

Phosphorus recovery from black water by chemical precipitation

*Master's Thesis in the International Master's Programme Applied Environmental
Measurement Techniques*

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Department of Civil and Environmental Engineering
Division of Water Environment Technology
CHALMERS UNIVERSITY OF TECHNOLOGY
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Abstract

Recycling of nutrients is becoming an important concept nowadays. The City of Gothenburg started a project whose main goal was recovery of nutrients from black water and their further application as a fertilizer. The project was initiated by building 110 villas and 17 apartments in the area named Skogaberg in Gothenburg, Sweden. The wastewater from the households is divided in 2 parts: black water and grey water. The black water includes food waste and water from toilets and the grey water incorporates water from dish washers and showers.

The aim of this study was to recover phosphorus from the black water by using calcium hydroxide, iron and aluminium salts as precipitants. The process was studied in beakers connected to a flocculator and the chemicals were also tested on a larger scale. A polymer was applied in some of the experiments. All the chemicals provided low residual phosphorus concentrations.

Phosphorus was recovered by more than 96% when using Fe salts. The Fe dose of 107.4 mg/l lowered the total P and PO₄-P concentrations below 0.9 and 0.4 mg/l. The lowest values were obtained between pH of 5.5 and 6. The Fe concentrations in the filtrated supernatants had values between 1.1 and 24 mg/l. The sludge volumes when achieving the highest phosphorus reductions were between 180 and 200 ml/l black water.

The Al salts decreased the total P and PO₄-P amounts below 1.6 and 0.8 mg/l respectively, providing reductions higher than 92% and sludge volume of 200ml/l black water. The highest reductions were achieved with a dose of 52.7 mg(Al)/l and the pH was between 6.2 and 7.4. Black water composition affects the Al concentrations and in the filtrated supernatants values between 0.096 and 3.3 mg/l were measured.

Precipitation with Ca(OH)₂ is highly pH dependent. The lowest residual P amounts (below 0.4 mg/l) were achieved at pH higher than 9.5 providing 90 ml(sludge)/l black water.

Further application of the sludges as fertilizers has to be investigated.

Key words: Recycling, nutrients, phosphorus, precipitants, residual phosphorus, pH, Fe, Al, sludge volume

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1. Introduction

The fast development of modern societies threatens the environment and the concept of recycling is becoming important nowadays. Recycling of nutrients probably will be a very important issue in the future. This process enables nutrients from urban areas to be brought back to agriculture and in this way a cycle is created.

The City of Gothenburg (Recycling Department) in cooperation with a real estate company (Egnahemsbolaget) started a project whose main goal was to recover nutrients from wastewater from households and return them to agriculture as a fertilizer. This project started by building 110 villas and 17 apartments in the area named Skogaberg on the island of Hisingen in Gothenburg, Sweden. (Karlsson 2005)

The wastewater from the households is divided in 2 parts: black water and grey water. The grey water incorporates the water from dishwashers and showers and it contains the biggest part of the heavy metals. The black water comprises of water from toilets (urine and faeces) mixed with food leftovers. Food disposers were installed in each household and after the food has been grinded it is mixed with the toilet water. (Karlsson 2005)

The idea of applying a fertilizer obtained from treatment of wastewater has not been widely accepted due to concerns of enriching the soil with heavy metals and other persistent organic pollutants. A few alternatives exist how to treat the black water and use the products of the treatment. The alternatives include combination of the following processes: reverse osmosis, anaerobic digestion of concentrated black water and chemical precipitation. (Meins 2005)

2. Study aims

In this study the processes of chemical precipitation of black water using calcium hydroxide, iron and aluminium salts as precipitants are investigated. The addition of the precipitants results in formation of Ca, Fe and Al phosphates and in this way phosphorus can be regained from the black water.

The investigations should give answers to the following issues:

- a) Percentage of regained phosphorus
- b) The amounts of the chemicals needed to achieve good removal degree of phosphorus
- c) Phosphorus concentration in the black water after precipitation
- d) Optimal mixing speed and time, flocculation speed and time and sedimentation time
- e) Optimal pH values
- f) Sludge volumes
- g) Application of the chemicals on a larger scale

3. Black water composition

The flow of the black water is around 20 m³/d with a maximum flow of 1.2 l/s for a period of 10 min. Phosphorus, nitrogen, potassium and sulphur can be recovered from the black water. The black water has a complex composition because besides urine and faeces it contains food leftovers. (Meins 2005)

The concentration of different parameters in two samples after the black water has passed the drum screen is given in Table 1.

Table 1: Black water composition (De Blois 2006)

Parameter	2006-01-23	2006-01-24	Mean value
TS (mg/l)	1080	1000	1040
Volatile solids (mg/l)	680	585	632.5
Suspended solids (mg/l)	730	540	635
TOC (mg/l)	440	350	395
COD (mg/l)	1800	1300	1550
BOD ₇ (mg/l)	710	620	665
Nitrogen, N (mg/l)	320	290	305
Ammonium, NH ₄ -N (mg/l)	130	130	130
Total P (mg/l)	23	20	21.5
Phosphate, PO ₄ -P (mg/l)	19	17	18
Potassium, K (mg/l)	50	48	49
Sulphur, S (mg/l)	26	26	26
Sulphate, SO ₄ (mg/l)	6.7	10.7	8.7

The composition of the black water varies during the day. In the mornings and evenings it is more concentrated and depending on cooking times there is more food waste in it. During the day it is very diluted and mostly contains urine. The high values of COD and BOD are result of the foodleftovers present in the black water. A big part of BOD and COD (40-50%) can be removed by filtration through the drum screen.

Approximately 80% of the total phosphorus is orthophosphate (HPO₄⁻) and it comes from the urine. Nitrogen is found in 3 different forms in the black water: organic nitrogen (which is bound to particles), urea and ammonium. Particle bound nitrogen and urea contribute with 30% each and the rest (40%) is ammonium. (De Blois 2006)

4. Chemical removal of phosphates

4.1 Phases of the chemical removal process

The process of chemical removal of phosphates consists of six different phases:

1. Dosing of a chemical and mixing
2. Precipitation
3. Coagulation
4. Flocculation
5. Sedimentation
6. Sludge removal

The first phase includes addition of a precipitant and mixing of the solution. After this, the processes which occur simultaneously and very quickly are precipitation and coagulation. (Henze et al. 2002)

In connection with precipitation dissolved orthophosphates are converted into solids with very low solubility. This is achieved by addition of metal salts or calcium hydroxide. Coagulation includes aggregation of colloids into primary particles. The process of precipitation is described in the next section and in this section more attention is paid on the process of coagulation.

If considering the particle diameter, particles can be placed in the following groups:

- Settleable flocs: $d_p > 100 \mu\text{m}$
- Primary particles: $1 \mu\text{m} < d_p < 100 \mu\text{m}$
- Colloids: $10^{-3} \mu\text{m} < d_p < 1 \mu\text{m}$
- Dissolved material: " d_p " $< 10^{-3} \mu\text{m}$

Formation of primary particles in the process of coagulation is a result of destabilisation of colloids. This process is not understood completely but it is meant that hydrophilic or hydrophobic properties of colloids, zeta potential and isoelectric point are responsible for that.

Hydrophilic colloids bind water, on the other hand hydrophobic colloids do not have this property. Water can be bound to hydrophilic particles because there are amino ($-\text{NH}_2$), carboxyl ($-\text{COOH}$) or hydroxyl ($-\text{OH}$) groups on their surface, which form hydrogen bonds with it. Due to these properties a water layer is formed around the particle. (Figure 4.1.1) (Henze et al. 2002)

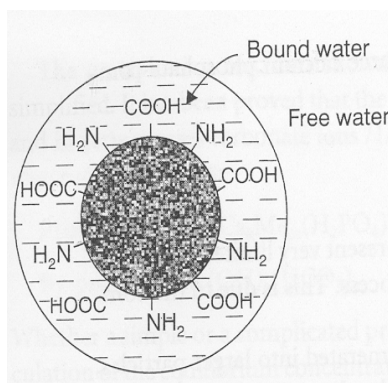


Figure 4.1.1: Layer of bound water around particle's surface

The colloids generally have a negative charge. Placing them in an electric field can prove this. The charge of the colloids is a result of dissociation of amino, hydroxyl and carboxyl groups or adsorption of ions from the water. The charge on the surface is known as primary charge. Oppositely charged particles are attracted by the primary charge and a fixed layer is formed close to the particle surface. As the distance from the particle increases the concentration of oppositely charged particles decreases and this layer is called diffuse layer. The plane of shear is the interface between the layer of water moving with the particle and the rest of the liquid. The electrostatic potential at this interface is called zeta

potential. Zeta potential gives an impression of the repelling forces among the particles.

The third important concept is isoelectric point. The isoelectric point is the pH value at which the net charge of the particle is 0. (see Figure 4.1.2) This point can be adjusted by addition of different ions to the water.

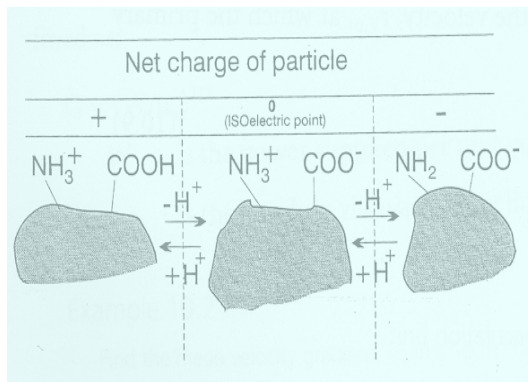


Figure 4.1.2: Changes of the surface charge of a particle

The surface charge of the particle can be reduced which means that forces of attraction (Van der Waals forces) dominate and the process of coagulation can start.

Destabilization of a colloid can be done by one of the following mechanisms:

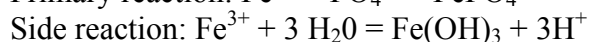
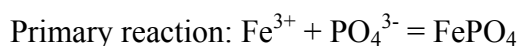
1. Bridging among the colloids – this can be achieved by addition of cationic, anionic or non-ionic polymers which will form "bridges" among the colloids.
2. Entrapment of the colloids – entrapment is a result of aggregation of the colloids with the precipitates.
3. Reduction of the particle's surface charge – this mechanism is a result of addition of different positive ions, acids or bases.

During the flocculation primary particles form larger particles – flocks. The formation of the flocks enables to separate the precipitated material after sedimentation. (Henze et al. 2002)

4.2 Precipitation of phosphates

4.2.1 Precipitation with Fe^{3+}

Fe^{3+} is frequently used as precipitant. The precipitation model when using Fe^{3+} can be shown by the following simple reactions:



The equilibrium diagram for FePO_4 shows that the lowest residual phosphate concentration can be achieved between pH of 5 and 6. Solubility of the by-product $\text{Fe}(\text{OH})_3$ is lowest in the pH region between 8 and 9. (see Figure 4.2.1.1 and Figure 4.2.1.2)

According to the side reaction the formation of the precipitate is accompanied by a reduction in alkalinity.

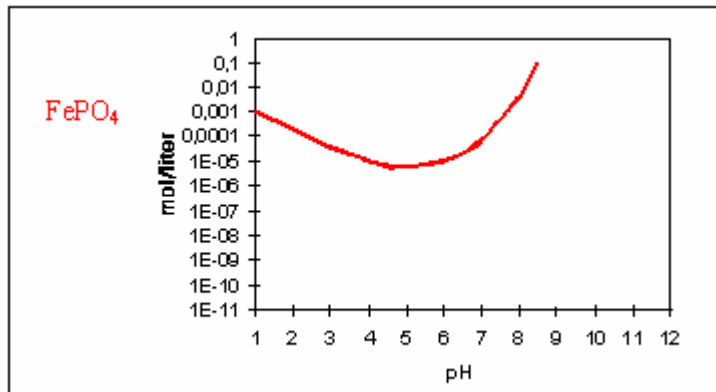


Figure 4.2.1.1: Solubility of FePO_4 (Levlin 2001)

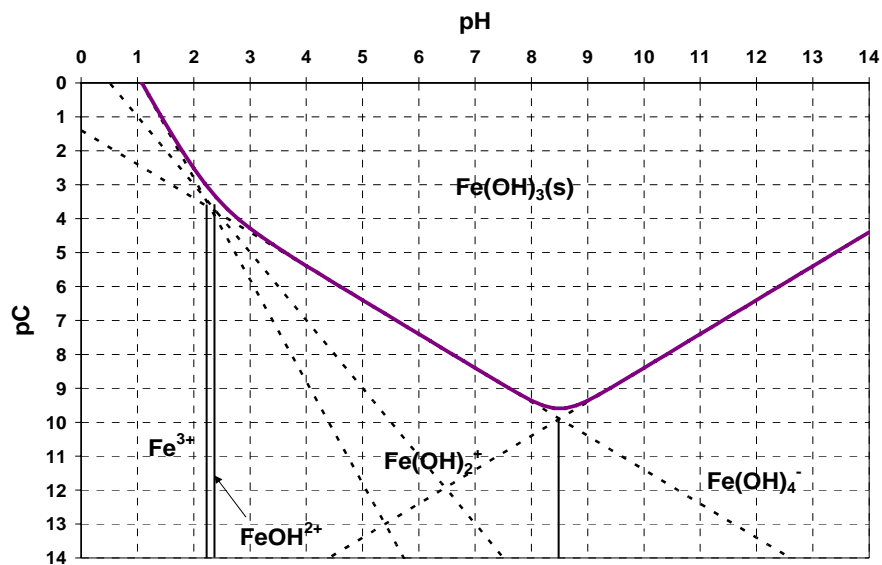


Figure 4.2.1.2: Solubility of Fe(OH)_3 (The Ohio state university 2007)

Previous scientific investigations proved that both precipitates contain calcium and carbonate ions which means that they have a complex composition. The composition of the primary and by product can be presented as follows:

Primary product: $\text{Ca}_A\text{Fe}_B(\text{H}_2\text{PO}_4)_C(\text{OH})_D(\text{HCO}_3)_E$

By product: $\text{Fe}_F(\text{OH})_G(\text{HCO}_3)_H$

Formation of the iron hydroxide contributes to the overall removal of phosphates in two ways. The first process is entrapment of particles by the voluminous particles of the precipitate and the other one is adsorption of phosphate ions on the particles surfaces. In literature more reactions that include formation of different complexes can be found. Equilibrium reactions include dissociation of phosphoric acid, hydrolysis of ferric ion and complex formation. (Jenkins et al. 1970)

Equilibrium reactions and pK values of these reactions are presented in Table 2.

Table 2: Equilibrium equations and pKa values (Jenkins et al.1970)

$\text{H}_3\text{PO}_4 = \text{H}^+ + \text{H}_2\text{PO}_4^-$	2.1
$\text{H}_2\text{PO}_4^- = \text{H}^+ + \text{HPO}_4^{2-}$	7.2
$\text{HPO}_4^{2-} = \text{H}^+ + \text{PO}_4^{3-}$	12.2
$\text{Fe}^{3+} + \text{H}_2\text{O} = \text{FeOH}^{2+} + \text{H}^+$	2.2
$\text{Fe}^{3+} + 2 \text{H}_2\text{O} = \text{Fe}(\text{OH})_2^{2+} + 2\text{H}^+$	5.7
$\text{Fe}^{3+} + 3 \text{H}_2\text{O} = \text{Fe}(\text{OH})_3(\text{aq}) + 3\text{H}^+$	12
$\text{Fe}^{3+} + 4 \text{H}_2\text{O} = \text{Fe}(\text{OH})_4^- + 4\text{H}^+$	21.6
$\text{Fe}^{3+} + \text{HPO}_4^{2-} = \text{FeHPO}_4^+$	-9
$\text{Fe}^{3+} + \text{H}_2\text{PO}_4^- = \text{FeH}_2\text{PO}_4^{2+}$	-
$\text{Fe}^{3+} + \text{SO}_4^{2-} = \text{FeSO}_4^+$	-3.9
$\text{Fe}^{3+} + 2 \text{SO}_4^{2-} = \text{Fe}(\text{SO}_4)_2^-$	-5.4
$2\text{Fe}^{3+} + 2 \text{H}_2\text{O} = \text{Fe}_2(\text{OH})_2^{4+} + 2\text{H}^+$	2.9
$\text{Fe}^{3+} + \text{PO}_4^{3-} = \text{FePO}_4(\text{s})$	-25.8
$\text{Fe}^{3+} + 3\text{OH}^- = \text{Fe}(\text{OH})_3(\text{s})$	-38
$\text{Fe}^{3+} + \text{PO}_4^{3-} + 2\text{H}_2\text{O} = \text{FePO}_4 \cdot 2 \text{H}_2\text{O}(\text{s})$	-26.4
$\text{Fe}^{3+} + 2 \text{H}_2\text{O} = \text{am FeOOH}(\text{s}) + 3\text{H}^+$	0.5
$2\text{Fe}^{3+} + 3\text{SO}_4^{2-} = \text{Fe}_2(\text{SO}_4)_3(\text{s})$	-3.7
$\text{Fe}^{3+} + 2 \text{H}_2\text{O} = 2\text{H}^+ + \text{Fe}(\text{OH})_2(\text{s})$	13.6

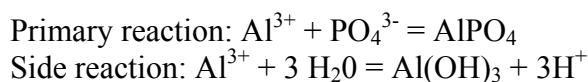
The primary reaction shows that 1 mol Fe reacts with 1 mol PO_4 and 1 mol FePO_4 is formed as a product of the reaction. Due to the formation of the complexes in the solution Fe/ PO_4 molar ratio must be higher than 1 if sufficient phosphorus removal has to be achieved. The total concentrations of iron and phosphate in the solution which are in equilibrium with the precipitates can be presented as follows:

$$C_{\text{PO}_4(\text{eq})} = [\text{H}_3\text{PO}_4] + [\text{H}_2\text{PO}_4^-] + [\text{HPO}_4^{2-}] + [\text{PO}_4^{3-}] + [\text{Fe HPO}_4^+] + [\text{Fe H}_2\text{PO}_4^{2+}]$$

$$C_{\text{Fe}(\text{eq})} = [\text{Fe}^{3+}] + [\text{FeOH}^{2+}] + [\text{Fe}(\text{OH})_2^{2+}] + [\text{Fe}(\text{OH})_3] + [\text{Fe}(\text{OH})_4^-] + [\text{FeH}_2\text{PO}_4^{2+}] + [\text{FeHPO}_4^+] + [\text{FeSO}_4^+] + [\text{Fe}_2(\text{OH})_2^{4+}] + [\text{Fe}(\text{SO}_4)_2^-]$$

4.2.2 Precipitation with Al^{3+}

Chemical reactions that take place when Al^{3+} is added are almost identical with those presented in the section for Fe^{3+} . The precipitation with Al^{3+} can simply be described with these reactions:



It can be concluded that the reactions of precipitation with Al salts are accompanied by an alkalinity reduction. The equilibrium diagram for AlPO_4 shows that the lowest phosphate concentrations can be accomplished between pH of 5 and 7. The lowest solubility of $\text{Al}(\text{OH})_3$ is registered between pH of 5.5 and 7.5. (see Figure 4.2.2.1 and Figure 4.2.2.2). A reduction in alkalinity accompanies the formation of $\text{Al}(\text{OH})_3$.

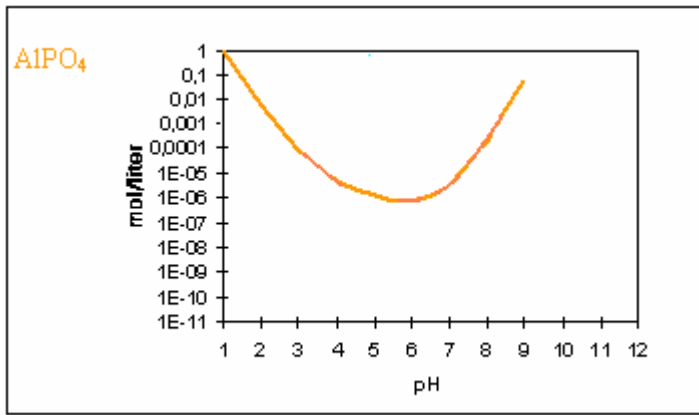


Figure 4.2.2.1 Solubility of AlPO_4 (Levlin 2001)

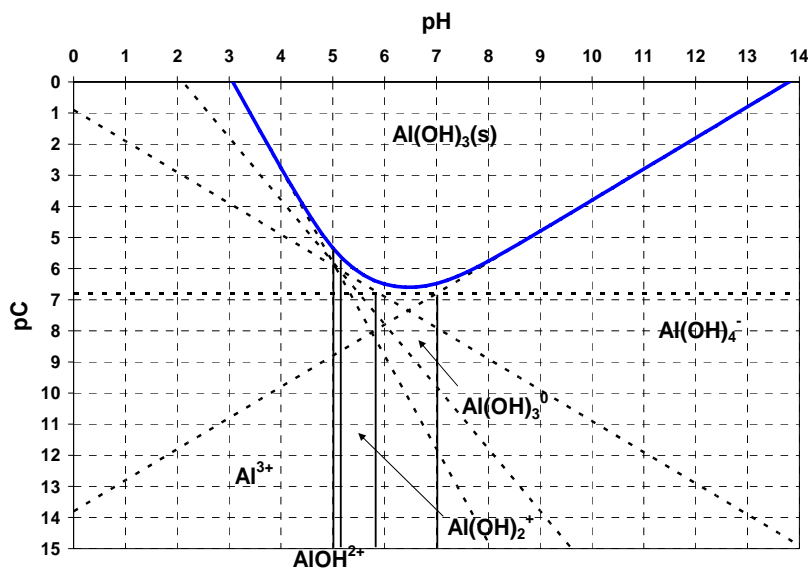
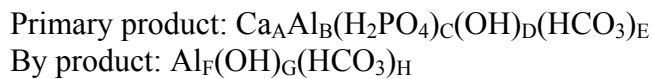


Figure 4.2.2.2: Solubility of Al(OH)_3 (The Ohio state university 2007)

Both precipitates contain calcium and carbonate ions and have complex composition as in the case with Fe. The complex composition of the precipitates can be given by the following formulas:



The processes of entrapment of particles and adsorption of phosphate ions on the particles surfaces are characteristic for these reactions also. (Jenkins et al. 1970)

Al^{3+} forms many complexes and due to this behaviour Al// PO_4 molar ratio needed for efficient phosphorus removal must be higher than 1. The equilibrium reactions and pK values are presented in Table 3.

Table 3: Equilibrium equations and pKa values (Faust and Aly 1998)

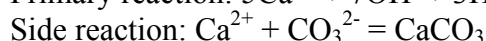
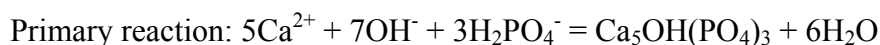
$\text{H}_3\text{PO}_4 = \text{H}^+ + \text{H}_2\text{PO}_4^-$	2.1
$\text{H}_2\text{PO}_4^- = \text{H}^+ + \text{HPO}_4^{2-}$	7.2
$\text{HPO}_4^{2-} = \text{H}^+ + \text{PO}_4^{3-}$	12.2
$\text{Al}^{3+} + \text{H}_2\text{O} = \text{AlOH}^{2+} + \text{H}^+$	4.97
$\text{Al}^{3+} + 2\text{H}_2\text{O} = \text{Al}(\text{OH})_2^+ + 2\text{H}^+$	9.3
$\text{Al}^{3+} + 3\text{H}_2\text{O} = \text{Al}(\text{OH})_3(\text{aq}) + 3\text{H}^+$	15
$\text{Al}^{3+} + 4\text{H}_2\text{O} = \text{Al}(\text{OH})_4^- + 4\text{H}^+$	23
$2\text{Al}^{3+} + 2\text{H}_2\text{O} = \text{Al}_2(\text{OH})_2^{4+} + 2\text{H}^+$	7.7
$3\text{Al}^{3+} + 4\text{H}_2\text{O} = \text{Al}_3(\text{OH})_4^{5+} + 4\text{H}^+$	13.9
$13\text{Al}^{3+} + 28\text{H}_2\text{O} = \text{Al}_3\text{O}_4(\text{OH})_{24}^{7+} + 32\text{H}^+$	98.7
$\alpha - \text{Al}(\text{OH})_3 + 3\text{H}^+ = \text{Al}^{3+} + 3\text{H}_2\text{O}$	-8.5
$\text{Al}(\text{OH})_3 + 3\text{H}^+ = \text{Al}^{3+} + 3\text{H}_2\text{O}$	-10.5
$\text{Al}^{3+} + \text{PO}_4^{3-} = \text{AlPO}_4(\text{s})$	-22.5
$\text{Al}^{3+} + 3\text{OH}^- = \text{Al}(\text{OH})_3(\text{s})$	-33

The equilibrium concentration of dissolved Al after sedimentation is a sum of the Al^{3+} ion and all the complexes:

$$C_{\text{Al}}(\text{eq}) = [\text{Al}^{3+}] + [\text{AlOH}^{2+}] + [\text{Al}(\text{OH})_2^+] + [\text{Al}(\text{OH})_3] + [\text{Al}(\text{OH})_4^-] + [\text{Al}_2(\text{OH})_2^{4+}] + [\text{Al}_3(\text{OH})_4^{5+}] + [\text{Al}_3\text{O}_4(\text{OH})_{24}^{7+}]$$

4.2.3 Precipitation with Ca

The precipitation with calcium is conducted by adding slaked or unslaked lime and it is highly pH dependent. An increase in both Ca concentration and alkalinity is achieved. This process can be described by the following reactions:



The phosphate compound produced in the first reaction is hydroxyapatite. Prior to the formation of the apatite more or less amorphous compounds are formed. In the system $\text{Ca-PO}_4\text{-CO}_3\text{-H}^+\text{-H}_2\text{O}$ precipitation of CaCO_3 competes with formation of calcium phosphate between pH of 8 and 11. Solubility of hydroxyapatite and calcium carbonate are shown in Figure 4.3.1 and Figure 4.3.2. Solubility of both solids decreases with increasing the pH.

