

THE INFLUENCE OF IRON-BASED CO-PRECIPITANTS ON ACTIVATED SLUDGE BIOMASS

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European Community legislation will increase the requirement for efficient phosphorus removal. The impact of chemical phosphorus removal by co-precipitation has only been considered from an effluent quality point of view. This paper considers the direct impact of two iron-based chemicals—iron (III) chloride and iron (II) sulphate—on an activated sludge pilot plant. Average chemical oxygen demand (COD) removals were 84.0% for iron (III) and 78.6% for iron (II). Nitrification appeared to be inhibited by the addition of high doses of iron (III). Ammonia removals of just 17.3% were observed compared with 88.7% for iron (II) addition. Suspended solids removal was poor (average removals = 69.5% for iron (II) and 48.0% for iron (III)) during the last few months of the trial due to problems with filamentous bacteria and denitrification in the sedimentation tanks. The oxygen uptake of the biomass dosed with iron (II) ($0.0121 \text{ mg O}_2 \text{ mg biomass}^{-1} \text{ h}^{-1}$) was on average 32.4% higher than that of iron (III); the latter averaged $0.0086 \text{ mg O}_2 \text{ mg biomass}^{-1} \text{ h}^{-1}$. Species diversity index (S_T) was similar for both chemicals; $S_T = 24.7$ with iron (II) addition and $S_T = 22.1$ with iron (III). There was a difference in the floc characteristics of the biomass. The flocs produced with iron (II) were larger and more dense than those with iron (III).

Keywords: co-precipitation; iron; phosphorus removal; respirometry; species diversity.

INTRODUCTION

Iron is commonly present in two oxidation states, iron (II) and iron (III). Iron sulphates and chlorides are effective precipitants for phosphorus (P) removal. Iron (III) is generally used more often in the water industry but iron (II) can be used if it is first oxidized to iron (III)¹. Iron (III) forms strong complexes with the condensed phosphates, pyrophosphate and tripolyphosphate, which may then be removed by adsorption onto iron (III) hydroxo-phosphate surfaces¹. The optimum pH for precipitation using iron (III) is approximately pH 5 and the theoretical optimum dosage is an Fe:P molar ratio of 1:1². Of the many complexes formed during precipitation, the most common is $[\text{Fe}(\text{HPO}_4)_2]^{+}$. Iron (II) is less pH dependent than iron (III) and when used to form iron (III) *in-situ* is a more efficient precipitant than a stock solution of iron (III)³. This may be because *in-situ* generation of iron (III) can promote a more effective contact between iron and the phosphate⁴. The main advantages that iron salts have over other precipitants are low cost and sludges which are easy to dewater⁵. In terms of cost, this is particularly true for iron (II) which can be used in the form of waste pickle liquor from the steel industry⁶.

Chemical precipitants can be added directly to the biomass of a biological system, known as co-precipitation. Past research has shown that co-precipitation processes are very effective. For example, P removal efficiencies ranging from 73–95% have been achieved using a variety of iron salts⁷. Iron salts have also been combined with polymers

to produce P removals of >85% during co-precipitation processes⁷. Other findings with iron-based compounds include the addition of iron (II) sulphate to a biological aerated filter⁶. At a dose of 1.25:1 (molar ratio Fe:P) P removals averaging 88.5% were produced.

The effect of iron-based precipitants on the biomass of the treatment system has been monitored only as deterioration in biological treatment in terms of effluent quality^{7,8}. Other effects on the biological system—such as changes in species diversity (S_T) and oxygen uptake—have not been examined.

This research aims to investigate the direct effects of adding two iron-based precipitants on both performance parameters (such as chemical oxygen demand (COD), ammonia and P removal) and also the oxygen uptake, S_T and floc characteristics of the biomass.

