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Pilot test operation of biological nitrogen removal of gold mill effluents

S. Morling[a,*], P. Moraeus[a], A. Unée[b]

[a]*Sweco Environment AB, P.O. Box 34044 S-100 26 Stockholm, Sweden*
Tel. +46 86966000; Fax: +46 86956290; email: stig.morling@sweco.se
[b]*Lappland Goldminers AB, Storgatan 36 S-921 31 Lycksele, Sweden*

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ABSTRACT

Effluents from gold mills are a potential environmental hazard, unless toxic substances such as nitrogen compounds (sodium cyanide, thiocyanate and ammonia) and heavy metals are removed. In northern Sweden, there are gold mines that are either already in operation, or to be opened, among them is the proposed Lappland Goldminers AB gold mill facility. The environmental permit for this new activity requires stringent removal levels of nitrogen. It was decided to operate a pilot plant to study the biological nitrogen removal performance. It has been based on a continuous activated sludge plant suited for biological nutrient removal. The plant has been operated at a neighbouring gold mill plant with an identical gold extraction process. The pilot study focused primarily on nitrification; however, also the denitrification performance and cyanide removal were monitored. The test results have been encouraging, showing up to a 100% nitrification capacity. The specific nitrification rate has been in the range (0.5) of 1.1–2.6 g N_{ox}/kg VSS/h, at temperatures ranging from 7.6 to 20°C. Different unintended process disturbances have been allowed for conclusions on the process stability and recovery potential. Low levels of total cyanide were recorded in the inlet, but virtually no traces were found in the treated water.

Keywords: Gold processing effluent; Biological nitrogen removal; Pilot plant operation; Load variations; Process stability

1. Introduction

The mining industry in Sweden has experienced a major recovery during the last decade. The international market for metals has developed in a very positive way for the industry, thanks to the demand for both construction metals and precious metals, mainly from the eastern hemisphere. As an example, the market price for gold over the last 10 years has increased by 294% and over the last five years by 179%. This in turn has resulted in a number of concessions for pilot- and full-scale activities. The mining enterprise Lappland Goldminers AB in Lycksele in northern Sweden has got the necessary environmental permit to open a gold mine as well as a gold extraction plant northwest of Lycksele town. The environmental permit stipulates high demands on the discharge of excess process water. The demands include for on *inter alia*, cyanide and ammonia nitrogen, as well as total nitrogen. These demands may be defined as rather new constraints for the mining industry. Traditionally, the use of sodium cyanide has been met with reluctance especially when the heap-leaching method has been

*Corresponding author.

used [1]. This model is not to be used at the Lappland Goldminers plant. A closed circuit model for the process water is foreseen. The focus on the different nitrogen compounds is related to the gold refining process, where the use of sodium cyanide is one of the important reagents. The end products of the process are *inter alia* thiocyanide and ammonia nitrogen. During the planning of the new facility for Lappland Goldminers AB, it was decided to investigate feasible models to treat the process wastewater from the gold extraction facility. Based on the results from the treatment of similar wastewater compositions, it was concluded that a biological treatment of the ammonia-rich wastewater would be possible and feasible. Examples from biological treatment with good results using trickling filter and rotating biological contactor have been reported [2]. The ability for a biological process to adsorb heavy metals and sustain a good microbial activity, even at demanding circumstances, was shown at the full-scale Sequencing Batch Reactor (SBR) plant in Nowy Targ, by treating influent wastewater with a high content of chromium (between 10 and 25 ppm) [3]. A typical application for biological treatment is found for coke oven plants [4–9]. These studies point out various limitations for a successful nitrogen removal. One obvious key factor is that the solids retention time is sufficiently high to allow for the acclimatization and safe nitrification. However, these studies focus on high temperature (25–38˚C) and high-strength wastewaters with respect to cyanides and thiocyanate. The studies point out the possibilities and constraints to run an activated sludge plant for nitrogen removal at specified conditions even on high-strength wastewater. As the neighbouring gold mill and extraction plant in Svartliden operates a similar process as the forthcoming one for Lappland Goldminers AB, it was concluded to run a pilot test at the Svartliden plant. The pilot plant facility is described in the following chapter. A pilot plant study was chosen rather than a laboratory-scale test facility, as a pilot plant is more likely to provide a better simulation of a full-scale plant. The variations in wastewater and other basic conditions may be studied and contribute to a more realistic evaluation. A laboratory-scale test on synthetic gold milling effluent showed that biological treatment is feasible [10].

