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Comparable Evaluation of Iron-based Coagulants for the Treatment of Surface Water and of Contaminated Tap Water

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Abstract: The aim of this study was to investigate the efficiency of various commercially-available coagulants based on iron, such as ferric or ferrous salts, or polyferric sulfate (PFS), for the treatment of surface (river) water in order to become drinkable. These coagulants were evaluated, considering the removal of suspended solids (SS), of natural organic matter (NOM) and of the residual iron concentration. Furthermore, the kinetics of flocculation was studied by using a Photometric Dispersion Analyzer (PDA), comparing the different flocculation rates. The optimum operational conditions, i.e. coagulant dosage, pH value, duration, and intensity of (initial) rapid-mixing rate, were determined for the examined cases. The results indicated that certain trivalent iron coagulants [Ferrisol-123 (based on $FeClSO_4$) – Ferrisol-Cl-130 (based on $FeCl_3$)] showed higher efficiency, than the divalent [Ferrosol-9 (based on a mixture of $FeSO_4$ and $FeCl_2$), presenting higher SS and NOM removal, higher flocculation rates, and lower residual iron concentration.

Keywords: Coagulant agents, ferric-ferrous coagulants, poly-ferric sulfate (PFS), photometric dispersion analyzer (PDA), drinking water

INTRODUCTION

Among the most widely applied processes for the production of potable water, as well as for the treatment of wastewaters, is the coagulation/flocculation (1, 2). This process is traditionally described as the destabilization of

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colloids, which are present in water supplies and it can be achieved by adding cations, which can interact specifically with the (mostly) negative colloids and therefore, to reduce (or neutralize) their charge. Typically, a small dosage of a poly-valent metal cation, such as iron or aluminum, is added to the water in order to destabilize and aggregate the colloidal particles and to adsorb the dissolved organic matter, facilitating their ultimate removal by means of subsequent sedimentation and/or filtration (3).

As convenient coagulant agents used for water treatment purposes compounds of polyvalent metal cations, such as iron or aluminum, are usually applied. In the case of ferric salts, trivalent ferric ions will hydrolyze rapidly, producing a range of iron species, which play an essential role in the coagulation process. Fe(III) coagulants were also found to be effective in a wide range of pH and temperature values for removing humic substances from water, which consist of the main trihalomethanes (THMs) precursor when coagulated water would be subsequently chlorinated. Given the subsequent health risks, it is necessary also to monitor the residual concentration of aluminium coagulants in the treated water and to take the appropriate measures in order to keep them below a certain concentration level. On the other side, the iron-based coagulants are expected to pose substantial lower health risks, than their aluminum counterparts; this is an important advantage of using ferric coagulants instead of the aluminum-based ones (4).

However, a major drawback in the use of conventional ferric salts is their inability to control the nature of coagulant species, formed rapidly during the addition/dilution stage, and resulting in their relatively poor performance. A possible way of improving the efficacy of coagulation is by the use of pre-polymerized coagulants. Generally, the respective polymers carry a higher cationic charge, which enhance their surface activity and charge-neutralizing capacity, making them more competitive, than the conventional coagulants (5).

Polyferric Sulphate (PFS) is a relatively new polymeric Fe(III) coagulant agent, with a molecular formula $[Fe_2(OH)_n(SO_4)_{3\text{-}n/2}]_m$ (where $n < 2$ *και* $m > 10$), carrying higher positive charge (i.e. $[Fe(OH)_x]^{(3-x)+}$ and $[Fe_m(OH)_x]_n^{(3m-x)n+}$) and having medium to large molecular weights (6). It was suggested that these PFS species are able to persist during the flocculation process and this may be the reason for their improved treatment performance (7). When comparing with the conventional coagulants, PFS is less corrosive and it can be applied effectively in a wider range of pH values, and it consumes less alkalinity, than $FeCl_3$ during the coagulation process. Also, the lower corrosive behavior shown by PFS is expected to lead to the large-scale application of this coagulant agent, especially for wastewater treatment in the near future (4).

Generally, PFS has been shown more efficient than the monomeric salt for removing turbidity, algae, colour and natural organic matter (8–10). Furthermore, according to Jiang and Graham, (1997) who studied the behavior of PFS and ferric sulfate, they found that PFS yields lower rate of hydroxide precipitates, and therefore a faster interaction with contaminants, when compared with the conventional ferric salts (11).

