

OPERATIONAL/PRACTICAL PAPER

Natural organic matter coagulation in Valencia water supply. Pilot plant studies

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ABSTRACT: To reduce disinfection by-product (DBP) formation in drinking water treatment, the presence of natural organic matter in surface waters must be minimised. This paper describes pilot plant studies carried out on two surface waters to assess the effectiveness of coagulation in organic matter removal, the Turia and Jucar rivers, which supply the city of Valencia (1m inhabitants). The experiments were conducted with different coagulants (iron sulphate, polyaluminium chloride (PACl)) and treatment schemes. Process effectiveness was evaluated in terms of effluent turbidity, presence of residual metal in final water, and organic matter removal. Four parameters were used to quantify organic matter concentration: total organic carbon (TOC), trihalomethane precursors, ultraviolet absorbance at 254 nm, and oxidability to permanganate. Iron sulphate yielded better results than PACl. Finally, some mathematical relationships were established between coagulant dosages and organic matter removal.

This paper involves a preliminary study on controlling DBP formation in conventional treatment. Future work will set out the results obtained using GAC filtration and ozonation.

INTRODUCTION

The coagulation process has traditionally been used to remove turbidity. However, coagulation has also been shown to remove some natural organic matter (NOM) fractions present in water [1–3]. NOM removal is of considerable significance due to the need to minimise disinfection by-product formation.

The four main factors affecting coagulation-driven NOM removal in water are: coagulant type and dosage, coagulation pH and water characteristics [4].

The most commonly used coagulants in water treatment are aluminium and iron salts, or prehydrolysed aluminium compounds (PACl). Aluminium salts have sometimes been more effective than iron salts in organic matter removal, but the opposite is usually true [5]. The coagulation of fulvic acids by alum and PACl was examined by Van Benchosten & Edzwald [6]. They found that although the aluminium species in solutions of alum and PACl are different, the dosage requirements of these coagulants are nearly identical for fulvic acid removal. However, aluminium hydrolysis and solubility are affected by temperature. PACl is prehydrolysed and therefore less sensitive to temperature variations.

The dependence of organic matter removal on coagulant

dosage follows one of the two different patterns illustrated in Fig. 1 [7].

In type 1, removal increases very sharply at a particular dosage. This behaviour is indicative of a precipitation mechanism and is associated with low pH and high concentrations of organic matter (especially fulvic and humic acids). Such removal has been shown to be stoichiometric [3,6–9].

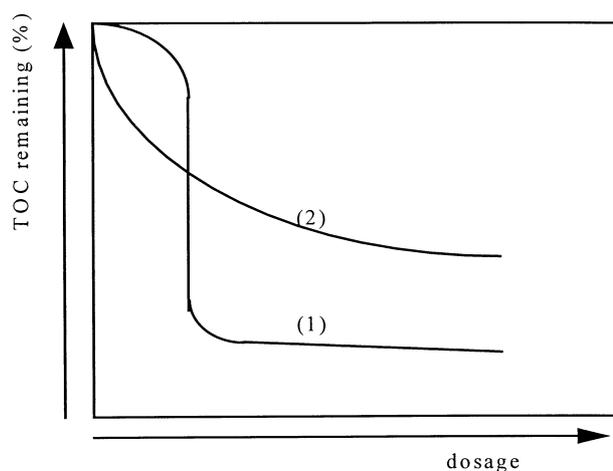


Fig. 1 Coagulation of organic matter: differing behaviour.

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In type 2, removal gradually increases with a rising coagulant dosage. This behaviour is most common for surface water supplies with moderate or high turbidity and alkalinity, and is associated with high pH and low concentrations of organic matter [1,10–12]. The main mechanism involved in this process is coprecipitation. In this case, the relationship is not necessarily stoichiometric.

According to the literature, the suitable pH for removing organic matter is 5–6 for aluminium salts and 4–5 for iron salts [1,2,9], though the removal mechanisms are different. At low pH the main removal mechanism is the formation of precipitates between organic matter and metal hydrolysis products [1,2,12,13]. At neutral or basic pH, the main mechanism is coprecipitation by adsorption onto the precipitate [10,11].

Water characteristics are an important factor in organic matter removal by coagulation. Alkalinity, the presence of divalent cations, sulphate and turbidity can affect the process [12–15].

Alkalinity is the most important factor, because it governs pH. If alkalinity is low, adequate pH for organics removal is achieved with low coagulant dosage. If alkalinity is high, acid pH to favour organics removal can be achieved by increasing coagulant dosage or lowering pH with chemical reactives (acids). This need for increased chemicals has many problems (increasing coagulant costs, sludge production and costs of adjustment and posterior readjustment of pH to avoid corrosion problems).

Using coagulation assays (jar-test) with different raw waters in an ample range of alkalinity, Chadik & Amy [12], showed that waters with low alkalinity require a lower coagulant dosage for good removal of organic matter than waters with high alkalinity; moreover, yields are higher.

