

SUPERCRITICAL OXIDATION OF WATER WORK SLUDGE FROM RINGSJÖVERKET

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Abstract

This paper presents how super critical water oxidation (SCWO) performs on the iron rich sludge from the drinking water treatment plant Ringsjöverket. The pilot-test shows that the SCWO-technique is capable of treating the sludge from Ringsjöverket. During the pilot- test all organic material were oxidized while the rest precipitated as ferric oxide crystals. Chromium and nickel in elevated concentrations were found in the ferric oxide. The nickel probably comes from corrosion of the pilot plant while most of the chromium probably comes from the raw water and pilot plant. Problems with scaling also occurred during the pilot-tests. These problems may be smaller in a full-scale plant solely constructed for treating sludge from Ringsjöverket.

The annual cost for a full scale SCWO-plant was estimated to be approximately 4.9 million SEK which include a saving of 1.3 M SEK in heating costs due to the surplus of energy produced by the process. The annual cost can be lowered with about 1 M SEK if the iron oxide can be reused for producing a new coagulant. If the iron oxide can be sold as a pigment the savings might be even greater.

Key words – SCWO, Supercritical oxidation, Sydvatten, Waterworks sludge

BACKGROUND

The process of making drinking water often includes a flocculation step where sludge is formed and taken out of the process. This sludge mainly consists of organic material and an inorganic coagulant which were added in the flocculation step. This coagulant would be a valuable resource for the production of new coagulants and perhaps also for other purposes if it could be separated from the organic fractions in the sludge. The Aqua Reci process, which has been developed by Chematur Engineering together with Feralco, uses super critical oxidation to achieve this separation. This is however a method which never has been used for treating water work sludge. Therefore Sydvatten AB, who provides 700 000 persons in the western part of Scania in south Sweden with drinking water, took an initiative for two Master

theses with the aim to investigate how this method would perform on sludge from the waterworks Ringsjöverket. This paper is the synthesis of the results and experiences gained from those studies.

THE CURRENT TREATMENT PROCESS AT RINGSJÖVERKET

Water treatment

Ringsjöverket is a surface water treatment plant which treats around 110 000 m³ water a day, (1 300 l/s). The maximum capacity is 205 000 m³ a day (2 400 l/s).

The raw water is taken from Lake Bolmen which is located in the province of Småland. With its low content of nutrients, Lake Bolmen is considered to be an oli-

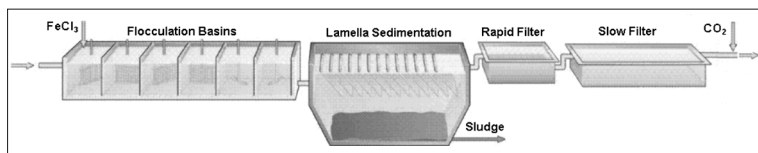


Figure 1. Treatment process at Ringsjöverket.

gotrofic lake. The catchment is 433 km² and consists mainly of forest which makes the water weakly colored and weakly humic.

The raw water is led from Lake Bolmen via an 80 kilometer long tunnel and through 25 kilometers of pipes to Ringsjöverket.

As a first stage in the treatment process (figure 1) an iron chloride solution (FeCl₃) is mixed into the raw water to induce flocculation. The average dose of iron chloride is 52 g/m³ (2 100 tonnes per year). Depending on the coagulant dose and the properties of the raw water sulphuric acid or caustic soda is added to achieve a suitable pH for flocculation. The flocs that forms are separated from the cleaning process in the following lamella sedimentation basins. In the next step the water passes through rapid sand filters containing fine-grained sand, where small remnants of flocs are retained. To remove odorous compounds the water is subjected to slow sand filtration which also reduces the chemical oxygen demand. This process requires the water to be slightly alkaline. The pH of the water is therefore raised by the addition of lime before the slow sand filtration. Finally the processed water is chlorinated for disinfection purposes.