MATERIALS AND METHODS

Pilot Plant System

An activated sludge plant consisting of two parallel test units was fed with primary settled sewage from Cranfield University Sewage Treatment Works, a wastewater comprising domestic and catering waste, and operated for a duration of approximately four months. The feed was supplied from a thermostatically controlled tank operated at 20°C. Each unit consisted of an aeration basin (capacity 330 l) followed by a settling tank (capacity 125 l). A dissolved oxygen (DO_2) concentration of 3–5 mg l^{-1} was

provided by four diffused air aerators submerged in the base of the aeration tank and monitored using a hand held DO₂ probe; the pH of the mixed liquor was also measured daily (Jenway Model 9071 portable DO₂ meter; Model 3071 pH and temperature meter). The plant was fed at a flow rate of 411 h⁻¹, delivered via a twin-headed peristaltic pump (Model 503S, Watson Marlow, Falmouth, UK). The return sludge was fed from the base of the settling column at 100% of the feed rate (twin-headed peristaltic pump, Model 501U, Watson Marlow, Falmouth, UK). The hydraulic retention time (HRT) of the plant was approximately 8 h. To achieve a sludge age of 8 d, mixed liquor was wasted directly from the aeration basin at a rate of 360 ml min⁻¹ for 5 min every hour (twin-headed peristaltic pump, Model 303S, Watson Marlow, Falmouth, UK). A scraper was placed in the clarifier to aid settling and was run at a speed of 30 rpm for 10 min every hour (240 V pump, RS Components Ltd, Northampton, UK). All tubing used was 12.5 mm internal diameter and made of PVC. The peristaltic pump tubing was Marprene (Watson Marlow, Falmouth, UK) for all pumps except the chemical dosing pump which used silicone tubing (Watson Marlow, Falmouth, UK).

Daily maintenance was required to keep the plant running constantly which included cleaning of the rim of the aeration tank and the weir of the settling column. All tubing and pipework was backwashed using tap water every month and all pumps were operated at maximum speed for 5 minutes every day. Samples for settled sewage, mixed liquor and final effluent were collected daily.

Chemical Addition

The chemicals were added to a pre-mixing zone (10 l) suspended at the top of the aeration basin at the same point as the recycled activated sludge and the sewage feed. An impeller was placed in the pre-mixing zone and operated at a speed of 200 rpm (240 V pump SD18M, RS Components Ltd, Northampton, UK) continuously to aid precipitation. The chemicals added were 3M iron (III) chloride and anhydrous iron (II) sulphate (industrial grades, Hays Chemicals Ltd, London, UK). Any dilutions were made using tap water. The chemicals were stored in calibrated lidded plastic vessels and the exact dose of chemical entering each tank was recorded every 24 h.

Analysis

Tests for COD, ammoniacal nitrogen, nitrate, soluble and total P were performed using the Hach Test'n'tube method (Camlab, Cambridge, UK). Parameters including five-day biological oxygen demand (BOD₅), mixed liquor suspended solids (MLSS), total solids and nitrogen (nitrite) were all measured according to standard methods⁹.

Respirometry and Species Diversity

Respirometry is a useful technique for indicating biomass inhibition or stimulation. The oxygen uptake of each biomass (dosed with either iron (II) or iron (III)) was measured every two days both prior to and during dosing. Respirometric measurements were made using an electrolytic manometer closed cell respirometer (Model 017, CES Ltd, Kent, UK). Species diversity and floc characteristics were

examined using a light microscope (New Quodmaster 100, Model M4000-D, (Swift, Japan) with 400× magnification. A qualitative and quantitative method adapted from Eikelboom and van Buijsen¹⁰ was used to record species number and type and floc shape and size as described by Clark *et al.*¹¹.