In this study four different types of commercially available iron-based coagulants (simple ferric salts: Ferrisol-123, Ferrisol-Cl-130 and Ferrosol-9, as well as the pre-polymerized PFS) were comparatively examined. The efficiency of these coagulants was mainly evaluated by considering the removal of suspended solids and dissolved organic matter, whereas the kinetics of coagulation was evaluated by using a specific experimental set-up (PDA 2000).

Photometric Dispersion Analyser (PDA-2000) can provide quick and continuous information in terms of flocculation index (correlated with the flocs size), regarding the aggregation state of particles taken place during a modified jar-test procedure. The degree of aggregation is influenced by the intensity of an initial rapid mixing step. Poor rapid mixing failed to induce aggregation and to effectively coagulate the small and difficult settling particles (12). Furthermore, a strong correlation was found between the removal efficiency of the turbidity and a specific parameter derived from the PDA-2000 obtained data, which is defined as the rate of the square root of the flocs size index and the maximum slope of the growing curve (13). The PDA-2000 instrument was used for the study of colloidal suspension flow and the respective coagulation in laboratory tests, as well as in industrial applications (14, 15).

EXPERIMENTAL

Methods

According to the relevant Standard Laboratory Practice, the respective coagulation-flocculation test was carried out in order to determine the chemicals, dosages, and conditions, required to achieve optimum removal results. This practice permits the evaluation of various coagulants and coagulant aids, used in the treatment of water and/or of wastewater.

Coagulation experiments were conducted using a jar-test apparatus (JP SELECTA, Barcelona, Spain). Initially, 1 L of sample was introduced into each of the jars, dosed with the appropriate dosage of coagulant, and the pH was adjusted to the desired value. After the coagulant addition, and before the addition of the polyelectrolyte, a sample of 30 ml was withdrawn for zeta-potential measurements, using a Laser Zee Meter (Model 501) from Pen Kem Inc (Bedford Hills, NY, USA). The suspensions were initially stirred rapidly at 160 rpm for 165 s, and for another 15 s during the polyelectrolyte addition; in all experiments concentration of polyelectrolyte 0.2 mg.L^{-1} was used and slow stirring at 45 rpm for 10 min was subsequently applied. After this period the paddles were withdrawn and the settling of created floc particles took place, lasting for 40 min. These experimental conditions were optimized during preliminary experiments. Following the sedimentation period a supernatant sample (50 ml) was withdrawn for turbidity determined in Nephelometric Turbidity Units-NTU (related to the Suspended Solids concentration), Natural Organic Matter (NOM, estimated by UV$_{254}$ absorbance), pH, conductivity, and residual iron concentration measurements. Turbidity and NOM measurements were carried out using a HACH RATIO/XR Turbidimeter and a Hitachi Spectrophotometer, respectively. Residual iron concentration was measured using the respective photometric test, whereas the pH values by using the pH-meter of Metrohm Herisau (16).

The kinetics of coagulation was evaluated by using PDA-2000. In Fig. 1 a schematic representation of experimental set-up of PDA-2000 is presented. This instrument is measuring the Ratio or Flocculation Index value (*R* or *FI*), which is related to the size of dispersed particles. The examined sample was flowing through a specifically designed cell, illuminated by a narrow light beam (of 850 nm wavelength) and the respective Ratio values were recorded and plotted in relation with the time. The *FI* curves obtained by the PDA-2000 have the typical form shown in Fig 2. At the beginning of coagulation step (i.e. after the addition of coagulant), a small change of *R* (or *FI*) values with time was observed. This phase can be considered as the "lag phase," during which the coagulation of suspension (or the destabilization of colloids) will take place. Then the value of *R* shows a distinct rise, as flocculation starts and the collisions of destabilized particles will result in aggregate formation. As the flocculation increases, the *R* value pass from a maximum and when it obtained a constant value, this means the establishment of an equilibrium between the formation and the breakage of flocs. The larger *R* values correspond to the merrier aggregation.