The presence of divalent cations (calcium and magnesium) enhances the removal of organic matter by coagulation [13–15].

The sulphate usually present in the water modifies coagulation, inhibiting the removal of organic matter [14,15].

Turbidity has generally been found to have little or no effect on the optimum conditions for NOM removal. However, the presence of organic compounds affects the removal of turbidity [16].

Natural waters exhibit different characteristics that can interact with the treatment process, leading to different results. Bench-scale and pilot plant experiments are required to allow selecting an appropriate treatment scheme.

MATERIALS AND METHODS

To select the most suitable coagulant and treatment scheme for the 'La Presa' treatment plant, several series of experiments were carried out in a pilot plant. For the treatment scheme and coagulant selected, natural organic matter removal was optimised. The experiments were carried out over a period of two years.

Pilot plant

The pilot plant was set up to study the coagulation and flocculation process and possible modifications to conventional treatment (GAC filtration and ozonation). The pilot plant consisted of the following stages: coagulation, flocculation, decantation, rapid sand filtration, ozonation and GAC filtration. A scheme of the pilot plant is shown in Fig. 2. The characteristics of the different treatment sections are summarised in Table 1. Historically presence of ammonium in the Turia river water has been a common problem, and so a nitrification step was included in the pilot plant. However, when an experiment was carried out, ammonium was not detected, hence it was decided not to consider nitrification in the study.

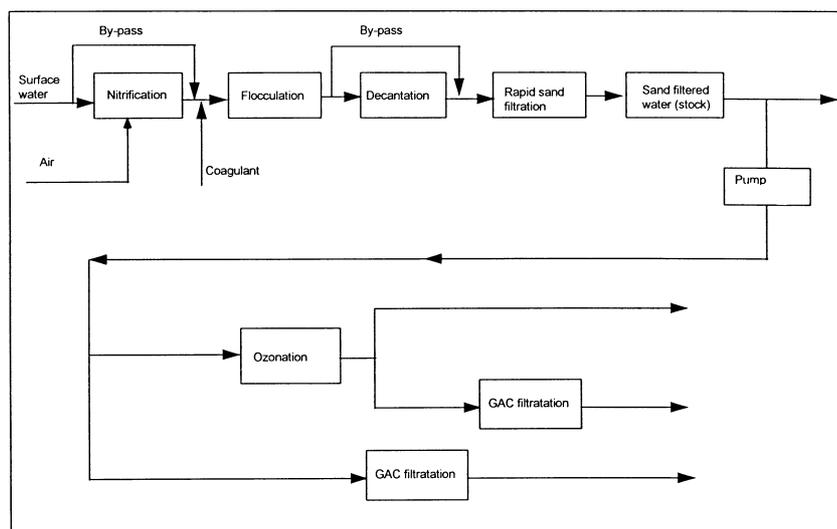


Fig. 2 Experimental set up.

Table 1 Characteristics of the different pilot plant sections

Stage	Q (m ³ /h)	V (m ³)	Characteristics
Flocculation	0.500	0.167	Residence time: 20 min
Decantation	0.500	1.50	Residence time: 3 h
Rapid sand filtration	0.500	0.24	Rise rate: 0.5 m/h Bed height: 1 m V filtration: 5 m/h
Ozonation	0.180	0.0911	
GAC filtration	0.090	0.0353	Bed height: 1.3 m V filtration: 5 m/h

The influent (Turia or Jucar river water) was pumped into the plant at a flow rate of 500 L/h using a centrifugal pump. To control the water flow, rotameters and fluid counters were used. For the coagulation, chemical reactive addition was performed in the influent pipe using a static mixer. To control the coagulant dosage a membrane metering pump was used. In the flocculator, to promote collisions between particles so that flocs were formed a slow (20 r.p.m.) mixer was used. The decantation process was carried out in a static decanter (effective volume: 1.5 m³). Water was clarified in a gravity filter, the bed consisting in silica sand (effective size: 1 mm, bed height: 1 m, filtration velocity: 5 m/h).

Coagulants

The coagulants used in this study were: iron sulphate (Ferriclar) with 44% Fe₂(SO₄)₃ and PACl (PAX 10) with 9.5% Al₂O₃. An anionic polyelectrolyte (Floerger AN910) was used as an adjuvant to coagulation.

To control the performance of the process, two samples from the different stages effluent were taken daily and physico-chemical parameters were determined (temperature, pH, conductivity, colour, turbidity, ammonium, nitrites, nitrates, iron, manganese, aluminium, sulphate, UV absorbance, hardness, alkalinity, calcium, magnesium and oxidability to permanganate). To estimate organics removal (TOC and THM precursors) one sample was analysed weekly.

Analytical techniques

Turbidity was measured with a HACH 2100 N turbidimeter (ISO 7027–1990).

Iron and aluminium concentrations were determined by spectrometric methods, ferrospectral (Merck) and Eriocromoi-cianine method respectively, using a Shimadzu UV-visible spectrometer.