2. Material and methods

As the Lappland Goldminers plant is still under planning, it was necessary to find a similar gold process plant with relevant discharge water qualities. The nearby Dragon Mine plant in Svartliden has a similar gold refining process. Effluent water from this plant was taken for analysis. Typical analysis results are presented in the next section.

The next step in the investigation was to run a pilot plant at the Svartliden plant. This test unit is based on a single-sludge activated sludge system, initially built for the development of nitrogen removal tests. It was used for a number of municipal projects in Scandinavia during the 1990s.

The pilot plant configuration is as follows:

- a mixing and flocculation compartment (not operated during the Svartliden tests);
- a pre-settling stage (not operated during the Svartliden tests);
- six units of bioreactors arranged in a series. The total reactor volume is 3.7 m^3. All reactors may be operated as aerobic or anoxic/anaerobic units by means of aeration or mixing. In the Svartliden tests, the plant was operated with the first two reactors with only mixing, while the remaining four reactors were aerated;
- a final clarifier for settling of activated sludge;
- return activated sludge (RAS) pumping is arranged with an eccentric screw pump with a variable capacity within the range of 0.1–0.8 m^3/h;
- the air supply is provided by a compressor;
- a dosing station with small membrane pumps is used to add phosphorus acid, an alkaline agent and ethanol in the Svartliden tests.

Phosphoric acid was added only twice during the operation period, in the mid of October when 12 mg phosphoric acid/h was added. For the rest of the operation period, no phosphorus was added.

Alkaline agent as $NaCO_3$ was added throughout the operation to prevent a pH drop due to nitrification. The dosage was directly proportional to the amounts of treated wastewater with a typical dose of 2.0 mL $NaCO_3$/L of wastewater treated.

Ethanol was added at an average of 0.17 ml/L of wastewater treated.

The untreated water is heated to 15˚C in order to mimic the forthcoming conditions at the Lappland Goldminers plant. The water temperature was recorded throughout the operation. The temperature variation is shown in Table 1.

The typical untreated process wastewater composition with respect to nitrogen compounds (NH_4–N, NO_3–N and N–tot) during the test period is presented in Table 2.

In Table 3, the other relevant pollutant variables in the untreated wastewater are presented. As only four analysis sets were performed during the test operation, the value range is presented in the table.

Table 1
Temperature variation during the test operation period of October–December 2006

Temperature (°C)
Number of observations: 24
Maximum: 24.1
Mean: 14.7
Median: 14.0
Minimum: 7.6

In Table 4, the pilot plant biological reactor dimensions and the theoretical hydraulic retention times (HRT) based on an influent flow of 400 L/h are presented. Table 5 shows the primary sedimentation and final sedimentation dimensions and hydraulic surface load at the same flow (400 L/h).

Fig. 1 shows a simplified process scheme of the pilot plant and sampling points.

The pilot plant was renovated and updated for the pilot operation in Svartliden.

The pilot plant tests were initially planned to cover a four-month period. During the test time, a successively increasing flow rate was planned. This would, principally, also increase the nitrogen load on the plant. The initial flow rate was 100 L/h and the final rate was 600 L/h. The objective with this operation mode was to identify the maximum nitrogen load level. The pilot operation was set into operation during the last week of September 2006. The initially intended capacity of 100 L/h was adjusted to 126 L/h, due to practical operational reasons.

The wastewater from the plant was taken downstream the tailing ponds. These ponds act along with the gold extraction process as transformers of the sodium cyanide. Low levels of cyanide were identified in the inlet process wastewater, typically <0.1 mg total Cyanide (CN).

