Simultaneous Treatment of Thiocyanates and Ammonia Nitrogen in Gold Mine Effluents Using Advanced Oxidation and Nitrification-Denitrification Processes

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Abstract Efficient treatment of gold mine effluents, with simultaneous removal of cyanides (CN^-) and their derivatives, including thiocyanates (SCN^-) and ammonia nitrogen (NH_3-N), remains a challenge. The present study assessed the removal efficiency of SCN^- and NH_3-N in gold mine effluents using an advanced oxidation process (ferrates) and a biological nitrification-denitrification (nit-denit) process. Results showed almost complete removal of SCN^- (>97%) using the two processes together. However, ferrates rapidly removed the SCN^-, NH_3-N concentrations increased up to 117 mg/L. Consequently, ferrate treatment alone was found inadequate to completely remove NH_3-N, which required a nit-denit process with a longer reaction time.

Keywords Ammonia nitrogen, ferrates, gold mine effluents, nitrification-denitrification, thiocyanates.

Introduction

Gold and silver extraction methods generate effluents that are contaminated with cyanides (CN^-), as well as with thiocyanates (SCN^-) (in sulfidic ores and at high alkalinity). Effective treatment technologies to remove CN^- are available, but they cannot simultaneously remove SCN^-. They also produce toxic by-products, such as ammonia nitrogen (NH_3-N) (Botz et al. 2005; Gould et al. 2012). Although SCN^-, NH_3-N, and nitrates (NO_3^-) are less toxic than CN^-, they are difficult to treat and environmentally persistent (Gould et al. 2012). Furthermore, excesses of NH_3-N and NO_3^- in receiving water can lead to eutrophication and acidification, thus resulting in subsequent toxic effects in aquatic ecosystems. Complementary treatment is therefore required after CN^- removal.

Recent findings indicate that advanced oxidation and biological processes transform SCN^- into sulfates (SO_4^{2-}) and intermediate by-products, such as NH_3-N, which is subsequently oxidized into NO_3^- (Gould et al. 2012; Oulego et al. 2015; Villemur et al. 2015). The advanced oxidation process uses strong oxidants, such as ferrate [Fe(VI)], which is also a coagulant, and is widely used to treat drinking water and wastewater (Yates et al. 2014; Goodwill et al. 2016). Recent studies showed that Fe(VI) is also an advantageous alternative for mine effluent treatment because it is environmentally friendly when transformed into Fe(III), which

is a non-toxic by-product (Waite 2015). The Fe(VI) can be synthesized through either a chemical or electrochemical process to produce dry Fe(VI) (K_2FO_4) or wet Fe(VI) (Na_2FeO_4) (Thompson et al. 1951).

Another economical option for SCN^-, NH_3-N, and NO_3^- removal is biological treatment. This alternative involves the use of bioreactors, such as the moving bed biofilm reactors (MBBRs), which provide optimal conditions for microorganism growth at a defined hydraulic retention time (HRT) (Kim et al. 2011; Villemur et al. 2015). Microorganisms (both chemolithotrophic and autotrophic) can be used to transform SCN^- into NH_3-N and SO_4^{2-} using SCN^- as an energy source. NH_3-N and NO_3^- can be treated by a nitrification-denitrification (nit-denit) process whereby NH_3N is oxidized into NO_3^- by nitrification and the NO_3^- is subsequently reduced to N_2 gas by denitrification (Jermakka et al. 2015).

The Fe(VI) and nit-denit processes could therefore provide a potentially advantageous SCN^- and NH_3-N treatment option (Sharma 2011; Waite 2015; Villemur et al. 2015; Gonzalez-Merchan et al. 2016). However, little is known about the oxidation by-products of SCN^- and NH_3-N, when these contaminants are simultaneously treated by either the Fe(VI) or biological nit-denit processes.

In this context, the objective of the present study was to assess the removal efficiency of SCN^-, NH_3-N, and NO_3^- using the Fe(VI) and biological nit-denit processes and to compare their performances.

Materials and methods

Site description and sampling

The performances of the Fe(VI) and nit-denit processes were assessed with an effluent collected from a gold mine treatment plant located in the province of Québec, Canada. The treatment was performed in two steps: first, the CN^- was removed by chemical oxidation (i.e., the Degussa process), and second, the SCN^- and NH_3-N were treated by a biological process. The treatment plant is described in detail by Laporte (2015) and Villemur et al. (2015). In the present study, the effluent was sampled at the inlet to the biological process where CN^- was absent. The effluents for the Fe(VI) treatability tests were collected in June 2015, and the effluents for the pilot-scale nitdenit process were collected every two weeks over a six-month period from July to December 2015. After characterization, the mean and standard deviation were calculated (n = 40) for all physicochemical parameters (Tab. 1).