MATERIALS

Jar tests experiments were performed for the treatment of model (simulated) suspensions using tap water, as well as river/surface water:

- Tap water suspensions: clay micro-particles (commercially available) and dissolved humic acid (obtained from Aldrich Chemicals) were dispersed

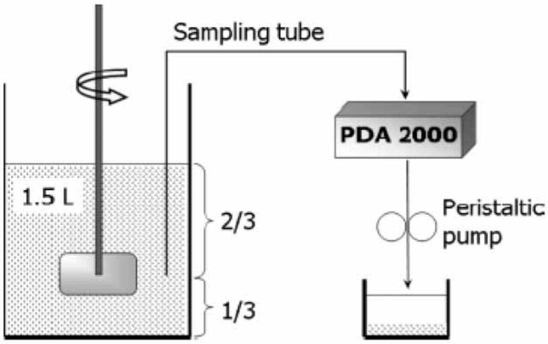

Figure 1. Schematic representation of the experimental set-up during PDA monitoring.

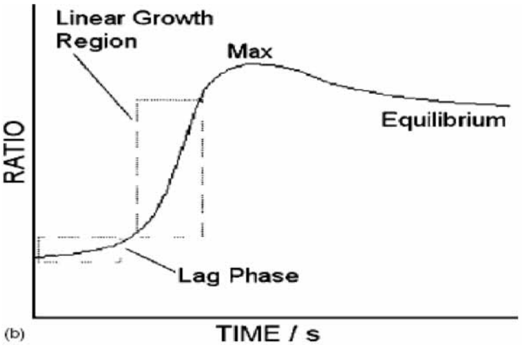

Figure 2. Typical ratio (or FI) curve.

in tap water with final concentrations 5 mg L^{-1} for each, in order to simulate the presence of suspended solids (turbidity) and of natural organic matter (NOM) in surface (natural) waters.

- Surface (river) water samples were also examined, by using water from a supply reservoir of Aliakmon River, located near Thessaloniki city (N. Greece).

The specific characteristics of these water types are presented in Table 1.

The following commercially-available iron-based coagulant reagents, examined in this study, were produced by the Greek Chemical Company Ferri-Tri Inc. (Thessaloniki, Greece) and were:

Ferrisol-123: solution of trivalent chloride sulfate iron $FeClSO_4$, containing 12.3% (w/w Fe^{3+}) and having density 1.52 kg · L^{-1}.

Ferrisol-Cl-130: solution of trivalent chloride iron $FeCl_3$, containing 13.9% (w/w Fe^{3+}) and having density 1.45 kg · L^{-1}.

Ferrosol-9: solution of divalent chloride iron $FeCl_2$, containing 9.0% (w/w Fe^{2+}) and having density 1.30 kg · L^{-1}.

Also, the following coagulation/flocculation reagents were used:

- PFS (PolyFerric Sulfate), obtained from China Chemical Company (Sanghai) with the following main properties (according to our

Table 1. Characteristics of examined waters

Water type	pH	Z-potential (mV)	Turbidity (NTU)	UV_{254}
Contaminated tap water	7.55	−16.8	11.00	0.126
River water	7.80	−14.4	8.00	0.045

measurements): solution of 1 g/L of this reagent was found to contain $Fe_{total} = 1.75$ g/L (25.5% of which is in polymeric form), pH of solution 0.9, having density 1.032 kg/L and ratio $[OH^-]/[Fe^{3+}] = 0.3$.

- "Pure" $FeCl_3 \cdot 6H_2O$ (for comparison reasons) obtained from the Chemical Company Panreac.
- Magnafloc LT-25: anionic polyelectrolyte, obtained from Ciba Speciality Chemicals Ltd (Basel, Switzerland) and used as a flocculant agent (to increase the size of previously produced micro-flocs).

RESULTS AND DISCUSSION

PDA 2000 Experiments

In order to determine the optimum conditions of initial stirring, the respective PDA experiments were conducted using the initially contaminated tap water and by adding $FeCl_3$ at different times of initial rapid mixing, combined with different speed (of initial stirring). Preliminary PDA experiments showed that the optimum rapid mixing time may last 3 min, and the optimum stirring speed must be kept at 160 rpm.

Coagulation of Contaminated Tap Water—Influence of pH on Flocculation Rate

Figure 3 displays the floc growth, regarding the coagulation of contaminated tap water by using Ferrisol-123 at different pH values, but without the addition of polyelectrolyte, in order to investigate only the efficiency of inorganic coagulant addition. The first 180 s corresponds to the rapid mixing phase of the flocculation. At pH 8 better results were obtained comparatively with the other pH values, which were confirmed also by the jar-tests experiments, in which the reduction of turbidity and NOM were effectively obtained.