For the analysis of the different nitrogen compounds (NH_4^+–N, NO_3^+–N and N–tot), a spectrophotometer, make HACH DR 2,800, was used at site. Although this equipment is not accredited, it was concluded that the advantage of obtaining rapid results was of paramount importance. Water samples were sent to an accredited laboratory for four times during the tests for the verification of the on-site results. A large majority of on-site results were confirmed by the laboratory analysis.

3. Results and discussion

In order to get a quick start of the pilot plant operation, the pilot plant was inoculated with nitrified sludge from a large municipal WasteWater Treatment Plant. A suitable plant was a little away from the site. Nevertheless, the start-up of the plant showed a continued nitrification which was inhibited when the pH dropped to pH < 5. The remaining alkalinity in the process water was consumed as the ammonia was rapidly oxidized. The need for the addition of an alkaline compound ($NaCO_3$) is presented in Section 2.

An alteration of the process mode was decided: the addition of alkali as well as organic carbon was started. The objective for the operation was accordingly shifted—the new operation mode would also give information on the denitrification capacity.

The operation mode for the plant thus was adjusted as follows:

- principal operation mode: a classical recirculation nitrifying/denitrifying activated sludge system was established;
- addition of external carbon source for pre-denitrification, by means of ethanol (typical dose as presented in Material and Methods), using the two first reactors as anoxic units, totally 1.0 m^3 reactor volume.

Table 2
Typical composition of the nitrogen compounds during the test period, inlet values

Period September–December 2006	NH_4^+–N In		NO_3^+–N In		N–tot In	
	mg/L	kg/d	mg/L	kg/d	mg/L	kg/d
Number of observations	25	25	25		24	24
Maximum value	59	0.61	1.9	<0.1	66	0.68
Average	43.7	0.3	0.1	0	49.1	0.3
Median value	47	0.28	0	0	52.5	0.34
Minimum value	27	0.05	0	0	30	0
Standard deviation	10.5	0.15	0.4	0	11.0	0.17

Table 3
Typical range of pollutant values during the test period, inlet values

Variable	Value
COD	17–33 mg/L
Tot CN	0.02–0.29 mg/L
Fe	0.161–0.368 mg/L
As	43.5–123 μg/L
Cd	<0.05–0.06 μg/L
Cr	<0.5–1.39 μg/L
Cu	42.5–123 μg/L
Pb	0.89–3.92 μg/L
Sb	1.18–2.56 μg/L
Zn	46.9–102 μg/L

Aeration for nitrification took place in the remaining four reactors, total volume of 2.7 m^3.

The actual flow loads could largely follow the planned program. However, it was even possible to increase the hydraulic load as the process performance showed good development. Fig. 2 demonstrates the flow variation into the plant during the test period. It also demonstrates the variation of the RAS flow during the same period.

The corresponding reactor performance with respect to nitrification during the test period is shown graphically in Fig. 3. It demonstrates that the nitrification was affected at four different times during the test operation. These disturbances during the test period have been identified as presented below:

(1) October 5
Nitrification efficiency went down to around 90%. At the same time, the pH was dropped to 6.0, a possible result of insufficient alkalinity in the bioreactors. Two different actions were taken simultaneously: the addition of alkali ($NaCO_3$) was increased and the addition of ethanol started to enhance a partial denitrification.
(2) October 7–19
Nitrification efficiency dropped to 60–70%. This was probably caused by a low pH in the reactor, around 5.0. The most likely reason for this was a temporary stoppage of the addition of $NaCO_3$ due to clogging in the dosage pipe. As may be seen from the figure, it took a couple of days before the nitrifiers were in a good shape again.
(3) November 10–14
Nitrification efficiency dropped to 65–70%. On the November 10, the ethanol dosage was very high in relation to the actual needs. This in turn limited the free oxygen level in the entire reactor system to <2 mg O_2/L. The available oxygen amount was evidently not sufficient to safeguard a full nitrification. The ethanol dosage was accordingly lowered the same day. On the following control day, the free oxygen level had been reestablished at around 3 mg O_2/L in the aeration reactors. However, the water temperature was dropped to 7.6°C due to the breakdown of the heating system. The matter was mitigated by a new type of heating system. As seen in the figure, the nitrification after these actions recovered swiftly.
(4) From the December 11 until the end of the test periodAt the end of the test period, the nitrification level decreased to 60–80%. No ethanol was added to the process at the end of the test. At the same time, the nitrogen load was increased due to a rather high flow and a higher concentration of nitrogen in the untreated water.