TOC was measured with a Dohrmann DC80 total organic carbon analyser, by persulphate-ultraviolet oxidation of organic carbon. The CO₂ formed in oxidation was measured by infrared spectrophotometric detection (ISO 8245–1987).

UV absorbance at 254 nm was measured using a Shimadzu UV-visible spectrometer and a 50 mm quartz cell.

Table 2 THMFP. Chlorine dosage used for the different TOC concentrations

TOC concentration (mg/L)	Chlorine dosage (mg/L)
0–3	10
3–7	20
7–10	30
> 10	40

Oxidability to permanganate was measured by a volumetric method (ISO 8467–1993).

To measure trihalomethane formation potential (THMFP), the samples were chlorinated according TOC concentration (Table 2) with chlorine solution, and incubated in darkness at 20 °C for 72 h. A Fisons Model GC8000, gas chromatographer with an electron capture detector and a Jm × 0.32 mm, 1.8 μm, capillary column were used to determine the THM formed. Volatile compounds were extracted by the ‘headspace’ procedure (ISO 1031–1994).

Water characteristics

The pilot plant was located in ‘La Presa’ water treatment plant. This location allows working with the two surface waters (Turia and Jucar rivers) used as water sources for the city of Valencia. Table 3 details the average values of the characteristics of the two waters involved in the study and the usual variation range.

Table 3 reveals that both waters presented similar characteristics, although the Jucar river water exhibited higher turbidity, with a higher colloidal character than the Turia river water. Moreover, the Turia river water yielded higher alkalinity, hardness and sulphate values than Jucar river water. Alkalinity, sulphates and hardness have opposite effects on organic matter removal by coagulation. Thus, these factors should be considered.

Although the major group of trihalomethane precursors are often humic and fulvic acids derived from plant material decomposition, algae are potential precursors of humic substances due to their biomass and extracellular products [17]. Therefore, during warm seasons, due to bloom algae growth, especially in waters controlled by open reservoirs, THMFP concentration exceeds normal values.

In Fig. 3, the evolution of THMFP for the two waters, over approximately two years (the period of the study) is shown. In both cases the maximum THMFP values were obtained during the warmer summer and autumn months. The lowest values were obtained during the winter months.

Table 3 Water characteristics

	Turia river water	Jucar river water
Temp. (°C)	—	—
pH	8.10 (8.22–7.95)	8.21 (8.22–8.18)
Conductivity (20 °C) (μS/cm)	1250 (1330–1190)	1010 (1027–1008)
Colour (Pt-C0 scale)	1 < > 5	1 < > 5
Turbidity (NTU)	13(18–2)	22 (34–13)
Ammonium (mg/L)	<0.1	<0.1
Nitrites (mg/L)	<0.01	<0.01
Nitrates (mg/L)	16–30	5
Iron (mg/L)	0.010	0.010
Manganese (mg/L)	N.D.	N.D.
Aluminium (mg/L)	N.D.	N.D.
Sulphate (mg/L)	310 (320–305)	290 (295–285)
UV abs (254 nm)	0.142 (0.220–0.120)	0.120 (0.174–0.112)
Alkalinity (mg CaCO ₃ /L)	200 (204–182)	182 (185–176)
Hardness (mg CaCO ₃ /L)	530 (560–524)	507 (508–496)
Calcium (mg/L)	155 (160–150)	129 (132–129)
Magnesium (mg/L)	40 (42–39)	41 (42–40)
Permang. Oxidability (mg O ₂ /L)	1.20 (2.10–0.96)	1.10 (1.80–0.90)
TOC (mg/L)	2.0 (2.2–1.8)	1.8 (2.1–1.5)