RESULTS

The maximum Fe (III) dose was originally set at 3:1 (molar ratio of Fe:P) following the results of previous jar testing experiments¹². However at this dose the resulting pH of the aeration tank was too low for biological treatment (pH < 4) and the maximum chemical doses added were 2.2:1 (molar ratio of Fe:total P) for iron (III) and 4:1 (molar ratio Fe:total P) for iron (II). Tables 1 and 2 show the optimum doses for both chemicals. An effluent P concentration limit of 2 mg l⁻¹ was difficult to obtain consistently, primarily due to the variability in the concentration of incoming P (range = 4–11 mg l⁻¹). For both chemicals, the consent may be met continuously at doses ranging from 1.5–2.0:1 (molar ratio Fe:P), allowing for standard deviations. Iron (II) performed less efficiently at the lower end of this scale so a dose of 2:1 would be preferable. Conversely, a dose ratio of < 2:1 would be optimum for iron (III) because the resulting low pH at this dose affects nitrification. At a dose of 1–1.5:1 (Fe:P) iron (III) did not consistently meet legislation, so an optimal dose of 1.75:1 (Fe:P) is suggested to satisfy all criteria (Table 1). The average effluent concentrations at dose ranges of 1.5–2:1 (Fe:P) were 2.2 mg l⁻¹ for iron (II) and 1.8 mg l⁻¹ for iron (III).

The BOD₅ and COD removal in both plants was reduced slightly during chemical addition. The average COD removal during chemical dosing was 83.8% for iron (II) and 80.4% for iron (III) (Table 2), compared to average pre-dosing values of 88.3% for iron (II) and 88.5% for iron (III). COD removals were not found to be significantly different (student t-test to 95% confidence) for the two chemicals. Suspended solids removal was adversely affected during chemical addition from 79.8% to 69.5% (iron (II)) and 85.1% to 66.5% (iron (III)) (Table 2). This was mostly due to presence of excess filamentous bacteria which resulted in sludge bulking. The performance of both chemicals was statistically the same (student t-test, 95% confidence limits). At the beginning of the trial, nitrification improved with ammonia removals increasing from 75.8% to 88.7% with iron (II) addition. The reverse effect was seen with iron (III), where the ammonia removal efficiency decreased from 76.9% to 17.3% (Table 2). This may be a direct effect of the low pH (mean pH = 4.80) resulting from the higher chemical dose (> 1.75:1 molar ratio Fe:P) (Table 1). Towards the end of this dosing period, a high proportion of filamentous bacteria was noticed in both mixed liquors (test and control). This resulted in poor settlement and high solids carryover in the final effluent, possibly resulting in a loss of the autotrophic bacteria responsible for nitrification. The chemical dosing was stopped for a period of 3 sludge ages whilst adjustments were made to reduce the filamentous bacteria (Tables 1–4). The adjustments made included changes in DO₂ concentration in the aeration tank, a slight reduction in sludge age (to 7 d) and an increased recycled activated sludge flow rate. The presence

Table 1. Phosphorus removal efficiencies at varying iron (II) and iron (III) doses.

Dose	No dose		1.0–1.5:1 Fe:P (Molar ratio)		1.5–2.0:1 Fe:P (Molar ratio)		Interrupted by filamentous bacteria		Interrupted by poor nitrification	
	Fe (II)	Fe (III)	Fe (II)	Fe (III)	Fe (II)	Fe (III)	Fe (II)	Fe (III)	Fe (II)	Fe (III)
Influent data										
Total P, mg l ⁻¹	8.3	8.3	6.8	6.8	8.6	8.6	6.8	6.8	7.9	7.9
Aeration tank pH	6.56	6.49	6.70	6.25	5.95	5.24	6.71	6.52	6.20	6.52
F:M ratio	0.16	0.20	0.13	0.12	0.15	0.11	0.10	0.09	0.16	0.22
MLSS, mg l ⁻¹	2287	1841	1808	1959	2165	3100	2052	2134	3048	2279
Organic fraction of biomass, %	66.9	69.1	60.6	57.8	57.2	59.5	59.9	58.7	52.7	49.0
Effluent data										
Total P, mg l ⁻¹	6.7	6.2	2.1	2.0	2.2	1.8	3.4	3.8	3.6	4.8
Soluble, mg l ⁻¹	5.3	4.9	0.4	0.2	0.3	0.1	1.2	1.3	1.9	0.9
Max P, mg l ⁻¹	11.8	8.4	2.7	3.2	3.1	2.5	10.6	6.9	6.7	7.6
Min P, mg l ⁻¹	3.6	4.0	1.6	1.2	0.9	1.6	1.8	1.4	2.0	2.3
Samples with > 70% P removal efficiency, %	0	0	66.6	66.6	86.0	100	6.0	12.5	6.0	0.0
Samples with > 80% P removal efficiency, %	0	0	0	8.0	14.0	38.0	0.0	0.0	0.0	0.0