Apart from these situations with identified disturbances, the nitrification has been complete, close to 100%. The recovery of complete nitrification in the reactors has also been swift as shown in the diagram Fig. 4.

Even though the main objective of the test was to find out the nitrification capacity, the denitrification was also possible to record. In Fig. 5, the total nitrogen at the inlet and outlet expressed as mg total N/L is shown. The figure demonstrates that the removal of total N could reach around 70% at stable conditions.

Suspended solids concentration in the reactor system has been within the range of 2,238–3,522 mg

Table 4
Pilot plant bioreactor dimensions; the HRT is based on a flow = 400 L/h

Bioreactor no.	Totally	Volume 1	Volume 2	Volume 3	Volume 4	Volume 5	Volume 6
Length (m)	3.3	0.5	0.5	0.7	0.6	0.6	0.5
Width (m)	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Height (m)	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Volume (m^3)	3.7	0.5	0.5	0.8	0.7	0.7	0.5
HRT (m)	9.25	1.25	1.25	2.0	1.75	1.75	1.25

Table 5
Pilot plant dimensions for primary and final sedimentation units; overflow rates are based on an influent flow of 400 L/h

	Primary sedimentation	Final sedimentation
Diameter (m)	0.85	0.99
Area (m^2)	0.57	0.77
Hydraulic surface load (m/h)	0.70	0.52

SS/L. Table 6 shows the statistical data for SS concentration during the operation period.

The ratio VSS/SS was assumed to be 0.7:1 as an average during the operation. Based on the performance, the following nitrification rates were calculated:

The variation of nitrification rate based on all occasions (25 nos) ranged from 0.5 to 2.6 mg N/h/g VSS, with an average rate of 1.25 mg N/h/g VSS. It should be underlined that low nitrogen loading of the plant resulted in complete nitrification (effluent values

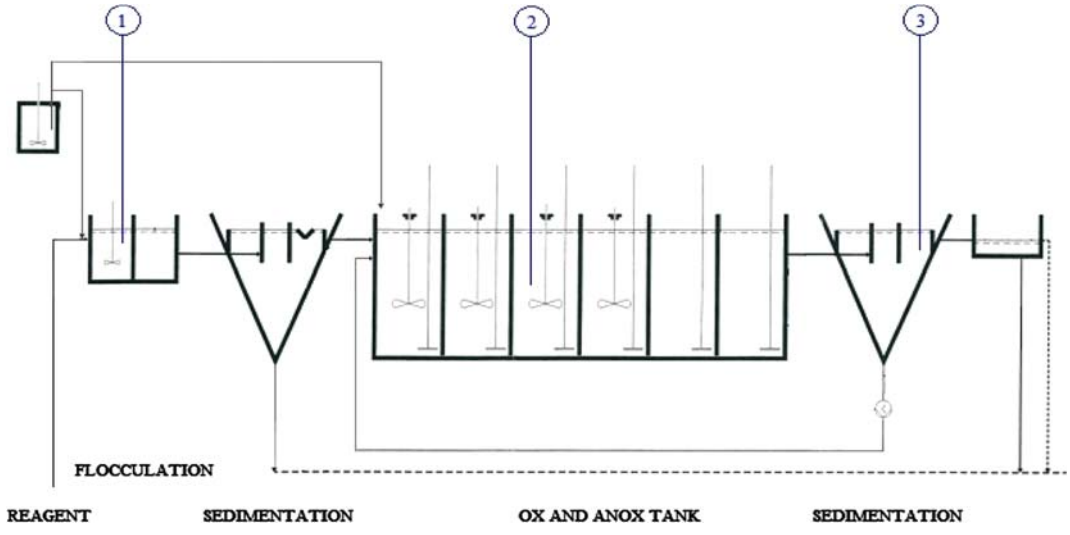

Fig. 1. Flow sheet of the pilot plant facility.

