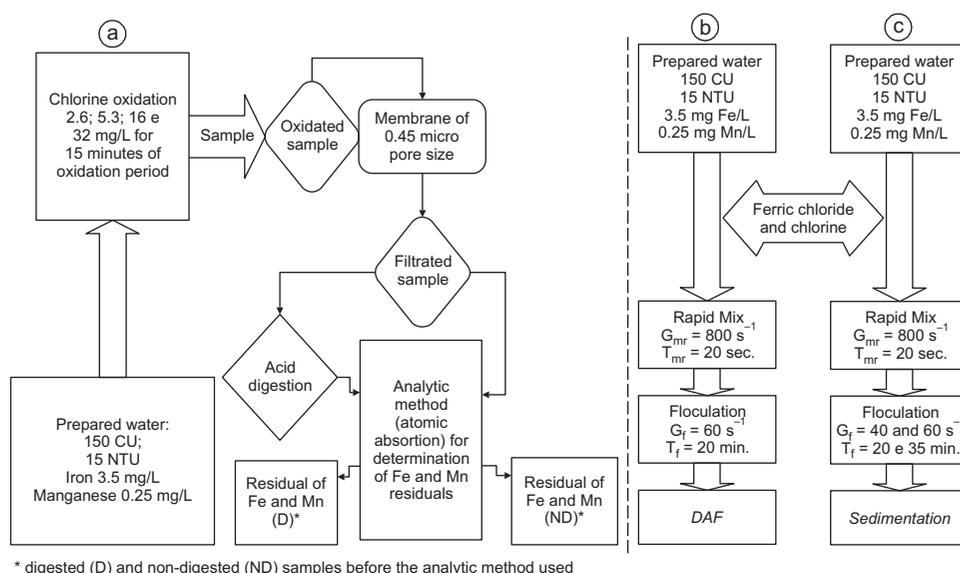


Figure 2 A scheme of the conducted essays.

**Table 1** Characteristics of synthetic raw water and operating conditions adopted during experiments.

Parameter	Synthetic water	Operating conditions	DAF	Sedimentation
Apparent color (CU-PtCo)	150	<i>Coagulation:</i> $D_{FeCl_3}$ (mg.L <sup>-1</sup> ) pH of coagulation <i>Rapid mix:</i> Time (sec)	0 to 35 5.8 to 9.3 20	30 and 45 3.8 to 8.7 20
True color (CU-PtCo)	40	Grm (s <sup>-1</sup> )	800	800
UV-254 nm	0.025	<i>Flocculation:</i> Time (min.)	20	20 and 35
DOC (mg.L <sup>-1</sup> )	2.5	Gf (s <sup>-1</sup> )	60	40 and 60
Conductivity (μs.cm <sup>-1</sup> ) Humic acid (mg.L <sup>-1</sup> )	55 5.0	<i>Clarification condition:</i> Velocity (cm.min <sup>-1</sup> )	12	2.5
Turbidity (NTU)	15	Apparent loading rate ratio	4.8	1
Total iron (mg.L <sup>-1</sup> )	3.50	Saturation pressure (KPa)	450	NA*
Total manganese (mg.L <sup>-1</sup> )	0.25	Recycle ratio percent (V/V)	10	NA*

\*Not applicable.

Another important point, referred to the results showed in Table 2, is that non-digested iron aliquots present always a very low residual; probably demonstrating that iron precipitated free forms were present even when no oxidant was added. Regarding manganese, the very close values between digested and non-digested forms probably indicated a weaker link between organic matter and manganese than that observed for iron.

#### **Coagulation and flocculation essays**

Preliminary DAF essays without oxidant were performed (Table 3). These essays demonstrate that a low oxidant dosage (2,6 mg/L) produce a slight increase on iron removal but a strong effect on manganese removal.

Thus, chlorine dosage of 2,6 mg/L was adopted during coagulation and flocculation essays.