of filamentous bacteria was reflected in the poor performance of both plants during this time (Tables 1–3). An inoculum of nitrifying bacteria was added to each aeration tank just prior to the last dosing period and high ammonia removals were obtained temporarily. The biomass dosed with iron (III) experienced increasing nitrite concentrations indicating only partial nitrification was being achieved. Nitrification recovered fully in the biomass dosed with iron (II); however, operational difficulties with the influent feed resulted in an increase in effluent nitrite concentrations (Table 4). The extent of nitrification during each dosing period is shown in Table 4, where the proportions of ammonia, nitrite and nitrate in the effluents are indicated.

Prior to dosing, the oxygen uptake of the active biomass was the same for each biomass (student t-test, 95% confidence) (Figure 1). However whilst dosing, the oxygen

uptake of the biomass dosed with iron (II) was significantly higher than for iron (III) and the student t-test indicated that the oxygen uptakes in the two aeration tanks were significantly different. At the start of the chemical dosing period (17/04/97), the oxygen uptake decreased significantly. Both biomasses recovered within 4 d, however, a reduction in oxygen uptake of 33.8% was observed in the biomass treated with iron (III). The average oxygen uptake rates (endogenous rates) for the dosing period were 0.0121 mg O₂ mg MLSS⁻¹ h⁻¹ for iron (II) and 0.0086 mg O₂ mg MLSS⁻¹ h⁻¹ for iron (III).

The S_T of the biomass dosed with iron (II) = 24.7 compared with an S_T = 22.1 for iron (III) addition (Figure 2) but these were not significantly different (student t-test at 95% confidence). In general, microscopic observations indicated that the biomass treated with iron (II)

Table 2. COD, BOD₅, nitrogen and suspended solids removal in the pilot plant at varying chemical doses.

Parameter	BOD ₅		COD		NH ₄		SS	
	Fe (II)	Fe (III)	Fe (II)	Fe (III)	Fe (II)	Fe (III)	Fe (II)	Fe (III)
No dose								
Influent, mg l ⁻¹	124	124	342	342	23.5	23.5	106	106
Effluent, mg l ⁻¹	4.8	5.0	39.4	39.6	5.6	5.4	19.5	14.1
% removal	96.2	96.0	88.3	88.5	75.8	76.9	79.8	85.1
1.0–1.5:1 Fe:P (Molar ratio)								
Influent, mg l ⁻¹	81.6	81.6	240	240	21.6	21.6	93.0	93.0
Effluent, mg l ⁻¹	4.5	3.8	28.3	26.0	2.0	2.2	10.3	18.8
% removal	94.5	95.3	88.1	88.8	92.3	91.6	89.0	79.6
1.5–2.0:1 Fe:P 2.0 (Molar ratio)								
Influent, mg l ⁻¹	110	110	324	324	32.3	32.3	96.7	96.7
Effluent, mg l ⁻¹	10.1	5.3	65.0	36.0	3.6	25.7	47.3	10.7
% removal	90.8	95.2	78.9	83.5	88.7	17.3	50.0	90.4
Interrupted by filamentous bacteria								
Influent, mg l ⁻¹	65.5	65.5	273	273	24.9	24.9	93.1	93.1
Effluent, mg l ⁻¹	6.7	7.5	45.1	48.2	8.7	5.8	24.5	28.4
% removal	94.6	94.1	84.5	83.0	62.8	71.2	73.2	68.9
Interrupted by poor nitrification								
Influent, mg l ⁻¹	166	166	315	315	24.4	24.4	98.8	98.8
Effluent, mg l ⁻¹	13.0	16.9	52.2	102.2	1.2	4.8	34.2	71.2
% removal	93.6	89.7	83.7	66.3	95.5	80.0	65.7	27.0

Table 3. Percentage total phosphorus removal at varying dose.