Similar results were also performed, by using Ferrisol-Cl-130 and Ferrosol-9 at different pH values (data not shown). As with the case of Ferrisol-123, at pH 8.0 rapid kinetic of coagulation was obtained, in comparison with the other pH values, especially with the acidic pH values.

In Fig. 4 a comparable diagram between the different examined coagulant agents is presented: Ferrisol-123, Ferrisol-Cl-130, and Ferrosol-9. As can be noticed the Ferrisol-Cl-130 is showing better coagulation behavior in comparison with the other two reagents, because it presents faster kinetics, which means quicker flocs formation and growth, an observation proved also by the respective jar-tests experiments.

The PDA experiments affirmed the outcome of jar-tests experiments. Observing the results of both experiments (PDA, jar tests), similar conclusions were noticed, i.e. that Ferrisol-Cl-130 was better coagulant agent, than Ferrisol-123 and Ferrosol-9, mainly because of the rapid coagulation

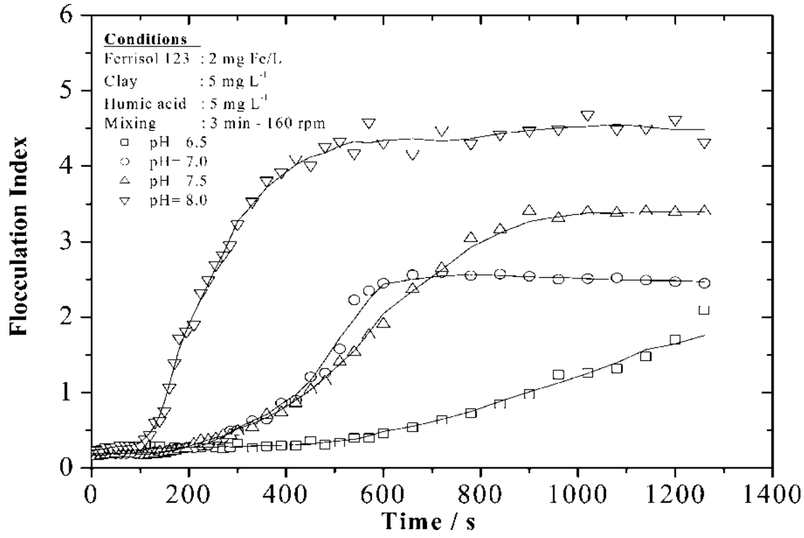

Figure 3. Coagulation of contaminated tap water: influence of different pH values, using Ferrosol-123.

kinetics, which Ferrisol-Cl-130 presented, resulting to better destabilization of colloidal particles and aggregation-flocculation behavior.

Although in this study the influence of temperature was not investigated during jar-tests or PDA experiments, relevant research showed that

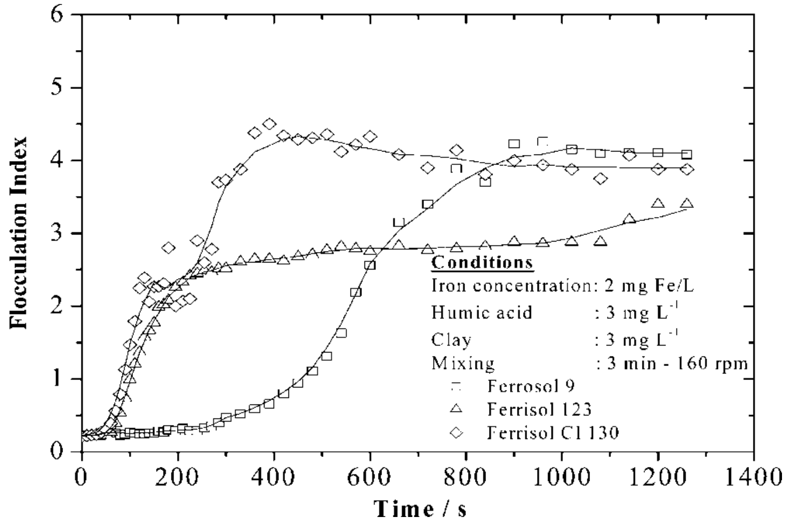

Figure 4. Comparison of iron-based (not-polymerized) commercial coagulants: Ferrosol-9, Ferrisol-123 and Ferrisol-Cl-130 at pH value 8.0.

temperature is known to affect the performance of flocculation, as floc formation is slower at lower temperatures for all the commonly applied coagulant agents. Breakage, in terms of floc size reduction, is also greater for higher temperatures, suggesting a weaker floc. Recovery after the application of increased shear is also greater at lower temperatures, implying that floc break-up is more reversible for lower temperatures (17).