Sludge treatment

The sludge which is taken out of the cleaning process in the lamella sedimentation basins normally has a dry content of about 0.5%. This sludge is pumped to a gravitation thickener where polymer is added. After the gravitation thickener the dry content of the sludge has increased to about 2.5%. The thickened sludge is then led to a centrifuge where the sludge is made even thicker with the help of a polymer. The result is a sludge which has a dry content of about 15%.

The sludge mainly consists of the added coagulant (FeCl₃) and organic materials like algae and humus that originate from Lake Bolmen.

SUPERCritical WATER OXIDATION

When certain compounds are heated above a certain temperature and placed under extremely high pressure they form a supercritical fluid. To put water in a supercritical state (figure 2) a temperature of at least 374.1°C and a pressure of 221 bars is needed.

When water is turned into a supercritical state, its properties change dramatically. The normally polar solvent then starts acting as a non-polar solvent where many organic compounds and gases like N₂, O₂ and CO₂ are highly soluble. (Smits *et al* 1998)

By adding oxygen to the supercritical fluid, an oxidation of the organic fractions is achieved. Because of the solvation properties of the supercritical water, the organic compounds and oxygen are brought into immediate molecular contact in the single homogeneous phase and the oxidation that occurs is complete and extremely fast (Smith *et al* 2002). The oxidation converts organic materials to CO₂, H₂O and N₂ while releasing energy. If operated properly, the conversion efficiency is often higher than 99% (Chematur Engineering 2005). Heavy metals are converted to their oxides, phosphorous is converted to phosphate and nitrogen containing compounds are converted to molecular nitrogen (nitrogen gas). The reason that no NO_x is produced is the relatively low temperature. NO_x only forms at temperatures above 850°C (Smits *et al* 1998). Salts and inorganic particles have a reduced solubility in supercritical water which means that they can precipitate out from the treated effluent (Smith *et al* 2002).

SUPERCritical OXIDATION OF THE SLUDGE FROM RINGSJÖVERKET

This study of the implementation of SCWO on the sludge from Ringsjöverket has been divided into two different parts. One part which involved simulating the

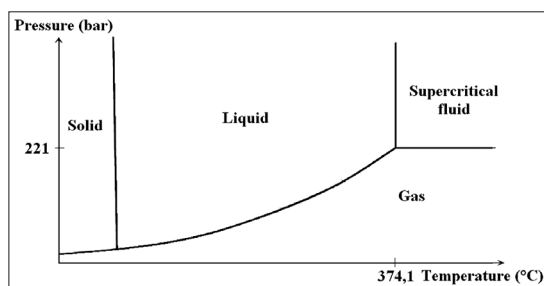


Figure 2. A simplified phase diagram for water.

SCWO process on water work sludge from Ringsjöverket. The results from these simulations were later compared with the results from the pilot scale runs in Karlskoga.

The other part of the study examined the feasibility of the SCWO method and how a full-scale plant could be implemented at Ringsjöverket. These examinations are based on the pilot scale run.

Pilot plant tests

The pilot plant tests were performed at Chematur's facilities in Karlskoga.

The pilot plant

The schematic figure of the pilot plant is shown in figure 3. The nominal capacity of the pilot plant is 225 kg/h.

Pretreatment

In the feed tank, the sludge can be diluted if the dryness is too high. To prevent too large particles from entering the high pressure pump a grinding pump is connected at the outlet of the feed tank. The high pressure pump raises the feed pressure to 230 bars before the sludge enters the economizer where it's heated to around 550°C by the reactor effluent. If the economizer can't provide enough heat, a gas heater can be used to heat the sludge. The gas heater uses liquefied petroleum gas.

The economizer and the gas heater are regularly washed with nitrogen acid due to fouling.