Dose	Iron (II)	Iron (III)
No dose	16.6 ± 11.0	20.8 ± 14.7
1.0–1.5:1 Fe:P (Molar ratio)	69.5 ± 3.2	69.5 ± 11.3
1.5–2.0:1 Fe:P 2.0 (Molar ratio)	75.0 ± 5.0	78.9 ± 6.8
Interrupted by filamentous bacteria	56.3 ± 11.8	47.5 ± 18.9
Interrupted by poor nitrification	54.6 ± 13.0	38.0 ± 22.1

produced denser flocs which were larger in size (small to medium sized, 150–500 μm) and very spherical in shape (Table 5). The biomass treated with iron (III) produced weak flocs which were very small in size and irregular in shape (Table 5). Prior to dosing both aeration tanks contained a small amount of filamentous bacteria (which is common for healthy mixed liquors¹⁰); however, a large increase was observed during dosing and their presence became a problem in sludge handling.

DISCUSSION

To meet European Community guidelines on P in effluents entering sensitive areas for a small wastewater treatment plant, an effluent concentration of 2 mg l^{-1} total P is required (or 80% removal). To achieve this effluent concentration using iron (II) sulphate, a dose of 2:1 (molar ratio Fe:P) should be sufficient and a dose of 1.75:1 (molar ratio of Fe:P) for iron (III) chloride. However the presence of excess filamentous bacteria during dosing have masked the effectiveness of these chemicals at the higher doses. Optimum doses of ferric chloride to a biological aerated filter (BAF) of 2:1 (molar ratio Fe:P) are reported¹³. However the addition of ferric chloride to aerated lagoons has required doses of >3:1 (molar ratio Fe:P)⁵. The use of ferrous sulphate for P removal in a BAF produced an optimum dose of 1.25:1 (molar ratio Fe:P)⁶. The lower doses required for BAFs could be related to the efficient suspended solids removal in this process.

At these doses, the BOD₅ discharge consent of 20 mg l^{-1} can be met for both chemicals. For iron (III), a suspended solids discharge consent of 30 mg l^{-1} can also be achieved but not for iron (II) (average effluent concentration at optimal dose = 47.3 mg l^{-1}). This poor performance may be due to the presence of filamentous bacteria affecting sludge settling. Nitrification appeared to be inhibited by the addition of iron (III). An examination of parameters such as the food:micro-organism (F:M) ratio and aeration tank pH (Table 1) indicated that low pH may have been responsible for the initial reduction in nitrification¹⁴.

The performance of the clarifier in the activated sludge

process is of paramount importance to the efficiency of the system¹⁵. The development of filamentous bacteria in both aeration tanks resulted in poor plant performance, due to poor sludge settling. Sludge bulking is one of the greatest operational difficulties facing nutrient removal plants¹⁶. Although the pilot plant contained an anoxic contact and pre-mixing zone, past research has shown the effectiveness of these zones to be questionable¹⁶, particularly in biological nutrient removal plants¹⁷. Sludge bulking is complicated by the variety of species responsible for this problem and the many different solutions, not all of which are effective in all situations¹⁸. Filamentous bacteria indicate a biologically stressed system, possibly due to a poor environment such as low dissolved oxygen and high F:M ratio¹⁹. Control of these conditions is not always sufficient to prevent the excess growth of filamentous bacteria as demonstrated by this trial. There is often a seasonal pattern which results in higher growth during colder periods and heavy rain²⁰; influent feed characteristics can also favour filamentous growth. These trials were performed during heavy local flooding in April/May 1998 which produced very weak influent sewage (F:M ratios generally <0.20 $\text{g BOD}_5\text{g MLSS}^{-1}\text{d}^{-1}$). It has been demonstrated that F:M ratios in the range of 0.25–0.60 $\text{g BOD}_5\text{g MLSS}^{-1}\text{d}^{-1}$ produce sludge volume index values typical of readily settleable sludge²¹. Also, the addition of iron for co-precipitation has been found to favour the growth of filamentous bacteria (particularly type 0041)¹⁷. It is not possible to prove that iron addition was directly responsible for the excess growth of filamentous bacteria.