N.D.: Not detected

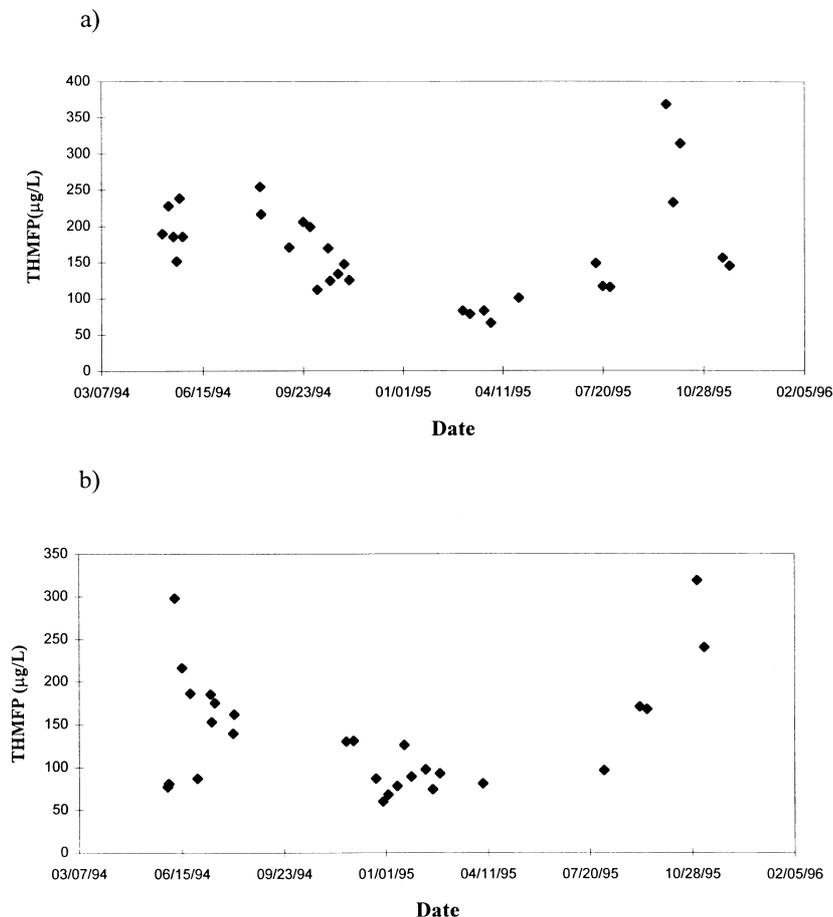


Fig. 3 THMFP evolution: (a) Turia river water; and (b) Jucar river water.

RESULTS AND DISCUSSION

Preliminary research

In order to select the most suitable coagulant and treatment scheme, several series of experiments were carried out with the two waters. The studied treatment schemes were: (a) coagulant addition (iron sulphate or PACl), flocculation, decantation and rapid sand filtration; (b) coagulant addition (iron sulphate or PACl), polyelectrolyte addition, flocculation, decantation and rapid sand filtration; and (c) coagulant addition (iron sulphate or PACl), flocculation and rapid sand filtration. The experiments were carried out for three weeks with each water, coagulant and treatment scheme. Since variations in the influent characteristics could not be controlled, the coagulant dosage was adjusted daily, sometimes different coagulant dosages were used.

The experiments were performed at natural water pH, being pH values continuously monitored. Although NOM removal increases at acid pH, the elevated alkalinity makes pH adjustment difficult. Moreover in industrial plant upgrading pH adjustment was not contemplated.

In the experiments with a decantation stage, the coagulant dosage used was determined daily in laboratory jar-test assays. In the experiments without decantation, the coagulant dosage was one-third of the dosage determined in the jar-test assays. Using lower coagulant dosages in these experiments prevented rapid clogging of the sand filter.

Turbidity removal

Table 4 details the data obtained on turbidity removal for the two waters with different coagulant and treatment schemes. It can be observed that a similar overall yield was obtained in all the experiments.

Presence of residual metal in final water (after sand filtration)

Table 5 lists the metal concentrations (according to the coagulant used). In all the experiments in which iron sulphate was used, the presence of iron in the final sample of water was not detected (detection limit 0.01 mg/L). When PACl was used, the presence of aluminium in final water was always detected, even at significant concentrations.

This fact can be attributed to the coagulation pH. In the equilibrium diagrams for iron and aluminium species, at the coagulation pH used (approaching 8), a high fraction of aluminium is present as a polymeric soluble species. At this pH most iron species are insoluble.

THM precursor removal

The results for THMFP obtained for the different coagulant and treatment schemes are summarised in Fig. 4 for Turia river water and Jucar river water. The coagulant dosages in p.p.m. are given in brackets. To avoid any influence of coagulant dosage on organic matter removal, the experiments which were conducted with approximately the same coagulant dosage were selected.

Table 4 Turia and Jucar river water turbidity and removal values

	Inlet turbidity (NTU)	Coagulant dose (p.p.m.)	Ccoagulation (pH)	Decanted turbidity (NTU)	Removal (%)	Filtered turbidity (NTU)	Removal (%)	Total removal (%)
Turia water								
IS	19.0	10	8.00	3.3	82.4	0.3	92.3	98.6
IS + poly.	7.4	10	8.00	1.9	74.3	0.4	80.3	94.9
IS without dec.	7.5	5	8.05	—	—	0.3	—	96.2
PACl	9.9	10	8.05	3.5	64.4	0.3	90.5	96.6
PACl + poly.	9.1	10	8.05	3.8	58.2	0.3	92.1	96.7
PACl without dec.	6.1	5	8.05	—	—	0.5	—	91.8
Jucar water								
IS	14.5	10	8.10	1.9	87.0	0.3	83.1	97.8
IS + poly.	15.2	10	8.10	2.3	84.9	0.4	84.4	97.6
IS without dec.	16.4	5	8.12	—	—	0.3	—	98.1
PACl	11.9	10	8.10	2.7	77.2	0.3	88.2	97.3
PACl + poly.	22.6	15	8.05	3.3	85.6	0.4	89.1	98.4
PACl without dec.	22.6	5	8.12	—	—	0.4	—	98.1

IS—Treatment with iron sulphate and decantation stage; PACl—Treatment with PACl and decantation stage; IS + poly.—Treatment with iron sulphate, polyelectrolyte, and decantation stage; PACl + poly.—Treatment with PACl, polyelectrolyte, and decantation stage; IS without dec.—Treatment with iron sulphate without a decantation stage; PACl without dec.—Treatment with PACl without a decantation stage.