Due to broad amount of results relating to coagulation and flocculation essays for both DAF and sedimentation (see Table 1) only the best results, for each technique, will be shown (Figure 3 and 4).

DAF results (Figure 3) demonstrate a removal higher than 98% for color (<2 CU), 95% for turbidity (0,7 NTU) and 93% for iron (<0,25 mgFe/L) when ferric chloride dosages of 15 mg/L (pH 6,3) and 30 mg/L (pH 5,8) were applied. A manganese removal around 50% (<0,16 mgMn/L) was verified for most of the dosages (except for  $D_{FeCl_3} = 0$  mg/L and  $D_{FeCl_3} = 35$  mg/L) in pH values much lower than the range usually considered adequate (pH above 8,5).

**Table2** Oxidation essays results followed by filtration in 0,45 μm membrane (no coagulant).

Adapted to: Moruzzi *et al.* (2004).

Oxidant dosages (mg/L)	Fe residual (mg/L) (D/ND) [%R]	Mn residual (mg/L) (D/ND) [%R]	Apparent color (uPtCo) (residual) [%R]
0.0	(1.59/<0.01) [54.5]	(0.25/0.25) [0.0]	20 [86.6]
2.6	(0.82/<0.01) [76.5]	(0.14/0.12) [44.0]	19 [87.3]
5.3	(0.45/<0.01) [87.1]	(0.11/0.07) [56.0]	15 [90.0]
16.0	(0.40/<0.01) [88.5]	(0.02/0.01) [92.0]	18 [88.0]
32.0	(0.36/<0.01) [89.7]	(0.02/0.01) [92.0]	18 [88.0]

Raw water characteristics: pH = 7.5; turbidity = 15 ± 01(NTU); apparent color = 150 ± 10 (CU-uPtCo); total iron = 3.5 mg/L; total manganese = 0.25 mg/L; humic acid = 5 mg/L; temperature = 20 ± 01(°C); oxidation time = 15 minutes (D: digested aliquots; ND: non-digested aliquots; [%R]: percentage of removal of the parameters).

A colloidal manganese formation containing favorable hydrophobic characteristic in these pH ranges can be a possible explanation to the unexpected removal. A high dosage of chlorine could be applied to increase manganese removal. In this point it is important to consider the use of other oxidants aiming to minimize THM formation risk. A higher pH value also could be advised but, this alternative was not investigated for DAF technique.

For iron results, Figure 3 shows that an adequate coagulation condition can promote an efficient removal of iron organic forms (>95% of efficiency, residual of 0.17 mgFe/l where  $D_{FeCl_3}$  of 15 mg/L in a pH value of 6.3 was investigated). Thus, the oxidation must not be the

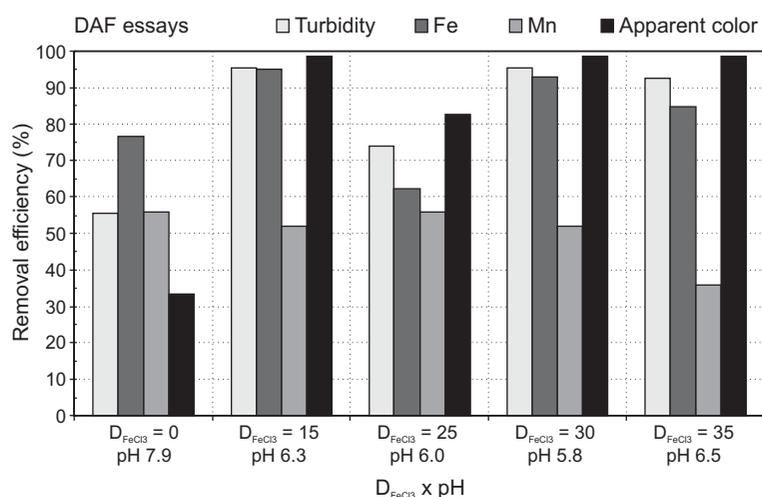
only focus to remove iron organic compounds but also the adjustment of coagulation and flocculation processes.