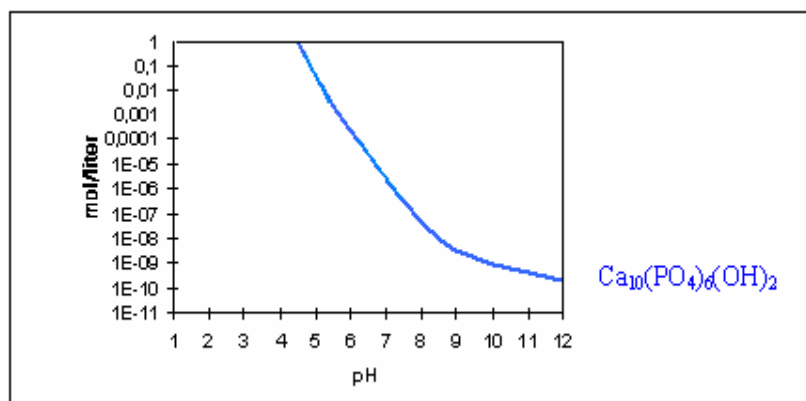


Figure 4.3.1. Solubility of hydroxyapatite (Levlin 2001)

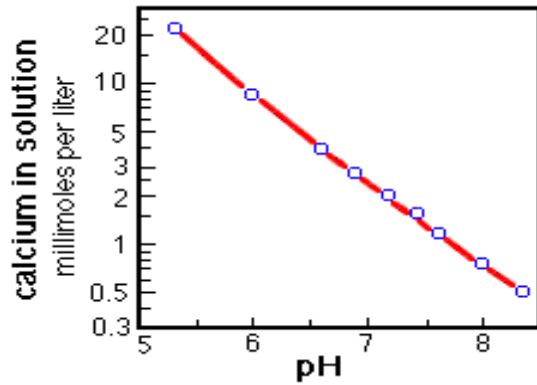


Figure 4.3.2: Solubility of CaCO_3 (University of Granada 2007)

Many different chemical reactions occur in this system. The equilibrium reactions and their pK values are presented in Table 4. (Henze et al. 2002)

Table 4: Equilibrium equations and pKa values (Jenkins et al. 1970)

$\text{H}_3\text{PO}_4 = \text{H}^+ + \text{H}_2\text{PO}_4^-$	2.1
$\text{H}_2\text{PO}_4^- = \text{H}^+ + \text{HPO}_4^{2-}$	7.2
$\text{HPO}_4^{2-} = \text{H}^+ + \text{PO}_4^{3-}$	12.2
$\text{Ca}^{2+} + \text{PO}_4^{3-} = \text{CaPO}_4^-$	- 6.5
$\text{Ca}^{2+} + \text{HPO}_4^{2-} = \text{CaHPO}_4$	-2.7
$\text{Ca}^{2+} + \text{H}_2\text{PO}_4^- = \text{CaH}_2\text{PO}_4^+$	-1.4
$\text{H}_2\text{CO}_3 = \text{HCO}_3^- + \text{H}^+$	-
$\text{HCO}_3^- = \text{CO}_3^{2-} + \text{H}^+$	10.25
$4\text{Ca}^{2+} + 3\text{PO}_4^{3-} + \text{H}^+ = \text{Ca}_4\text{H}(\text{PO}_4)_3$	- 46.9
$\text{Ca}^{2+} + \text{CO}_3^{2-} = \text{CaCO}_{3(\text{aq})}$	-3.2
$\text{Ca} + \text{H}_2\text{O} = \text{H}^+ + \text{Ca}(\text{OH})^+$	12.69
$5\text{Ca}^{2+} + 7\text{OH}^- + 3\text{H}_2\text{PO}_4^- = \text{Ca}_5\text{OH}(\text{PO}_4)_3(\text{s}) + 6\text{H}_2\text{O}$	- 44.3
$\text{Ca}^{2+} + \text{CO}_3^{2-} = \text{CaCO}_{3(\text{s})}$	- 8.48

The following equation gives the equilibrium concentration of dissolved Ca species after sedimentation.

$$C_{\text{Ca}(\text{eq})} = [\text{Ca}^{2+}] + [\text{CaPO}_4^-] + [\text{CaHPO}_4] + [\text{CaH}_2\text{PO}_4^+] + [\text{Ca}_4\text{H}(\text{PO}_4)_3] + [\text{CaCO}_{3(\text{aq})}] + [\text{Ca}(\text{OH})^+]$$

5. Equipment and experiments

The black water from the system is collected in a sump and it is transported by a grinder pump to the drum screen in the laboratory. The level in the sump is controlled by floaters and after the liquid reaches a certain level the pump starts. The black water enters the inner side of the drum screen and passes holes with a diameter of 0.6 mm. The thick material from this first pre-treatment from the inner side of the screen is transported to the local sewage plant (Gryaab). Then, the black water goes to a sedimentation tank where sedimentation of sand and other settleable particles takes place. Black water samples were taken after the drum screen in different periods of the day. In order to have a more representative sample before

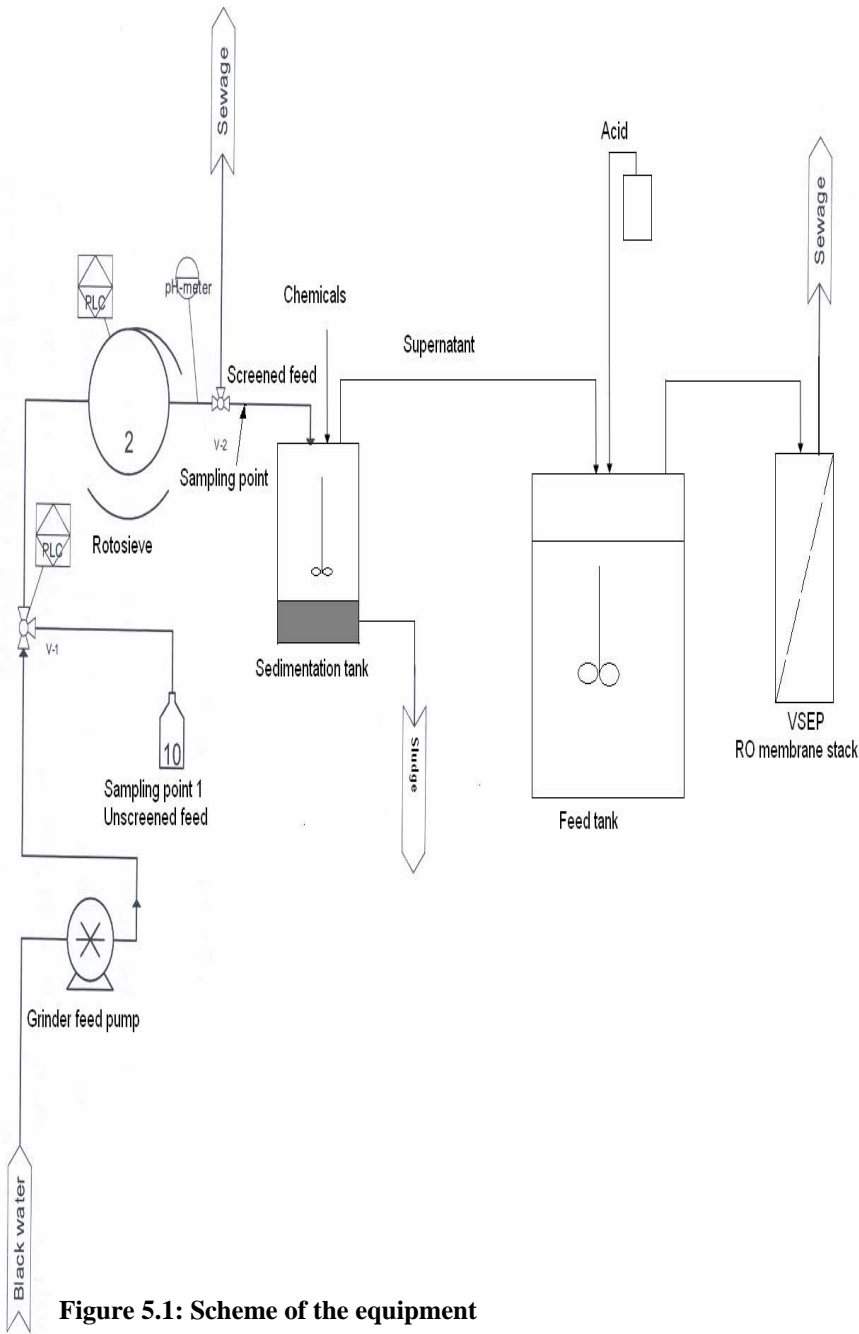


Figure 5.1: Scheme of the equipment

some experiments, black water was collected in a small tank during night. When applying the chemicals on a larger scale a sedimentation tank with a capacity of 270l was used. After the sedimentation, treated black water was directed to a feed tank where pH was adjusted to avoid scaling on the membranes and then it entered the RO system. The scheme of the equipment is presented in Figure 5.1.

The process of chemical precipitation was studied in beakers connected to a flocculator. The following parameters can be adjusted on the flocculator: mixing speed and time, flocculation speed and time and sedimentation time.

In order to precipitate phosphates from the black water different chemicals such as iron chloride (FeCl_3), iron sulphate ($\text{Fe}_2(\text{SO}_4)_3$), iron chloride sulphate (FeClSO_4), aluminium chloride (AlCl_3) and calcium hydroxide ($\text{Ca}(\text{OH})_2$) were used.

In Table 5. concentrations and densities of iron and aluminium salts are presented.

Table 5: Concentrations and densities of iron and aluminium salts

Chemical	Concentration (% Fe)	Density (g/cm^3)
FeCl_3 (PIX 111)*	13.7	1.42
$\text{Fe}_2(\text{SO}_4)_3$ (PIX 113)*	11.5	1.55
FeClSO_4 (PIX 118)*	11.6	1.5
FeCl_3 (ciba)	13.8	1.42
AlCl_3 (Pluspac 1465)*	7.4	1.35
AlCl_3 (Pluspac 1800)*	9	1.39

*Specifications of the chemicals are presented in Appendix K.

In the beginning the following adjustments on the flocculator were used:

1. Mixing time – 15s
2. Mixing speed – 350 rpm
3. Flocculation time – 10 min
4. Flocculation speed – 40 rpm
5. Sedimentation time – 10 min

The experiments started by dosing the chemicals with a micropipette and this was followed by fast mixing. Some experiments were conducted with addition of an anionic polymer and the mixing time was extended to 30s. 2% polymer solution (v/v) was prepared by dissolving the polymer in distilled water and the amount of the solution that was added was 5 ml/l black water. In these experiments the polymer solution was added 20s after dosing the precipitant.

In the laboratory at Skogaberg pH, alkalinity, conductivity and sludge volume were measured. pH was measured in the feed, during flocculation and after sedimentation. Conductivity was measured in the feed and after sedimentation.

pH and conductivity were measured with portable instruments. Carbonate alkalinity was determined by titration with 0.1 M HCl down to pH=5.4 and total alkalinity by titration down to pH=4.5.

The following formula is used to calculate the alkalinity:

$$\text{Alk} = V_{\text{HCl}} * C_{\text{HCl}} / V_{\text{SAMPLE}}$$

After the sedimentation the supernatant was decanted and the sludge was transferred in a funnel and the sludge volume was registered after 30 min settling.

The supernatants were first filtered through 2.7 µm glass fibre filter using Buchner funnel and then the filtrate was passed through 1 µm glass fibre filter. Filtrated and non-filtrated supernatants were sent for further analysis to Al Control laboratories. The following parameters were measured at Alcontrol: PO₄-P, total P, Fe, Al, Ca and suspended solids.

Phosphorus concentration was preliminary assumed to be 20 mg/l. FeCl₃, Fe₂(SO₄)₃ and FeClSO₄ were added to the black water in amounts needed to achieve different Fe/P molar ratios. The following assumed Fe:P molar ratios were achieved: 0.5:1, 0.75:1, 1:1, 1.5:1, 2:1 and 3:1.

The supernatant was still turbid after applying the following assumed molar ratios of 0.5 and 0.75. The experiments with aluminium salts were continued with the following Al/P ratios: 1, 1.5, 2 and 3.

Ca(OH)₂ was added to the black water in amounts needed to achieve the following pH values: 8, 8.5, 9, 9.5, 10, 10.5 and 11. Unmeasured amount of Ca(OH)₂ was dissolved in water in 1l volumetric flask and the solution was titrated with HCl down to pH=4.5 in order to determine the concentration of Ca(OH)₂. After the preparation of Ca(OH)₂ solution, 50 ml of black water were titrated and the amount of Ca(OH)₂ needed to increase the pH was determined.

Fe₂(SO₄)₃ (PIX 113), FeCl(SO₄) (PIX 118) and AlCl₃ (Pluspac 1800) were used in one series of experiments. Three different series of experiments were conducted with FeCl₃ (PIX 111), FeCl₃ (ciba) and AlCl₃ (Pluspac 1465). The first series were performed by addition of different dosings of the salts to different feeds; in the second series phosphate removal degrees and SVI (Sludge Volume Indices) were determined and in the third series these chemicals were applied in conjunction with a polymer. (see Table 6)

All the results for each experiment are presented in tables in the Appendix. (from Appendix A to Appendix J)

Table 6: Different experiments and used chemicals

	Used chemicals	Goal
Experiment 1	FeCl ₃ (PIX 111), Fe ₂ (SO ₄) ₃ (PIX 113), FeCl(SO ₄) (PIX 118), FeCl ₃ (ciba), AlCl ₃ (Pluspac 1800), AlCl ₃ (Pluspac 1465)	To determine the amounts needed for efficient precipitation
Experiment 2	FeCl ₃ (PIX 111), FeCl ₃ (ciba), AlCl ₃ (Pluspac 1465), Ca(OH) ₂	Calculation of removal degrees of phosphorus
Experiment 3	FeCl ₃ (PIX 111), FeCl ₃ (ciba), AlCl ₃ (Pluspac 1465), Magnafloc 336	To find out the difference in the process with and without polymer addition

6. Results

6.1 $Fe_2(SO_4)_3$ (PIX 113)

The experiments with $Fe_2(SO_4)_3$ were conducted by achieving the assumed Fe/P molar ratios presented in Table 7. The goal was to determine the Fe amount needed for efficient phosphate removal.

Table 7: Assumed Fe/P molar ratios and dosed amounts of Fe

Assumed Fe/P molar ratio	0.5	0.75	1	1.5	2	3
mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4

The amounts of PO_4 -P, total P and Fe in the non-filtrated and filtrated supernatants are given in Figure 6.1.1 and Figure 6.1.2. The concentrations of PO_4 -P and total P in the non-filtrated supernatant after dosing 17.9 mg(Fe)/l were 9 and 14 mg/l. The concentrations decreased down to 0.015 and 0.69 mg/l after addition of 107.4 mg(Fe)/l. The Fe concentration was high in the non-filtrated sample and at the peak it was 21 mg/l (after dosing 53.7 and 71.6 mg/l) and decreased down to 11 mg/l.

The Fe, PO_4 -P and total P concentrations in non-filtrated supernatants are higher compared to filtrated supernatants because particles of $FePO_4$ and $Fe(OH)_3$ which are not settled contribute to the Fe and PO_4 amounts. Another reason is possible adsorption of Fe and PO_4 on particles.

After adding 17.9 mg(Fe)/l the residual PO_4 -P and total P concentrations in the filtrated supernatant were 6.3 and 9.7 mg/l and declined to 1.3 and 2.3 mg/l when the Fe dose was 53.7 mg/l. The lowest concentrations were detected in the last experiment (107.4 mg/l) and they were 0.027 and 0.15 mg/l.

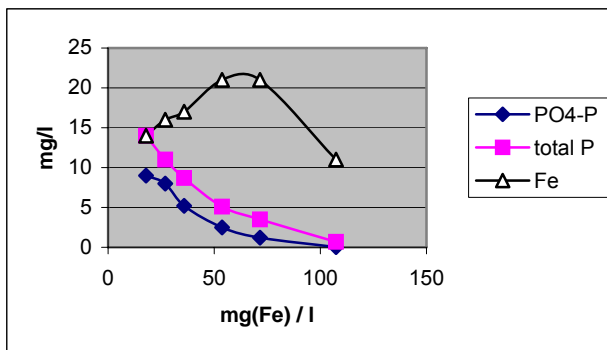


Figure 6.1.1 Concentrations of PO_4 -P, total P and Fe in a non-filtrated supernatant

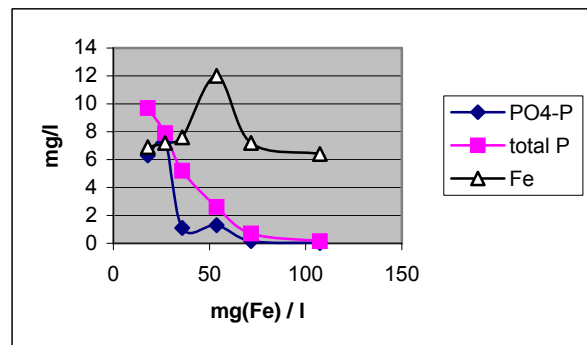


Figure 6.1.2 Concentrations of PO_4 -P, total P and Fe in a filtrated supernatant

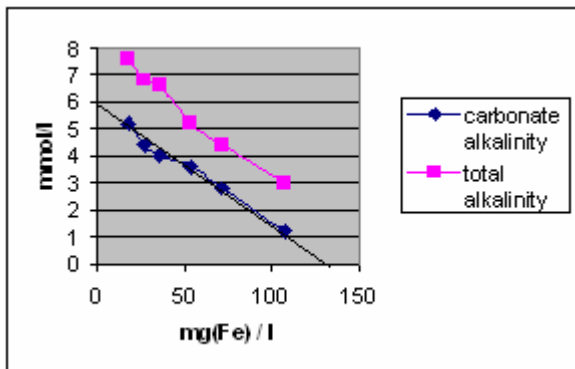


Figure 6.1.3: Carbonate and total alkalinity

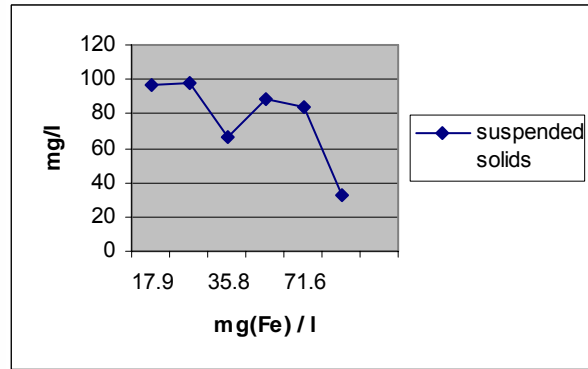


Figure 6.1.4: Concentration of suspended solids

The decrease in carbonate and total alkalinity (see Figure 6.1.3) was almost linear. These parameters had values of 5.2 and 7.6 mmol/l after adding 17.9 mg/l and decreased down to 3.6 and 5.2 mmol/l when the Fe dose was 53.7 mg/l. When applying the highest dose the carbonate and total alkalinity were 1.2 and 3 mmol/l. The decrease in alkalinity can be determined by calculating the slope of the line in Figure 6.1.3.

$$\tan(\alpha) = - 6 / 2.36 = -2.5$$

Approximately, the decrease in alkalinity was 2.5 mmol/mmol Fe.

In Figure 6.1.4 the change in the concentration of suspended solids is shown. The curve shows that there were fluctuations in the concentration of suspended solids and after adding 107.4 mg/l the concentration of suspended solids decreased to 33 mg/l. The sludge volume increased from 30 ml (in the first experiment) to 180 ml when the Fe dose was 107.4 mg/l.

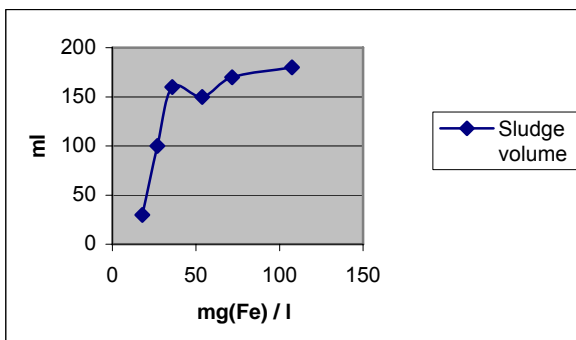


Figure 6.1.5: Sludge volume

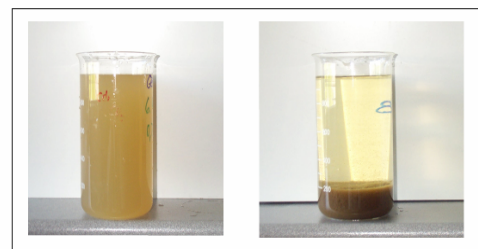


Figure 6.1.6: Black water before and after addition of 107.4 mg(Fe) / l

The addition of $\text{Fe}_2(\text{SO}_4)_3$ provided good results and in Figure 6.1.6 clarity difference between the feed sample and treated black water with 107.4 mg(Fe)/l can be observed.

6.2 FeCl(SO₄) (PIX 118)

The amounts of Fe that were added to the feed sample and assumed Fe/P molar ratios are shown in Table 8. The experiments were performed to find out the Fe amount needed for efficient precipitation of phosphates.

Table 8: Assumed Fe/P molar ratios and dosed amounts of Fe

Assumed Fe/P molar ratio	0.5	0.75	1	1.5	2	3
mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4

The concentrations of PO₄-P, total P and Fe in the non-filtrated and filtrated supernatants are presented in Figure 6.2.1 and Figure 6.2.2. After dosing 17.9 mg/l the concentrations of PO₄-P and total P in the non-filtrated supernatant were 8.6 and 12 mg/l. An increase in these concentrations was noticed after adding 26.9 mg/l because other feed sample was used and it contained much higher amount of phosphorus and suspended solids.

The amounts of PO₄-P and total P declined down to 8.3 and 14 mg/l for the Fe dose of 71.6 mg/l. The residual PO₄-P and total P concentrations after applying Fe dose of 107.4 mg/l were 0.24 and 0.49 mg/l.

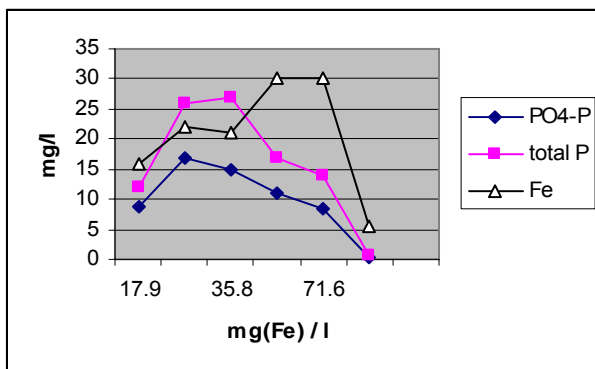


Figure 6.2.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

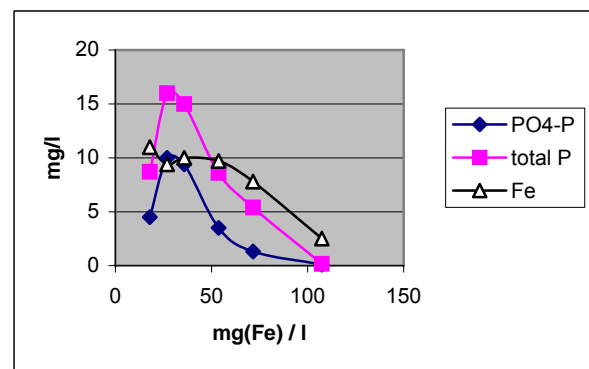


Figure 6.2.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

Fe concentration of 30 mg/l was measured after adding 53.7 and 71.6 mg/l. In the filtrated supernatant the PO₄-P and total P concentrations were 10 and 16 mg/l when the Fe dose was 26.9 mg/l and for the highest dose decreased to 0.084 and 0.17 mg/l.

After applying the lowest Fe dose the carbonate and total alkalinity were 4.2 and 5.8 mmol/l and at the peak higher values were detected (11.8 and 14.8 mmol/l). (see Figure 6.2.3)

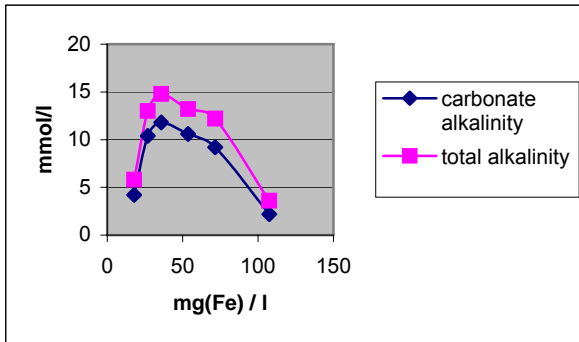


Figure 6.2.3: : Carbonate and total alkalinity

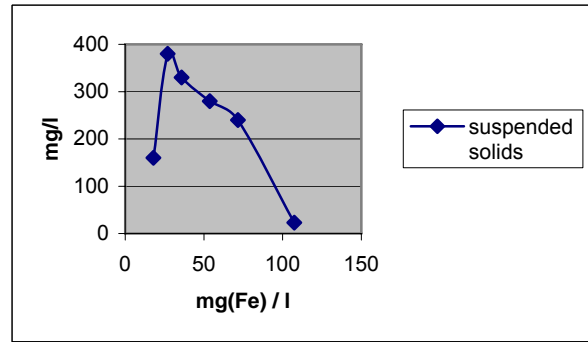


Figure 6.2.4: : Concentration of suspended solids

In the first experiment with this chemical the amount of suspended solids was 160 mg/l and rose to 380 mg/l after addition of the highest Fe amount because the feed sample had much higher concentration of suspended solids than the previous one.

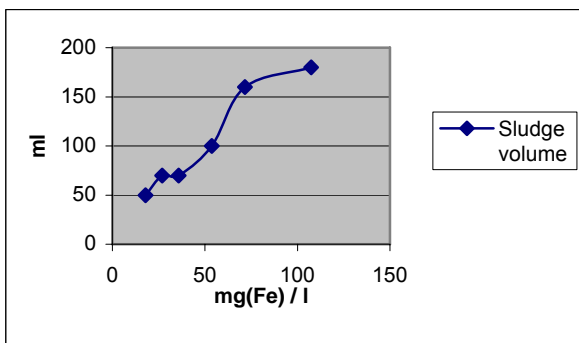


Figure 6.2.5: Sludge volume

Gradual increase in the sludge volume was observed. Addition of 17.9 mg(Fe)/l resulted in formation of 50 ml of sludge and the highest dose provided 180 ml of sludge. (Figure 6.2.5)

In Figure 6.2.6 a visual comparison of a feed sample and treated black water with 107.4 mg/l is given.