In the Reactor

The reactor is where the supercritical oxidation takes place. The reactor consists of a long steel pipe which can endure the high pressure and temperature needed to reach the supercritical state. In the beginning of the reactor, oxygen can be injected to start the oxidation which takes place in approximately 30–60 seconds. If the COD-content in the sludge is high, oxygen can be injected in a second point further away in the reactor to fully carry out the oxidation. When the oxidation is performed in two steps water is added to cool down the reactor and make the oxygen addition possible without exceeding the thermal limit of the construction material.

After the Reactor

The hot effluent is cooled through the economizer which also heats the incoming sludge. The temperature is cooled to ambient temperature and the pressure is lowered through a capillary network.

The gas which is generated in the process is separated in a gas/liquid separator. The separated gas is analyzed

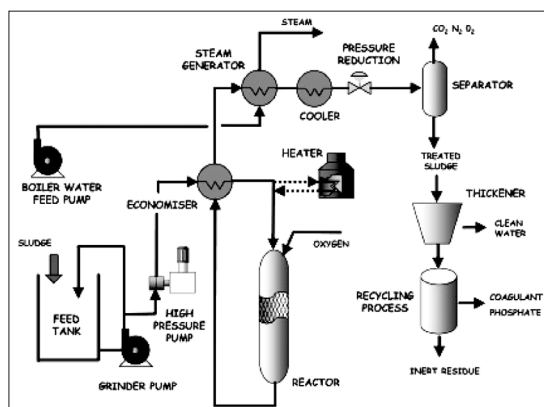


Figure 3. *The pilot plant.* (Chematur, 2005.)

for oxygen, carbon dioxide and carbon monoxide to assess if the oxidation is optimal. If the oxygen content is higher than 25 % nitrogen is added to dilute the gas as a safety precaution.

Pilot tests

The centrifuged sludge was transported to Chematur in 1000 kg bags which were emptied into the feed tank. But since the mixer in the feed tank couldn't handle the viscous sludge it had to be diluted with tap water.

If the process is to be self-supported on heat, the energy content in term of COD in the sludge should be above 70 g/l. Otherwise the gas heater has to be used to preheat the sludge. The centrifuged sludge with a dry substance around 15 % fulfils this criterion. But due to the dilution which had to be made, the COD-content in all samples were below 70 g/l.

The COD-content was measured on seven diluted sludge samples. The results are presented in table 1. The drop in COD-content for sample 4, 5 and 6 is due to a valve which wasn't properly closed which led to an unwanted dilution of the sludge.

Table 1. *Dry solids (DS) and COD content in centrifugated sludge.*

Sample	Sample moment	Ds [%]	COD [g/l]
1	2005-06-07, 13:45	11.2	66,5
2	2005-06-07, 19:45	11.6	64,7
3	–	11.8	69,5
4	2005-06-08, 08:15	8.7	49
5	–	9.2	54
6	2005-06-08, 17:45	9.4	55,6
7	–	10.5	60

Due to the relatively low COD-content, the gas heater was used initially to reach sufficiently high temperature. Complete oxidation of the organic content in the diluted sludge was achieved in one step. If the sludge hadn't been diluted the oxidation would probably have to be done in two steps. After three hours of operation, the gas heater was by-passed to see if the process was self-supported on heat but after 1.5 hours the temperature inside the reactor had decreased too much and the gas heater was turned on again.

After about 9 hours of operation the pressure drop in the economizer, due to scaling, was too high and a switch to a clean economizer was made.

After 14.5 hours the pressure drop over the gas heater had risen so high that the operation had to be turned down. The heater was cooled down and cleaned with nitric acid for almost one hour until the washing pump broke down. The process was started again but after a while it was obvious that the washing only had had a negligible effect and the operation was stopped again. The heater was cooled down and washed but after approximately one hour of washing the heater clogged due to incruster which de-attached from the heater walls. This led to the operation being terminated after 33 hours of actual operation.

During the 33 hours of operation a total of 7600 kg diluted sludge where treated in the pilot plant.

Properties of the effluent gas

The amount of oxygen and carbon dioxide in the effluent gas and the gas flow were measured. The total gas effluent from the operation was 694 kg of which 139 kg (20%) where oxygen. In a full-scale commercial plant the oxygen content in the effluent should be around 5% (Mallol, 2005).