The failure of the biomass dosed with iron (III) to nitrify fully after a seed of nitrifying bacteria had been added may be due to differences in floc characteristics. The less dense flocs produced by iron (III) dosing may have not been sufficient for the nitrifiers to bind to and settle with. This would result in the nitrifiers being lost from the system. A gradual decrease in *Nitrobacter* spp. (the bacteria responsible for the conversion of nitrite to nitrate) was observed. *Nitrobacter* spp. have a slower growth rate than *Nitrosomonas* spp. (the bacteria responsible for the conversion of ammonia to nitrite) and will therefore be lost from the system at a greater rate (Table 4). Conversely, the denser flocs produced by iron (II) dosing may have helped to settle the nitrifiers and thereby return them to the aeration tank. Adverse effects of iron addition on the settling characteristics of activated sludge have been observed previously²². The sudden decline of *Nitrobacter* spp. in the biomass dosed with iron (II) was due to operational problems with the influent.

Another reason for the poor general performance could be the microbial reduction of iron (III) in sludge flocs

Table 4. Distribution of ammonia, nitrate and nitrite in effluent produced by the pilot plant (mg l^{-1}).

Dose	Iron (II)			Iron (III)		
	NH ₄ –	NO ₃ –	NO ₂ –	NH ₄ –	NO ₃ –	NO ₂ –
No dose	5.6	21.4	0.1	5.4	22.2	0.4
1.0–1.5:1 Fe:P (Molar ratio)	2.0	19.4	0.6	2.2	20.1	0.1
1.5–2.0:1 Fe:P 2.0 (Molar ratio)	3.6	23.1	1.6	25.7	3.5	0.0
Interrupted by filamentous bacteria	8.7	14.8	2.2	5.8	14.5	2.0
Interrupted by poor nitrification	1.2	18.7	4.3	4.8	11.0	9.2

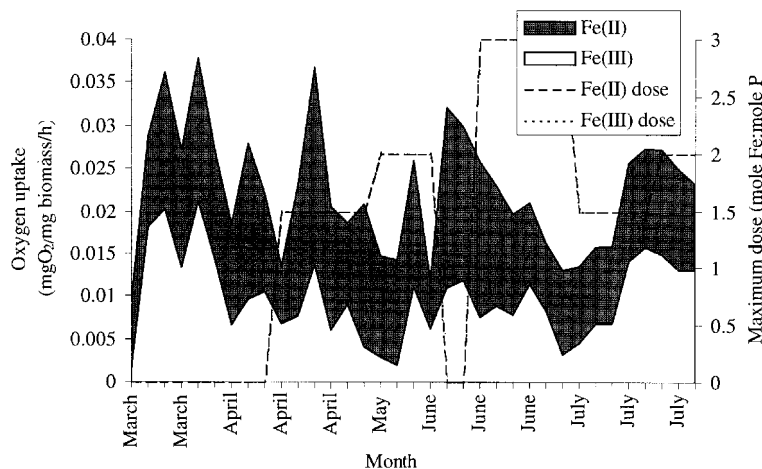


Figure 1. Variations in oxygen uptake rate with increasing chemical dose.

which results in an increase in bulk water turbidity²³. Iron (III) generated *in-situ* from iron (II) is more efficient at P removal than the use of a iron (III) stock solution³. Any excess iron (III) from a stock solution may undergo microbial reduction in the final settlement tank. It is also possible that denitrification can occur with the re-oxidation of iron (II) to (III) in anoxic conditions (nitrite or nitrate are used as electron acceptors)²⁴. However background levels of 8–15 mg Fe(II) g⁻¹ solids were found in a biological nutrient removal plant²⁵ indicating a high concentration of iron (II) is required before this conversion takes place.