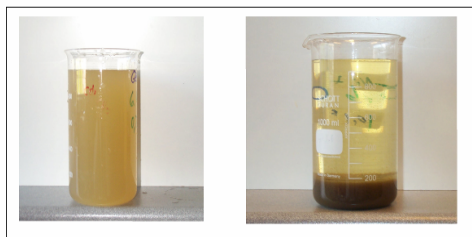


Figure 6.2.6: Black water before and after addition of 107.4 mg(Fe) / l

6.3 FeCl₃ (PIX 111)

6.3.1 FeCl₃ (PIX 111) (Experiment 1)

The goal of these experiments was to determine the conditions for good precipitation of FePO₄. The amounts of Fe needed to achieve different Fe/P molar ratios were calculated and the values are given in Table 9.

Table 9: Assumed Fe/P molar ratios and dosed amounts of Fe

Assumed Fe/P molar ratio	0.5	0.75	1	1.5	2	3
mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4

As the amount of Fe increased the concentrations of PO₄-P and total P decreased significantly. The decreases in these parameters are illustrated in Figure 6.3.1. and Figure 6.3.2.

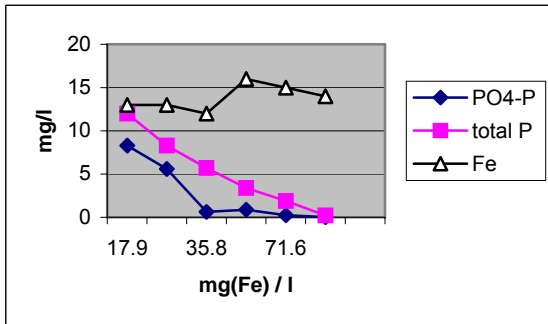


Figure 6.3.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

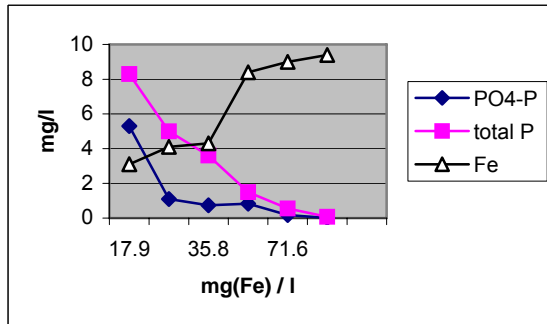


Figure 6.3.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

After adding 17.9 mg/l the concentrations of PO₄-P and total P in the non-filtrated supernatant were 8.3 and 12 mg/l respectively. The concentrations decreased down to 0.63 and 5.7 mg/l after adding 35.8 mg/l. The lowest concentrations were noticed in the filtrated supernatant after addition of 107.4 mg/l and those were <0.01 and 0.07 mg/l.

The Fe concentration in the non-filtrated supernatant was high and it ranged between 12 and 16 mg/l. After adding 17.9 mg(Fe)/l the Fe concentration in the filtrated supernatant was 3.1 and increased to 9.4 mg/l.

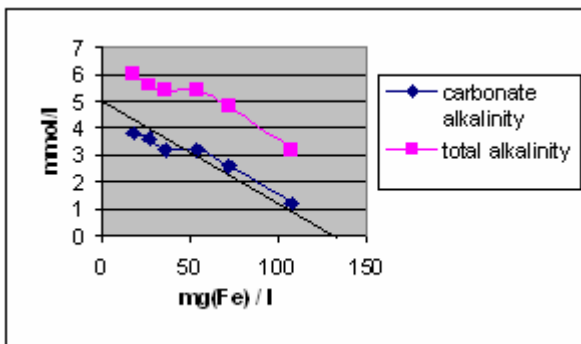


Figure 6.3.3: Carbonate and total alkalinity

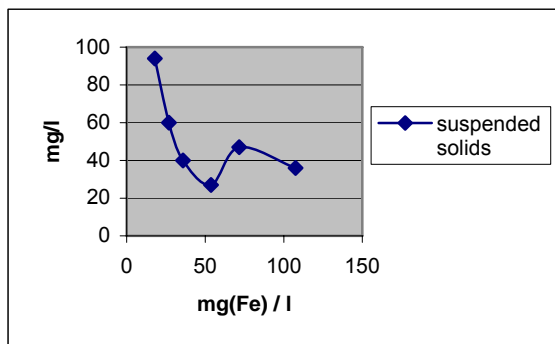


Figure 6.3.4: Suspended solids

After addition of the lowest amount the carbonate and total alkalinity were 3.8 and 6 mmol/l and after dosing 107.4 mg/l declined to 1.2 and 3.2 mmol/l. (see Figure 6.3.3)

The slope of the line is:

$$\tan(\alpha) = - 5 / 2.4 = -2.08$$

The decrease in alkalinity was approximately 2.08 mmol/mmol (Fe)

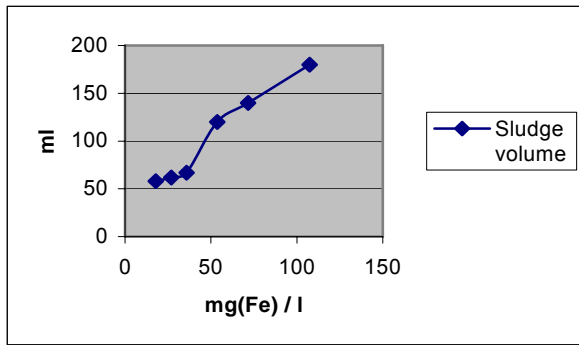


Figure 6.3.5: Sludge volume

Due to the different compositions of the feed samples there was no gradual decrease in the amount of suspended solids as illustrated in Figure 6.3.4. In the first experiment the amount was 94 mg/l and it decreased to 27 mg/l.

The sludge volume that was formed in each experiment is shown in Figure 6.1.5. After applying the lowest amount of Fe the sludge volume was 58 ml and increased to 180 ml after adding 107.4 mg/l.

As a result of the good removal of phosphates and suspended solids the turbidity of the supernatants decreased and there was a clear difference compared to the feed sample. In Figure 6.3.6 beakers with a feed sample and black water after adding 107.4 mg(Fe)/l are shown.

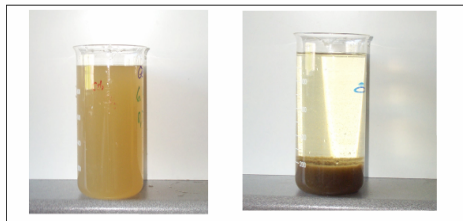


Figure 6.3.6: Black water before and after addition of 107.4 mg(Fe) / l

6.3.2 FeCl₃ (PIX 111) (Experiment 2)

Considering that the phosphorus concentration was determined in the feed samples it was possible to determine some parameters such as removal degree of phosphorus, suspended solids, decrease in alkalinity, SVI.

The amounts of Fe that were added to the feed samples were: 53.7, 71.6 and 107.4 mg/l. In the first two experiments the PO₄-P and total P amounts in the non-filtrated feed sample were 12 and 16 mg/l and in the last experiment these values were 14 and 17 mg/l.

The PO₄-P, total P and Fe concentrations in the non-filtrated and filtrated supernatants are given in Figure 6.3.2.1 and Figure 6.3.2.2. The Fe dose of 53.7 mg/l decreased the total P amount to 3.8 mg/l which corresponds to 76% reduction. Increasing the Fe dose to 71.6 and 107.4 mg/l caused decreases in the total P concentrations to 1.4 and 0.5 mg/l which represent 91% and 97% reductions respectively. These Fe doses produced the following residual PO₄-P concentrations in the non-filtrated supernatants: 3, 1 and 0.14 mg/l. These were the reduction degrees in PO₄-P: 75, 91.7 and 99%.

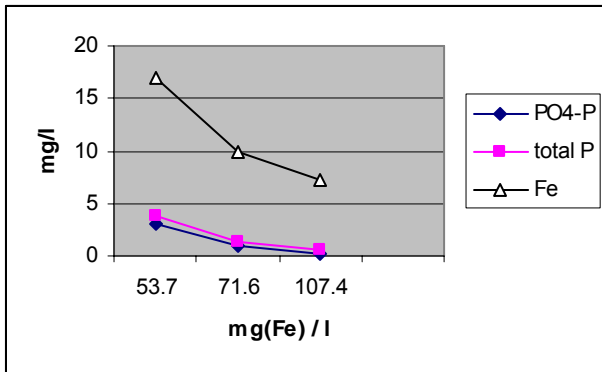


Figure 6.3.2.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

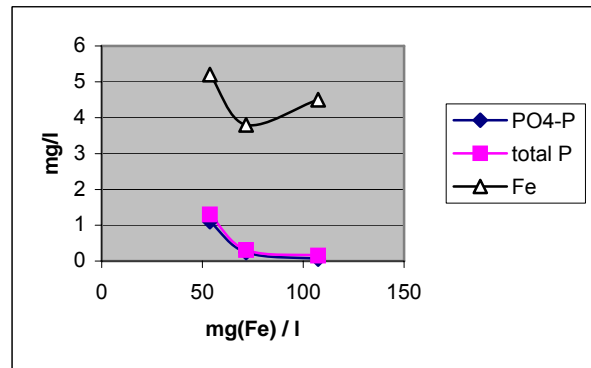


Figure 6.3.2.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

The Fe concentration decreased from 17 mg/l to 7.2 mg/l in the non-filtrated supernatant.

Converted into moles the amounts of Fe added to the black water were: 0.98, 1.3 and 1.95 mmol/l. The amount of PO₄ in the filtrated feed sample before adding 0.98 and 1.3 mmol(Fe)/l was 0.35 mmol hence the following Fe/PO₄ molar ratios were achieved: 2.8 and 3.7. In the next experiment (107.4 mg(Fe)/l) the ratio was 4.6. The lowest Fe/PO₄ molar ratio caused a reduction of 89.2% in the amount of total P. The Fe/PO₄ molar ratio of 3.7 lowered the total P concentration by 97.4 % and the reduction with the highest molar ratio was 99%.

The lowest Fe dose removed 90% of PO₄-P. For the ratio of 3.7 the PO₄-P concentration decreased by 98%. The highest ratio of 4.6 caused 99.6% reduction in the amount of PO₄-P.

Alkalinity was measured in the feed and in the non-filtrated supernatants and this enabled to calculate the decrease in alkalinity. (see Table 10)

Table 10: Decrease in alkalinity

mmol(Fe)/l	0.98	1.3	1.95
Decrease in alkalinity (mmol/mmol(Fe))	2.86	2.9	2.77

In Figure 6.3.2.3 the decrease in carbonate and total alkalinity can be observed.

The amounts of suspended solids decreased to 13, 7.9 and 4.3% of the concentrations in

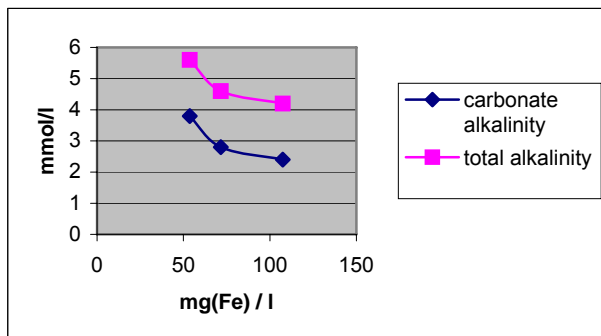


Figure 6.3.2.3: Carbonate and total alkalinity

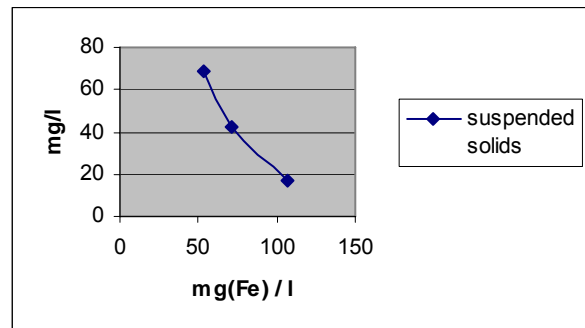


Figure 6.3.2.4: Concentration of suspended solids

the feeds which represent very good removal degrees.

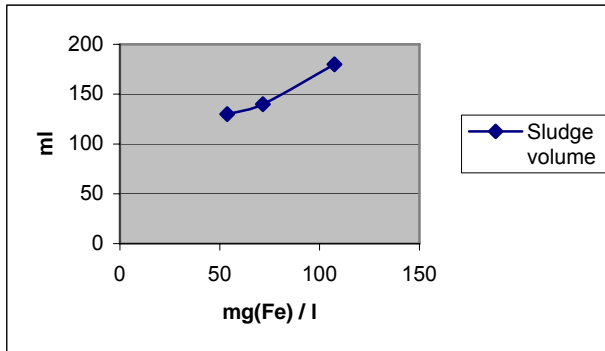


Figure 6.3.2.5: Sludge volume

The increase in the sludge volume can be seen in Figure 6.3.2.5. After adding 53.7 mg/l the sludge volume was 130 ml and increased to 180 ml after dosing 107.4 mg/l. The amounts of FePO_4 and $\text{Fe}(\text{OH})_3$ can be calculated because the PO_4 and Fe concentrations before adding the chemical and after the sedimentation are known.

SVI is calculated after applying the Fe dose of 107.4 mg/l. The processes of entrapment and adsorption of PO_4 and Fe ions on particles are neglected.

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.42 - 0.002$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.418 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = [\text{Fe}]_{(\text{initial})} - [\text{Fe}]_{(\text{after sedimentation})}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.08 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.87 \text{ mmol}$$

1 mol PO_4 reacts with 1 mol Fe and 1 mol FePO_4 is produced hence, 0.418 mmol PO_4 produced 0.418 mmol FePO_4 . In this experiment the amount of FePO_4 was 62.7 mg. 0.418 mmol of Fe entered this reaction and the rest of the Fe – 1.45 mmol produced 1.45 mmol $\text{Fe}(\text{OH})_3$. The mass of $\text{Fe}(\text{OH})_3$ produced was 153.7 mg.

The amount of suspended solids in the feed is a sum of the amounts in the sludge and supernatant.

$$m_{(\text{s.s. in sludge})} = m_{(\text{s.s. in feed})} - m_{(\text{s.s. in supernatant})}$$

$$m_{(\text{s.s. in sludge})} = 390 - 17 = 373 \text{ mg}$$

The amount of total solids (TS) in the sludge is a sum of the masses of FePO_4 , $\text{Fe}(\text{OH})_3$ and suspended solids.

$$m_{(\text{TS in sludge})} = m_{(\text{FePO}_4)} + m_{(\text{Fe}(\text{OH})_3)} + m_{(\text{s.s. in sludge})}$$

$$m_{(\text{TS in sludge})} = 589.4 \text{ mg}$$

$$V_{\text{sludge}} = 180 \text{ ml}$$

By knowing all these parameters the Sludge Volume Index (SVI) can be calculated. By definition it is a sludge volume occupied by 1g of dry sludge solids after 30 min settling. (Norweco 2007)

$$\text{SVI} = V_{\text{sludge}} / m_{(\text{TS in sludge})}$$

$$\text{SVI} = 180 / 0.59 = 305 \text{ ml(sludge)/g TS}$$

6.3.3 FeCl₃ (PIX 111) and a polymer

The aim of these experiments was to realize the difference in the results after polymer addition. The addition of the polymer Magnafloc 336 did not have a big influence on the reductions in the phosphorus amount. In these experiments the same Fe/PO₄ molar ratios as in the previous section were reached: 2.8, 3.7 and 4.6. The concentrations of PO₄-P, total P and Fe in the non-filtrated and filtrated supernatants are presented in Figure 6.3.3.1 and Figure 6.3.3.2.

In the non-filtrated supernatant the amounts of PO₄-P and total P were 1.9 and 2.2 mg/l

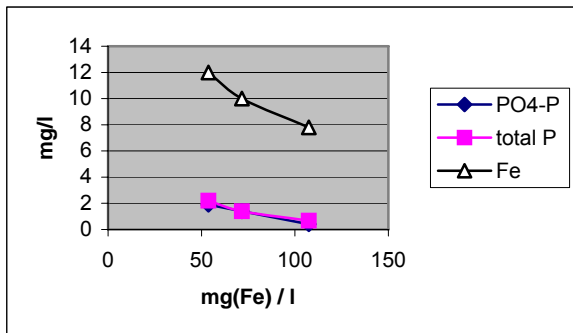


Figure 6.3.3.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

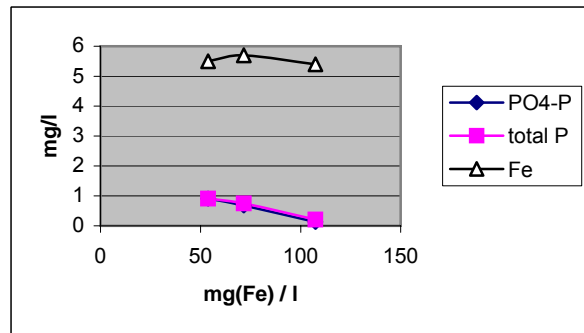


Figure 6.3.3.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

when the Fe/PO₄ molar ratio was 2.8. These values are 1.1 and 1.6 mg lower compared to the concentrations when the polymer was not added. In the experiment with the highest Fe/PO₄ molar ratio of 4.6 the amounts of PO₄-P and total P were 0.4 and 0.67 mg/l and these values are higher compared to those in the experiments conducted without polymer addition. (Figure 7.2.1)

Only in the first experiment for the lowest Fe/PO₄ molar ratio the Fe concentration was lower than the one detected when the polymer was not added. The other values for the Fe concentrations were the same as in experiments described in Chapter 6.3.2.

In the filtrated supernatant the amounts of PO₄-P and total P in the first experiment were 0.91mg/l which is 0.2 mg difference compared to the previous experiments without the polymer. For the Fe/PO₄ molar ratio of 4.6 the amounts of PO₄-P and total P were 0.13 and 0.21 mg/l. These values were higher than those in the experiments when the polymer was not added and the differences were 0.074 and 0.06 mg.

The polymer does not have influence on the alkalinity and the values that were

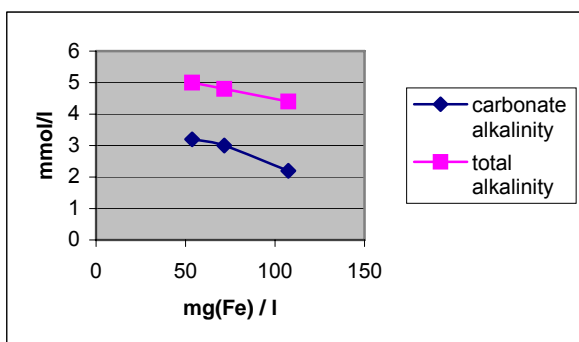


Figure 6.3.3.3: Carbonate and total alkalinity

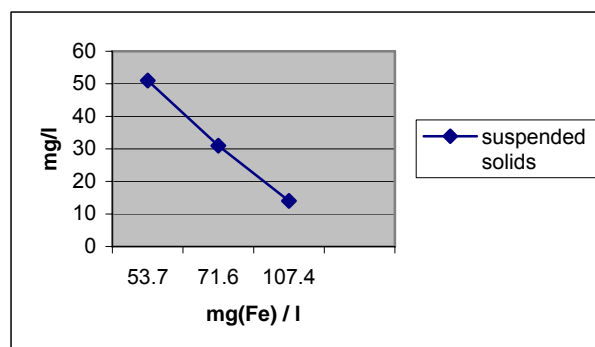
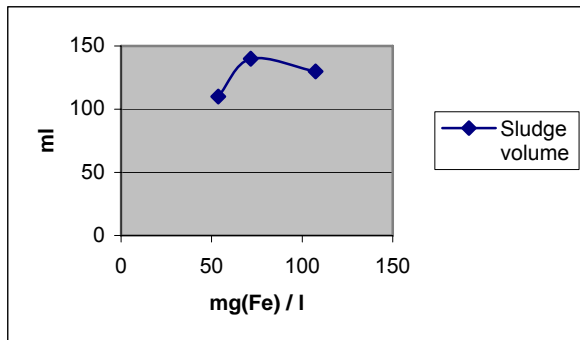


Figure 6.3.3.4: Concentration of suspended solids

measured were same as in Chapter 6.3.2.

The addition of the polymer produced supernatants with lower amounts of suspended solids than in the experiments without polymer addition (concentrations were 3-18 mg lower). When the Fe/PO₄ molar ratio was 2.8 the reduction in the amount of suspended solids was 90.4%. For the Fe/PO₄ molar ratios of 3.7 and 4.6 the amounts of suspended solids decreased by 94.2 and 96.4%.

The anionic polymer that was added forms "bridges" among the particles during the coagulation, improves the process and in this way the sludge becomes more compact. The sludge that was formed was less voluminous than the sludge in the experiment when the polymer was not added.



Calculations for the amounts of FePO₄, Fe(OH)₃ and SVI value are made below.

Figure 6.3.3.5: Sludge volume

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.42 \text{ mmol} - 0.004 \text{ mmol}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.38 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = [\text{Fe}]_{(\text{initial})} - [\text{Fe}]_{(\text{after sedimentation})}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.098 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.852 \text{ mmol}$$

In this experiment 57 mg of FePO₄ and 156 g of Fe(OH)₃ were produced.

$$m_{(\text{s.s. in sludge})} = m_{(\text{s.s. in feed})} - m_{(\text{s.s. in supernatant})}$$

$$m_{(\text{s.s. in sludge})} = 390 - 14 = 376 \text{ mg}$$

$$m_{(\text{TS in sludge})} = m(\text{FePO}_4) + m(\text{Fe}(\text{OH})_3) + m_{(\text{s.s. in sludge})}$$

$$m_{(\text{TS in sludge})} = 589 \text{ mg}$$

$$V_{\text{sludge}} = 130 \text{ ml}$$

$$\text{SVI} = 220.3 \text{ ml}(\text{sludge})/\text{g TS}$$

6.4 FeCl₃ (ciba)

6.4.1 FeCl₃ (ciba) (Experiment 1)

It was concluded from the previous experiments that the assumed Fe/P molar ratios less than 1 were not sufficient for good phosphorus removal and the next experiments were conducted by beginning with assumed molar ratio of 1. The experiments were performed to determine the optimal Fe dose. The assumed Fe/P molar ratios and amounts of Fe added are presented in the following Table.

Table 11: Assumed Fe/P molar ratios and amounts of Fe

Assumed Fe/P molar ratio	1	1.5	2	3
mg (FeCl ₃)/l	35.8	53.7	71.6	107.4

After dosing 35.8 mg/l the residual PO₄-P and total P concentrations in the non-filtrated supernatant were: 6.5 and 9.1 mg/l. These concentrations dropped down to 2.7 and 3.1 mg/l when the Fe dose increased to 107.4 mg/l. (Figure 6.4.1)

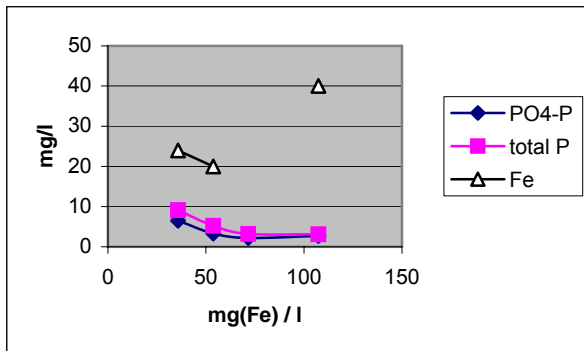


Figure 6.4.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

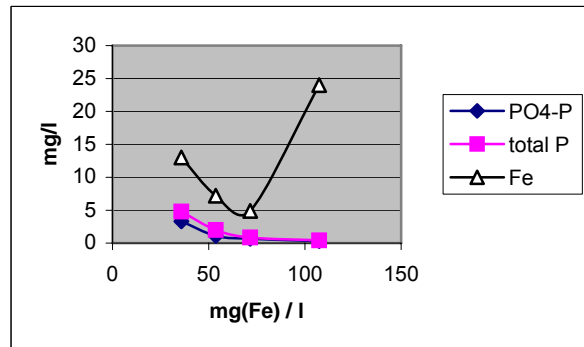


Figure 6.4.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

High Fe concentrations in the non-filtrated and filtrated supernatants were measured. When the Fe dose was 35.8 mg/l the Fe amount in the non-filtrated supernatant was 24 mg/l. A decrease in the Fe concentration was noticed when increasing the Fe dose. However for the highest Fe dose the Fe concentration rose to 40 mg/l. In the filtrated supernatant the Fe concentration followed the same trend and for the Fe dose of 107.4 mg/l increased to 24 mg/l. The addition of 35.8 mg(Fe)/l decreased the amounts of PO₄-P and total P to 3.3 and 4.8 mg/l. Low residual PO₄-P and total P amounts were registered after addition of the highest dose - 0.31 and 0.42 mg/l.

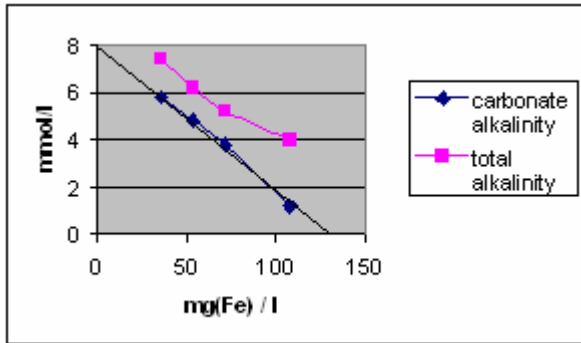


Figure 6.4.3: Carbonate and total alkalinity

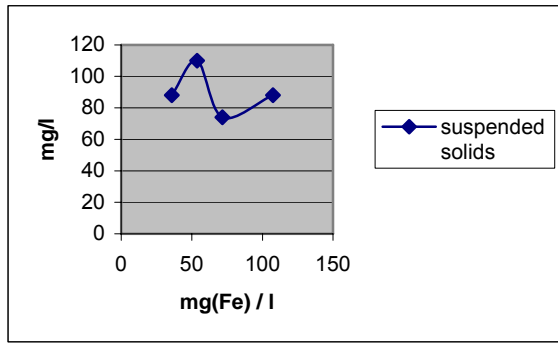


Figure 6.4.4: Suspended solids

Significant decrease in the carbonate and total alkalinity was noticed and the values were 1.2 and 4 mmol/l respectively. Because the decrease in alkalinity was almost linear the slope of the line is calculated as follows:

$$\tan(\alpha) = -8 / 2.36 = -3.4$$

Addition of 1mmol(Fe) caused a decrease in alkalinity by 3.4 mmol.

Different feed compositions were a reason for the fluctuations in the amount of suspended solids (see Figure 6.4.4). Addition of an Fe dose of 71.6 mg/l caused a decline of this parameter to 74 mg/l but when the highest Fe dose was applied the amount of suspended solids was 88 mg/l.