Table 5 Turia and Jucar river water iron and aluminium concentrations and removals by the studied treatment schemes

	Inlet iron (mg/L)	Decanted iron (mg/L)	Filtered iron (mg/L)	Filtered water (pH)	Removal (%)
IS + Turia water					
IS	0.06	0.97	0.01	8.00	99.3
IS + poly.	0.10	0.38	0.01	8.00	93.3
IS without dec.	0.02	–	0.01	8.05	–
IS + Jucar water					
IS	0.03	0.45	N.D.	8.10	99.4
IS + poly.	0.03	0.56	N.D.	8.10	93.6
IS without dec.	0.03	–	N.D.	8.10	–
	Inlet aluminium (mg/L)	Decanted aluminium (mg/L)	Filtered aluminium (mg/L)	Filtered water (pH)	Removal (%)
PACl + Turia water					
PACl	N.D.	0.53	0.27	8.00	48.3
PACl + poly.	N.D.	0.48	0.19	8.00	60.6
PACl without dec.	N.D.	–	0.13	8.05	–
PACl + Jucar water					
PACl	N.D.	0.32	0.19	8.10	40.6
PACl + poly.	N.D.	0.33	0.14	8.05	58.8
PACl without dec.	N.D.	–	0.27	8.15	–

N.D.: not detected (detection limit 0.01 mg/L)

IS—Treatment with iron sulphate and decantation stage; PACl—Treatment with PACl and decantation stage; IS + poly.—Treatment with iron sulphate, polyelectrolyte, and decantation stage; PACl + poly.—Treatment with PACl, polyelectrolyte, and decantation stage; IS without dec.—Treatment with iron sulphate without a decantation stage; PACl without dec.—Treatment with PACl without a decantation stage.

In both figures it can be seen that iron sulphate yielded better results than PACl for the same treatment schemes with the two waters.

The lowest performances for the two coagulants were obtained in the experiments without decantation. This was due to the use of lower coagulant dosages and the high pH of coagulation.

The experiments run with a polyelectrolyte and decantation stage for the two waters did not significantly improve on the results obtained in the experiments without the polyelectrolyte. When polyelectrolyte is used as coagulant aid its function is agglomerate the flocs forming stronger and denser flocs. As the adsorption of NOM occurs essentially instantaneously, polyelectrolyte will not enhance significantly NOM removal by coprecipitation.

TOC removal

In this case, similar results to THM precursor removal were obtained (Fig. 4), THM precursors being an important part of TOC.

Therefore, the best treatment scheme for the two waters was:

addition of coagulant (iron sulphate), flocculation, decantation and rapid sand filtration. This scheme was used in all the following experiments.

Removal of natural organic matter with iron sulphate

In this case, for data analysis, the averaged organic matter removal for the different coagulant dosages was used. The measurement of UV absorbance at 254 nm, oxidability to permanganate, TOC and THMFP were used to evaluate organic matter removal. The experiments were carried out at the natural pH of the water.

Figures 5–8 show organic matter removal in terms of oxidability to permanganate, UV absorbance, TOC and THMFP. This removal is expressed as outlet/inlet to the treatment: coagulation, flocculation, decantation and rapid sand filtration.

The curves obtained have an asymptotic tendency. Such behaviour is common in surface water supplies with moderate or high turbidity and alkalinity, and a low organic matter content. In the Turia river water (Figs 5a, 6a, 7a and 8a), dosages of iron sulphate above 10 p.p.m. did not increase