In the same way, sedimentation results (Figure 4) demonstrate that the adjustment of coagulation and flocculation can promote good removal efficiency for color (86%, residual of 21 CU), iron (62%, residual of 1.33 mgFe/L) and turbidity (86%, residual of 2.1 NTU) while using  $D_{FeCl_3}$  of 30 mg/L and pH value of 6.2 (flocculation: Gf around  $40\text{ s}^{-1}$  and Tf of 20 min.). However, the best results regarding manganese removal (>84%, residual <0.04 mgMn/L) occurred in higher coagulant dosages and pH values ( $D_{FeCl_3} = 45\text{ mg/L}$  and pH around 8.0, respectively), where turbidity efficiency decreases.

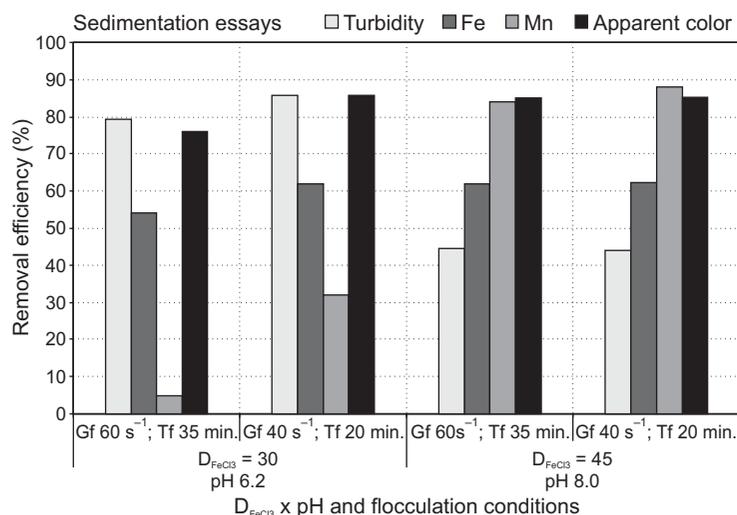
**Table 3** DAF essays with and without chlorine applied together with ferric chloride for dosages of 15 and 30 mg/L in a pH value of 6.3.

$D_{FeCl_3}$ (mg/L)	$D_{Cl_2}$ (mg/L)	Fe (mg/L) (D/ND) [R%]	Mn (mg/L) (D/ND) [R%]
15	0	(0.34/0.06) [90]	(0.20/0.17) [20]
15	2.6	(0.17/0.02) [95]	(0.12/0.08) [52]
30	0	(0.46/0.35) [87]	(0.23/0.21) [8]
30	2.6	(0.33/nd) [90]	(0.10/0.08) [60]

Raw water characteristics: pH = 7.5; turbidity =  $15 \pm 01$ (NTU); apparent color =  $150 \pm 10$  (CU-uPtCo); total iron = 3.5 mg/L; total manganese = 0.25 mg/L; humic acid = 5 mg/L; temperature =  $20 \pm 01$ (°C); oxidation time = 20 minutes ( $D_{FeCl_3}$  ferric chloride dosage;  $D_{Cl_2}$  chlorine dosage; D: digested aliquots; ND: non-digested aliquots; [R%]: percentage of removal of the parameters; nd: not detected level).