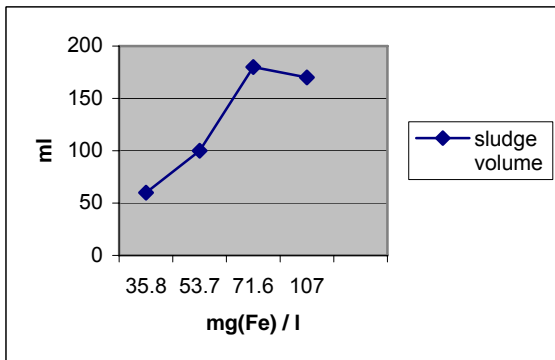


Figure 6.4.5: Sludge volume

Due to the different feed samples the amount of produced sludge did not increase gradually. The sludge volume increase to 180 ml when the Fe dose was 71.6 mg/l and then for the highest dose a lower value was measured – 170 ml. Beakers with a feed sample and treated black water with 107.4 mg(Fe)/l are presented in Figure 6.4.6.

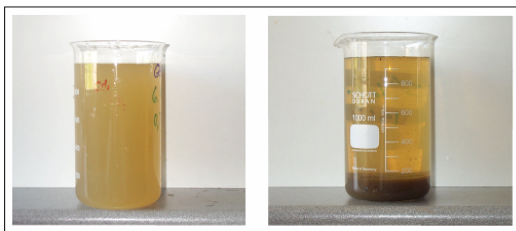


Figure 6.4.6: Black water before and after addition of 107.4 mg(Fe) / l

6.4.2 FeCl₃ (ciba) (Experiment 2)

The amounts of Fe that were added to the feed samples were: 53.7, 71.6 and 107.4 mg/l. The PO₄-P and total P concentrations in the non-filtrated feed sample before adding 53.7 and 71.6 mg/l were 11 and 16 mg/l. In the third experiment the PO₄-P and total P amounts were 14 and 20 mg/l.

Expressed in moles the amounts of Fe added to the feed samples were: 0.98, 1.3 and 1.95 mmol. According to these values the Fe/PO₄ molar ratios were 2.8, 3.7 and 4.3. The changes in the concentrations of PO₄-P, total P and Fe in the non-filtrated and filtrated supernatants can be observed in Figure 6.4.2.1 and Figure 6.4.2.2.

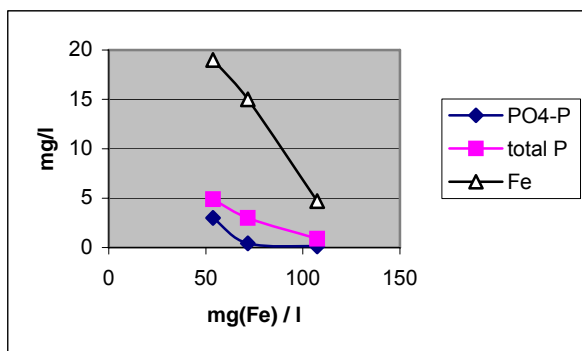


Figure 6.4.2.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

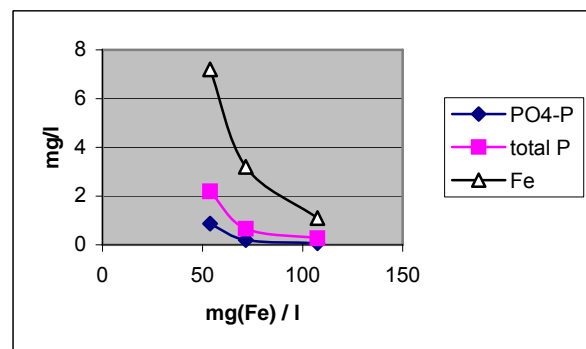


Figure 6.4.2.2: Concentrations of PO₄-P, total P and Fe in a filtrated supernatant

Residual PO₄-P and total P concentrations of 3 and 4.9 mg/l in the non-filtrated supernatant for the Fe/PO₄ of 2.8 were obtained. The reductions in these parameters were 72.7 and 69.4%. Increasing the Fe dose caused a decrease in the residual concentrations and for the Fe/PO₄ molar ratio of 4.3 they were 0.11 and 0.89 mg/l. The PO₄-P and total P amounts were reduced by 99.2 and 95.5%.

Big decrease in the concentration of Fe was noticed as the Fe dose increased. The Fe concentration in the non-filtrated supernatant was 19 mg/l in the first experiment and declined to 4.7 mg/l when the Fe/PO₄ molar ratio was 4.3.

In the filtrated supernatant PO₄-P residual concentrations that were obtained for all the experiments were below 1 mg/l and the lowest PO₄-P amount was 0.065 mg/l corresponding to Fe/PO₄ molar ratio of 4.3. In these experiments the Fe concentrations were below 8 mg/l and the highest molar ratio provided Fe concentration of 1.1 mg/l.

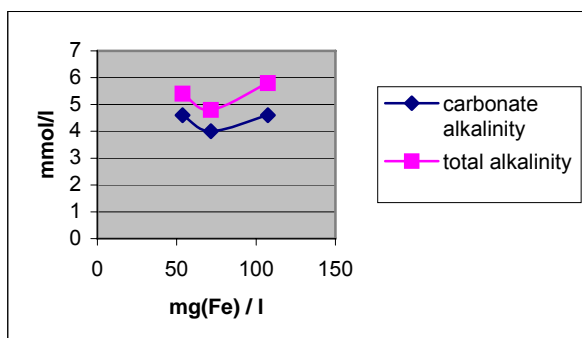


Figure 6.4.2.3: Carbonate and total alkalinity

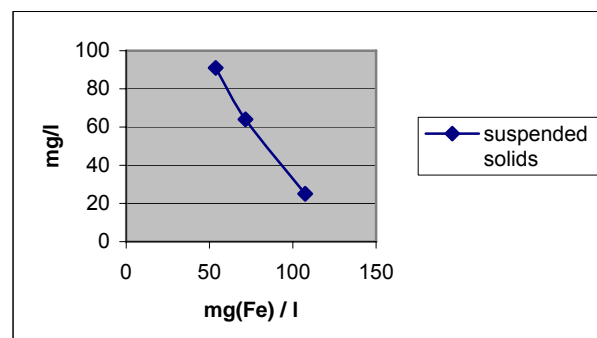


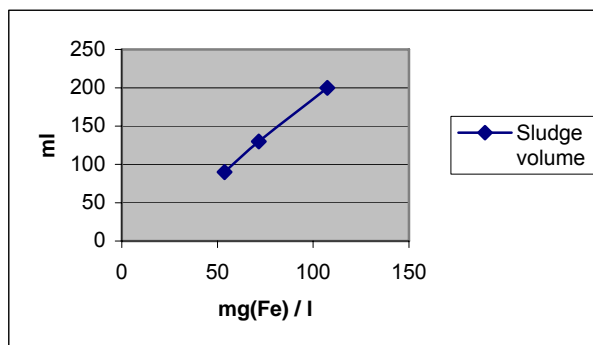
Figure 6.4.2.4: Concentration of suspended solids

Figure 6.4.2.3 shows that the values of carbonate and total alkalinity after applying the Fe/PO₄ molar ratio of 3.7 were lower than those for the Fe/PO₄ molar ratio of 4.3. This is due to the fact that other feed sample was used for the third experiment and it had higher alkalinity. In Table 12 the decrease in alkalinity caused by addition of 1 mmol(Fe) is presented.

Table 12: Decrease in alkalinity

mmol(Fe)/l	0.98	1.3	1.95
Decrease in alkalinity (mmol/mmol(Fe))	2.4	2.3	2.8

The reduction in the amount of suspended solids was linear. The lowest Fe/PO₄ molar ratio of 2.8 provided 54.5% reduction in the amount of suspended solids. The amount of suspended solids declined to 64 mg/l which represents 32% of the concentration in the feed. The application of the highest Fe/PO₄ molar ratio provided 88% reduction in the amount of suspended solids.



Linear increase in the sludge volume was noticed during the experiments. As a result of addition of 53.7 mg(Fe)/l, 90 ml of sludge were formed. The highest sludge volume that was produced was 200 ml. Calculations for the amounts of produced FePO₄, Fe(OH)₃ and SVI value is made below for the highest Fe dose applied (107.4 mg/l)

Figure 6.4.2.5: Sludge volume

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.45 \text{ mmol} - 0.002 \text{ mmol}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.448 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = [\text{Fe}]_{(\text{initial})} - [\text{Fe}]_{(\text{after sedimentation})}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.02 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.93 \text{ mmol}$$

0.448 mmol PO₄ reacted with 0.448 mmol Fe and produced 0.448 mmol FePO₄ corresponding to 67.2 mg. 1.48 mmol produced 1.48 mmol Fe(OH)₃ which is equal to 157 mg.

$$m_{(\text{s.s. in sludge})} = m_{(\text{s.s. in feed})} - m_{(\text{s.s. in supernatant})}$$

$$m_{(\text{s.s. in sludge})} = 210 - 25 = 185 \text{ mg}$$

$$m_{(\text{TS in sludge})} = m_{(\text{FePO}_4)} + m_{(\text{Fe(OH)}_3)} + m_{(\text{s.s. in sludge})}$$

$$m_{(\text{TS in sludge})} = 409.2 \text{ mg}$$

$$V_{\text{sludge}} = 200 \text{ ml}$$

$$\text{SVI} = 500 \text{ ml(sludge)/g TS}$$

6.4.3 FeCl₃ (ciba) and a polymer

The use of FeCl₃ (ciba) in conjunction with Magnafloc 336 was investigated to realize how it affects the process. As in the experiments without a polymer addition the same amounts of Fe were used: 0.98, 1.3 and 1.95 mmol/l. The PO₄-P in the filtrated feed sample was the same as in the previous Chapter, hence the following Fe/PO₄ molar ratios were achieved: 2.8, 3.7 and 4.3. Residual PO₄-P, total P and Fe concentrations in the non-filtrated and filtrated supernatants are presented in Figure 6.4.3.1 and Figure 6.4.3.2.

In the non-filtrated supernatant for the Fe/PO₄ molar ratio of 2.8 the PO₄-P and total P concentrations were 1.9 and 3.8 mg/l (1.1 mg less compared to the experiments without a polymer). When the highest Fe/PO₄ molar ratio of 4.3 was achieved the residual PO₄-P concentration was 0.25 mg/l which is 0.14 higher compared to the concentration without applying the polymer.

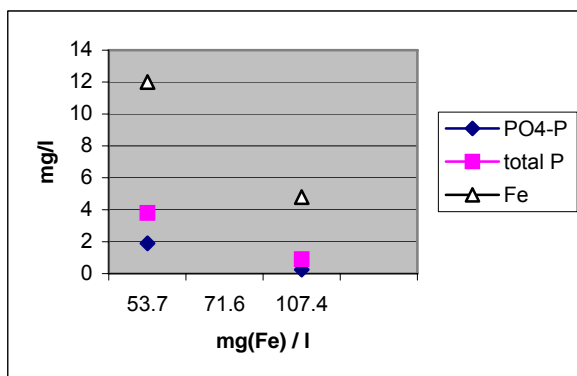


Figure 6.4.3.1: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

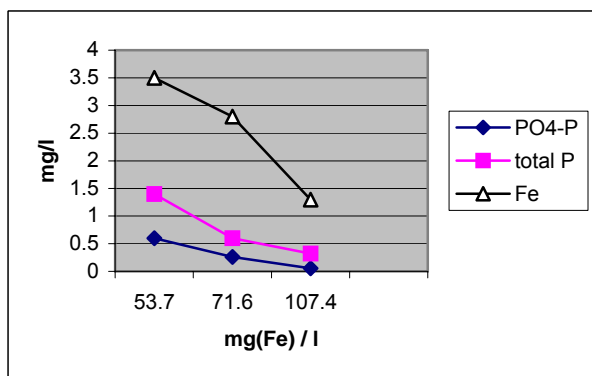


Figure 6.4.3.2: Concentrations of PO₄-P, total P and Fe in a non-filtrated supernatant

For the lowest Fe/PO₄ molar ratio of 2.8 the PO₄-P and total P concentrations in the filtrated supernatant were 0.6 and 1.4 mg/l. These values are 0.2 and 0.8 mg lower compared to those presented in Chapter 6.4.2. Almost the same values were obtained for the highest Fe/PO₄ molar ratio of 4.3.

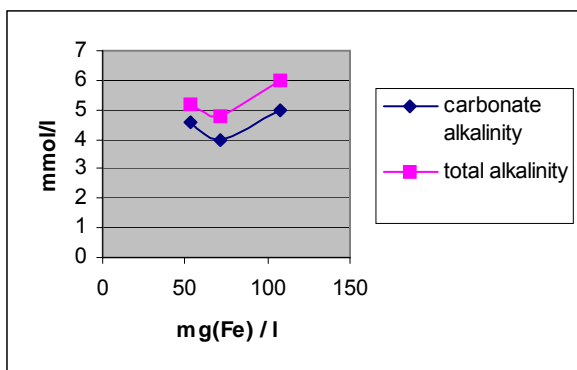


Figure 6.4.3.3: Carbonate and total alkalinity

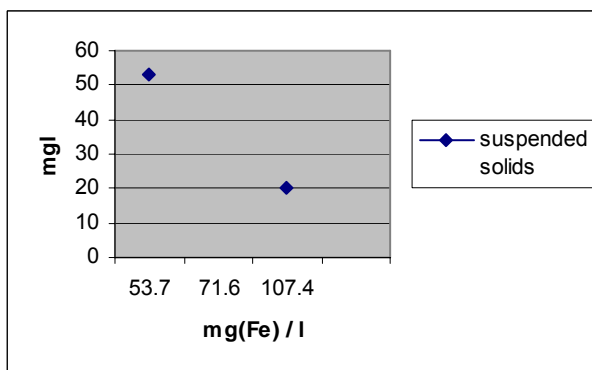
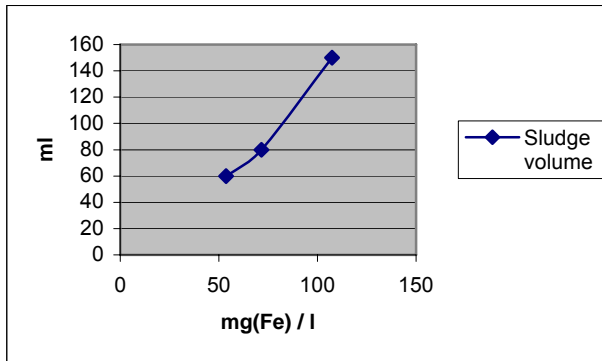


Figure 6.4.3.4: Concentration of suspended solids

Achieving the Fe/PO₄ molar ratio of 2.8 caused a reduction in the amount of suspended solids by 73.5% and the highest molar ratio provided reduction of 90.5%.

When applying the lowest Fe dose the amount of suspended solids was 40 mg lower compared to the Experiment 1. Approximately the same results were obtained for the highest Fe dose.



The addition of a polymer improves the coagulation process by forming bonds among the particles and in this way more compact sludge is made. The highest sludge volume was 150 ml. (Figure 7.4.5)

Figure 6.4.3.5: Sludge volume

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.45 \text{ mmol} - 0.002 \text{ mmol}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.448 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = [\text{Fe}]_{(\text{initial})} - [\text{Fe}]_{(\text{after sedimentation})}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.02 \text{ mmol}$$

$$[\text{Fe}]_{(\text{converted into precipitate})} = 1.93 \text{ mmol}$$

0.448 mmol PO₄ reacted with 0.448 mmol Fe and produced 0.448 mmol FePO₄ corresponding to 67.2 mg. 1.48 mmol produced 1.48 mmol Fe(OH)₃ which is equal to 157 mg.

$$m_{(\text{s.s. in sludge})} = m_{(\text{s.s. in feed})} - m_{(\text{s.s. in supernatant})}$$

$$m_{(\text{s.s. in sludge})} = 210 - 20 = 190 \text{ mg}$$

$$m_{(\text{TS in sludge})} = m(\text{FePO}_4) + m(\text{Fe}(\text{OH})_3) + m_{(\text{s.s. in sludge})}$$

$$m_{(\text{TS in sludge})} = 414.2 \text{ mg}$$

$$V_{\text{sludge}} = 150 \text{ ml}$$

$$\text{SVI} = 362.3 \text{ ml}(\text{sludge})/\text{g TS}$$

6.5 AlCl₃ (Pluspac 1800)

The experiments with AlCl₃ (Pluspac) were performed by using the amounts shown in Table 13. Al amounts needed for efficient precipitation of AlPO₄ were determined.

Table 13: Assumed Al/P molar ratios and amounts of Al

Assumed Al/P molar ratio	1	1.5	2	3
mg(Al)/l	17.6	26.3	35.1	52.7

The amounts of PO₄-P, total P and Fe in the non-filtrated and filtrated supernatants are illustrated in Figure 6.5.1 and Figure 6.5.2. The PO₄-P and total P amounts in the non-filtrated supernatant decreased from 10 and 14 mg/l (when 17.6 mg(Al)/l were added) to 1.8 and 2.9 mg/l when the highest amount of Al was applied.

In the filtrated supernatant after addition of 35.1 mg/l the PO₄-P and total P concentrations were 2.4 and 4 mg/l respectively and the concentrations dropped down to 0.87 and 1.7 mg/l for the highest dose of 52.7 mg/l. The Al concentrations were 2 mg/l and 0.78 mg/l after applying 35.1 and 52.7 mg(Al)/l. The Al concentration in the supernatants declined with increasing the Al dose.

In Figure 6.5.3 the decline in alkalinity can be observed. In the experiments with Al salts the decrease in alkalinity was lower compared to experiments with Fe salts because the solutions of Al salts contain OH⁻ ions which have a buffering effect.

The carbonate and total alkalinity were 8.4 and 10.2 mmol/l after adding 17.6 mg/l and decreased to 6.8 and 8.6 mmol/l when the highest dose was added.

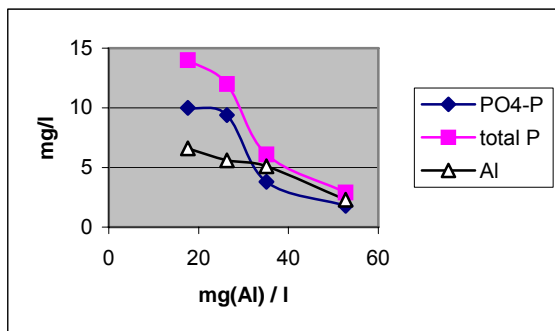


Figure 6.5.1: Concentration of PO₄-P, total P and Al in a non-filtrated supernatant

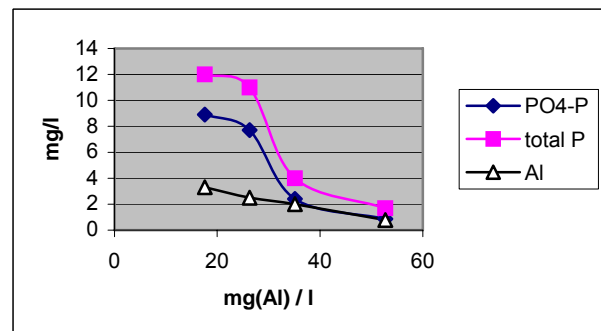


Figure 6.5.2: Concentration of PO₄-P, total P and Al in a filtrated supernatant

When the Al dose was 17.6 mg/l the amount of suspended solids was 150 mg/l and for

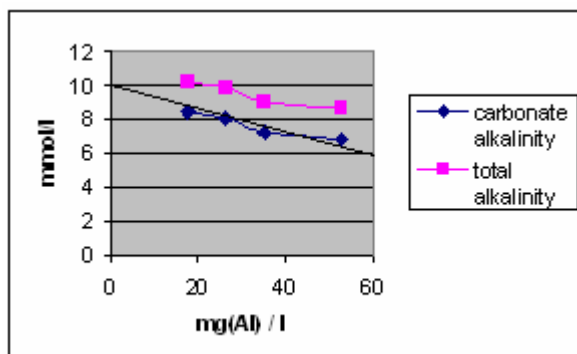


Figure 6.5.3: Carbonate and total alkalinity

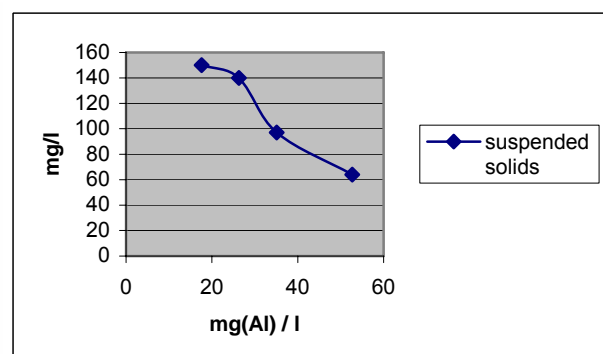


Figure 6.5.4: Concentration of suspended solids

the highest dose this value was 64 mg/l. (Figure 6.5.4)

The slope of the line in Figure 6.5.3 is calculated below:

$$\tan(\alpha) = - 4/2.2 = - 1.8$$

The decrease in alkalinity was approximately 1.8 mmol/mmol(Al).

The increase in the sludge volume when increasing the Al dose can be observed in Figure 6.6.5. The sludge volume that was formed in the first experiment (17.6 mg(Al)/l) was 70 ml and increased to 200 ml when adding the highest Al dose.

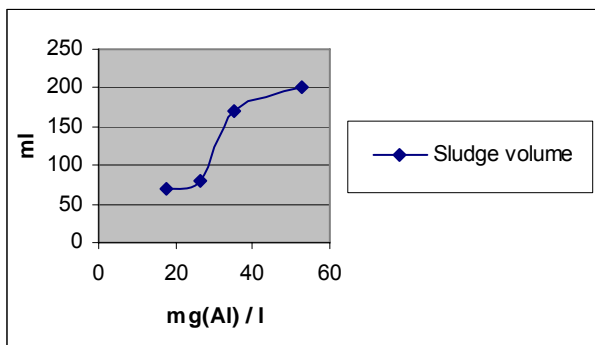


Figure 6.5.5: Sludge volume

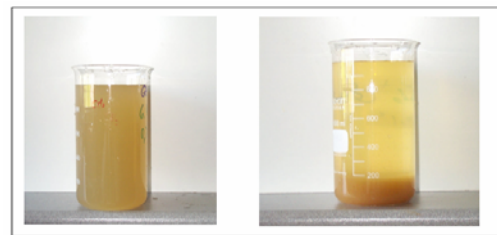


Figure 6.5.6: Black water before and after addition of 52.7 mg(Al) / l

The addition of 52.7 mg/l provided a clear supernatant which can be seen in Figure 6.5.6.

6.6 AlCl₃ (Pluspac 1465)

6.6.1 AlCl₃ (Pluspac 1465) (Experiment 1)

The amounts of Al that were used in the experiments with AlCl₃ (Pluspac 1465) are given in Table 14.

Table 14: Assumed Al/P molar ratios and amounts of Al

Assumed Al/P molar ratio	1	1.5	2	3
mg(Al)/l	17.6	26.3	35.1	52.7

The changes in concentrations of PO₄-P, total P and Fe in the non-filtrated and filtrated supernatants are given in Figure 6.6.1.1 and Figure 6.6.1.2. In the non-filtrated supernatant, after dosing 17.6 mg/l the PO₄-P and total P amounts were 6.2 and 7.8 mg/l. These concentrations decreased to 0.11 and 0.41 mg/l for the highest amount of 52.7 mg/l. In the

non-filtrated supernatant the Al concentration was 4.6 mg/l in the first experiment (17.6 mg(Al)/l). Increasing the Al dose to 52.7 mg/l caused a decrease in the Al concentration to 1 mg/l.

In the filtrated supernatant the PO₄-P and total P concentrations were 4.7 and 4.8 mg/l in the first experiment and decreased to 0.092 and 0.17 mg/l after addition of 52.7 mg/l.

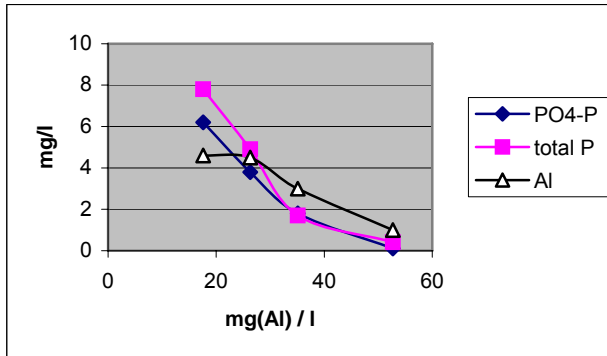


Figure 6.6.1.1: Concentrations of PO₄-P, total P and Al in a non-filtrated supernatant

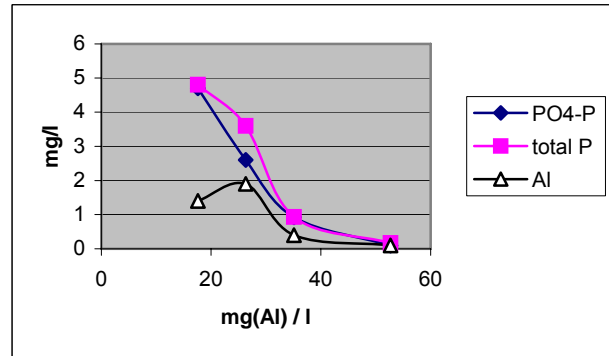


Figure 6.6.1.2: Concentrations of PO₄-P, total P and Al in a filtrated supernatant

The Al concentration in the filtrated supernatant in the first experiment was 1.4 mg/l and a very low Al concentration was detected in the fourth experiment – 0.096mg/l.