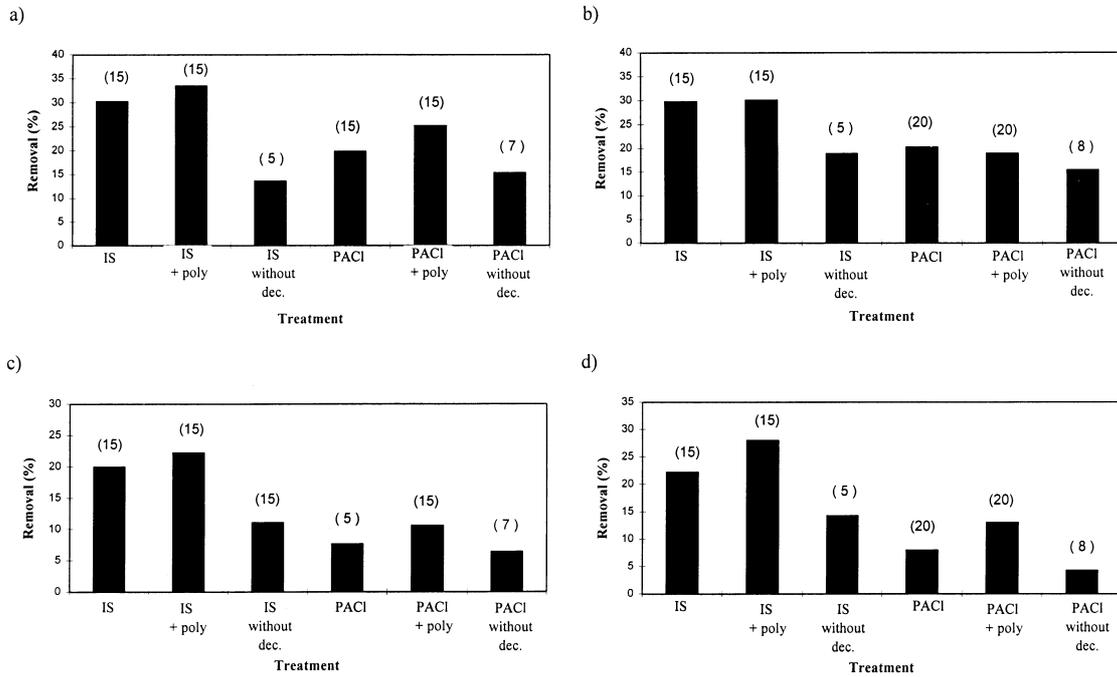


Fig. 4 Organic matter removal for different treatment schemes: (a) THM precursor removal in Turia river water; (b) THM precursor removal in Jucar river water; (c) TOC removal in Turia river water; and (d) TOC removal in Jucar river water.

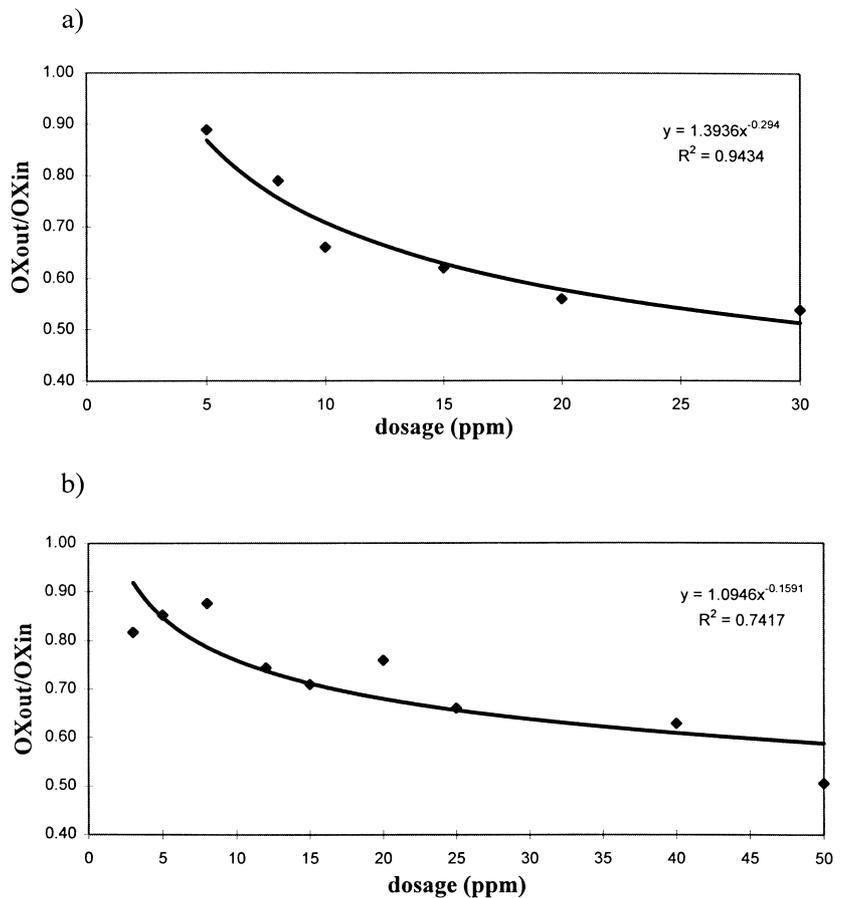


Fig. 5 Removal for oxidability to permanganate with iron sulphate: (a) Turia river water; and (b) Jucar river water.