Table 1 *Physicochemical composition of the real gold mine effluent (in mg/L, except for pH)*

Parameter	pH	Eh (mV)	DO	SCN^-	OCN^-	SO_4^{2-}	NH_3-N	NO_2^-	NO_3^-
Value	7.5 – 8.4	325±38	8.7±1.1	435±53	54±1	2 362±320	41±8	5.2±3.1	65±37

Mean ± standard deviation

Batch treatability testing with Fe(VI)

The treatability tests were performed with wet Fe(VI), which was synthesized as described by Thompson et al. (1951), Ciampi et al. (2009), and Gonzalez-Merchan et al. (2016). The preparation required an appropriate oxidant and alkaline conditions to ensure Fe(VI) stability (Eq. 1).

$$2\,Fe(NO_3)_3 + 3\,NaOCl + 10\,NaOH \rightarrow 2\,Na_2FeO_4 + 3\,NaCl + 6\,NaNO_3 + 5\,H_2O \quad (1)$$

The treatability tests were performed using a jar test with a one-hour reaction time. The assessed Fe(VI) doses were 100, 200, 300, 350, 400, and 500 mg/L. More details on the treatability testing with ferrates are provided elsewhere (Gonzalez-Merchan et al. 2016).

Treatability testing with a nit-denit process using a pilot-scale system

The nit-denit process was tested in the laboratory using a pilot-scale system over a 140-day period under continuous flow, which was ensured with six pre-calibrated peristaltic pumps (Masterflex), at room temperature (15–25°C). For each reactor, the effective volume was 17.5 L (Fig. 1). The HRT varied from 5.5 to 10 hours for reactors A to D, whereas the HRT varied from 8 to 14 hours for reactor E.

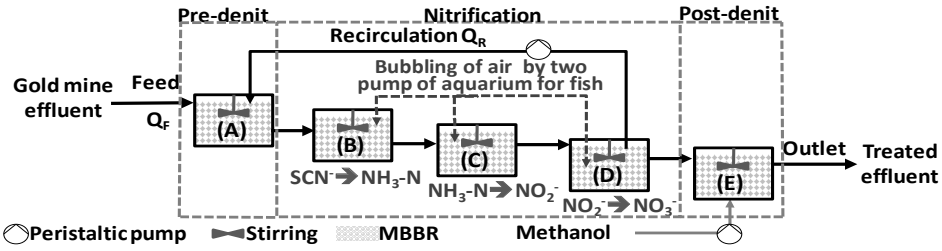

Figure 1 *Pilot-scale system with continuous flow*

During the nitrification process, pH was maintained at ~7.5 using a metering pump to add soda ash (Na_2CO_3). Two air pumps per container were also installed to maintain the aerobic conditions, and the stirring agitators were set at 200 RPM. Reactor A (pre-nitrification step) was fed with nitrified effluent from reactor D and gold mine effluent, with the dilution factor varying from 1.4 to 1.8. These dilution factors were estimated considering the feed (Q_F) and recirculation (Q_R) flows (Eq. 2). In reactor E (post-denitrification step), methanol (99%) was used as an external carbon source (Fig. 1).

$$Dilution\,factor = \frac{Q_F - Q_R}{Q_F} \times 100 \quad (2)$$

Physicochemical and microbiological characterization

Physicochemical parameters of the effluent, including pH, redox potential (Eh), dissolved oxygen (DO), NH_3-N, nitrites (NO_2^-), NO_3^-, SO_4^{2-}, cyanates (OCN^-), and SCN^-, were analyzed before and after each treatment step. The analysis methods for all physicochemical

parameters are described in detail in Gonzalez-Merchan et al. (2016) and Tanabene (2016). The presence of different functional groups of nitrifying and denitrifying microorganisms was verified by polymerase chain reaction (PCR) amplification. Bacterial biofilms were then harvested from the carriers at 0.3 g (wet weight) and used for DNA extraction with a MoBio Power Soil DNA extraction kit following the manufacturer's protocol. In the present study, the genetic markers used included the genes *amoA*, *nirK, nirS*, *norB,* and *nosZ* (Rotthauwe et al. 1997; Braker et al. 1998; Geets et al. 2007).

Data processing

Treatment performance was assessed according to the oxidation efficiency of SCN^-, NH_3-N, NO_2^-, and NO_3^-, as calculated with Eq. (3), where Ci and Cf are the concentrations before and after each treatability test. Efficiencies were considered when the calculated value was > 0.

$$Efficiency\,(\%) = \frac{Ci - Cf}{Ci} \times 100 \qquad (3)$$

Results and discussion

In gold mine effluent, SCN^- concentrations decreased with increasing Fe(VI) doses, whereas in the nit-denit process, SCN^-, NH_3-N, and NO_3^- were removed at HRT ≥ 8 hours. The evolutions of treated effluent quality using the Fe(VI) and nit-denit processes are discussed below.

SCN- and NH_3-N oxidation with Fe(VI)

Fe(VI) preparation requires strong alkaline and oxidizing conditions (Thompson et al. 1951; Ciampi et al. 2009). These conditions were present in the treated effluents, which showed high pH and Eh values (up to ~12 and ~515 mV, respectively; Tab. 2). Even though the Eh increased, high pH values were maintained after effluent treatment. These findings could be explained by the excess NaOH used to prepare the Fe(VI) (Eq. 1). These high Eh values are consistent with previous studies, where the values were ~700 mV under alkaline conditions (Sharma 2011).