The reason for the high amount of used oxygen was to ensure that the oxidation was complete during the pilot tests and to prevent the formation of CO. Despite the oxygen surplus some CO could be found in the effluent. During the test 2.41 kg (0.073 kg/h) of CO where released into the air.

Properties of the effluent liquid

From the trial a total volume of 10 m³ of liquid effluent were obtained. When the solid parts in the effluent had sedimentated the concentration of different metals in the water phase were analyzed. The results are presented in table 2.

The content of copper, mercury and nickel is too high when compared to the warning values set by the municipality of Eslöv, where Ringsjöverket is located. This means that the liquid effluent in this case can't be led to

the waste water treatment plant without further treatment.

The variations of the concentrations are quite high. This is probably due to the different dry substances of the incoming sludge and to the fact that it takes some time before the process conditions has stabilized (table 1).

Properties of the effluent dry fraction

The dry substance of the liquid effluent was 2%. The dry fraction of the effluent consists of inorganic substances, mainly iron oxide (63% mass fraction) that precipitates inside the reactor. The dry fraction was examined for physical properties, metals and biological rests.

Physical properties

The size of the iron oxide particles was determined with a field emission scanning electron microscope at the National Center for High Resolution Electro Microscopy at the Center for Chemistry and Chemical Engineering at Lund University.

The most dominating sizes varied between 50–500 nm. Two types of particles were dominating, small rounder boards and larger angular boards.

Because of the strong oxidizing environment that prevails in the reactor all of the iron ought to be oxidized to its highest oxidation state which means that Fe₂O₃ should be formed. An x-ray diffraction investigation showed that 85% were hematite and 15% were magnetite.

Table 2. Concentration (mg/l) of metals in the water phase after 3, 9, 21 and 31 hours.

Sample	1	2	4	6
Al	2.3	2.0	0.56	0.17
As	<0.04	<0.03	<0.01	<0.01
Ca	60	62	48	32
Cd	0.0053	0.0058	0.00036	0.00027
Co	0.061	0.11	0.0016	0.0013
Cr	0.010	0.012	0.097	0.010
Cu	0.68	0.95	0.0033	0.0037
Fe	0.17	0.69	0.25	0.031
Hg	0.0034	0.0032	0.0017	0.00037
K	7.8	7.7	5.4	2.5
Mn	8.4	13	660	0.24
Mo	0.0077	0.00064	<0.0005	<0.0005
Ni	3.2	4.7	0.097	0.069
Pb	0.094	0.11	0.00051	<0.0002
Ti	0.000051	0.00022	<0.000010	0.00010
Zn	1.8	1.7	0.030	0.049
P	<0.01	<0.01	<0.01	<0.01

Metals

The metal content in the dry fraction is presented in table 3.

715 000 mg/kg dry substance were metals and 97.9% of the metals were iron. 208 180 mg/kg dry substance derives from the weight of the oxygen fraction in Fe_2O_3 and Fe_3O_4 . The main part of the remaining 77 133 mg/kg is probably due to the oxygen fraction in other metal oxides.

Biological rest

No organic impurities could be detected in the dry fraction of the effluent.

Energy

In the economizer the temperature of the effluent was lowered from a mean temperature of 461°C to a mean temperature of 297°C. At the pilot plant the excess heat after the economizer was cooled to a mean temperature of 10°C.

The amount of energy cooled away after the economizer during the whole operation was 3333 kWh. In a full-scale commercial plant it will be possible to reutilize this excess amount of energy.

Simulations

Since the pilot plant is considerably smaller than a potential full-scale SCWO-plant the super critical process has been simulated to investigate how a full-scale plant would perform on the sludge from Ringsjöverket. The model was built using MATLAB for the reaction model and HYSYS for simulation of an entire SCWO-plant.