**Figure 3** Bests results from DAF essays. ( $D_{FeCl_3}$ : ferric chloride dosage). Raw synthetic water characteristics: pH = 7.5; turbidity =  $15 \pm 01$ (NTU); apparent color =  $150 \pm 10$  (CU = uPtCo); total iron = 3.5 mg/L; total manganese = 0.25 mg/L; humic acid = 5 mg/L; temperature =  $20 \pm 01$  (°C); oxidation time = 20 minutes; chlorine dosage = 2.6 mg/L. Conditions during DAF essays: rapid mixture: velocity gradient:  $800\text{ s}^{-1}$ , time: 20 sec.; flocculation: velocity gradient:  $60\text{ s}^{-1}$ , time: 20 min; dissolved air flotation: recirculation: 10%(v/v); saturation pressure:  $450 \pm 10$  kPa; flotation velocity: 12 cm/min. Conditions of sedimentation essays: rapid mixture: velocity gradient:  $800\text{ s}^{-1}$ , time: 20 sec.; flocculation: indicated on graph; sedimentation velocity: 2.5 cm/min.



**Figure 4** Bests results from sedimentation essays. ( $D_{FeCl_3}$ : ferric chloride dosage). *Raw synthetic water characteristics*: pH = 7.5; turbidity =  $15 \pm 01$ (NTU); apparent color =  $150 \pm 10$  (CU = uPtCo); total iron = 3.5 mg/L; total manganese = 0.25 mg/L; humic acid = 5 mg/L; temperature =  $20 \pm 01$  (°C); oxidation time = 20 minutes; chlorine dosage = 2.6 mg/L. *Conditions during DAF essays*: rapid mixture: velocity gradient:  $800 \text{ s}^{-1}$ , time: 20 sec.; flocculation: velocity gradient:  $60 \text{ s}^{-1}$ , time: 20 min.; dissolved air flotation: recirculation: 10% (v/v); saturation pressure:  $450 \pm 10$  kPa; flotation velocity: 12 cm/min. *Conditions of sedimentation essays*: rapid mixture: velocity gradient:  $800 \text{ s}^{-1}$ , time: 20 sec.; flocculation: indicated on graph; sedimentation velocity: 2.5 cm/min.

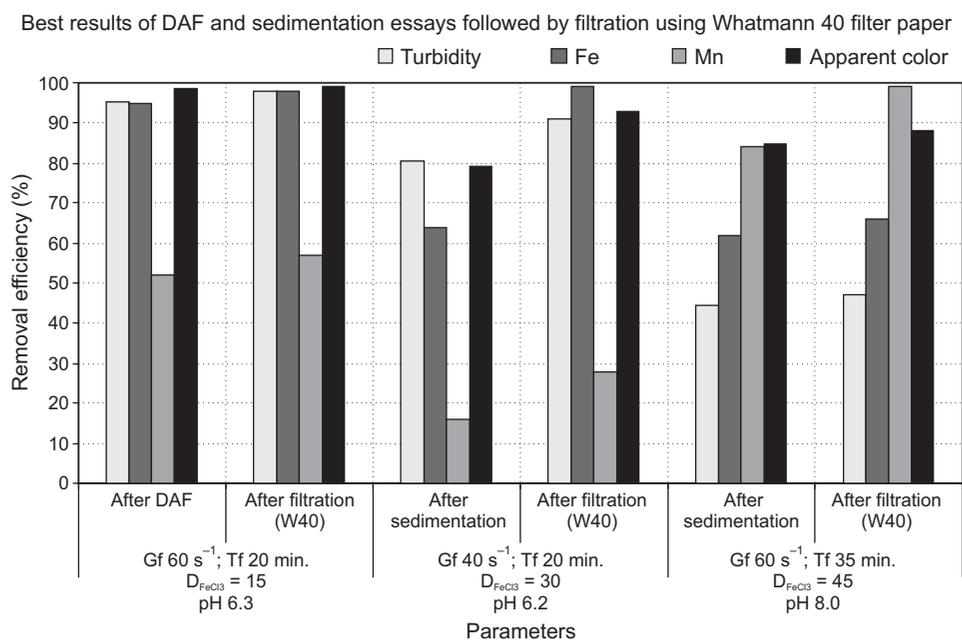
Figure 5 presents, side by side, the best results of DAF and sedimentation followed, each one, by filtration using paper filter Whatman 40 ( $8 \mu\text{m}$  medium pore size). Comparing results between DAF and sedimentation it is possible to verify high removal efficiency for turbidity, iron and color when DAF was applied (flotation velocity of 7.2 m/h). On the other hand, manganese best condition was achieved in a high pH value and coagulant dosage during sedimentation essay (sedimentation velocity of 1.5 m/h) but, in this case, turbidity efficiency decreases considerably (<45%, corresponding to 8.3 NTU of residual). Unfortunately, no DAF essays were carried out by using high coagulant dosages ( $D_{FeCl_3}$  45 mg/L) and pH around 8.0 in order to compare with the sedimentation results so, further studies are recommended. However, when comparing DAF and sedimentation in lower coagulation pH range (around 6.2), it can be observed that DAF is far more efficient than sedimentation for all analyzed parameters even when using lower dosages of coagulant (15 mg/L) than that applied during the sedimentation essay (30 mg/L).