Commercial Coagulants used for the Treatment of (Aliakmon) River Water

Jar-tests of surface (Aliakmon) river water were carried out in order to evaluate the available commercial coagulants and to determine the optimum conditions for the application of them.

Ferrisol-123

The results of the jar-tests experiments using Ferrisol 123 as coagulant are presented in Table 2. The optimum dosage of Ferrisol-123 was found to be 3 mg $\cdot$ L^{-1}, achieving relatively better removal for turbidity.

It can be also observed that as the pH value decrease (Fig. 5) the removals of turbidity and of UV_{254} were also decreased, meaning that the coagulant becomes less active in the acidic area. Therefore, this coagulant is performing better in the alkaline, as at the pH value over 7.5 the amorphous hydrous hydroxide of iron would be formed, helping into the destabilization and aggregation of colloidal particles, thereby facilitating their ultimate removal by means of sedimentation and filtration, which are the usual subsequently applied solids separation processes. In addition, the respective z-potential measurements showed that Ferrisol-123 was slightly reducing the negative surface charge of colloids (Table 2).

Ferrisol-Cl-130

This coagulant was found active in both pH areas (alkaline and acidic) by using a smaller dosage (2 mg . L^{-1} as Fe), as it was shown in the respective results (Table 3). The removal of turbidity was always over 90%, achieving

Table 2. Coagulation of contaminated water at pH 7.5, using Ferrisol-123 as coagulant agent

$[Fe^{3+}]$ mg $\cdot$ L^{-1}	3	5	7	9
Z-potential (mV)	−9.5	−10.5	−11.2	−10.9
Turbidity (NTU)	0.7	0.85	0.9	1.1
UV_{254}	0.041	0.040	0.043	0.044

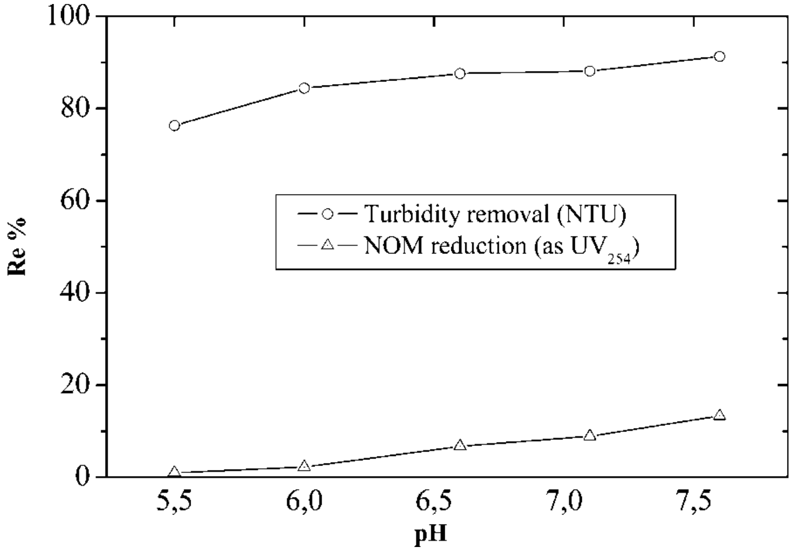

Figure 5. Surface (Aliakmon river) water treatment; turbidity and NOM (as UV_{254} measurements) removals in different pH values. Coagulant (Ferrisol-123) and polyelectrolyte dosages were kept constant at 2 mg . L^{-1} and 0.2 mg . L^{-1}, respectively.

also higher reduction of NOM in comparison with Ferrisol-123, which may be devoted to the slightly lower values of z-potential (Table 3).

Ferrosol-9

In this case, insufficient reduction of turbidity and NOM were obtained for the majority of examined dosages and values of pH. Satisfactory removals of turbidity were obtained only at the acidic pH values (Table 4).