Oxidation reaction model

Initially the main functional groups and molecules in the sludge were determined using data found in literature and analysis performed on the sludge. Based on these studies, model molecules were derived, which represents the water work sludge.

The oxidation reactions of the model molecules were studied in order to establish a reaction model to be used in the reactor. This model mainly involves acetic acids and ammonia. The kinetic data for the non nitrogenous sludge were chosen as for sewage sludge, as no water work sludge kinetic could be found in literature.

The reaction model gave good results when compared with the experimental results achieved from the pilot plant.

Table 3. Metals in the dry fraction of the effluent.

	mg/kg DS
Ag	0.2
As	20
Bi	0.2
Cd	<0.06
Co	7.5
Cr	140
Cu	74
Fe	700 000
Hg	0.20
Mn	1 800
Mo	86
Ni	420
Pb	21
Sb	5.4
Se	6.7
Si	12 000
Sn	3.6
Tl	<0.1
Te	0.11
Zn	44
SUM	715 000

Simulation of the entire SCWO-plant

The findings from the oxidation reaction model were used in the HYSYS software to simulate the entire plant. The scale up was done with the assumption that the sludge will keep a dry substance of 15%.

According to the simulations, two separated oxygen injections will be needed along the length of the reactor to completely oxidize all the organic material without exceeding the thermal limit of the reactor material. The best option in terms of volume required for temperature stabilization is to use one cooling stage placed after the second oxygen injection.

The simulations show that the process would be self sufficient in terms of energy and that the reaction would produce enough energy to cover the overall heating demand of Ringsjöverket.

A problem which was encountered during the simulations was the low conversion of ammonia. The conversion of ammonia was, however, higher during the pilot tests which can be explained by the fact that sewage sludge hasn't got the same kinetics as water work sludge. The reaction pathway and kinetic of ammonia is also very hard to simulate.

The calculations did not take fouling and scaling problems into account and therefore represents an optimal steady-state of the process.

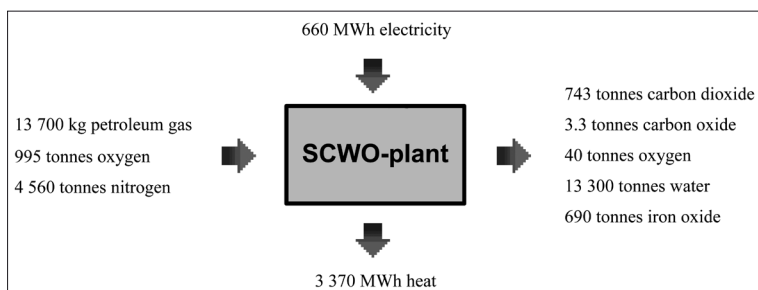


Figure 4. Material and mass balance for a full-scale SCWO-plant.

SUPER CRITICAL OXIDATION AT RINGSJÖVERKET, A CASE STUDY

Material and energy balance sheet

The results from the pilot tests have been recalculated to estimate the size, flow rates and costs of a full-scale SCWO-plant located at Ringsjöverket. The involved parameters are assumed to increase linearly.

The calculations are based on the design maximum capacity of Ringsjöverket (2 400 l/s). The sludge production is estimated to vary between 900–1 400 m³/h (15% Ds) with an average of 1 160 m³/h. The total running time for the SCWO-plant is expected to be around 7 500 hours per year. The COD in the sludge is assumed to be above 70 g/l.

Figure 4 describes the total flows for the parameters needed to establish the supercritical reaction in a full-scale SCWO-plant at Ringsjöverket.

A full-scale SCWO-plant also requires 4 560 m³ of nitrogen to dilute the effluent gas at start and stop. Approximately 5 200 kg of nitric acid will be needed every year for washing the gas heater and the economizer. Finally about 6 650 tonnes of water will be used for pressure reduction and cooling.

Iron oxide

According to the pilot tests about 690 tonnes of iron oxide can be extracted with a full-scale SCWO-plant every year.