After adding the lowest Al dose the carbonate and total alkalinity were 4.8 and 7.4 mmol/l and after applying the highest amount of Al decreased to 3.6 and 6.4 mmol/l. (see Figure 6.6.1.3)

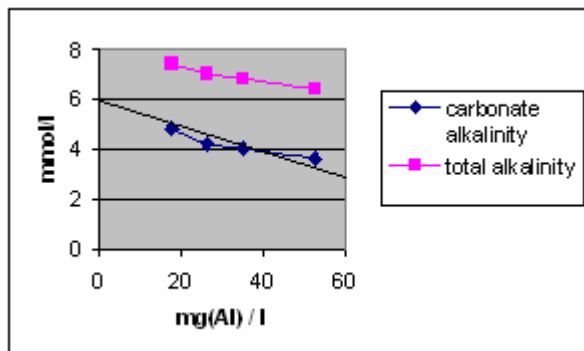


Figure 6.6.1.3: Carbonate and total alkalinity

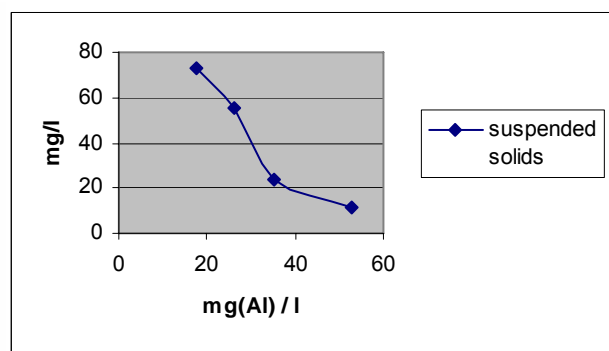


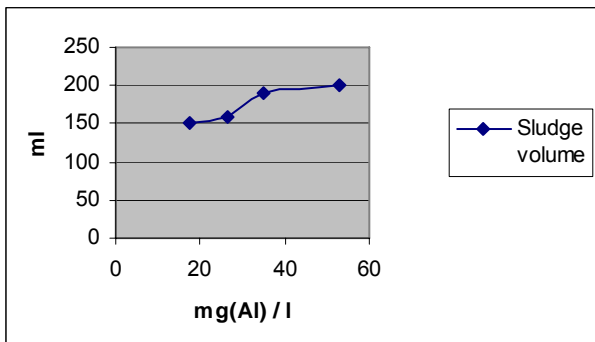
Figure 6.6.1.4: Concentration of suspended solids

The decrease in alkalinity can be calculated as follows:

$$\tan(\alpha) = -3/2.2 = 1.36$$

Compared to decrease in alkalinity when applying Fe salts in this case lower value is obtained. 1 mmol Al decreased the alkalinity by 1.36 mmol.

Addition of this chemical provided a good removal degree of suspended solids. After dosing 35.1 mg/l the amount of suspended solids in the non-filtrated supernatant was 24 mg/l and it was 11 mg/l after dosing 52.7 mg/l.



The volume of sludge that was formed in the first experiment was 150 ml and increased to 200 ml. The clarity of the supernatant after applying the highest Al dose was evident. (Figure 6.5.6)

Figure 6.6.1.5: Sludge volume

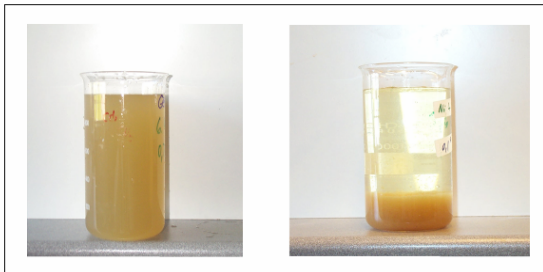


Figure 6.6.1.6: Black water before and after addition of 52.7 mg(Al) / l

6.6.2 AlCl₃ (Pluspac 1465) (Experiment 2)

The Al amounts that were added to the feed samples were: 0.98, 1.3 and 1.95 mmol/l. The PO₄ concentration in the filtrated feed sample was 0.45 mmol/l. The Al/PO₄ molar ratios were: 2.2, 2.9 and 4.3. Residual PO₄-P, total P and Al concentrations in the non-filtrated and filtrated supernatants are given in Figure 6.6.2.1 and Figure 6.6.2.2. The amounts of PO₄-P and total P in the non-filtrated supernatant for Al/PO₄ molar ratio of 2.2 declined to 6 and 8.1 mg/l corresponding to the following reductions: 57.1 and 59.5%. Achieving the Al/PO₄ molar ratio of 2.9 decreased these parameters to 1.5 and 3.4 mg/l and the reductions in the PO₄-P and total P amounts were 89.3 and 83%. The amounts of PO₄-P and total P declined by 99 and 95% when achieving the highest Al/PO₄ molar ratio of 4.3.

The lowest Al dose decreased the PO₄-P and total P concentrations in the filtrated supernatant to 5.3 and 13 mg/l and the reductions in these parameters were 62.1 and 23.5%. The residual PO₄-P and total P concentrations after applying the highest Al dose were 0.046 and 0.67 mg/l hence decreased by 99.7 and 96%.

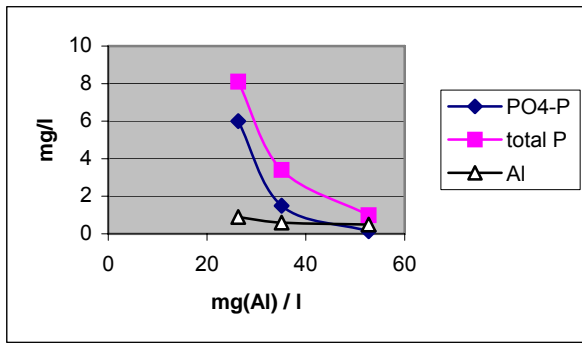


Figure 6.6.2.1: Concentrations of PO₄-P, total P and Al in a non-filtrated supernatant

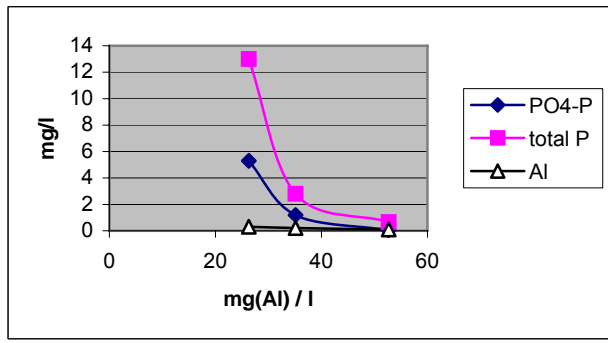


Figure 6.6.2.2: Concentrations of PO₄-P, total P and Al in a filtrated supernatant

Attaining the highest Al/PO₄ molar ratio of 4.3 decreased the carbonate and total alkalinity to 6.8 and 7.8 mmol/l. (Figure 6.6.2.3) In Table 15 the decrease in alkalinity in each of the experiments is presented:

Table 15: Decrease in alkalinity

mmol(Al)/l	0.98	1.3	1.95
Decrease in alkalinity mmol/mmol(Al)	0.61	0.77	1

In Figure 6.6.2.4 the decrease in the amount of suspended solids is shown. Before adding 0.98 and 1.3 mmol(Al)/l the concentration of suspended solids in the feed sample was 210 mg/l. After achieving the Al/PO₄ molar ratios of 2.2 and 2.9 the amounts of suspended solids decreased down to 43 and 41 mg/l. These concentrations represent around 20.4 and 19.5% of the initial concentrations. For the highest dose the reduction in the concentration of suspended solids was 89.2%.

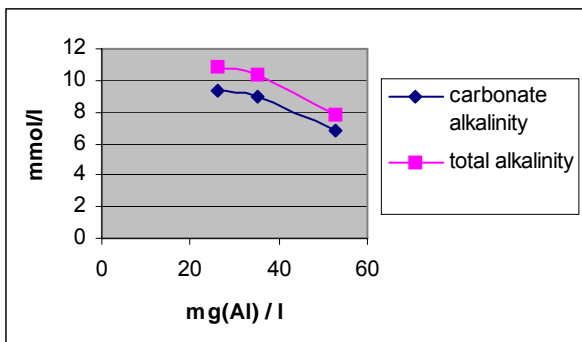


Figure 6.6.2.3: Carbonate and total alkalinity

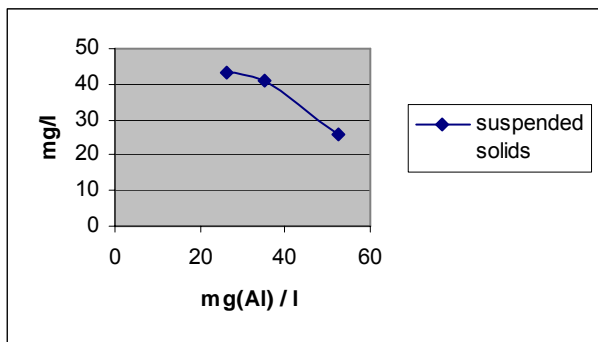
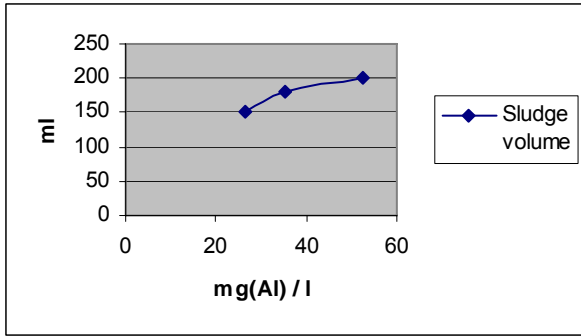


Figure 6.6.2.4: Concentration of suspended solids



The sludge volume obtained after dosing 26.3 mg(Al)/l was 150ml. Increasing the Al dose to 53.7 mg/l caused an increase in the sludge volume to 200ml. Considering the phosphate, aluminium concentrations and the amount of removed suspended solids SVI can be determined.

Figure 6.6.2.5: Sludge volume

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.48 \text{ mmol} - 0.001 \text{ mmol}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.479 \text{ mmol}$$

$$[\text{Al}]_{(\text{converted into precipitate})} = [\text{Al}]_{(\text{initial})} - [\text{Al}]_{(\text{after sedimentation})}$$

$$[\text{Al}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.004 \text{ mmol}$$

$$[\text{Al}]_{(\text{converted into precipitate})} = 1.946 \text{ mmol}$$

1 mol Al reacts with 1mol PO_4 and 1 mol AlPO_4 is produced. 0.479 mmol PO_4 produced 0.479 mmol AlPO_4 which corresponds to 58.4 mg AlPO_4 . 1.47 mmol Al produced 1.47 mmol $\text{Al}(\text{OH})_3$ and the mass of the precipitate was 114.7 mg.

$$m_{(\text{s.s. in sludge})} = m_{(\text{s.s. in feed})} - m_{(\text{s.s. in supernatant})}$$

$$m_{(\text{s.s. in sludge})} = 240 - 26 = 214 \text{ mg}$$

$$m_{(\text{TS in sludge})} = m(\text{FePO}_4) + m(\text{Fe}(\text{OH})_3) + m_{(\text{s.s. in sludge})}$$

$$m_{(\text{TS in sludge})} = 387.1 \text{ mg}$$

$$V_{\text{sludge}} = 200 \text{ ml}$$

$$\text{SVI} = 516.8 \text{ ml}(\text{sludge})/\text{g TS}$$

6.6.3 AlCl_3 (Pluspac 1465) and a polymer

The addition of the polymer Magnafloc 336 provided almost the same removal degree of phosphates as in the experiments conducted without polymer addition. In the non-filtrated supernatant for the Al/ PO_4 molar ratio of 2.2 the amount of PO_4 -P was 5.8 mg/l and it was 0.2 mg lower compared to the one without polymer addition (see Figure 6.6.3.1)

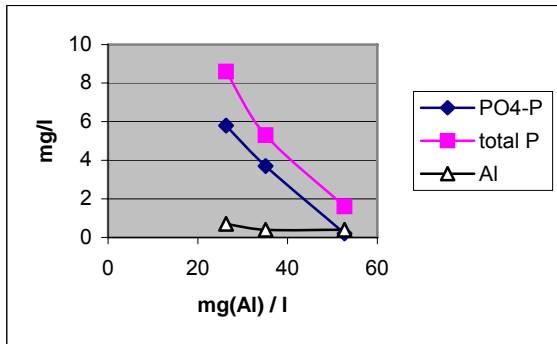


Figure 6.6.3.1: Concentrations of PO₄-P, total P and Al in a non-filtrated supernatant

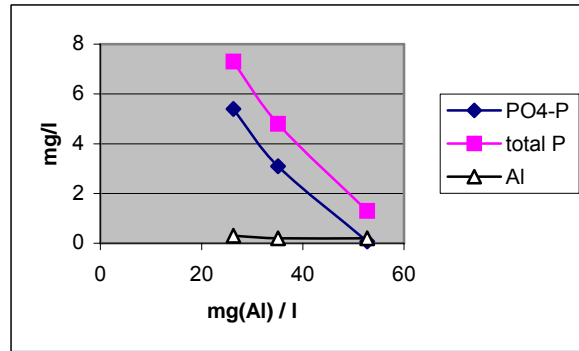


Figure 6.6.3.2: Concentrations of PO₄-P, total P and Al in a filtrated supernatant

Achieving the Al/PO₄ molar ratio of 4.3 caused a decrease in the amount of PO₄-P and this parameter had a value of 0.21 mg/l which is higher than in the experiment without a polymer. When the Al doses were 26.3 and 31.5 mg/l the Al concentrations in the non-filtrated supernatant were equal to those when the same Al doses were applied but without polymer addition.

In the filtrated supernatant the amounts of PO₄-P and total P were lower than those in the previous section. The polymer did not change the alkalinity of the supernatant and as can be seen in the Figure 6.6.3.3 almost identical values were obtained.

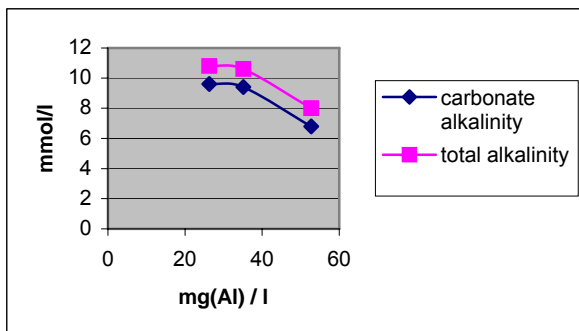


Figure 6.6.3.3: Carbonate and total alkalinity

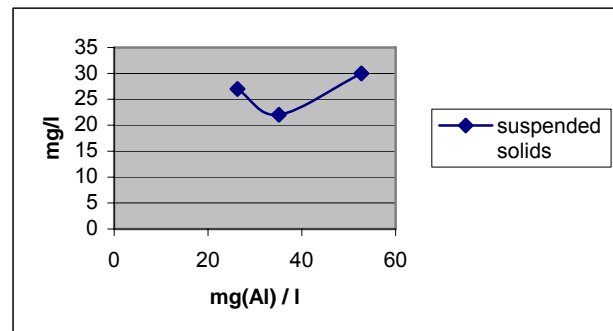


Figure 6.6.3.4: Concentration of suspended solids

The change in the concentration of suspended solids is given in Figure 6.6.3.4. Achieving the lowest Al/PO₄ molar ratio of 2.2 decreased the amount of suspended solids by 87%. The reduction was 87.5% when the Al/PO₄ molar ratio of 4.3 was applied.

It is characteristic for the experiments with polymer addition that the sludge volume is lower compared to the experiments conducted without a polymer. The highest sludge volume that was obtained was 170 ml.

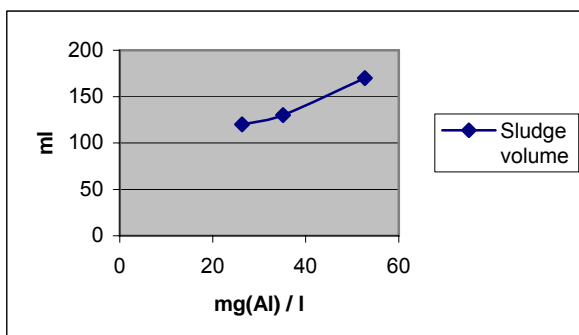


Figure 6.6.3.5: Sludge volume

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.48 \text{ mmol} - 0.002 \text{ mmol}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.478 \text{ mmol}$$

$$[\text{Al}]_{(\text{converted into precipitate})} = [\text{Al}]_{(\text{initial})} - [\text{Al}]_{(\text{after sedimentation})}$$

$$[\text{Al}]_{(\text{converted into precipitate})} = 1.95 \text{ mmol} - 0.007$$

$$[\text{Al}]_{(\text{converted into precipitate})} = 1.943 \text{ mmol}$$

0.478 mmol PO_4 reacted with 0.478 mmol Al and produced 0.478 mmol AlPO_4 which corresponds to 58.3 mg. 1.47 mmol Al were converted into 1.47 mmol $\text{Al}(\text{OH})_3$ and the mass of this precipitate was 114.7 mg.

$$m(\text{s.s. in sludge}) = m(\text{s.s. in feed}) - m(\text{s.s. in supernatant})$$

$$m(\text{s.s. in sludge}) = 240 - 30 = 210 \text{ mg}$$

$$m(\text{TS in sludge}) = m(\text{FePO}_4) + m(\text{Fe}(\text{OH})_3) + m(\text{s.s. in sludge})$$

$$m(\text{TS in sludge}) = 383 \text{ mg}$$

$$V_{\text{sludge}} = 170 \text{ ml}$$

$$\text{SVI} = 443.9 \text{ ml}(\text{sludge})/\text{g TS}$$

6.7 $\text{Ca}(\text{OH})_2$

In order to have a more representative sample, black water was collected in a tank during night. $\text{PO}_4\text{-P}$, total P and suspended solids were determined before and after adding $\text{Ca}(\text{OH})_2$ and this gave a good impression of the ability of this chemical to remove phosphorus and suspended solids. The amounts of $\text{Ca}(\text{OH})_2$ needed to reach different pH are given in Table 16.

Table 16: Dosed amounts of $\text{Ca}(\text{OH})_2$

Amount of $\text{Ca}(\text{OH})_2$ (mmol/l)	0.54	0.96	2	4	6.7	8.4	9.3
pH	7.7	8.3	8.96	9.45	10.1	10.61	11.4

The $\text{PO}_4\text{-P}$ and total P amounts in the feed were 10 and 13 mg/l. At pH of 7.7 the $\text{PO}_4\text{-P}$ concentration in the non-filtrated supernatant was lowered to 9.9 mg/l. Bigger decrease in the amounts of $\text{PO}_4\text{-P}$ and total P was noticed at pH=8.3. At this pH, $\text{PO}_4\text{-P}$ and total P concentrations were 8.2 mg/l and 11mg/l. Another sample of black water was used for continuation of the experiments. The sample was also collected during night and the concentrations of $\text{PO}_4\text{-P}$ and total P were 11 and 14 mg/l respectively. At pH=9 significant removal of P was noticed. The $\text{PO}_4\text{-P}$ concentration dropped down to 3.3 mg/l which was 30 % of the initial concentration. The best results were obtained at pH=9.45. This pH was reached by adding 4 mmol($\text{Ca}(\text{OH})_2$)/l and the concentration of $\text{PO}_4\text{-P}$ was 0.033mg/l corresponding to reduction of 99.7%.

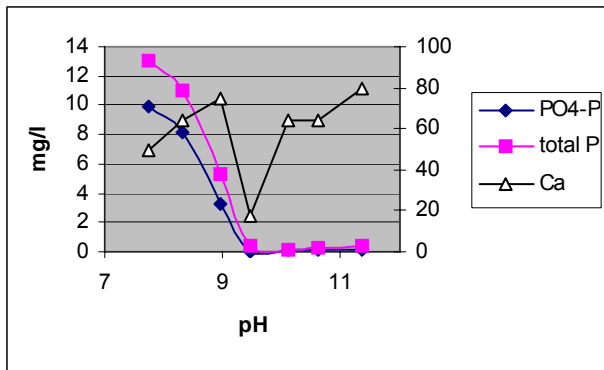


Figure 6.7.1: Concentrations of PO₄-P, total P and Ca depending on the pH in a non-filtrated sample

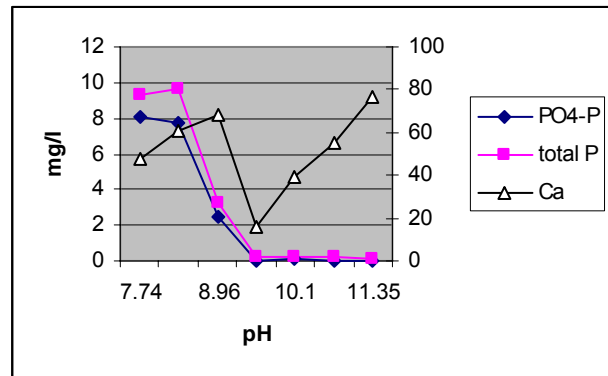


Figure 6.7.2: Concentration of PO₄-P, total P and Ca depending on the pH in a filtrated sample

The Ca concentration in the supernatants increased until reaching pH of 9 because very small amount of Ca was converted into precipitate. When the pH reached 9.5 the Ca concentration decreased due to removal of the Ca present in the feed and the Ca that was added. Further increase in the Ca(OH) dose increased the Ca concentration due to excess addition.

The amount of PO₄ in the filtrated feed sample before adjusting the pH to 7.74 and 8.33 was 0.25 mmol/l and it was 0.31 mmol/l before reaching the higher pH values. At pH=7.74 the Ca/PO₄ molar ratio was 2.1 and this ratio was 3.8 at pH=8.33. Small removal of phosphorus was noticed.

The Ca/PO₄ molar ratio applied to adjust the pH to 8.96 was 6.5. At this pH the amount of total P in the filtrated sample decreased to 3.3 mg/l which represents 25.4% of the initial concentration. The Ca/PO₄ molar ratio was 13 when reaching a pH of 9.5. In Figure 6.7.1 and 6.7.2 can be observed that the PO₄-P and total P concentrations at pH values higher than 9.5 had low values. The amounts of these parameters declined approximately by 98 and 97%

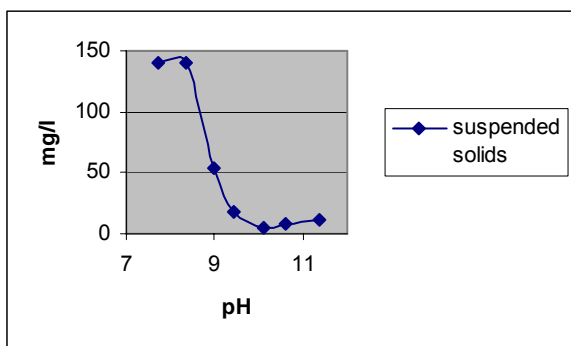


Figure 6.7.3: Concentration of suspended solids depending on the pH

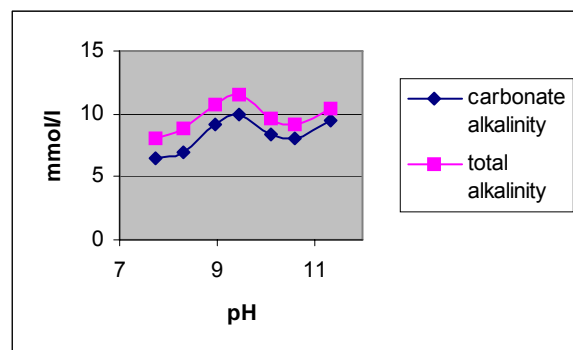
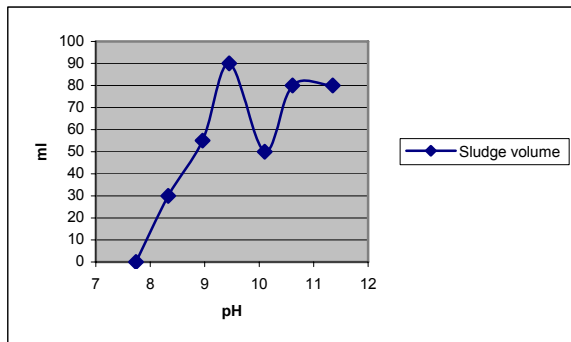


Figure 6.7.4: Carbonate and total alkalinity depending on the pH

The values for carbonate and total alkalinity reached the peak at pH=9.45 (10 and 11.6 mmol/l respectively). This was unexpected because at this pH the lowest Ca concentrations in the supernatant were measured and a decrease in alkalinity due to conversion of bicarbonate and carbonate ions into CaCO₃ is expected.

Addition of Ca(OH)₂ provided low amounts of suspended solids in the supernatants at pH values higher than 9.5. The lowest value was registered at pH=10.1 and the reduction in the amount of suspended solids was 96.6%.

The precipitates started to form at pH of 8.3 and the sludge volume reached the peak at pH=9.45. (Figure 6.7.5)



The highest value for the sludge volume was detected at pH=9.45 (90 ml) when dosing 4 mmol(Ca(OH)₂)/l. A visual comparison between the feed sample and treated black water is shown in Figure 6.7.6.

Figure 6.7.5: Sludge volume depending on the pH

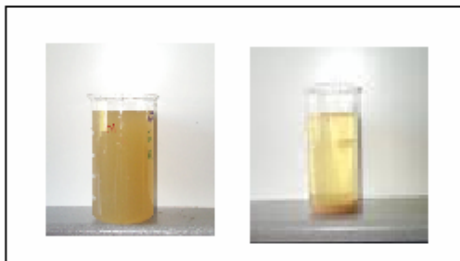
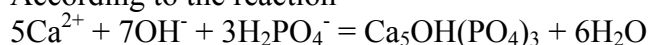


Figure 6.7.6: Feed sample and a black water treated with Ca(OH)₂, pH=9.45

The Ca in the feed was not measured but according to previous measurements it was around 30 mg/l and this value is used in the calculations. The SVI at pH of 9.5 and 10.1 are calculated below:

1. pH=9.5

According to the reaction



1 mol Ca reacts with 0.6 moles PO₄ to produce 0.2 moles hydroxiapatite

$$\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.3 - 0.00061$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.299 \text{ mmol}$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = [\text{Ca}]_{(\text{feed})} + [\text{Ca}]_{(\text{added})} - [\text{Ca}]_{(\text{after sedimentation})}$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = 0.75 + 4 - 0.4$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = 4.35 \text{ mmol}$$

0.3 mmol PO_4 reacted with 0.5 mmol Ca and produced 0.1 mmol $\text{Ca}_5\text{OH}(\text{PO}_4)_3$ which corresponds to 50.2 mg. 3.85 mmol Ca were converted into 3.85 mmol CaCO_3 and the mass of this precipitate was 385 mg.

$$m(\text{s.s. in sludge}) = m(\text{s.s. in feed}) - m(\text{s.s. in supernatant})$$

$$m(\text{s.s. in sludge}) = 150 - 18 = 132 \text{ mg}$$

$$m(\text{TS in sludge}) = m(\text{Ca}_5\text{OH}(\text{PO}_4)_3) + m(\text{CaCO}_3) + m(\text{s.s. in sludge})$$

$$m(\text{TS in sludge}) = 567.2 \text{ mg}$$

$$V_{\text{sludge}} = 90 \text{ ml}$$

$$\text{SVI} = 157.9 \text{ ml}(\text{sludge})/\text{g TS}$$

2. pH=10.1

$$[\text{PO}_4]_{(\text{converted into precipitate})} = [\text{PO}_4]_{(\text{initial})} - [\text{PO}_4]_{(\text{after sedimentation})}$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.3 - 0.005$$

$$[\text{PO}_4]_{(\text{converted into precipitate})} = 0.295 \text{ mmol}$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = [\text{Ca}]_{(\text{feed})} + [\text{Ca}]_{(\text{added})} - [\text{Ca}]_{(\text{after sedimentation})}$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = 0.75 + 6.7 - 1$$

$$[\text{Ca}]_{(\text{converted into precipitate})} = 6.45 \text{ mmol}$$

0.3 mmol PO_4 reacted with 0.5 mmol Ca and produced 0.1 mmol $\text{Ca}_5\text{OH}(\text{PO}_4)_3$ which corresponds to 50.2 mg. 5.95 mmol Ca were converted into 5.95 mmol CaCO_3 and the mass of this precipitate was 595 mg.

$$m(\text{s.s. in sludge}) = m(\text{s.s. in feed}) - m(\text{s.s. in supernatant})$$

$$m(\text{s.s. in sludge}) = 150 - 5.1 = 144.9 \text{ mg}$$

$$m(\text{TS in sludge}) = m(\text{Ca}_5\text{OH}(\text{PO}_4)_3) + m(\text{CaCO}_3) + m(\text{s.s. in sludge})$$

$$m(\text{TS in sludge}) = 790.1 \text{ mg}$$

$$V_{\text{sludge}} = 50 \text{ ml}$$

$$\text{SVI} = 63.3 \text{ ml}(\text{sludge})/\text{g TS}$$

The SVI at pH=10.1 is around 2 times lower compared to the value at pH=9.5 because the sludge volume that was obtained at the higher pH was approximately 2 times lower.