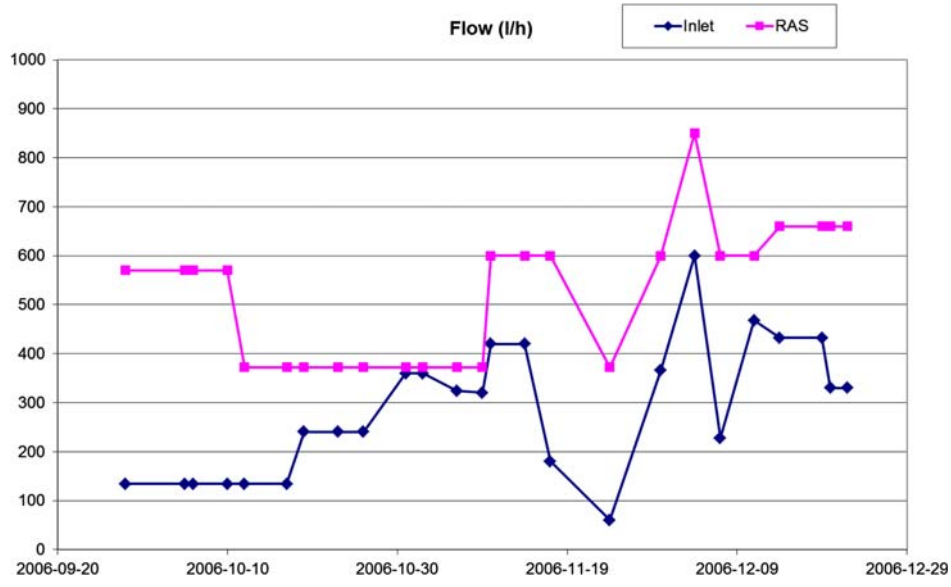

Fig. 2. Variations in inlet flows and RAS flows during the pilot operation.

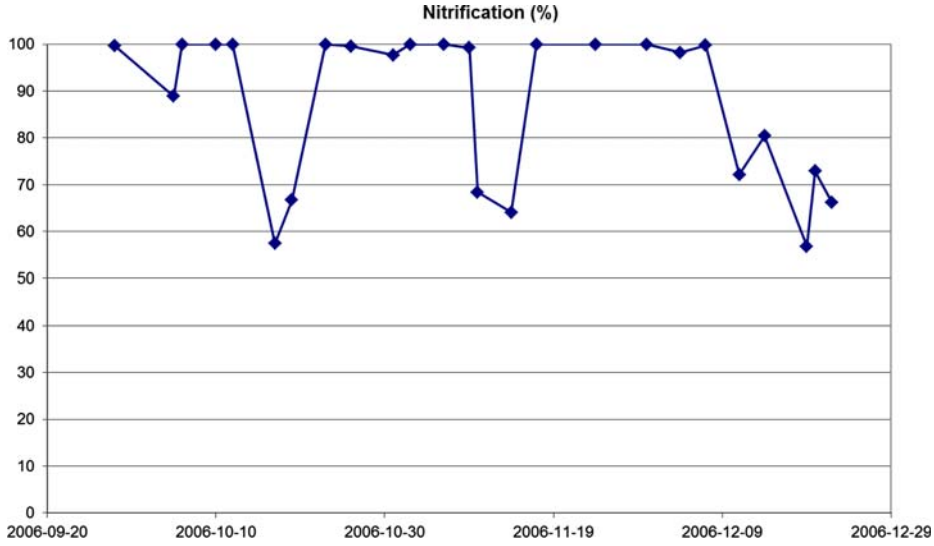

Fig. 3. Variation in nitrification level during the test period.