This iron oxide can be used for making a new coagulant which can be reused at Ringsjöverket. The new coagulant can be created by dissolving the iron oxide in hydrochloric acid (HCl).

This is only possible if the produced iron chloride solution fulfils the regulations regarding metal content which is stated by the Swedish National Food Administration (SLV). There is also a European CEN-standard which divides the coagulant into three different levels, all are allowed to use for drinking water production. The threshold values are presented in table 4.

Table 5 shows the calculated metal content in a recycled iron chloride solution. The calculations are based on the analysis which was made on the iron oxide (table 3).

When compared to the National Food Administration all metals, except chromium, are present in concentrations below the threshold values stated by the National Food Administration. When compared to the CEN-standard the levels of both arsenic and nickel are above the limits for type 3.

Table 4. Threshold values (mg/kg active substance) for metal content in coagulants intended for drinking water production.

	Threshold values, SLV	CEN-standard type 1	CEN-standard type 2	CEN-standard type 3
As	10	2	2	2
Cd	1	0,1	2,5	5
Cr	20	5	35	50
Hg	0.5	0,03	0,5	1
Mn	–	500	1000	2000
Ni	–	6	35	50
Pb	10	3,5	10	40

Table 5. *Content of regulated metals in a recycled coagulant.*

Content in 1 liter recycled coagulant (mg)	
As	6.1
Cd	<0.019
Cr	43
Hg	0.064
Mn	547
Ni	131
Pb	6.6

Most of the nickel probably originates from corrosion of the SCWO-plant, especially from the pressure reducing capillary network which is built of steel with higher content of nickel than the rest of the plant. Most of the chromium originates from Lake Bolmen but a considerable part probably also comes from corrosion of the plant. The other parameters most likely originate mainly from the raw water.

Energy

The plant will generate approximately 3 370 MWh of heat every year which can be utilized for electricity or heat production. This could replace the current heat source which varies between oil, electricity and gas from a nearby landfill. During 2004 and 2005 the energy consumption for heating at Ringsjöverket was approximately 1 400 MWh.

Surveillance

A well adjusted SCWO-plant will when working properly have to be supervised via displays in a control room. Some personnel will still be needed for manual work and cleaning. Except for the personnel handling the sludge today approximately 0.5 fulltime posts more will be needed.

Dimensions

The building dimensions depend on example numbers, size of storage tanks and how the iron oxide will be handled. Double economizers will be necessary to make continuous operation possible and the reactor is estimated to be about 250–350 meters long and have a diameter of between one and four inches. With this in consideration a building area of 300 m² is a reasonable building size. (Nilsson, 2005)

Economy

The total investment cost for a full-scale SCWO-plant would be approximately 36 850 000 SEK, which with a depreciation time of 20 years and an interest of 4 % gives

Table 6. *Total costs (SEK) for a full-scale SCWO-plant at Ringsjöverket.*

	Total investment	Yearly cost
Capital costs		
Building	1 800 000	132 000
Aqua Critox	35 000 000	2 600 000
Concrete plate oxygen	50 000	4 000
Operational costs		
Oxygen		2 500 000
LPG		310 000
Nitrogen		37 000
Water		5 000
Electricity		390 000
Nitric acid		10 000
Staff		175 000
Saving income		
Heat		-1 252 245
Total	36 850 000	4 910 755

an yearly capital cost of about 2 700 000 SEK. The yearly moving costs will be about 3 427 000 SEK. All heating expenses (2005) at a cost of 1 252 245 SEK is produced from the SCWO-plant.

Table 6 summarizes the costs for a full-scale plant at Ringsjöverket.

The cost per cubic meter produced drinking water will be about 0.065 SEK, roughly 10 % of the operational costs including pumping.

The saving in heating costs is based on the electricity price during 2005. This parameter has shown a clearly increasing trend during the last years and is therefore an uncertain factor.