The substantial lower removal of turbidity in comparison with the other iron-based coagulants and under similar experimental conditions can be devoted to the respective hydrolysis products, which seem to contain significant amounts of gelatinous hydrous amorphous iron hydroxides (difficult settling) and as a result, the turbidity was found to increase, instead of the

Table 3. Coagulation of contaminated water, using Ferrisol-Cl-130 at different pH values

$[Fe^{3+}]$ mg · L^{-1}	2	2	2	2	2	2
pH	8.5	8.0	7.5	7.0	6.5	6.0
Z-potential (mV)	−8.5	−8.2	−9.7	−9.8	−10.8	−11.2
Turbidity (NTU)	0.17	0.48	0.5	0.39	0.70	0.75
UV_{254}	0.038	0.032	0.028	0.023	0.033	0.022

Table 4. Coagulation of contaminated water, using Ferrosol-9 at acidic pH values

$[Fe^{3+}]$ mg · L^{-1}	4	4	4	4	4
pH	3.5	4.5	5.5	6.5	7.8
Z-potential	−10.2	−10.9	−11.5	−11.0	−12.8
Turbidity (NTU)	0.89	1.5	14.8	20.0	20.0
UV_{254}	0.228	0.352	0.380	0.400	0.389

expected decrease. Although the other coagulants showed better performance at the neutral or slightly alkaline pH range, Ferrosol-9 was found to perform better at the acidic pH range, reducing (slightly) the surface charge of colloids; as a consequence, relatively better removals of turbidity and NOM were obtained, but in the acidic pH range only (3.5–4.5).

Pfs

This polymeric inorganic coagulant was also examined for the treatment of surface water. In Table 5 and in Fig. 6 the results of jar tests experiments at different pH values and for various dosages of PFS are presented, regarding the removal of turbidity and NOM. Contrary to what Jiang and Graham (5) referred about the behavior of z-potential, in our experiments the opposite trend was observed, i.e. by increasing the pH values towards the alkaline region, the z-potential measurements were found to be slightly decreased.

In these experiments, PFS was found to be less active, than the respective Ferrisol-123 or Ferrisol-Cl-130 agents, as the later reagents were found capable to remove effectively turbidity (i.e. below the achievement goal of 1.0 NTU), with a relatively smaller addition of coagulant in the alkaline pH range. It is also noted that in the acidic pH area similar good results were also obtained, by using Ferrisol-123 or Ferrisol-Cl-130, as compared with those obtained by using PFS.

Comparable Results for the Coagulants used for Treatment of Surface (River) Raw Water

Comparable jar-test experiments were conducted at pH 8.0, using 3 mg·L^{-1} of each coagulant, as these concentrations were previously found optimal

Table 5. Coagulation of contaminated water, using PFS at different pH values

$[Fe^{3+}]$ (mg · L^{-1})	4	4	4	4
pH	7.5	7.0	6.5	6.0
Z-potential (mV)	−9.8	−9.9	−10.4	−11.7
Turbidity (NTU)	0.40	0.70	1.00	1.50
UV_{254}	0.028	0.032	0.038	0.031

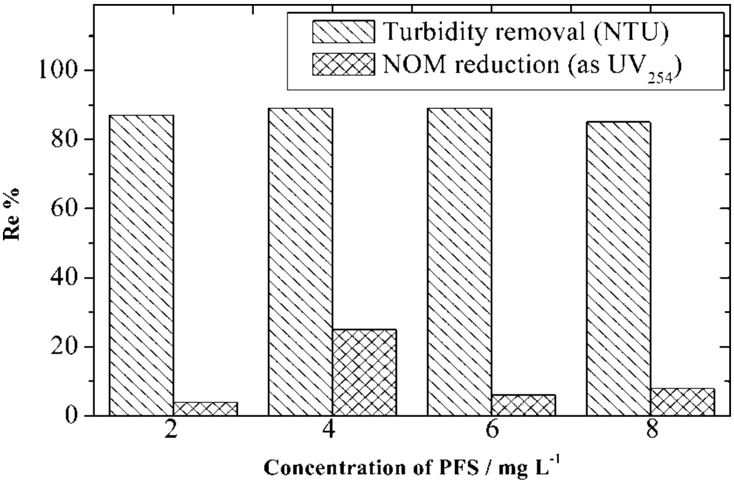

Figure 6. Surface (river) water treatment by using different concentrations of (pre-polymerized) PFS coagulant at pH 7.8; the results are expressed as turbidity and NOM (as UV_{254} measurements) percentage removals, whereas the polyelectrolyte dosage was kept constant at 0.2 mg · L^{-1}.