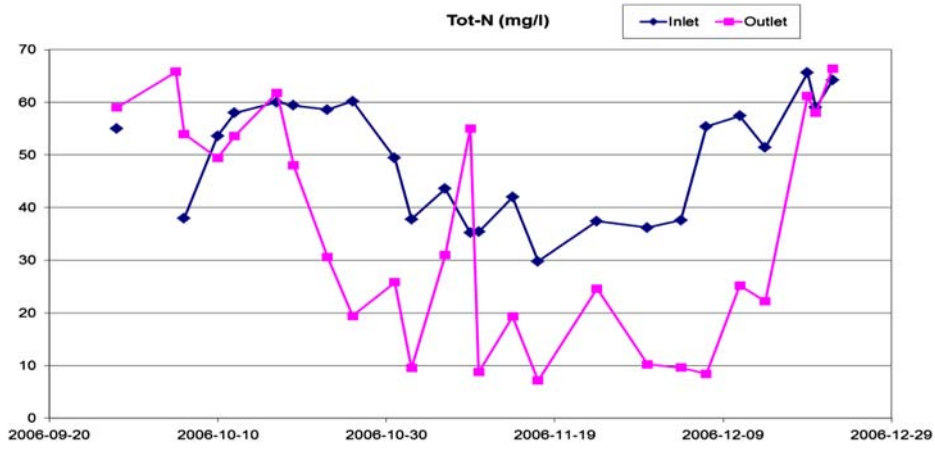

Fig. 4. Total nitrogen inlet and outlet concentrations during the test operation.

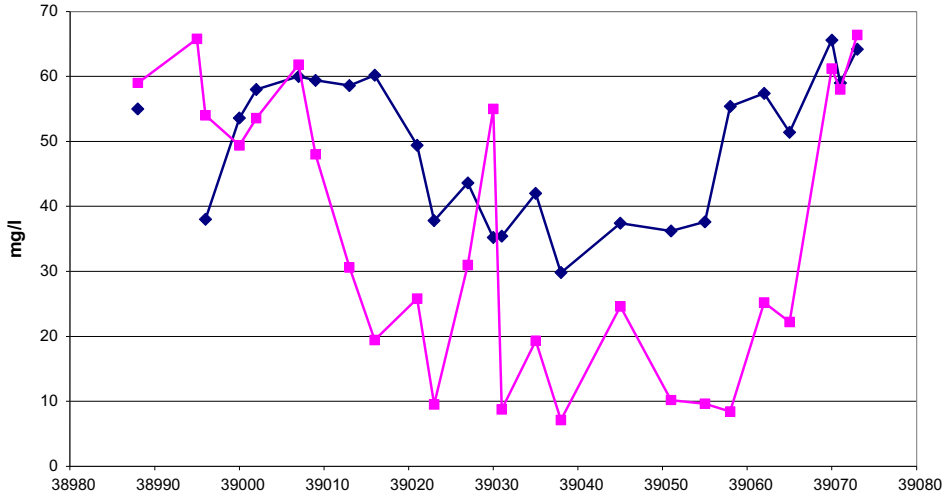

Fig. 5. Inlet and outlet nitrogen concentrations during test period.

Table 6
SS concentration in the reactor during the operation

Number of observations	25	nos
Maximum value	3,522	mg SS/L
Mean value	3,207	mg SS/L
Median value	3,300	mg SS/L
Minimum value	2,238	mg SS/L
Standard deviation	319	

close to 0 mg NH_4^+–N/L). This in turn means that the actual nitrification rate may have been higher. By excluding the observations when complete nitrification was found, the minimum nitrification rate was 1.1 mg N/h/g VSS. The identified disturbances with respect to nitrification were not related to high-strength nitrogen in the untreated wastewater. As described above, "practical" operation failures were the main cause for the drop of nitrification efficiency.

In Table 7, the amount of oxidized ammonia is shown along with the specific nitrification rate for all observations during the period.