If the iron oxide can be used for producing a new iron chloride chemical further savings can be made. The yearly cost for iron chloride (2004) was about 2 100 000 SEK. If 50 % of the iron chloride can be replaced by a recycled coagulant about 1 000 000 SEK would be saved each year.

Another alternative is if the iron oxide could be sold as a pigment. The price for hematite varies between 10 and 20 SEK/kg depending on quality and amounts. The market for magnetite is smaller and the price varies between 30 and 80 dollars per ton (Nilsson, 2005).

DISCUSSION

The SCWO-technique is capable of treating on the sludge from Ringsjöverket. During the pilot test all of the organic material were oxidized and the inorganic material, mainly iron, precipitated. Unfortunately the

pilot plant had problems with handling the sludge and due to this the test had to be terminated after 33 hours.

The test shows problems with the ability to handle viscous sludge and fouling are the biggest problems with the technique. The levels of nickel and chromium in the iron oxide are also a considerable problem if the iron is supposed to be used for producing a new coagulant. Most of the nickel probably comes from corrosion of the pressure reducing part of the pilot plant while most of the chromium probably comes from the raw water and pilot plant.

If these problems can be solved and the costs can be lowered, the SCWO-technique would be an attractive alternative for treating water work sludge.

Oxidized organic matter

The most interesting property of the SCWO-technique is that all the organic matter can be oxidized at the same time as the inorganic matter precipitates. For a water work like Ringsjöverket this means that the raw material of the coagulant can be separated from the sludge at the same time as the organic material is fully oxidized. The two performed studies also show that the SCWO-method works with the iron-rich sludge from Ringsjöverket. This makes the technique to an interesting alternative for Sydvaatten who buys large amounts of iron chloride.

The SCWO-technique, however, is a method which so far commercially mainly has been used for purposes like recycling expensive catalysts and destroying chemical weapons. Therefore the number of SCWO-plants in the world today is quite limited and they are seldom in continuous operation for longer periods of time.

When it comes to treating water work sludge with SCWO the knowledge is very limited and there are several question marks and problems which need to be solved before a full scale SCWO-plant can be constructed.

One of the biggest problems is the impurity of the iron oxide with regards to nickel and chromium. The results so far indicate that it's not possible to recycle the iron oxide and make a new coagulant without adventuring the quality of the new product. An alternative can be to use the coagulant in a sewage treatment plant which hasn't got as strict threshold values for coagulants. The metal content in the liquid effluent also needs to be lowered before it can lead to a sewage treatment plant.

Problems with scaling occurred during the pilot tests which eventually led to the termination of the test after 33 hours. This problem will likely be smaller due to the bigger pipe diameter of the reactor in a larger plant, but still not insignificant.

Another big question with this method is, of course, the economy. The investment cost as well as the yearly cost is high even if the savings which can be made in heating up Ringsjöverket and recycling the coagulant are taken into account. To make this method to an economically reasonable alternative for Sydvaatten the iron oxide has to generate an income which can lower the annual costs more than recycling the coagulant would. This aspect makes it very interesting to find out if the iron oxide can be sold as for example a pigment.

Suggested future work

The SCWO-process needs to be tested during a longer period of time to get more results from analyses and to find out how the plant works when the SCWO-process has reached a steady state. Future tests should also be made in a bigger pilot plant to make the problems with fouling smaller. Tests with an oxygen dose which correlates to the dose which would be used in a full scale plant should also be performed.

Further investigations need to be done to find out if there is an interest to use the iron oxide for other purposes than producing a new coagulant.

A proper life cycle analysis needs to be done to enable a comparison of the SCWO-method with other alternatives.

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This report is a compilation and a summary of two master theses which were performed by Corinne Mallol and Magnus Nilsson in cooperation with Chalmers, Chematur Engineering AB, Feralco Nordic AB, Lund University and Sydvaatten AB during 2005. In June, 2007, Chematur Engineering Group sold their SuperCritical Water technology to the Irish enterprise Super Critical Fluids International. www.scfi.eu

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