for the surface (river) water treatment. The rest of the experimental conditions were the same as previously mentioned, i.e. initial rapid mixing at 160 rpm for 165 s, followed by slow stirring at 45 rpm for 10 min and finally by 40 min for gravity settling of the flocs. Table 6 presents the comparable experimental results, following the coagulation treatment by using different iron-based coagulants.

It can be observed that the residual iron concentration exceeded the upper permissible limit for drinking water (200 μg · L^{-1}, according to E.U. Dir. 98/83/EC), when using PFS, Ferrosol-9 and FeCl3, whereas the use of Ferrisol-123 or Ferrisol-Cl-130 produced generally lower residual iron concentrations than this concentration limit. The respective z-potential measurements indicate

Table 6. Comparison between different iron-based coagulants at pH 8 for the treatment of surface (river) water

Coagulant type	Ferrisol-123	Ferrisol-Cl-130	PFS	$FeCl_3$	Ferrosol 9
$[Fe^{3+}]$ (mg · L^{-1})	3	3	3	3	3
Z-potential (mV)	−9.0	−8.5	−10.6	−10.1	−15.4
Turbidity (NTU)	0.40	0.30	1.70	0.65	>20
UV_{254}	0.039	0.037	0.044	0.029	0.300
Residual Fe concentration (μg · L^{-1})	195	175	320	215	740

that the most efficient coagulant, i.e. producing the higher reduction of the negative charge and therefore, the better destabilization of colloidal particles, were achieved by using mainly Ferrisol-Cl-130, followed by Ferrisol-123.

Figure 7 presents the influence of different iron-based coagulants at pH 8.5 for the percentage removal of turbidity and of NOM. It can be observed that better treatment results were obtained by using Ferrisol-123 or Ferrisol-Cl-130, followed closely by $FeCl_3$ which present rather similar behavior, i.e. around or over 40% reduction of UV absorbance and over 90% removal of turbidity. Ferrosol-9 and PFS agents showed the worst treatment efficiency, as the final turbidity values were around 1-3 NTU, therefore in this case the removal goal for turbidity (<1 NTU) could not be achieved.

Effect of pH on the Treatment of Surface (River) Water

The results presented in Table 6 were obtained at the natural pH value of surface water (i.e. 8). As it was expected from the relevant literature studies (18). The NOM and turbidity removals were not found satisfactory at the acidic pH values, probably because of the insufficient reduction of the negative surface charge of colloids, as indicated by the respective z-potential measurements. Therefore, the role of pH is significant, because it can substantially influence the surface charge of organic and inorganic colloids. Higher (more alkaline) pH values (8.5) may improve the removal

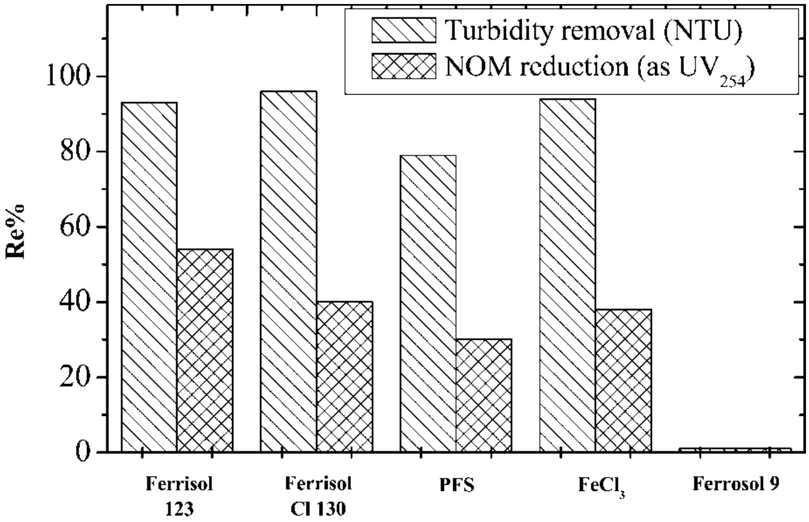

Figure 7. Treatment of surface (river) water, using different types of iron-based coagulants under comparable experimental conditions: pH 8.5, coagulant and polyelectrolyte dosage kept constant at 2 mg · L^{-1} and 0.2 mg · L^{-1}, respectively. The results were expressed as percentage removal of turbidity and of NOM (UV_{254} measurements).