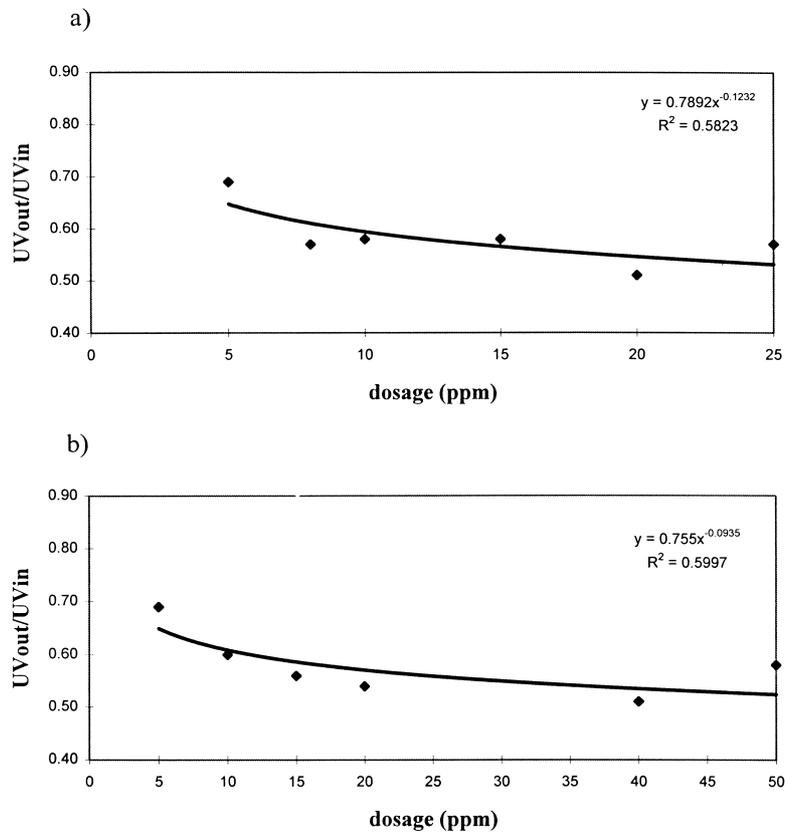


Fig. 6 Removal for UV absorbance at 254 nm with iron sulphate: (a) Turia river water; and (b) Jucar river water.

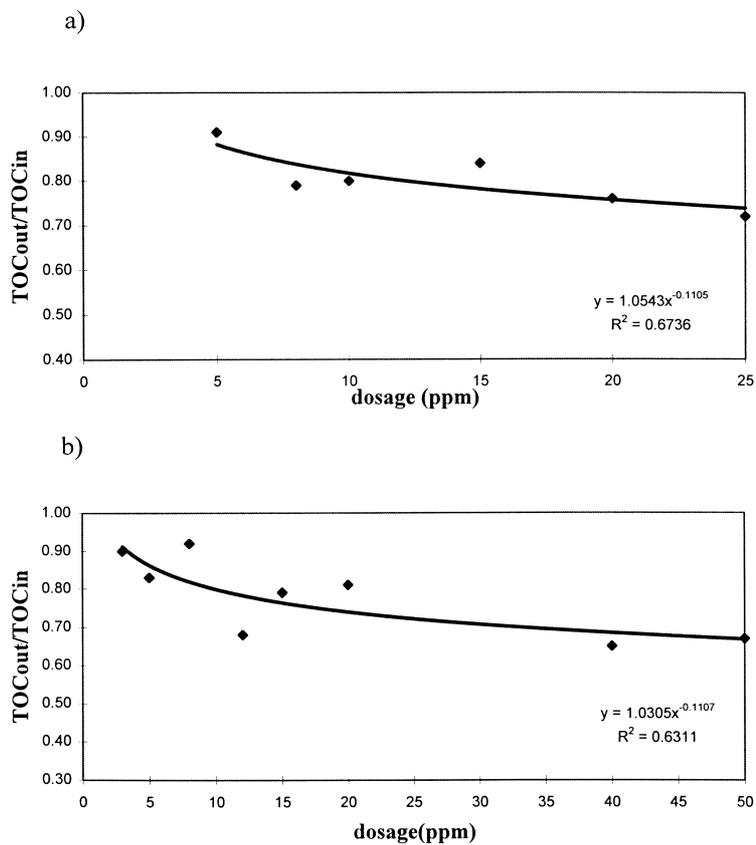


Fig. 7 TOC removal with iron sulphate: (a) Turia river water; and (b) Jucar river water.