7. Tank experiments

The tank experiments were performed in order to realize the application of FeCl₃ (PIX 111) on a larger scale. A sedimentation tank with a capacity of 270 l was used. The black water entered the sedimentation tank after passing the drum screen. Because the composition of the feed differs during the day 11 of feed from the tank was taken and it was titrated with FeCl₃ (PIX 111) down to pH between 5.5 and 6. By doing this the risk of decreasing the pH below 5 or adding an Fe dose not sufficient to achieve good removal of phosphate and suspended solids was avoided.

It was not possible to programme the mixer running in 2 different modes and only fast mixing was applied. The following parameters were used for all the experiments:

- mixing speed: 1500rpm
- mixing time: 10 min
- sedimentation time: 1h

The VSEP was run in this regime for one month. 47 experiments were conducted and the mean value of FeCl₃ amounts that were added to the feed samples is 0.69 ml/l. Samples from the feed, non-filtrated supernatant and sludge were taken after performing one of the experiments. The feed volume was 200l and 60l of sludge were produced.

Many parameters were determined in the samples and the results are presented in Table 17.

Table 17: Parameters in the feed, non-filtrated supernatant and sludge

Parameter	Feed	Non-filtrated supernatant	Sludge
Sulphur, S (mg/l)	16	22	38
pH	6.6	5.5	5.4
Alkalinity, HCO ₃ (mg/l)	420	22	34
Conductivity			
COD (Cr) (mg/l)	4300	690	13000
TOC (mg/l)	980	240	2900
BOD ₇ (mg/l)	2000	400	4300
Nitrogen, N (mg/l)		100	970
PO ₄ -P (mg/l)	28	0.063	0.91 ?
Total P (mg/l)	48	0.22	110
NH ₄ -N (mg/l)	140		
Sulphate, SO ₄ (mg/l)	<10	< 10	
Suspended solids (mg/l)	1800	11	8200
Aluminium, Al (µg/l)	1100	53	2400
Potassium, K (mg/l)	54		
Iron, Fe (mg/l)	1.3	7.5	580
Calcium, Ca (mg/l)	63	57	93
Fluoride, F (mg/l)	<12	< 12	
Barium, Ba (µg/l)	250	26	170 ?
Silicon, Si (mg/l)	4.1	1.7	5
Strontium, Sr(µg/l)	100	110	150

The amount of a substance in the feed is a sum of the amounts of the substance in the supernatant and in the sludge.

$$M_{\text{feed}} = M_{\text{sludge}} + M_{\text{supernatant}}$$
$$C_{\text{feed}} * V_{\text{feed}} = C_{\text{sludge}} * V_{\text{sludge}} + C_{\text{supernatant}} * V_{\text{supernatant}}$$

Where

C – concentration (mg/l)

V – volume (l)

The results which are not reliable with respect to this mass balance are marked (?).

The Fe dose that was added to the feed sample was 4.2 mmol/l. The PO₄ amount in the feed sample was 0.9 mmol hence the Fe/PO₄ molar ratio achieved in the experiment was 4.7.

The PO₄-P and total P concentrations in this feed were higher than those noticed while conducting the beaker experiments. Very high concentration of suspended solids in the feed was measured – 1800 mg/l.

The total P concentration in the non-filtrated supernatant was 0.22 mg/l corresponding to 99.6% reduction. Achieving the Fe/PO₄ molar ratio of 4.7 lowered the PO₄-P concentration to 0.063 mg/l which is 99.8% reduction. Good removal of COD and BOD₇ was achieved in this experiment. COD was lowered by 84% and BOD₇ by 80%. After sedimentation lower concentrations of Ba and Si were measured in the supernatant.

Turbidity was measured in 19 supernatant samples and values between 5 and 64 FAU (formazine attenuation units) were obtained.

Treating the black water in the tank showed that same removal degrees of phosphates and supernatant clarity were achieved as in the beaker experiments.

8. Discussion

In order to get a better understanding of the results the Chemical Equilibrium Modelling System (MINEQL+) was used to calculate the Saturation Indices (SI) for different solids. The saturation index is calculated as follows:

$$SI = \log (Q/K_{sp})$$

where Q – ion product for the solid

K_{sp} – solubility constant for the solid

The saturation indices can be obtained if the programme does not consider the formation of the solids. When calculating the SI values for iron containing solids the input parameters were: Fe, PO₄³⁻, H⁺ and H₂O. The input parameters that were used for determination of the SI for aluminium containing solids included Al, PO₄³⁻, H⁺, H₂O and for calcium containing solids the programme considered Ca, CO₃²⁻, PO₄³⁻, H⁺ and H₂O. A simplified model was used to calculate the saturation indices. The SI gives an image for the state of a system and the following states are possible: undersaturated (SI < 0), equilibrium (SI = 0) and oversaturated (SI > 0). The input parameters for Fe, Al, Ca and PO₄³⁻ that were used in the programme were those measured in the filtrated supernatants.

In this Chapter comparison among the obtained results is made and the values for SI are discussed.

8.1 Experiment 1

The residual PO₄-P concentrations in the non-filtrated and filtrated supernatants after addition of different Fe amounts are presented in Table 18.

Table 18: PO₄-P concentrations in the non-filtrated and filtrated supernatants (mg/l)

	mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4
FeCl₃ (PIX 111)	non-filtrated	8.3	5.6	0.63	0.88	0.24	< 0.01
	filtrated	5.3	1.1	0.74	0.83	0.18	< 0.01
Fe₂(SO₄)₃	non-filtrated	9	8	5.2	2.5	1.2	0.015
	filtrated	6.3	7.3	1.1	1.3	0.16	0.027
FeCl(SO₄)	non-filtrated	8.6	17	15	11	8.3	0.24
	filtrated	4.5	10	9.4	3.5	1.3	0.084
FeCl₃ (ciba)	non-filtrated			6.5	3.3	2.2	2.7
	filtrated			3.3	1.1	0.65	0.31

Except in the experiments with FeCl(SO₄) and FeCl₃ (ciba) the residual PO₄-P concentrations in the non-filtrated and filtrated supernatants decreased as the Fe dose increased. The Fe dose of 17.9 mg/l decreased the PO₄-P concentrations around 9 mg/l in the non-filtrated supernatants. In the filtrated supernatants the PO₄-P concentrations ranged between 4.5 and 6.3 mg/l. The pH values of the black water samples during the flocculation ranged between 6.6 and 6.9.

The Fe dose of 26.9 mg/l when using FeCl₃ (PIX 111) and Fe₂(SO₄)₃ decreased the PO₄-P amounts in both non-filtrated and filtrated supernatants. An increase in the PO₄-P amount was noticed when treating the black water with FeCl(SO₄). This resulted from much higher

orthophosphate concentration in the feed sample and the other reason is that the experiment was conducted at pH=7.8 which is above the optimal pH value for formation of FePO₄.

Increasing the Fe dose to 35.8 mg/l led to low residual concentrations when using FeCl₃ (PIX 111) - below 1 mg/l. Treating the black water with Fe₂(SO₄)₃ and FeCl₃ (ciba) provided residual amounts between 5.2 and 6.5 mg/l. High residual amounts were noticed when adding FeCl(SO₄).

After adding 71.6 mg(Fe)/l when using FeCl₃ (PIX 111), Fe₂(SO₄)₃ and FeCl₃ (ciba) the pH values of the black water during the flocculation ranged between 6.1 and 6.4. In the experiment with FeCl(SO₄) the pH of the black water was higher – 6.9.

The Fe dose of 107.4 mg/l caused very low residual PO₄-P amounts when using any of the salts. The lowest concentrations were measured after addition of FeCl₃. The experiments when applying the highest Fe dose were conducted at pH values between 5.7 and 6.1.

The total P concentrations in the non-filtrated and filtrated supernatants are presented in Table 19. Small removals of total P were achieved when adding 17.9 mg(Fe)/l. The addition of this amount when using FeCl₃, Fe₂(SO₄)₃ and FeCl(SO₄) caused total P amounts between 12 and 14 mg/l in the non-filtrated supernatants. The total P concentrations in the filtrated supernatants ranged between 8.3 and 9.7 mg/l.

Table 19: Total P concentrations in the non-filtrated and filtrated supernatants (mg/l)

	mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4
FeCl₃ (PIX 111)	non-filtrated	12	8.3	5.7	3.4	1.9	0.22
	filtrated	8.3	5	3.6	1.5	0.55	0.07
Fe₂(SO₄)₃	non-filtrated	14	11	8.7	5.1	3.5	0.69
	filtrated	9.7	7.9	5.2	2.6	0.7	0.15
FeCl(SO₄)	non-filtrated	12	26	27	17	14	0.49
	filtrated	8.7	16	15	8.6	5.4	0.17
FeCl₃ (ciba)	non-filtrated			9.1	5.2	3.2	3.1
	filtrated			4.8	2	0.84	0.42

A decrease in the total P concentrations was registered after adding 26.9 mg(Fe)/l with FeCl₃ and Fe₂(SO₄)₃. Very high total P amount (26 mg/l) was registered when applying the same Fe dose with FeCl(SO₄) due to a high phosphorus concentration in the feed.

Gradual decrease in the total P concentrations was detected with increasing the Fe dose with each of the salts. Addition of the highest Fe dose when using FeCl₃ (PIX 111), Fe₂(SO₄)₃ and FeCl(SO₄) decreased the total P amounts below 0.7 mg/l in the non-filtrated supernatants and in the experiment with FeCl₃ (ciba) this value was higher – 3.1 mg/l. In the filtrated supernatants the total P concentrations ranged between 0.1 and 0.4 mg/l.

The Fe concentrations in the non-filtrated supernatants ranged between 12 and 40 mg/l and in the filtrated supernatants between 2.5 and 24 mg/l. (see Table 20)

Table 20: Fe concentrations in the non-filtrated and filtrated supernatants (mg/l)

	mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4
FeCl₃ (PIX 111)	non-filtrated	13	13	12	16	15	14
	filtrated	3.1	4.1	4.3	8.4	9	9.4
Fe₂(SO₄)₃	non-filtrated	14	16	17	21	21	11
	filtrated	6.9	7.2	7.6	12	7.2	6.4
FeCl(SO₄)	non-filtrated	16	22	21	30	30	5.4
	filtrated	11	9.4	10	9.7	7.8	2.5
FeCl₃ (ciba)	non-filtrated			24	20		40
	filtrated			13	7.2	4.9	24

The Fe concentrations in the non-filtrated and filtrated supernatants increased with increasing the Fe dose and then decreased. In the experiment with FeCl₃ (ciba) a big increase in the Fe concentration was noticed when the highest Fe dose was applied. Increasing the Fe dose with FeCl₃ (PIX 111) and FeCl₃ (ciba) caused an increase in the Fe concentrations in the filtrated supernatant. The high Fe amounts probably resulted from formation of complexes among Fe and dissolved compounds in the black water.

In Table 21 the SI for FePO₄ · 2H₂O and Fe(OH)₃ (using the simplified model that was described) are presented. The SI for FePO₄ · 2H₂O had lower values at lower pH and the systems were closer to equilibrium states. In the pH interval between 5.8 and 6.2 after applying the highest Fe dose with FeCl₃ (PIX 111), Fe₂(SO₄)₃ and FeCl(SO₄) the SI had values around 1 which is 10 times higher than the solubility product. The lowest residual phosphate concentration with FeCl₃ (ciba) was achieved at pH=5.67. At this pH the saturation index was 2.84 which resulted from higher Fe and PO₄ concentrations compared to other experiments.

The SI for Fe(OH)₃ indicate that if considering only the parameters that were used by the programme the systems would be oversaturated with respect to Fe(OH)₃. Values between 3 and 5 were calculated. This indicates that Fe was bound to dissolved compounds in the black water.

Table 21: SI for FePO₄ · 2H₂O and Fe(OH)₃

	mg(Fe)/l	17.9	26.9	35.8	53.7	71.6	107.4
	pH	6.6	6.5	6.4	6.32	6.13	5.78
FeCl₃ (PIX 111)	FePO ₄ · 2H ₂ O	3.05	2.54	2.41	2.75	2.15	0.95
	Fe(OH) ₃	3.49	3.55	3.46	3.63	3.53	3.22
	pH	6.65	6.54	6.45	6.6	6.29	5.86
Fe₂(SO₄)₃	FePO ₄ · 2H ₂ O	3.52	3.63	2.86	2.97	1.96	1.22
	Fe(OH) ₃	4.02	3.92	3.89	3.85	3.51	3.11
	pH	6.96	7.83	7.55	7.22	6.9	6.15
FeCl(SO₄)	FePO ₄ · 2H ₂ O	3.44	2.89	3.18	3.03	2.70	1.23
	Fe(OH) ₃	4.43	5.04	4.87	4.61	4.23	2.90
	pH			6.94	6.65	6.42	5.67
FeCl₃ (ciba)	FePO ₄ · 2H ₂ O			3.31	2.70	2.36	2.84
	Fe(OH) ₃			4.42	3.88	3.50	3.48

The experiments with Al salts were carried out at higher pH values compared to those with Fe salts. The results show that there was a gradual decrease in the PO₄-P and total P concentrations when both Al salts were used. (see Table 22)

Table 22: PO₄-P and total P concentrations in the non-filtrated and filtrated supernatants

	mg(Al)/l	17.6	26.3	35.1	52.7	
AlCl ₃ (Pluspac 1465)	non-filtrated	6.2	3.8	1.8	0.11	PO ₄ -P (mg/l)
	filtrated	4.7	2.6	0.92	0.092	
AlCl ₃ (Pluspac 1800)	non-filtrated	10	9.4	3.8	1.8	
	filtrated	8.9	7.7	2.4	0.87	
AlCl ₃ (Pluspac 1465)	non-filtrated	7.8	4.9	1.7	0.41	Total P (mg/l)
	filtrated	4.8	2.6	0.93	0.17	
AlCl ₃ (Pluspac 1800)	non-filtrated	14	12	6.1	2.9	
	filtrated	12	11	4	1.7	

AlCl₃ (Pluspac 1465) provided lower residual PO₄-P and total P concentrations. Applying the highest Al dose when using Pluspac 1465 decreased the pH to 6.2 and after applying the same amount with Pluspac 1800 the pH was 6.9.

The optimal precipitation region for aluminium phosphate is between pH of 6 and 7 and this was one of the reasons why AlCl₃ (Pluspac 1800) provided higher residual phosphorus concentrations than AlCl₃ (Pluspac 1465). The other reason was more concentrated feed sample that was used in the experiments with AlCl₃ (Pluspac 1800).

The Al concentrations in the non-filtrated and filtrated supernatants (see Table 23) decreased with increasing the Al dose.

Table 23: Al concentrations in the non-filtrated and filtrated supernatants

	mg(Al)/l	17.6	26.3	35.1	52.7	
AlCl ₃ (Pluspac 1465)	non-filtrated	4.6	4.5	3	1	Al (mg/l)
	filtrated	1.4	1.9	0.4	0.096	
AlCl ₃ (Pluspac 1800)	non-filtrated	6.6	5.6	5.1	2.3	
	filtrated	3.3	2.5	2	0.78	

The Al concentrations in the non-filtrated supernatants ranged between 1 and 6.6 mg/l. In the filtrated supernatants values between 0.096 and 3.3 mg/l were measured. Probably Al does not form complexes to such an extent as Fe which explains lower Al concentrations.

The SI for AlPO₄ when using AlCl₃ (Pluspac 1465) had lower values at lower pH which means that the systems were closer to the equilibrium states. In the experiments with AlCl₃ (Pluspac 1800) almost no change in the SI for AlPO₄ was noticed. The SI for Al(OH)₃ had values between 2.1 and 3.3 and the systems were oversaturated. (Table 24)

Table 24: SI for AlPO₄ and Al(OH)₃

	mg(Al)/l	17.6	26.3	35.1	52.7
	pH	6.44	6.37	6.35	6.23
AlCl ₃ (Pluspac 1465)	AlPO ₄	4.27	4.22	3.10	1.57
	Al(OH) ₃	3.32	3.44	2.77	2.11
	pH	7.76	7.55	7.16	6.94
AlCl ₃ (Pluspac 1800)	AlPO ₄	2.13	2.46	2.80	2.40
	Al(OH) ₃	2.86	2.92	3.14	2.89

8.2 Experiment 2

The results from the experiments when determining the Fe/PO₄ and Al/PO₄ molar ratios are presented in the following tables. The removal degrees of PO₄-P (expressed in %) after achieving different Fe/PO₄ and Al/PO₄ molar ratios are given in Table 25.

Table 25: Removal degree of PO₄-P and PO₄-P concentrations

	mg(Fe)/l	Removal degree (%)			PO ₄ -P (mg/l)		
		53.7	71.6	107.4	53.7	71.6	107.4
	Fe/PO₄ molar ratio	2.8	3.7	4.6	2.8	3.7	4.6
FeCl₃ (PIX 111)	non-filtrated	75	91.7	99	3	1	0.14
	filtrated	90	97.8	99.6	1.1	0.24	0.056
FeCl₃ (PIX 111) and a polymer	non-filtrated	84.2	88.3	97.1	1.9	1.4	0.4
	filtrated	91.7	93.8	99	0.91	0.68	0.13
	Fe/PO₄ molar ratio	2.8	3.7	4.3	2.8	3.7	4.3
FeCl₃ (ciba)	non-filtrated	72.7	96.2	99.2	3	0.42	0.11
	filtrated	-	-	99.5	0.87	0.2	0.065
FeCl₃ (ciba) and a polymer	non-filtrated	82.7	-	98.2	1.9	-	0.25
	filtrated	-	-	99.6	0.6	0.26	0.056
	Al/PO₄ molar ratio	2.2	2.9	4.3	2.2	2.9	4.3
AlCl₃ (Pluspac 1456)	non-filtrated	57.1	89.3	99	6	1.5	0.14
	filtrated	62.1	91.4	99.7	5.3	1.2	0.046
AlCl₃ (Pluspac 1456) and a polymer	non-filtrated	58.6	73.6	98.5	5.8	3.7	0.21
	filtrated	61.4	77.9	99.5	5.4	3.1	0.073

The lowest Fe/PO₄ molar ratios that were achieved with FeCl₃ (PIX 111) and FeCl₃ (ciba) had the same values and the removal degrees in the non-filtrated and filtrated supernatants had close values. The Fe/PO₄ molar ratio of 2.8 when using FeCl₃ (PIX 111) and FeCl₃ (ciba) provided removal degrees of total P higher than 70%. The removal degree that was obtained when achieving the Al/PO₄ molar ratio of 2.2 was around 60% but this ratio was lower compared to the ratios achieved with the Fe salts. The highest molar ratios produced low residual phosphorus concentrations and removed 95% of the total phosphorus. (see Table 26)

The use of the anionic polymer Magnafloc did not have a big influence on the removal degrees as can be noticed in the Tables.

Comparing these results with those in Tables 18 and 19 when applying the same Fe doses with FeCl₃ (PIX 111) and FeCl₃ (ciba) shows that similar values were obtained. The difference in the values resulted from using different feed samples in the experiments.

Table 26: Removal degree of total P and residual total P concentrations

		Removal degree (%)			P concentration (mg/l)		
		2.8	3.7	4.6	2.8	3.7	4.6
	Fe/PO₄ molar ratio						
FeCl₃ (PIX 111)	non-filtrated	76.3	91.3	97	3.8	1.4	0.5
	filtrated	89.2	97.4	98.9	1.3	0.31	0.15
FeCl₃ (PIX 111) and a polymer	non-filtrated	86.3	91.3	96	2.2	1.4	0.67
	filtrated	92.4	93.8	98.5	0.91	0.75	0.21
	Fe/PO₄ molar ratio	2.8	3.7	4.3	2.8	3.7	4.3
FeCl₃ (ciba)	non-filtrated	69.3	81.3	95.6	4.9	3	0.89
	filtrated	-	-	98.4	2.2	0.66	0.27
FeCl₃ (ciba) and a polymer	non-filtrated	76.3	-	95.5	3.8	-	0.9
	filtrated	-	-	98.1	1.4	0.6	0.32
	Al/PO₄ molar ratio	2.2	2.9	4.3	2.2	2.9	4.3
AlCl₃ (Pluspac 1465)	non-filtrated	59.5	83	95	8.1	3.4	0.99
	filtrated	-	83.5	95.8	-	2.8	0.67
AlCl₃ (Pluspac 1465) and a polymer	non-filtrated	57	73.5	92	8.6	5.3	1.6
	filtrated	57	71.8	91.9	7.3	4.8	1.3

Table 27: SVI for different Fe/PO₄ and Al/PO₄ molar ratios

Fe/PO₄ = 4.6		
FeCl₃ (PIX 111)	305	SVI (ml (sludge)/g TS)
FeCl₃ (PIX 111) and a polymer	220.3	
Fe/PO₄ = 4.3		
FeCl₃ (ciba)	500	
FeCl₃ (ciba) and a polymer	362.3	
Al/PO₄ = 4.3		
AlCl₃ (Pluspac 1465)	516.8	
AlCl₃ (Pluspac 1465) and a polymer	443.9	
Ca(OH)₂ pH=9.5	157.9	
Ca(OH)₂ pH=10.1	68.5	

In Table 27 SVI values for the highest Fe/PO₄ and Al/PO₄ molar ratios are presented for the different chemicals tested. The sludge volumes that were obtained in the experiments with the polymer were lower and due to this lower SVI were calculated.

The use of Ca(OH)₂ provided lower sludge volumes and this was the reason for significantly lower SVI.

The Fe concentrations in the filtrated supernatants had values between 4.7 and 17 mg/l on the other hand the Al concentrations were below 0.9 mg/l. (see Table 28)

Table 28: Fe and Al concentrations in the non-filtrated and filtrated supernatants

	Fe/PO₄ molar ratio	2.8	3.7	4.6		
FeCl₃ (PIX 111)	non-filtrated	17	10	7.2	Fe (mg/l)	
	filtrated	5.2	3.8	4.5		
FeCl₃ (PIX 111) and polymer	non-filtrated	12	10	7.8		
	filtrated	5.5	5.7	5.4		
	Fe/PO₄ molar ratio	2.8	3.7	4.3		
FeCl₃ (ciba)	non-filtrated	19	15	4.7		
	filtrated	7.2	3.2	1.1		
FeCl₃ (ciba) and a polymer	non-filtrated	12		4.8		
	filtrated	3.5	2.8	1.3		
	Al/PO₄ molar ratio	2.2	2.9	4.3		
AlCl₃ (Pluspac 1465)	non-filtrated	0.9	0.6	0.5		Al (mg/l)
	filtrated	0.3	0.2	<0.1		
AlCl₃ (Pluspac 1465) and a polymer	non-filtrated	0.7	0.4	0.4		
	filtrated	0.3	0.2	0.2		

The Fe concentrations presented had lower values compared to those when using the same chemical in the previous experiments (see Table 20). As shown in the Table a gradual decrease in the Fe concentrations was registered and this was not characteristic for the first series of experiments. This leads to the conclusion that the residual Fe amounts depend on the composition of the black water and formation of complexes between Fe and dissolved compounds.

The Al concentrations measured in these experiments were much lower compared to those presented in the previous Chapter. Feed composition highly affects the Al concentration in the supernatants.

The SI for FePO₄ had lower values at lower pH and the systems were closer to equilibrium. There was no big difference in the SI for Fe(OH)₃ and according to the obtained values the ion product of the solid was approximately 1000 times higher than the solubility product. The SI for Fe(OH)₃ ranged between 2.9 and 3.66 and these values were close to those presented in the previous section. (Table 29)

Table 29: SI for FePO₄ · 2H₂O and Fe(OH)₃

	mg(Fe)/l	53.7	71.6	107.4
	pH	6.49	6.31	6.06
FeCl₃ (PIX 111)	FePO ₄ · 2H ₂ O	2.51	1.75	1.26
	Fe(OH) ₃	3.27	2.95	2.90
	pH	6.38	6.34	6.04
FeCl₃ (PIX 111) and a polymer	FePO ₄ · 2H ₂ O	2.48	2.35	1.71
	Fe(OH) ₃	3.20	3.12	2.98
	pH	6.8	6.56	6.58
FeCl₃ (ciba)	FePO ₄ · 2H ₂ O	2.55	1.67	0.66
	Fe(OH) ₃	3.96	3.43	2.94
	pH	6.79	6.58	6.59
FeCl₃ (ciba) and a polymer	FePO ₄ · 2H ₂ O	2.08	1.71	0.68
	Fe(OH) ₃	3.66	3.41	3.04

As shown in Table 30 values close to 0 for SI for AlPO₄ were obtained in the three experiments with AlCl₃ (Pluspac 1465) which means that the systems were slightly undersaturated and precipitation of AlPO₄ principally can not occur. The SI for Al(OH)₃ were lower compared to Experiment 1 and the systems were closer to equilibrium. In these experiments the SI ranged between 1.3 and 2.