After filtration by using paper filter (Whatman 40) all samples presented better results than after the pre-clarification step (DAF or Sedimentation). However, the lowest difference between pre-clarification technique and filtration was obtained during DAF essays. Although DAF

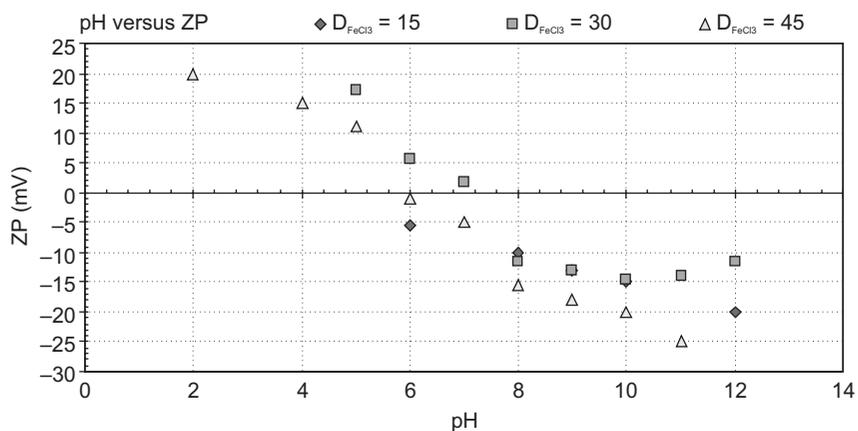
essay followed by filtration presented the lowest residual of color (<2 CU), turbidity (0.60 NTU) and iron (0.10 mg/L), the filtration step enhanced the quality slightly. As a consequence, in a water treatment system incorporating DAF, the filter runs would be improved due to the lower load of solids in the influent of the filters.

Due to manganese's lower connection with NOM than iron forms (inferred through the difference between Digested and Non Digested samples), the removal of this metal was better when pH value was higher (manganese residual of 0.03 mg/L for pH value around 8.0). However, it is interesting to observe that DAF was able to remove part of Mn forms in a lower pH value (6.2) than the recommended ones (>8.0). A possible explanation is that Mn forms in pH around 6.0, after oxidation and coagulation, presented some favorable condition that permits them to be removed (in some extent) by DAF better than by sedimentation.

Figure 6 presents some results of Zeta Potential analyses after rapid mix for ferric chloride dosages of 15, 30 and 45 mg/L in several pH values. For all coagulant dosages, the Zeta Potential values were near the neutral when the pH values were in the range of 6.0 to 7.0. This agrees with the best results obtained during the DAF and sedimentation essays, as shown in Figures 3 and 4 considering iron, color and turbidity removals.