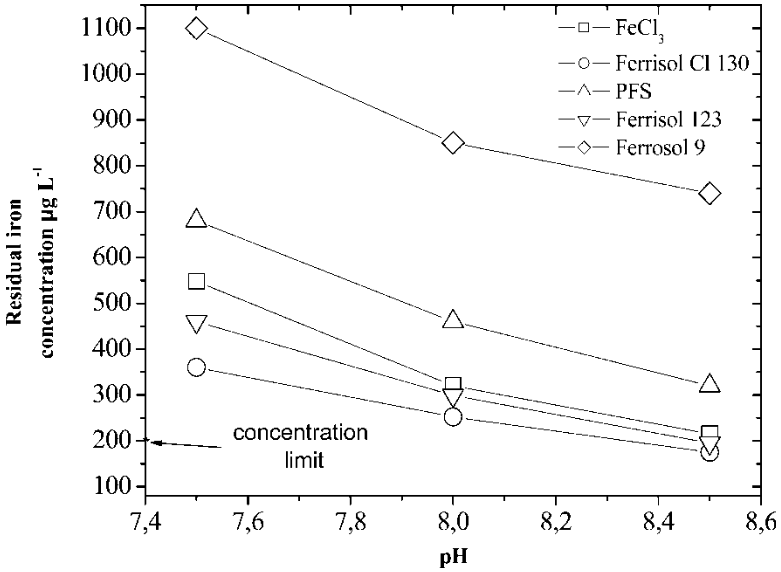

Figure 8. Treatment of surface (river) water treatment; effect of initial pH value on the residual iron concentration, using different iron coagulant types: the dosage of coagulant and polyelectrolyte were kept constant at 3 mg · L^{-1} and 0.2 mg . L^{-1}, respectively.

of soluble and colloidal matter, as well as of turbidity (Fig. 7). The optimum pH value, regarding the higher efficiency of coagulation, depends mainly on the content of the organic compounds and on the type of the applied coagulant.

The effect of pH on the residual iron concentration can be clearly noticed in Fig. 8. As the pH value was increased towards more alkaline pH values, the residual iron concentration was found to decrease.

CONCLUSIONS

In this study the results of comparable evaluation between different iron-based coagulants were presented, regarding the treatment of surface (river) water. Better coagulants for water treatment were found to be Ferrisol-123 and Ferrisol-Cl-130, as they were able to remove higher percentages of turbidity (around or more than 90%) and of NOM (around or more than 40%), respectively. In addition, relatively lower z-potential values were obtained for the negatively charged colloidal particles at higher (slightly alkaline) pH values. Furthermore, for both of these coagulants in the pH range 8–8.5 lower residual iron concentration was found, as compared with the respective legislative concentration limit (200 μg · L^{-1}).

On the contrary, Ferrosol-9 which contains divalent iron was not found effective for water treatment at the pH values 5.5–8.5, although at the pH value 3.5–4.5 sufficient removal of SS was observed. This behavior was also confirmed by the respective z-potential values. As the pH value was decreased, the negative charge of the colloids was reduced, and consequently, the aggregation may be more satisfactory, facilitating the further coagulation and the significant removal of turbidity.

Also, the specific PFS agent, examined in this study did not show good results, as was expected from the relevant literature (6, 11). The monomeric salts of iron showed better removal in comparison with PFS. It was found that PFS was more effective at the pH values (7.5–8.5), as in this area the removal of SS and NOM is higher, than in the acidic area.

The study of coagulation kinetics by using the PDA 2000 experimental set-up, showed that higher coagulation rate was also obtained by the addition of Ferrisol-Cl-130. Several parameters, such as pH, mixing time and intensity of the initial rapid mixing stage of coagulation process, were found to influence the removals of turbidity and of NOM, as achieved by the coagulation process. The flocculation rate was found to be sufficient at the pH value about 8 (i.e. in the slightly alkaline area) for the simulated (test) suspensions and for the examined coagulant types.

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