A comparison with other studies on nitrification performance where the wastewater composition is regarded as hazardous in relation to nitrification is given below:

A follow up study of a full-scale SBR plant treating leachate at a Swedish land fill site where the water temperature was kept at +15˚C demonstrated a nitrification rate ranging from 0.9 to 1.9 g N_{ox}/kg VSS/h. The actual rate was mainly related to the actual nitrogen loading of the plant. Another Swedish leachate treatment facility, also based on SBR technology showed a variation in the nitrification capacity from 0.5 to 1.1 mg $N_{oxidised}$/g VSS/h [11]. In this latter case, the plant does not operate with the temperature control of the process water.

The reduction of Chemical Oxygen Demand (COD) has been insignificant, which may be related to the addition of organic carbon (ethanol). No analysis of COD fractions was done, and the SS concentration in treated water was not followed. On the other hand, the sludge volume and SS concentration in the reactors were measured and recorded regularly. The calculated Sludge Volume Index (SVI) levels were consistently high throughout the test period. The variation is shown in Table 8.

Based on the high SVI values and visual observations during the test operation, it is more than likely that a rather high SS concentration was occurred in the effluent. This in turn would explain the insignificant COD reduction, as well as the discharge levels of phosphorus between 0.3 and 6.4 mg total *P*/*L*. The untreated wastewater had a very low total *P* concentration < 0.2 mg/L.

The heavy metal content in the inlet wastewater was recorded four times during the tests. Good removals of copper, lead and zinc were found. Other hazardous metals such as arsenic and cadmium remained more or less unchanged in terms of concentration. Table 9 summarizes the discharge variation of heavy metals from the plant. The copper content in the biological sludge was found at high levels of about 1,360 mg/kg TS.

Table 8
SVI variation in the reactor during the operation

Number of observations	24	nos
Maximum value	211.0	mL/g
Mean value	168.5	mL/g
Median value	168.2	mL/g
Minimum value	100.0	mL/g
Standard deviation	28.7	

Table 7
Nitrification performance and specific nitrification rate throughout the test period

	NH_4^+–N In		NH_4^+–N Out		NH_4^+–N Red. rate amounts/nitrification	
	mg/L	kg/d	mg/L	kg/d	kg/d	mg N/g VSS h*
Number of observations	25	25	25	25	25	25
Maximum value	59	0.61	25,3	0.26	0.46	2.6
Average	43.68	0.30	6.20	0.05	0.24	1.25
Median	47	0.28	0.2	0	0.27	1. 2
Minimum value	27	0.05	0	0	0.05	0.2
Standard deviation	10.5	0.15	8.4	0.07	0.11	0.67

Table 9
Typical range of pollutant values during the test period, discharge values

Variable	Value
COD	19–72 mg/L
Tot CN	0.0 mg/L
Fe	0.18–1.3 mg/L
As	26.9–114 μg/L
Cd	<0.05–0.103 μg/L
Cr	<0.5–0.693 μg/L
Cu	14.1–35.1 μg/L
Pb	<0.2–0.434 μg/L
Sb	1.22–2.36 μg/L
Zn	7.33–31.5 μg/L

4. Conclusions

The test plant has been operated during a three-month period, from the last week of September until the 22 December. The main task for the study was to investigate whether it was feasible for an ammonia-rich process wastewater from a gold extraction to be treated by a biological nitrogen removal process. The test demonstrated that it was possible to perform a complete nitrification of the ammonia nitrogen. It does not seem likely that the presence of heavy metals or the remaining CN levels have caused a significant inhibition of the nitrification.

The test also demonstrated that it is possible to achieve a good denitrification, provided that a suitable carbon source is added. Although the other nitrogen fractions were low in incoming water, it was also found that these were either transformed or reduced. The CN content in the incoming water was completely removed by the biological process.

An important finding from the test is that the process demonstrated a stable performance, with a capacity to rapidly overcome disturbances, caused by pH-drop, temperature fall or lack of organic carbon.

A potentially high nitrification rate was found. During stable operation, this rate was found to be in the range of 1–2.6 mg NH_4^+–N/g VSS/h. The actual nitrification rate was comparable with what has been found in biological leachate treatment plants at similar conditions.

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