Table 30: SI for AlPO₄ and Al(OH)₃

	mg(Al)/l	26.3	35.1	52.7
	pH	8.24	8	7.34
AlCl₃ (Pluspac 1465)	AlPO ₄	-0.47	-0.71	-0.35
	Al(OH) ₃	1.38	1.39	1.81
	pH	8.25	8.05	7.39
AlCl₃ (Pluspac 1465) and a polymer	AlPO ₄	-0.54	-0.30	-0.02
	Al(OH) ₃	1.35	1.39	2.05

For the Ca(OH)₂ experiments, the PO₄-P, total P, Ca concentrations and removal degrees after reaching different pH values are presented in Table 31.

Table 31: Removal degrees and concentrations

	mmol(Ca(OH)₂)/l	0.54	0.96	2	4	6.7	8.4	9.3
	pH	7.74	8.33	8.96	9.45	10.1	10.61	11.35
Removal degree of PO₄-P (%)	non-filtrated	1	18	70	99.7	98.2	98.8	99
	filtrated	-	2.5	74.2	99.8	98.4	99.4	99.8
Removal degree of P (%)	non-filtrated	-	15.4	62.1	97.1	98.7	98.1	97.5
	filtrated	-	26.2	74.6	98.3	98	98.7	99.2
PO₄-P (mg/l)	non-filtrated	9.9	8.2	3.3	0.033	0.2	0.13	0.11
	filtrated	8.1	7.7	2.5	0.019	0.16	0.056	0.017
Total P(mg/l)	non-filtrated	13	11	5.3	0.4	0.18	0.26	0.35
	filtrated	9.3	9.6	3.3	0.22	0.26	0.17	0.11
Ca (mg/l)	non-filtrated	50	64	75	17	64	64	80
	filtrated	48	61	68	16	39	55	77

Removal degrees of phosphates were around 70% when increasing the pH to 9. Above this pH the removal degrees of PO₄-P and total P were above 98%.

The Ca concentrations in the non-filtrated supernatants had values between 50 and 80 mg/l and in the filtrated supernatants slightly lower amounts were measured. (48-77 mg/l).

At pH=9.45 the system was undersaturated with respect to Ca₃(PO₄)₂ and the saturation index for apatite had the lowest value. Very high SI for apatite (above 10) were calculated at the other pH values. The high SI values even in this case indicate that Ca was bound by dissolved compounds which were not taken into consideration in the equilibrium program. Gradual increase in the SI for calcite can be noticed. (see Table 32)

Table 32: SI for Ca₃(PO₄)₂, Ca₅OH(PO₄)₃ and CaCO₃

	pH	7.74	8.33	9.45	10.1	10.6	11.3
Ca(OH)₂	Ca ₃ (PO ₄) ₂	2.32	3.73	-1.39	2.16	1.34	0.41
	Apatite	10.06	12.83	5.98	12.24	11.49	10.87
	Calcite	0.29	1.00	1.47	2.25	2.28	2.36

9. Conclusions and recommendations

FeCl_3 (PIX 111), $\text{Fe}_2(\text{SO}_4)_3$, $\text{FeCl}(\text{SO}_4)$, FeCl_3 (ciba), AlCl_3 (Pluspac 1465), AlCl_3 (Pluspac 1800) and $\text{Ca}(\text{OH})_2$ have proved to be efficient in the phosphorus recovery from the black water.

The following conclusions were drawn when using the Fe salts:

- The total P concentration can be reduced below 0.9 mg/l and the $\text{PO}_4\text{-P}$ concentration declined below 0.4 mg/l. The removal efficiency was at least 96%.
- The lowest $\text{PO}_4\text{-P}$ and total P residual values were obtained after adding 107 mg(Fe) at pH values between 5.5 and 6 and those were <0.01 and 0.22 mg/l.
- An Fe/ PO_4 molar ratio of 4.6 was attained when applying 107.4 mg(Fe)/l
- Depending on the feed composition the Fe concentrations in the filtrated supernatants ranged between 1.1 and 24 mg/l.
- 200 ml(sludge)/l black water were obtained when achieving the lowest P concentrations

The experiments with Al salts led to the following conclusions:

- The amounts of total P and $\text{PO}_4\text{-P}$ were reduced below 1.6 and 0.8 mg/l corresponding to removal degree around 92%.
- 52.7 mg(Al)/l had to be added to achieve the mentioned values and the pH was between 6.2 and 7.4
- The highest A dose provided an Al/ PO_4 molar ratio of 4.3
- The Al concentration measured in the filtrated supernatants were between 0.096 and 3.3 mg/l
- The sludge volume that was obtained after applying the highest dose was 200 ml/l black water.

FeCl_3 (PIX 111) and AlCl_3 (Pluspac 1465) provided the lowest residual P concentrations. The amount of the residual P concentration is highly dependent on the composition of the feed sample.

The experiments with $\text{Ca}(\text{OH})_2$ showed the following:

- The total P and $\text{PO}_4\text{-P}$ concentrations decreased below 0.4 and 0.2 mg/l by increasing the pH above 9.5 with $\text{Ca}(\text{OH})_2$ doses from 4 to 9.3 mmol/l.
- The Ca/ PO_4 molar ratio achieved at a pH of 9.5 was 13.3
- The sludge volume at pH of 9.5 was 90 ml/l black water and decreased to 80 ml/l black water with higher doses.

Fe, Al salts and $\text{Ca}(\text{OH})_2$ provided very low residual phosphorus concentrations and further analyses should be focused on the sludge treatment. The treatment could include thickening of the sludge by sedimentation in another tank and digestion of the sludge. After doing this conclusions can be drawn about its further applicability as a fertilizer. Suitability of the iron and aluminium phosphates as fertilizers has to be investigated due to possible negative effects on biota.

Furthermore, $\text{Mg}(\text{OH})_2$ could also be tested as precipitant.

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Appendix A

Parameters in non-filtrated and filtrated supernatants after addition of FeCl₃ (PIX 111)

Parameters in the non-filtrated and filtrated supernatants after adding 17.9 mg(Fe)/l

pH (during flocculation)	6.62	
t (during flocculation) (°C)	20.2	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.65	
t (after sedimentation) (°C)	22.1	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.8	
Total alkalinity (after sedimentation) (mmol/l)	6	
Conductivity (after sedimentation) (µS/cm)	1229	
PO ₄ -P (after sedimentation) (mg/l)	8.3	5.3
Total P (after sedimentation) (mg/l)	12	8.3
Fe (after sedimentation) (mg/l)	13	3.1
Suspended solids (after sedimentation) (mg/l)	94	
V _{sludge} (after sedimentation) (ml)	58	

Parameters in the non-filtrated and filtrated supernatants after adding 26.9 mg(Fe)/l

pH (during flocculation)	6.5	
t (during flocculation) (°C)	21.4	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.52	
t (after sedimentation) (°C)	22.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.6	
Total alkalinity (after sedimentation) (mmol/l)	5.6	
Conductivity (after sedimentation) (µS/cm)	1249	
PO ₄ -P (after sedimentation) (mg/l)	5.6	1.1
Total P (after sedimentation) (mg/l)	8.3	5
Fe (after sedimentation) (mg/l)	13	4.1
Suspended solids (after sedimentation) (mg/l)	60	
V _{sludge} (after sedimentation) (ml)	62	

Parameters in the non-filtrated and filtrated supernatants after adding 35.8 mg(Fe)/l

pH (during flocculation)	6.4	
t (during flocculation) (°C)	21.2	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.42	
t (after sedimentation) (°C)	22.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.2	
Total alkalinity (after sedimentation) (mmol/l)	5.4	
Conductivity (after sedimentation) (µS/cm)	1256	
PO ₄ -P (after sedimentation) (mg/l)	0.63	0.74
Total P (after sedimentation) (mg/l)	5.7	3.6
Fe (after sedimentation) (mg/l)	12	4.3
Suspended solids (after sedimentation) (mg/l)	40	
V _{sludge} (after sedimentation) (ml)	67	

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)/l

pH (during flocculation)	6.32	
t (during flocculation) (°C)	20.4	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.36	
t (after sedimentation) (°C)	22.2	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.2	
Total alkalinity (after sedimentation) (mmol/l)	5.4	
Conductivity (after sedimentation) (µS/cm)	1356	
PO ₄ -P (after sedimentation) (mg/l)	0.88	0.83
Total P (after sedimentation) (mg/l)	3.4	1.5
Fe (after sedimentation) (mg/l)	16	8.4
Suspended solids (after sedimentation) (mg/l)	27	
V _{sludge} (after sedimentation) (ml)	120	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l

pH (during flocculation)	6.13	
t (during flocculation) (°C)	21.7	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.2	
t (after sedimentation) (°C)	22.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	2.6	
Total alkalinity (after sedimentation) (mmol/l)	4.8	
Conductivity (after sedimentation) (µS/cm)	1375	
PO ₄ -P (after sedimentation) (mg/l)	0.24	0.18
Total P (after sedimentation) (mg/l)	1.9	0.55
Fe (after sedimentation) (mg/l)	15	9
Suspended solids (after sedimentation) (mg/l)	47	
V _{sludge} (after sedimentation) (ml)	140	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l

pH (during flocculation)	5.78	
t (during flocculation) (°C)	22	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	5.8	
t (after sedimentation) (°C)	22.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	1.2	
Total alkalinity (after sedimentation) (mmol/l)	3.2	
Conductivity (after sedimentation) (µS/cm)	1421	
PO ₄ -P (after sedimentation) (mg/l)	<0.01	<0.01
Total P (after sedimentation) (mg/l)	0.22	0.07
Fe (after sedimentation) (mg/l)	14	9.4
Suspended solids (after sedimentation) (mg/l)	36	
V _{sludge} (after sedimentation) (ml)	180	
COD (mg/l)	470	
S _{total} (mg/l)	14	
N _{total} (mg/l)	110	

Appendix B

Parameters in non-filtrated and filtrated supernatants after addition of $\text{Fe}_2(\text{SO}_4)_3$

Parameters in the non-filtrated and filtrated supernatants after adding 17.9 mg(Fe)/l

pH (during flocculation)	6.65	
t (during flocculation) (°C)	23.4	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.69	
t (after sedimentation) (°C)	24.3	
Carbonate alkalinity (after sedimentation) (mmol/l)	5.2	
Total alkalinity (after sedimentation) (mmol/l)	7.6	
Conductivity (after sedimentation) ($\mu\text{S}/\text{cm}$)	1235	
$\text{PO}_4\text{-P}$ (after sedimentation) (mg/l)	9	6.3
Total P (after sedimentation) (mg/l)	14	9.7
Fe (after sedimentation) (mg/l)	14	6.9
Suspended solids (after sedimentation) (mg/l)	97	
V_{sludge} (after sedimentation) (ml)	30	

Parameters in the non-filtrated and filtrated supernatants after adding 26.9 mg(Fe)/l

pH (during flocculation)	6.54	
t (during flocculation) (°C)	23.5	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.59	
t (after sedimentation) (°C)	24.3	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.4	
Total alkalinity (after sedimentation) (mmol/l)	6.8	
Conductivity (after sedimentation) (µS/cm)	1256	
PO ₄ -P (after sedimentation) (mg/l)	8	7.3
Total P (after sedimentation) (mg/l)	11	7.9
Fe (after sedimentation) (mg/l)	16	7.2
Suspended solids (after sedimentation) (mg/l)	98	
V _{sludge} (after sedimentation) (ml)	100	

Parameters in the non-filtrated and filtrated supernatants after adding 35.8 mg(Fe)/l

pH (during flocculation)	6.45	
t (during flocculation) (°C)	24.3	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.51	
t (after sedimentation) (°C)	24.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	4	
Total alkalinity (after sedimentation) (mmol/l)	6.6	
Conductivity (after sedimentation) (µS/cm)	1275	
PO ₄ -P (after sedimentation) (mg/l)	5.2	1.1
Total P (after sedimentation) (mg/l)	8.7	5.2
Fe (after sedimentation) (mg/l)	17	7.6
Suspended solids (after sedimentation) (mg/l)	66	
V _{sludge} (after sedimentation) (ml)	160	

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)/l

pH (during flocculation)	6.6	
t (during flocculation) (°C)	15.3	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.46	
t (after sedimentation) (°C)	18.4	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.6	
Total alkalinity (after sedimentation) (mmol/l)	5.2	
Conductivity (after sedimentation) (µS/cm)	1234	
PO ₄ -P (after sedimentation) (mg/l)	2.5	1.3
Total P (after sedimentation) (mg/l)	5.1	2.6
Fe (after sedimentation) (mg/l)	21	12
Suspended solids (after sedimentation) (mg/l)	89	
V _{sludge} (after sedimentation) (ml)	150	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l

pH (during flocculation)	6.29	
t (during flocculation) (°C)	19.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.32	
t (after sedimentation) (°C)	21.3	
Carbonate alkalinity (after sedimentation) (mmol/l)	2.8	
Total alkalinity (after sedimentation) (mmol/l)	4.4	
Conductivity (after sedimentation) (µS/cm)	1325	
PO ₄ -P (after sedimentation) (mg/l)	1.2	0.16
Total P (after sedimentation) (mg/l)	3.5	0.7
Fe (after sedimentation) (mg/l)	21	7.2
Suspended solids (after sedimentation) (mg/l)	84	
V _{sludge} (after sedimentation) (ml)	170	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l

pH (during flocculation)	5.86	
t (during flocculation) (°C)	21.2	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	5.92	
t (after sedimentation) (°C)	22.1	
Carbonate alkalinity (after sedimentation) (mmol/l)	1.2	
Total alkalinity (after sedimentation) (mmol/l)	3	
Conductivity (after sedimentation) (μS/cm)	1347	
PO ₄ -P (after sedimentation) (mg/l)	0.015	0.027
Total P (after sedimentation) (mg/l)	0.69	0.15
Fe (after sedimentation) (mg/l)	11	6.4
Suspended solids (after sedimentation) (mg/l)	33	
V _{sludge} (after sedimentation) (ml)	180	

Appendix C

Parameters in non-filtrated and filtrated supernatants after addition of FeCl(SO₄)

Parameters in the non-filtrated and filtrated supernatants after adding 17.9 mg(Fe)/l

pH (during flocculation)	6.96	
t (during flocculation) (°C)	21.5	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.94	
t (after sedimentation) (°C)	22.3	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.2	
Total alkalinity (after sedimentation) (mmol/l)	5.8	
Conductivity (after sedimentation) (µS/cm)	1109	
PO ₄ -P (after sedimentation) (mg/l)	8.6	4.5
Total P (after sedimentation) (mg/l)	12	8.7
Fe (after sedimentation) (mg/l)	16	11
Suspended solids (after sedimentation) (mg/l)	160	
V _{sludge} (after sedimentation) (ml)	50	

Parameters in the non-filtrated and filtrated supernatants after adding 26.9 mg(Fe)/l

pH (during flocculation)	7.83	
t (during flocculation) (°C)	20	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.8	
t (after sedimentation) (°C)	21.1	
Carbonate alkalinity (after sedimentation) (mmol/l)	10.4	
Total alkalinity (after sedimentation) (mmol/l)	13	
Conductivity (after sedimentation) (µS/cm)	2020	
PO ₄ -P (after sedimentation) (mg/l)	17	10
Total P (after sedimentation) (mg/l)	26	16
Fe (after sedimentation) (mg/l)	22	9.4
Suspended solids (after sedimentation) (mg/l)	380	
V _{sludge} (after sedimentation) (ml)	70	

Parameters in the non-filtrated and filtrated supernatants after adding 35.8 mg(Fe)/l

pH (during flocculation)	7.55	
t (during flocculation) (°C)	20.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.6	
t (after sedimentation) (°C)	21.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	11.8	
Total alkalinity (after sedimentation) (mmol/l)	14.8	
Conductivity (after sedimentation) (µS/cm)	2240	
PO ₄ -P (after sedimentation) (mg/l)	15	9.4
Total P (after sedimentation) (mg/l)	27	15
Fe (after sedimentation) (mg/l)	21	10
Suspended solids (after sedimentation) (mg/l)	330	
V _{sludge} (after sedimentation) (ml)	70	

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)/l

pH (during flocculation)	7.22	
t (during flocculation) (°C)	21.6	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.23	
t (after sedimentation) (°C)	22.1	
Carbonate alkalinity (after sedimentation) (mmol/l)	10.6	
Total alkalinity (after sedimentation) (mmol/l)	13.2	
Conductivity (after sedimentation) (μS/cm)	2300	
PO ₄ -P (after sedimentation) (mg/l)	11	3.5
Total P (after sedimentation) (mg/l)	17	8.6
Fe (after sedimentation) (mg/l)	30	9.7
Suspended solids (after sedimentation) (mg/l)	280	
V _{sludge} (after sedimentation) (ml)	100	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l

pH (during flocculation)	6.9	
t (during flocculation) (°C)	22.3	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.93	
t (after sedimentation) (°C)	22.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	9.2	
Total alkalinity (after sedimentation) (mmol/l)	12.2	
Conductivity (after sedimentation) (μS/cm)	2360	
PO ₄ -P (after sedimentation) (mg/l)	8.3	1.3
Total P (after sedimentation) (mg/l)	14	5.4
Fe (after sedimentation) (mg/l)	30	7.8
Suspended solids (after sedimentation) (mg/l)	240	
V _{sludge} (after sedimentation) (ml)	160	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l

pH (during flocculation)	6.15	
t (during flocculation) (°C)	19.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.14	
t (after sedimentation) (°C)	20.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	2.2	
Total alkalinity (after sedimentation) (mmol/l)	3.6	
Conductivity (after sedimentation) (µS/cm)	1616	
PO ₄ -P (after sedimentation) (mg/l)	0.24	0.084
Total P (after sedimentation) (mg/l)	0.49	0.17
Fe (after sedimentation) (mg/l)	5.4	2.5
Suspended solids (after sedimentation) (mg/l)	23	
V _{sludge} (after sedimentation) (ml)	180	

Appendix D

Parameters in non-filtrated and filtrated supernatants after addition of FeCl₃ (ciba)

Parameters in the non-filtrated and filtrated supernatants after adding 35.8 mg(Fe)/l

pH (during flocculation)	6.94	
t (during flocculation) (°C)	20	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.95	
t (after sedimentation) (°C)	20.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	5.8	
Total alkalinity (after sedimentation) (mmol/l)	7.4	
Conductivity (after sedimentation) (µS/cm)	1382	
PO ₄ -P (after sedimentation) (mg/l)	6.5	3.3
Total P (after sedimentation) (mg/l)	9.1	4.8
Fe (after sedimentation) (mg/l)	24	13
Suspended solids (after sedimentation) (mg/l)	88	
V _{sludge} (after sedimentation) (ml)	60	

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)/l

pH (during flocculation)	6.65	
t (during flocculation) (°C)	20.1	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.67	
t (after sedimentation) (°C)	20.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.8	
Total alkalinity (after sedimentation) (mmol/l)	6.2	
Conductivity (after sedimentation) (µS/cm)	1492	
PO ₄ -P (after sedimentation) (mg/l)	3.3	1.1
Total P (after sedimentation) (mg/l)	5.2	2
Fe (after sedimentation) (mg/l)	20	7.2
Suspended solids (after sedimentation) (mg/l)	110	
V _{sludge} (after sedimentation) (ml)	100	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l

pH (during flocculation)	6.42	
t (during flocculation) (°C)	20.5	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.45	
t (after sedimentation) (°C)	20.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.8	
Total alkalinity (after sedimentation) (mmol/l)	5.2	
Conductivity (after sedimentation) (µS/cm)	1534	
PO ₄ -P (after sedimentation) (mg/l)	2.2	0.65
Total P (after sedimentation) (mg/l)	3.2	0.84
Fe (after sedimentation) (mg/l)		4.9
Suspended solids (after sedimentation) (mg/l)	74	
V _{sludge} (after sedimentation) (ml)	180	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l

pH (during flocculation)	5.67	
t (during flocculation) (°C)	21.2	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	5.69	
t (after sedimentation) (°C)	21.2	
Carbonate alkalinity (after sedimentation) (mmol/l)	1.2	
Total alkalinity (after sedimentation) (mmol/l)	4	
Conductivity (after sedimentation) (µS/cm)	1572	
PO ₄ -P (after sedimentation) (mg/l)	2.7	0.31
Total P (after sedimentation) (mg/l)	3.1	0.42
Fe (after sedimentation) (mg/l)	40	24
Suspended solids (after sedimentation) (mg/l)	88	
V _{sludge} (after sedimentation) (ml)	170	

Appendix E

Parameters in non-filtrated and filtrated supernatants after addition of AlCl₃ (Pluspac 1465)

Parameters in the non-filtrated and filtrated supernatants after adding 17.6 mg(Al)/l

pH (during flocculation)	6.44	
t (during flocculation) (°C)	21.5	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.47	
t (after sedimentation) (°C)	21.4	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.8	
Total alkalinity (after sedimentation) (mmol/l)	7.4	
Conductivity (after sedimentation) (μS/cm)	1400	
PO ₄ -P (after sedimentation) (mg/l)	6.2	4.7
Total P (after sedimentation) (mg/l)	7.8	4.8
Al (after sedimentation) (μg/l)	4600	1400
Suspended solids (after sedimentation) (mg/l)	73	
V _{sludge} (after sedimentation) (ml)	150	

Parameters in the non-filtrated and filtrated supernatants after adding 26.3 mg(Al)/l

pH (during flocculation)	6.37	
t (during flocculation) (°C)	21.5	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.42	
t (after sedimentation) (°C)	21.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.2	
Total alkalinity (after sedimentation) (mmol/l)	7	
Conductivity (after sedimentation) (µS/cm)	1444	
PO ₄ -P (after sedimentation) (mg/l)	3.8	2.6
Total P (after sedimentation) (mg/l)	4.9	3.6
Al (after sedimentation) (µg/l)	4500	1900
Suspended solids (after sedimentation) (mg/l)	55	
V _{sludge} (after sedimentation) (ml)	160	

Parameters in the non-filtrated and filtrated supernatants after adding 35.1 mg(Al)/l

pH (during flocculation)	6.35	
t (during flocculation) (°C)	21.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.37	
t (after sedimentation) (°C)	22.2	
Carbonate alkalinity (after sedimentation) (mmol/l)	4	
Total alkalinity (after sedimentation) (mmol/l)	6.8	
Conductivity (after sedimentation) (µS/cm)	1482	
PO ₄ -P (after sedimentation) (mg/l)	1.8	0.92
Total P (after sedimentation) (mg/l)	1.7	0.93
Al (after sedimentation) (µg/l)	3000	400
Suspended solids (after sedimentation) (mg/l)	24	
V _{sludge} (after sedimentation) (ml)	190	

Parameters in the non-filtrated and filtrated supernatants after adding 52.7 mg(Al)/l

pH (during flocculation)	6.23	
t (during flocculation) (°C)	22.1	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.28	
t (after sedimentation) (°C)	22.5	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.6	
Total alkalinity (after sedimentation) (mmol/l)	6.4	
Conductivity (after sedimentation) (µS/cm)		
PO ₄ -P (after sedimentation) (mg/l)	0.11	0.092
Total P (after sedimentation) (mg/l)	0.41	0.17
Al (after sedimentation) (µg/l)	1000	96
Suspended solids (after sedimentation) (mg/l)	11	
V _{sludge} (after sedimentation) (ml)	200	

Appendix F

Parameters in non-filtrated and filtrated supernatants after addition of AlCl₃ (Pluspac 1800)

Parameters in the non-filtrated and filtrated supernatants after adding 17.6 mg(Al)/l

pH (during flocculation)	7.76	
t (during flocculation) (°C)	20.1	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.72	
t (after sedimentation) (°C)	20.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	8.4	
Total alkalinity (after sedimentation) (mmol/l)	10.2	
Conductivity (after sedimentation) (µS/cm)	1489	
PO ₄ -P (after sedimentation) (mg/l)	10	8.9
Total P (after sedimentation) (mg/l)	14	12
Al (after sedimentation) (µg/l)	6600	3300
Suspended solids (after sedimentation) (mg/l)	150	
V _{sludge} (after sedimentation) (ml)	70	

Parameters in the non-filtrated and filtrated supernatants after adding 26.3 mg(Al)/l

pH (during flocculation)	7.55	
t (during flocculation) (°C)	20.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.53	
t (after sedimentation) (°C)	21.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	8	
Total alkalinity (after sedimentation) (mmol/l)	9.8	
Conductivity (after sedimentation) (µS/cm)	1564	
PO ₄ -P (after sedimentation) (mg/l)	9.4	7.7
Total P (after sedimentation) (mg/l)	12	11
Al (after sedimentation) (µg/l)	5600	2500
Suspended solids (after sedimentation) (mg/l)	140	
V _{sludge} (after sedimentation) (ml)	80	

Parameters in the non-filtrated and filtrated supernatants after adding 35.1 mg(Al)/l

pH (during flocculation)	7.16	
t (during flocculation) (°C)	21.4	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.16	
t (after sedimentation) (°C)	21.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	7.2	
Total alkalinity (after sedimentation) (mmol/l)	9	
Conductivity (after sedimentation) (µS/cm)	1606	
PO ₄ -P (after sedimentation) (mg/l)	3.8	2.4
Total P (after sedimentation) (mg/l)	6.1	4
Al (after sedimentation) (µg/l)	5100	2000
Suspended solids (after sedimentation) (mg/l)	97	
V _{sludge} (after sedimentation) (ml)	170	

Parameters in the non-filtrated and filtrated supernatants after adding 52.7 mg(Al)/l

pH (during flocculation)	6.94	
t (during flocculation) (°C)	22.1	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.95	
t (after sedimentation) (°C)	22.7	
Carbonate alkalinity (after sedimentation) (mmol/l)	6.8	
Total alkalinity (after sedimentation) (mmol/l)	8.6	
Conductivity (after sedimentation) (µS/cm)	1628	
PO ₄ -P (after sedimentation) (mg/l)	1.8	0.87
Total P (after sedimentation) (mg/l)	2.9	1.7
Al (after sedimentation) (µg/l)	2300	780
Suspended solids (after sedimentation) (mg/l)	64	
V _{sludge} (after sedimentation) (ml)	200	

Appendix G

Parameters in non-filtrated and filtrated supernatants after addition of Ca(OH)₂

Parameters in the non-filtrated and filtrated feed samples before adjusting the pH to 7.74 and 8.33 by adding Ca(OH)₂