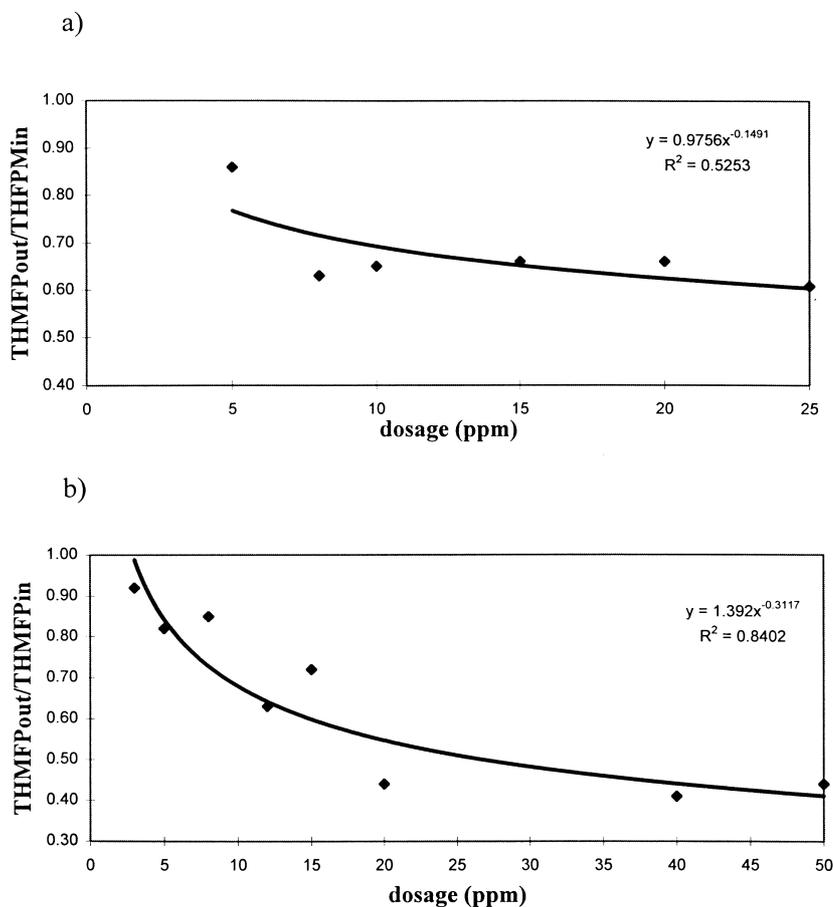


Fig. 8 THM precursor removal with iron sulphate: (a) Turia river water; and (b) Jucar river water.

organic matter removal. In Jucar river water (Figs 5b, 6b, 7b and 8b), the lowest yield was obtained for coagulant dosages below 10 p.p.m. Dosages above 20 p.p.m. did not increase organic matter removal. For oxidability higher dosages are needed, but to control disinfection by-products formation, THMFP is the most important parameter to be considered.

Although the 10 and 20 p.p.m. dosages needed for removing organic matter in Turia and Jucar water, respectively, were higher than the required dosages for removing turbidity, they allowed the satisfactory elimination of turbidity.

In the Turia river water the maximum removals obtained were $\approx 20\%$ for TOC (Fig. 7a), 40% for UV absorbance (Fig. 6a), 45% for oxidability to permanganate (Fig. 5a) and 35% for THMFP (Fig. 8a). In Jucar river water, the maximum removals were 30% for TOC (Fig. 7b) and 40% for UV absorbance (Fig. 6b), while removal was 50% (Fig. 5b) for oxidability to permanganate and 60% (Fig. 8b) for THMFP.

Organic matter removal in the Jucar river water was higher than in the Turia river water. This is attributable to the lower alkalinity and sulphate concentration of the Jucar river water. High alkalinity values decrease NOM removal for a given coagulant dose because the pH does not decrease as much. Moreover, high concentrations of sulphates can deteriorate NOM removal by coagulation. The metal hydrolysis originated

higher pH reduction in Jucar river water (Table 6), promoting the precipitation mechanism. However, no great reduction of pH was achieved (only about 0.3 units) for both waters, due to their high alkalinity.

With regard to hardness, the high concentrations of Ca^{2+} and Mg^{2+} in both waters, were expected to favour organic matter removal by coagulation. But the results obtained in Turia river water, show that alkalinity was the prevailing

Table 6 Lowering of pH caused by coagulant dosage

Iron sulphate dosage (p.p.m.)	Jucar water	Turia water
5	0.10	0.02
10	0.20	0.05
12	0.20	0.10
15	0.25	0.10
20	0.30	0.15
25	0.40	0.20
30	0.50	0.25
40	0.55	0.35
50	0.70	0.40

factor. In natural waters with moderate or high alkalinity values, hardness is not an important parameter to be accounted for, but alkalinity is the most important factor. Although alkalinity and hardness normally are well correlated, hardness is not only associated with alkalinity. In this case both waters presented high concentrations of sulphates. Thus, alkalinity and hardness can be considered separately.

Finally, one important result of the study was that UV absorbance and oxidability to permanganate (rapid determination parameters) could be used to estimate treatment performance. Nevertheless, caution must be exercised when they are used as surrogate parameters for THMFP.

SUMMARY

From the results of the study carried out in the pilot plant with the two waters, it can be concluded that:

- 1 Coagulant type and treatment scheme had no influence on the turbidity of sand-filtered water.
- 2 Using PACl yielded final water with a high aluminium concentration at the pH used.
- 3 The use of iron sulphate as a coagulant in treatment with a decantation stage yielded better results than PACl in removing organic matter. Using a polyelectrolyte did not enhance coagulant effectiveness.
- 4 Coagulant dosage influenced organic matter removal. This influence is higher for THM precursors removal.
- 5 Relationships were obtained between iron sulphate dosage and organic matter removal for the two waters.
- 6 The iron sulphate dosages needed for organic matter removal were higher than the dosages required to remove turbidity, but they allowed satisfactorily eliminating turbidity.
- 7 In natural waters with high values of alkalinity, hardness is not an important parameter to account for, alkalinity is the most important factor.

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