Respirometric measurements indicate the biomasses were not stimulated by the addition of either iron-based chemical. The average oxygen uptake rates during dosing of 0.0121 mg O₂ mg MLSS⁻¹ h⁻¹ for iron (II) and 0.0086 mg O₂ mg MLSS⁻¹ h⁻¹ for iron (III) are comparable with findings for activated sludge by Mayhew and Stephenson²⁶ which were approximately 0.0035 mg O₂ mg MLSS⁻¹ h⁻¹. It is important to note that these are endogenous respiration rates, without added oxygen or substrate.

Biological wastewater treatment requires a wide range of species for complete degradation^{27,28}. Observations of species present in activated sludge have been used to indicate both the stage in the development of the treatment process and the prevalent environmental conditions in the

system¹⁹. Curtis and Craine²⁸ highlighted the need to examine both S_T and species richness to obtain a better picture, as often high diversity is only indicative of a high level of population heterogeneity. Using these two parameters as a basis for the examination of the microbiology of the systems, it was shown that the S_T in both aeration tanks were very similar. The values produced are comparable with indices produced during the additions of an aluminium-based polyelectrolyte (ABP) and aluminium sulphate ($S_T = 30.5$ and 23.3 respectively)¹¹. The slightly higher S_T produced by iron (II) addition could be linked to a higher average pH (6.4) compared to iron (III) (pH = 5.9) or differences in floc characteristics. The flocs produced by the addition of iron (II) were more dense and larger than that produced by iron (III), which were similar to flocs produced by aluminium sulphate addition¹¹. Species diversity among the ciliate population in activated sludge has previously shown little variation but seasonal trends were apparent²⁹.

CONCLUSIONS

- (1) A total P effluent concentration of 2 mg l⁻¹ should be achieved with doses of 2:1 (molar ratio Fe:P) for Fe (II) and 1.75:1 (molar ratio Fe:P) for Fe (III).
- (2) At these doses the efficiency of BOD₅ and COD

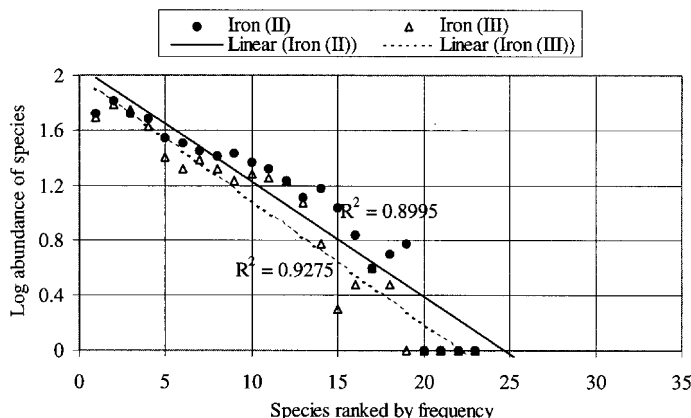


Figure 2. Mean S_T indices for mixed liquor treated with iron (II) and iron (III).

Table 5. Changes in floc characteristics during addition of iron-based chemicals.

	Pre-dosing, %	Iron (II), %	Iron (III), %
Floc description	(no 'weak, irregular' or 'weak, round' flocs observed)		
Firm, irregular	89	21	84
Firm, round	11	79	16
Floc size			
Small	36	59	78
Medium	61	40	16
Large	3	1	6

removal decreased slightly with both chemicals. Suspended solids removal and nitrification were both adversely affected during chemical addition, to a greater extent with iron (III) addition. This was primarily due to the excess growth of filamentous bacteria experienced.

(3) The oxygen uptake rates were increased by the addition of iron (II) but not by the addition of iron (III). Improved biological treatment was not observed by iron (II) addition so stimulation was not indicated.

(4) During chemical addition, the biomasses produced similar S_T indices.

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