Before precipitation	non-filtrated solution	filtrated solution
pH	7.13	
t (°C)	17.9	
Carbonate alkalinity (mmol/l)		
Total alkalinity (mmol/l)		
Conductivity (μS/cm)		
PO ₄ -P (mg/l)	10	7.9
Total P (mg/l)	13	13
Suspended solids (mg/l)	160	

Parameters in the non-filtrated and filtrated supernatants at pH=7.74 and pH=8.33

Ca(OH) ₂	pH=7.74		pH=8.33	
pH (during flocculation)	7.8		8.39	
t (during flocculation) (°C)	14.7		15	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.74		8.33	
t (after sedimentation) (°C)	17.5		17.7	
Carbonate alkalinity (after sedimentation) (mmol/l)	6.4		7	
Total alkalinity (after sedimentation) (mmol/l)	8		8.8	
Conductivity (after sedimentation) (μS/cm)	1193		1195	
PO ₄ -P (after sedimentation) (mg/l)	9.9	8.1	8.2	7.7
Total P (after sedimentation) (mg/l)	13	9.3	11	9.6
Ca (after sedimentation) (mg/l)	50	48	64	61
Suspended solids (after sedimentation) (mg/l)	140		140	
V _{sludge} (after sedimentation) (ml)	-		30	

Parameters in the non-filtrated and filtrated feed samples before adjusting the pH to 8.96, 9.45, 10.1, 10.61 and 11.35 by adding Ca(OH)₂

Before precipitation	non-filtrated solution	filtrated solution
pH	7.36	
t (°C)	14.6	
Carbonate alkalinity (mmol/l)	7.2	
Total alkalinity (mmol/l)	8.8	
Conductivity (μS/cm)	1167	
PO ₄ -P (mg/l)	11	9.7
Total P (mg/l)	14	13
Suspended solids (mg/l)	150	

Parameters in the non-filtrated and filtrated supernatants at pH=8.96 and pH=9.45

Ca(OH) ₂	pH=8.96		pH=9.45	
pH (during flocculation)	9.02		9.51	
t (during flocculation) (°C)	15.6		16.2	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	8.96		9.45	
t (after sedimentation) (°C)	17.5		17.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	9.2		10	
Total alkalinity (after sedimentation) (mmol/l)	10.8		11.6	
Conductivity (after sedimentation) (μS/cm)	1206		1048	
PO ₄ -P (after sedimentation) (mg/l)	3.3	2.5	0.033	0.019
Total P (after sedimentation) (mg/l)	5.3	3.3	0.4	0.22
Ca (after sedimentation) (mg/l)	75	68	17	16
Suspended solids (after sedimentation) (mg/l)	54		18	
V _{sludge} (after sedimentation) (ml)	55		90	

Parameters in the non-filtrated and filtrated supernatants at pH=10.1 and pH=10.61

Ca(OH) ₂	pH=10.1		pH=10.61	
pH (during flocculation)	10.12		10.69	
t (during flocculation) (°C)	17.1		17.2	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	10.1		10.61	
t (after sedimentation) (°C)	18.2		18.3	
Carbonate alkalinity (after sedimentation) (mmol/l)	8.4		8	
Total alkalinity (after sedimentation) (mmol/l)	9.6		9.2	
Conductivity (after sedimentation) (µS/cm)	767		656	
PO ₄ -P (after sedimentation) (mg/l)	0.2	0.16	0.13	0.056
Total P (after sedimentation) (mg/l)	0.18	0.26	0.26	0.17
Ca (after sedimentation) (mg/l)	64	39	64	55
Suspended solids (after sedimentation) (mg/l)	5.1		8	
V _{sludge} (after sedimentation) (ml)	50		80	

Parameters in the non-filtrated and filtrated supernatants at pH=11.35

Ca(OH) ₂	pH=11.35	
pH (during flocculation)	11.38	
t (during flocculation) (°C)	17.8	
	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	11.35	
t (after sedimentation) (°C)	18.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	9.4	
Total alkalinity (after sedimentation) (mmol/l)	10.4	
Conductivity (after sedimentation) (μS/cm)	898	
PO ₄ -P (after sedimentation) (mg/l)	0.11	0.017
Total P (after sedimentation) (mg/l)	0.35	0.11
Ca (after sedimentation) (mg/l)	80	77
Suspended solids (after sedimentation) (mg/l)	11	
V _{sludge} (after sedimentation) (ml)	80	

Appendix H

Parameters in non-filtrated and filtrated supernatants after addition of FeCl₃ (PIX 111)

Parameters in the non-filtrated and filtrated feed samples before adding 53.7 mg(Fe)l

Before precipitation	non-filtrated solution	filtrated solution
pH	7.21	
t (°C)	15.8	
Carbonate alkalinity (mmol/l)	6.6	
Total alkalinity (mmol/l)	8.6	
Conductivity (μS/cm)	1242	
PO ₄ -P (mg/l)	12	11
Total P (mg/l)	16	12
Suspended solids (mg/l)	530	

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.49		6.38	
t (during flocculation) (°C)	13		13.5	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.52		6.42	
t (after sedimentation) (°C)	15.4		15.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	3.8		3.2	
Total alkalinity (after sedimentation) (mmol/l)	5.6		5	
Conductivity (after sedimentation) (μS/cm)	1307		1327	
PO ₄ -P (after sedimentation) (mg/l)	3	1.1	1.9	0.91
Total P (after sedimentation) (mg/l)	3.8	1.3	2.2	0.91
Fe (after sedimentation) (mg/l)	17	5.2	12	5.5
Suspended solids (after sedimentation) (mg/l)	69		51	
V _{sludge} (after sedimentation) (ml)	130		110	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.31		6.34	
t (during flocculation) (°C)	13		12.5	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.38		6.37	
t (after sedimentation) (°C)	17.2		15.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	2.8		3	
Total alkalinity (after sedimentation) (mmol/l)	4.6		4.8	
Conductivity (after sedimentation) (µS/cm)	1388		1333	
PO ₄ -P (after sedimentation) (mg/l)	1	0.24	1.4	0.68
Total P (after sedimentation) (mg/l)	1.4	0.31	1.4	0.75
Fe (after sedimentation) (mg/l)	10	3.8	10	5.7
Suspended solids (after sedimentation) (mg/l)	42		31	
V _{sludge} (after sedimentation) (ml)	140		140	

Parameters in the non-filtrated and filtrated feed samples before adding 107.4 mg(Fe)l

Before precipitation	non-filtrated solution	filtrated solution
pH	7.16	
t (°C)	17.3	
Carbonate alkalinity (mmol/l)	7.8	
Total alkalinity (mmol/l)	9.8	
Conductivity (µS/cm)	1448	
PO ₄ -P (mg/l)	14	13
Total P (mg/l)	17	14
Suspended solids (mg/l)	390	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.06		6.04	
t (during flocculation) (°C)	15.9		16.3	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.18		6.08	
t (after sedimentation) (°C)	17.7		17.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	2.4		2.2	
Total alkalinity (after sedimentation) (mmol/l)	4.2		4.4	
Conductivity (after sedimentation) (µS/cm)	1622		1620	
PO ₄ -P (after sedimentation) (mg/l)	0.14	0.056	0.4	0.13
Total P (after sedimentation) (mg/l)	0.5	0.15	0.67	0.21
Fe (after sedimentation) (mg/l)	7.2	4.5	7.8	5.4
Suspended solids (after sedimentation) (mg/l)	17		14	
V _{sludge} (after sedimentation) (ml)	180		130	

Appendix I

Parameters in non-filtrated and filtrated supernatants after addition of FeCl₃ (ciba)

Parameters in the non-filtrated and filtrated feed samples before adding 53.7 and 71.6 mg(Fe)/l

Before precipitation	non-filtrated solution
pH	8.27
t (°C)	18.7
Carbonate alkalinity (mmol/l)	7
Total alkalinity (mmol/l)	8
Conductivity (μS/cm)	952
PO ₄ -P (mg/l)	11
Total P (mg/l)	16
Suspended solids (mg/l)	200

Parameters in the non-filtrated and filtrated supernatants after adding 53.7 mg(Fe)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.8		6.79	
t (during flocculation) (°C)	18.5		18.9	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.86		6.82	
t (after sedimentation) (°C)	19.7		19.9	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.6		4.6	
Total alkalinity (after sedimentation) (mmol/l)	5.4		5.2	
Conductivity (after sedimentation) (μS/cm)	1134		1158	
PO ₄ -P (after sedimentation) (mg/l)	3	0.87	1.9	0.6
Total P (after sedimentation) (mg/l)	4.9	2.2	3.8	1.4
Fe (after sedimentation) (mg/l)	19	7.2	12	3.5
Suspended solids (after sedimentation) (mg/l)	91		53	
V _{sludge} (after sedimentation) (ml)	90		60	

Parameters in the non-filtrated and filtrated supernatants after adding 71.6 mg(Fe)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.56		6.58	
t (during flocculation) (°C)	19.6		20.3	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.62		6.63	
t (after sedimentation) (°C)	20.6		20.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	4		4	
Total alkalinity (after sedimentation) (mmol/l)	4.8		4.8	
Conductivity (after sedimentation) (μS/cm)	1226		1218	
PO ₄ -P (after sedimentation) (mg/l)	0.42	0.2		0.26
Total P (after sedimentation) (mg/l)	3	0.66		0.6
Fe (after sedimentation) (mg/l)	15	3.2		2.8
Suspended solids (after sedimentation) (mg/l)	64			
V _{sludge} (after sedimentation) (ml)	130		80	

Parameters in the non-filtrated and filtrated feed samples before adding 107.4 mg(Fe)l

Before precipitation	non-filtrated solution	filtrated solution
pH	8.55	
t (°C)	18.8	
Carbonate alkalinity (mmol/l)	10	
Total alkalinity (mmol/l)	11.2	
Conductivity (μS/cm)	1292	
PO ₄ -P (mg/l)	14	14
Total P (mg/l)	20	17
Suspended solids (mg/l)	210	

Parameters in the non-filtrated and filtrated supernatants after adding 107.4 mg(Fe)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	6.58		6.59	
t (during flocculation) (°C)	18.9		19.2	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	6.63		6.65	
t (after sedimentation) (°C)	19.8		20.1	
Carbonate alkalinity (after sedimentation) (mmol/l)	4.6		5	
Total alkalinity (after sedimentation) (mmol/l)	5.8		6	
Conductivity (after sedimentation) (μS/cm)	1615		1645	
PO ₄ -P (after sedimentation) (mg/l)	0.11	0.065	0.25	0.056
Total P (after sedimentation) (mg/l)	0.89	0.27	0.9	0.32
Fe (after sedimentation) (mg/l)	4.7	1.1	4.8	1.3
Suspended solids (after sedimentation) (mg/l)	25		20	
V _{sludge} (after sedimentation) (ml)	200		150	

Appendix J**Parameters in non-filtrated and filtrated supernatants after addition of AlCl₃ (Pluspac 1465)****Parameters in the non-filtrated and filtrated supernatants after adding 26.3 mg(Al)/l and a polymer**

	Without a polymer		Magnafloc 336	
pH (during flocculation)	8.24		8.25	
t (during flocculation) (°C)	19.5		19.9	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	8.25		8.2	
t (after sedimentation) (°C)	20.2		20.6	
Carbonate alkalinity (after sedimentation) (mmol/l)	9.4		9.6	
Total alkalinity (after sedimentation) (mmol/l)	10.8		10.8	
Conductivity (after sedimentation) (μS/cm)	1530		1573	
PO ₄ -P (after sedimentation) (mg/l)	6	5.3	5.8	5.4
Total P (after sedimentation) (mg/l)	8.1	13	8.6	7.3
Al (after sedimentation) (mg/l)	0.9	0.3	0.7	0.3
Suspended solids (after sedimentation) (mg/l)	43		27	
V _{sludge} (after sedimentation) (ml)	150		120	

Parameters in the non-filtrated and filtrated supernatants after adding 35.1 mg(Al)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	8		8.05	
t (during flocculation) (°C)	20.8		20.7	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.99		8.03	
t (after sedimentation) (°C)	20.9		21.4	
Carbonate alkalinity (after sedimentation) (mmol/l)	9		9.4	
Total alkalinity (after sedimentation) (mmol/l)	10.4		10.6	
Conductivity (after sedimentation) (µS/cm)	1659		1647	
PO ₄ -P (after sedimentation) (mg/l)	1.5	1.2	3.7	3.1
Total P (after sedimentation) (mg/l)	3.4	2.8	5.3	4.8
Al (after sedimentation) (mg/l)	0.6	0.2	0.4	0.2
Suspended solids (after sedimentation) (mg/l)	41		22	
V _{sludge} (after sedimentation) (ml)	180		130	

Parameters in the non-filtrated and filtrated feed samples before adding 52.7 mg(Al)l

Before precipitation	non-filtrated solution	filtrated solution
pH	8.09	
t (°C)	19.2	
Carbonate alkalinity (mmol/l)	8.8	
Total alkalinity (mmol/l)	10.2	
Conductivity (µS/cm)	1453	
PO ₄ -P (mg/l)	14	15
Total P (mg/l)	20	16
Suspended solids (mg/l)	240	

Parameters in the non-filtrated and filtrated supernatants after adding 52.7 mg(Al)/l and a polymer

	Without a polymer		Magnafloc 336	
pH (during flocculation)	7.34		7.39	
t (during flocculation) (°C)	17.8		18.4	
	non-filtrated supernatant	filtrated supernatant	non-filtrated supernatant	filtrated supernatant
pH (after sedimentation)	7.28		7.43	
t (after sedimentation) (°C)	18.5		18.8	
Carbonate alkalinity (after sedimentation) (mmol/l)	6.8		6.8	
Total alkalinity (after sedimentation) (mmol/l)	7.8		8	
Conductivity (after sedimentation) (µS/cm)	1440		1594	
PO ₄ -P (after sedimentation) (mg/l)	0.14	0.046	0.21	0.073
Total P (after sedimentation) (mg/l)	0.99	0.67	1.6	1.3
Al (after sedimentation) (mg/l)	0.5	<0.1	0.4	0.2
Suspended solids (after sedimentation) (mg/l)	26		30	
V _{sludge} (after sedimentation) (ml)	200		170	

Appendix K Specifications of the chemicals

PIX-III

Kemwater PIX-I 11 — 2006-11

Kemwater PIX-I 11, järnklorid, är ett flytande fälningsmedel för vattenrening och innehåller aktiva 3-värda järn-föreningar. PIX-I 11 lämpar sig för avtoppsvattenrening, yt- och grundvattenrening samt vattenmjukning i de flesta reningsprocesser. PIX-III är också utmärkt att använda vid svaveivätebekämpning.

Kemiska data

Järn (Fe ³)	13,8±0,2%
Spec. vikt vid medelhalt	1,42 g/cm ³
Viskositet (25°C)	ca 40 mPas pH <1
Fri syra	5-15 g/kg
Kloridhalt	260-280 g/kg
Sulfathalt	ca 0,5%
Lägsta lagringstemp.	-1 5°C
Vatten olösligt	<0,05 %
Aktiv substans (Me ₃)	ca 2,45 mol/kg

Spårämnen

Kadmium (Cd)	<0,0028 mg/kg
Kobolt (Co)	5 mg/kg
Krom (Cr)	7 mg/kg
Koppar (Cu)	1 mg/kg
Kvicksilver (Hg)	<0,005 mg/kg
Nickel (Ni)	7 mg/kg
Bly (Pb)	0,037 mg/kg
Zink (Zn)	5 mg/kg

*) I-lalternerna baseras på föregående års utfall.

Dosering

Dosering av PIX- 111 sker med pumpar i korrosionsskyddat utförande. Även rörledningar och ventiler skall vara utförda i plast eller gummertat stål. PIX-III doseras lämpligast utan utspädning direkt från lagertank.

Säkerhet

PIX-1 11 är svagt frätande. Vid öppen hantering av PIX-III skall skyddsglasögon användas. Skyddshandskar och annan skyddsbeklädnad används vid behov. Kroppsdelar som kommer i kontakt med vätskan skall sköljas rikligt med vatten. Vid varaktig irritation på hud, kontakta läkare. Vid stänk i ögon, skölj rikligt med vatten ur ögon flaska och kontakta läkare. Vid spill, spola med vatten och neutralisera med kalk eller kalkstensmjöl.

För ytterligare information avseende hantering, klassificering och märkning - se PIX-1 11 säkerhetsdatablad.

Produkten uppfyller kraven enligt SLV ES 2001 :30.

Förvaring

Hållbarhet minst 12 månader.

Lagertankar och behållare skall märkas enligt

AFS 2000:4, Märkning 40 §:

I närheten av lossningsplatsen skall nöddusch finnas.

PIX-113

KemwaterPIX-1 13— 2006-11

Kemwater PIX-1 13, järnsuffat, är ett flytande fällningsmedel för vattenrening och innehåller aktiva 3-värda järnföreningar. PIX-113 lämpar sig för avloppsvattenrening i de flesta reningsprocesser. PIX-1 13 är också utmärkt att använda vid svavelvätebekämpning.

Kemiska data

Järn (Fe ³)	11,4 ± 0,4%
Spec. vikt vid medelhalt	ca 1,55 g/cm ³
pH	<1
Sulfathalt	<330 g/kg
Lägsta lagringstem.	ca-10°C
Aktiv substans (Me)	ca 2,08 mol/kg

Spårämnen Typanalys

Kadmium (Cd)	<0,05 mg/kg
Kobolt (Co)	24 mg/kg
Krom (Cr)	2,4 mg/kg
Koppar (Cu)	1 mg/kg
Kvicksilver (Hg)	<0,05 mg/kg
Nickel (Ni)	26 mg/kg
Bly (Pb)	0,15 mg/kg
Zink (Zn)	28 mg/kg

*) Halterna baseras på föregående års utfall.

Dosering

Dosering av PIX-1 13 sker med pumpar i korrosionsskyddat utförande. Även rörledningar och ventiler skall vara utförda i plast eller gummerat stål. PIX-113 doseras lämpligast utan utspädning direkt från lagertank.

Säkerhet

PIX-1 13 är svagt frätande. Vid öppen hantering av PIX-1 13 skall skyddsglasögon användas! Skyddshandskar och annan skyddsbeklädnad används vid behov. Kroppsdelar som kommer i kontakt med vätskan skall sköljas rikligt med vatten. Vid varaktig irritation på hud, kontakta läkare. Vid stänk i ögon, skölj rikligt med vatten ur ögonflaska och kontakta läkare. Vid spill, spola med vatten och neutralisera med kalk eller kalkstensmjöl.

För ytterligare information avseende hantering, klassificering och märkning se PIX-113 säkerhetsdatablad.

Produkten uppfyller kraven enligt SLV ES 2001:30.

Förvaring

Hållbarhet minst 6 månader.

Lagertankar och behållare skall märkas enligt

AES 2000:4, Märkning 40 §:

I närheten av lossningsplatsen skall nöddusch finnas.

PIX-118

KemwaterPIX-1 18— 2006-11

Kemtster *PIX-1 18*, järnkloridsulfat, är ett flytande fällningsmedel för vattenrening och innehåller aktiva 3-värda järn-föreningar. *PIX-1 18* lämpar sig för avtoppsvattenrening i de flesta reningsprocesser. *PIX-118* är också utmärkt att använda vid sva valvätebekämpning.

Dosenng

Dosering av PIX- 118 sker med pumpar i korrosionsskyddat utförande. Även rörledningar och ventiler skall vara utförda i plast eller gummerat stål. PIX-118 doseras lämpligast utan utspädning direkt från lagertank.

Kemiska data

Järn (Fe ³)	11,6±0,3%
Spec. vikt vid medelhalt	ca 1,48 g/cm ³
Viskositet (25°C)	ca 20 mPas
pH	<1
Fri syra	10-15 g/kg
Kloridhalt	max 250 g/kg
Sulfathalt	max 250 g/kg
Lägsta lagringstem.	+5°C
Aktiv substans (Me)	ca 2,07 mol/kg
Spårämnen	
Kadmium (Cd)	<0,007 mg/kg
Kobolt (Co)	11 mg/kg
Krom (Cr)	27 mg/kg
Koppar(Cu)	2 mg/kg
Kvicksilver (Hg)	<0,005 mg/kg
Nickel (Ni)	23 mg/kg
Bly (Pb)	0,2 mg/kg
Zink (Zn)	29 mg/kg

Säkerhet

PIX-1 18 är svagt frätande. Vid öppen hantering av PIX-118 skall skyddsglasögon användas! Skyddshandskar och annan skyddsbeklädnad används vid behov. Kroppsdelar som kommer i kontakt med vätskan skall sköljas rikligt med vatten. Vid varaktig irritation på hud, kontakta läkare. Vid stänk i ögon, skölj rikligt med vatten ur ögon flaska och kontakta läkare. Vid spill, spola med vatten och neutralisera med kalk eller kalkstensmjöl. För ytterligare information avseende hantering, klassificering och märkning - se PIX-1 18 säkerhetsdatablad.

Förvaring

Hållbarhet minst 12 månader.
Lagertankar och behållare skall märkas enligt
AFS 2000:4, Märkning 40 §:

I närheten av lossningsplatsen skall nöddusch finnas.

PlusPAC 1465 är en högkoncentrerad polyaluminiumkloridlösning med hög prestanda. Den aktiva komponenten utgörs i huvudsak av ett polynukleärt högladdat sjuvärdigt aluminiumkomplex. Produktens allmänna formel kan skrivas:



PlusPAC 1465 neutraliserar laddningen av kolloidal och suspenderad substans och kompakta flockar bildas. Flockarna kan enkelt avlägsnas från vattnet genom sedimentering eller flotation.

PlusPAC 1465 har upp till 40 % högre laddningstäthet per aluminiumjon än de flesta på marknaden förekommande polyaluminiumkloridprodukter.

Produktegenskaper

- Hög reduktion av turbiditet och suspenderad substans
- Hög basicitet ger mindre pH-sänkning
- Effektiv för slamkonditionering
- Effektiv även vid låga temperaturer
- Lag slamproduktion

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PlusPAC 1465 möter de krav som ställs i SS-EN 883 svensk standard för processkemikalier för beredning av dricksvatten.

Applikationer

- Dricks- och avloppsvattenrening
- Industriell avloppsvattenrening
- Processvattenrening
- Slamkonditionering
- Laddningsneutralisation inom pappers- och massa- industrin
- Rening av oljeförorenade vatten

Dosering

Produkten doseras med lämplig doserpump, t.ex. av membrantyp direkt i råvattnet eller avloppsvattnet. Tack vare produktens reaktivitet bör doserpunkten väljas så att en god inblandning av koagulanten erhålls. Optimal dos kan bestämmas med flockningsförsök i bägarskala.

Fällnings-pH beror på användningsområdet.

Lagring

Lagertankar skall vara av glasfiberarmerad polyester eller gummerat stal. Undvik detaljer i mässing eller kolstal. Rörledningar och ventiler bör vara i PVC, glasfiberarmerad polyester eller annat syra- och kloridresistent material.

Regelbunden inspektion och rengöring av lagertankar och doseringsutrustning rekommenderas.

Leverans

PlusPAC 1465 levereras med tankbil eller i plastbehållare (IBC 800-1000 liter).

PlusPAC 1465

Polyaluminiumkloridlösning

Ciba ::»

Value beyond chemistry

Rev.datum 2005-11 -07

Produktspecifikation	
Utseende:	gulaktig vätska
Egenskap:	irriterande
Al:	7,3% ±0,2%
Basicitet:	64 % ± 3 %
Densitet, 20°C:	1,33 g/ml
Viskositet:	21 cP
pH, 200:	1,7 ± 0,4
Frys punkt:	- 35°C
Spårämnen, medel	
Arsenik (As):	< 0,05 mg/kg
Kadmium (Cd):	< 0,04 mg/kg
Kobolt (Co):	< 0,1 mg/kg
Krom (Cr):	0,3 mg/kg
Koppar (Cu):	0,1 mg/kg
Kvicksilver (Hg):	< 0,002 mg/kg
Nickel (Ni):	0,2 mg/kg
Bly (Pb):	< 0,3 mg/kg
Zink (Zn):	1,3 mg/kg

Technical Information

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Productname **CIBA®MAGNAFLOC®336**

Anionic flocculant

A Product of Ciba Specialty Chemicals

Description

Magnafloc®336 is a high molecular weight anionic polyacrylamide flocculant supplied as a free flowing granular powder.

Principal Uses

Magnafloc®336 has found application in a wide variety of mineral processing operations including the following:

1. Base metal sulphide and oxide concentrates thickening and filtration
2. Sedimentation of coal tailings
3. Sedimentation of coal fines
4. Filtration of coal fines
5. Sedimentation and filtration of metal hydroxides
6. Sedimentation of fine sands and clay
7. Tailings dewatering
8. Brine clarification
9. Phosphate slimes thickening

Dosage depends on application but normally lies in the range 50 – 200 g/tonne of dry substrate flocculated.

Packaging

Magnafloc®336 is supplied in 25 kg nett plastic bags shrinkwrapped onto a pallet suitable for export shipment. The product can also be supplied via intermediate big bags or bulk tanker. Specific details of

bag and tanker sizes can be obtained on request. Corrosivity towards most standard materials of construction is low, but aluminium and galvanised equipment should be avoided. **Magnafloc®336** spills are a hazard due to the inherent slipperiness of this type of product. Spills should be cleaned up immediately. Dry spills should be left dry and swept up. If the polymer becomes wet, an absorbant material should be applied to the spill, then swept up and discarded.

Technical Service

Advice and assistance in the running of laboratory and plant tests to select the correct flocculant and determine the best application is given by representative of Ciba Specialty Chemicals, who are experienced in mineral processing applications.

Magnafloc 336

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Health & Safety

Magnafloc®336 has a low order of oral toxicity and does not present any abnormal handling problems. Detailed information on handling and any precautions to be observed in the use of the the product(s) described in this leaflet can be found in our relevant Health and Safety Information sheet.

Typical Properties

Physical Form White granular powder

Particle Size 98 % < 1000 µm

Bulk Density 0.75 g/cm³

pH of 1 % solution at 25°C 7.0

Application & Storage

Recommended solution concentration

Stock solution 0.25 – 0.5 % max.

Feed solution 0.025 – 0.1 % max.

Recommended storage periods

Solid up to two years

Stock solution 1 – 2 days

Storage of polymer should be in a cool, dry place. Details on preparation and feeding can be obtained from a Ciba Specialty Chemicals Representative.

Warranty

The information contained in this leaflet is given in good faith but no liability is assumed nor is freedom from any patent owned by Ciba Specialty Chemicals or other implied. This information should not be taken to represent a specification for the product.

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