**Figure 5** Comparison between the best results after DAF and sedimentation followed by filtration Whatmann 40 paper filter (8 μm pore medium diameter) for each technique. (D<sub>FeCl<sub>3</sub></sub>: ferric chloride dosage). *Raw synthetic water characteristics:* pH = 7.5; turbidity = 15 ± 01 (NTU); apparent color = 150 ± 10 (CU = uPtCo); total iron = 3,5mg/L; total manganese = 0.25 mg/L; humic acid = 5 mg/L; temperature = 20 ± 01 (°C); oxidation time = 20 minutes; chlorine dosage = 2.6 mg/L. *Conditions during DAF essays:* rapid mixture: velocity gradient: 800 s<sup>-1</sup>, time: 20 sec.; flocculation: velocity gradient: 60 s<sup>-1</sup>, time: 20 min; dissolved air flotation: recirculation: 10%(v/v); saturation pressure: 450 ± 10 kPa; flotation velocity: 12 cm/min. *Conditions of sedimentation essays:* rapid mixture: velocity gradient: 800 s<sup>-1</sup>, time: 20 sec.; flocculation: indicated on graph; sedimentation velocity: 2.5 cm/min.



**Figure 6** Zeta Potential values concerning ferric chloride dosages of 15, 30 e 45 mg/L for different pH values of the studied water (obtained by applying lime). Samples collected after rapid mix and analyzed with Zetamaster model ZEM5000 (Malvern).

In general terms, DAF presented much better results than sedimentation not only for iron removals but also for color removal and turbidity. The best group of results obtained in the sedimentation study was: 82% for color removal (21 CU of residual); 62% for iron removal (1.3 mgFe/L of residual) and 32% for manganese removal (0.12

mgMn/L of residual). On the other hand, the best group of results from DAF investigations was: 98% for color removal (<2 CU of residual); 95% for iron removal (0.17 mgFe/L of residual); 95% for turbidity removal (0.7 NTU of residual) and 52% for manganese removal (0.12 mgMn/L of residual).

The manganese residual of the filtered effluent of the DAF essay carried out by applying 15 mgFeCl<sub>3</sub>/L at pH around 6.3 was 0.10 mg/L, which is exactly the limit of the Brazilian drinking water standard. Therefore, when using DAF associated with a low coagulation pH an alternative to improve even more the manganese removal would be the application of an additional oxidant dosage just after the DAF step. In this way, it would be possible to promote the manganese oxidation and its subsequent removal in the filtration step. Thus, even using chlorine, the risk of THM formation would be minimized due to the very high color removal efficiency of the prior DAF step. Anyway, additional research is recommended to confirm this assumption.

The determinations of the THM concentrations in the DAF and sedimentation effluents were performed (only for the best conditions of both kinds of essays). After the oxidation (2.6 mgCl<sub>2</sub>/L)/sedimentation essay the THM concentration was 4.0 µg/L and after the oxidation (2.6 mgCl<sub>2</sub>/L)/DAF essay was 6,5 µg/L. Thus, for these conditions the final THM concentration values were both below the Brazilian drinking water standard limit (100 µg/L).

DAF alternative was considered an attractive alternative treating the synthetic water with a high efficiency in a higher loading rate than sedimentation did. Besides, decreasing the THM formation risks with a low consume of chemicals and producing less sludge. Furthermore, DAF was the best alternative to remove organic iron, turbidity and color.

## Conclusions

- Iron and manganese analyses in samples digested and non-digested showed that iron was linked in a stronger way than manganese did.
- Zeta Potential essays showed that the pH range between 6.0 and 7.0 presented values by -5 to +5 mV and this range was in agreement with the best results of coagulation for both DAF and sedimentation.
- DAF/Filtration processes carried out by applying low dosage chlorine oxidation and low coagulation pH (around 6.3) constitute a more attractive alternative than that for high coagulation pH/oxidation/sedimentation systems to effectively clarify and simultaneously remove iron and manganese ions from this kind of water.

## Recommendation

It is recommended for future investigations the use of alternative oxidants to promote effective low pH Mn oxidation and to minimize the THM formation risk.

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