Table 2 *Physicochemical composition of effluents treated with Fe(VI)*

Fe(VI) (mg/L)	pH	Eh (mV)	NH_3-N (mg/L)	OCN^- (mg/L)	SO_4^{2-} (mg/L)	NO_3^- (mg/L)	SCN^- (mg/L)	Efficiency (%)
Raw	10.1	291	85	-	2 396	67	475	-
100	12.9	449	94	124	2 607	1049	338	29
200	13.1	479	96	143	2 720	1 542	204	57
300	13.2	500	99	159	2 805	2 180	130	73
350	13.2	508	97	167	2 847	2 321	116	76
400	13.3	509	116	174	2 889	2 857	59	88
500	13.3	515	117	169	2 903	3 323	5	99

The strongly oxidizing conditions of the Fe(VI) treatment decreased the initial SCN^- (475 mg/L) concentrations, whereas SO_4^{2-} increased with increasing Fe(VI) doses. The final SO_4^{2-} concentration was 1.2 times higher in the treated vs. the raw effluent. This result was interpreted in the sense that SCN^- was transformed into SO_4^{2-}, as corroborated by previous findings (Sharma et al. 2002).

In addition, SCN^- oxidation (~99%) was confirmed by the formation of NH_3-N and NO_3^-, with concentrations increasing from 85 to 117 mg/L and from 67 to 3 323 mg/L, respectively. At the same time, OCN^- concentrations rose to 174 mg/L, whereas NO_2^- concentrations were below the detection limit. The absence of NO_2^- indicates that in the presence of a strong oxidant, such as Fe(VI), the NO_2^- was rapidly transformed into NO_3^-, as demonstrated previously by Sharma et al. (1998). Consequently, the SCN^- oxidation produced intermediate by-products, such as OCN^-, which was hydrolyzed into NH_3-N, and subsequently oxidized into NO_3^- (Sharma et al. 2002; Oulego et al. 2014). In the present study, the very high NO_3^- concentrations could be related to NH_3-N oxidation as well as the $Fe(NO_3^-)$ that was used as the Fe(III) source for Fe(VI) preparation (Eq. 1). Although the SCN^- was successfully treated, these results indicate that the reaction time was too short to allow complete NH_3-N removal. This can be attributed to the slow oxidation kinetics of NH_3-N in the presence of SCN^- (Sharma et al. 1998; Gonzalez-Merchan et al. 2016).

These results justify the need for new processes in Fe(VI) treatment for NH_3-N and NO_3^-.

Removal efficiency of SCN-, NH_3-N and NO_3- using the biological nit-denit process

A pilot-scale system was used to assess the nit-denit process, in which the concentrations of SCN, NH_3-N, and NO_3^- decreased with increasing dilution factor and HRT duration (Tab. 3). In the pre- and post-denit steps, the conditions were anoxic, as confirmed by the low DO concentrations (< 0.5 mg/L), and the pH values varied from 6.4 to 9.2. In contrast, for the nitrification process, conditions were aerobic (as reflected by DO > 5 mg/L), with consistent alkaline pH values of 7.5 due to the presence of bubbling air and the Na_2CO_3 which was added to control the pH. In this nitrification process, the activity of microorganisms requires the presence of DO as well as pH regulation (Lay-Son et al. 2008; Kim et al. 2011). These results are also related to the Eh values, which were higher for the nitrification than for the pre- and post-denitrification steps (> 70 mV vs. < -50 mV). Thus, the aerobic conditions during the nitrification process could be appropriate for SCN^-, NH_3-N, and NO_2^- oxidation, whereas during the pre- and post-denitrification processes, the anoxic conditions could have contributed to the NO_3^- reduction, as indicated by previous findings (Kim et al. 2011; Lay-Son et al. 2008; Villemur et al. 2015).

In the present study, the biological nit-denit process was performed in three main steps: First, in reactor A (pre-denitrification), at HRT ≥ 8 hours, the initial SCN^- concentrations were removed (> 53%) at all dilution factors. However, at HRT = 5.5 hours, SCN^- removal was significantly related to the dilution factor (Tab. 3). For example, at dilution factors of 1.5 and 1.6, the removal efficiency increased from 37 to 54%. Despite these differences, the SCN^- concentrations were on average ~1.6 times higher in the feed than in reactor A at a dilution

factor of 1.5. Moreover, the increasing NH_3-N concentrations (from 41 to 83 mg/L) reflected the SCN^- oxidation in reactor A. These results confirmed that the contaminants were diluted and that SCN^- can be oxidized with high HRT and dilution factors. Consequently, as shown by Kim et al. (2011), pre-denitrification contributes mainly to reducing contaminant concentrations. However, in reactor A, NO_3^- concentrations were only slightly diminished (< 20%), and the concentrations actually increased at a dilution factor of 1.8, because the recycled effluent (from reactor D; Fig. 1) produced strong concentrations (up to 450 mg/L). Second, the nitrification process involved the oxidation of SCN^-, NH_3-N, and NO_2^- in reactors B, C, and D, respectively. In reactor B, SCN^- was efficiently removed (> 77%) at HRT ≥ 8 hours, whereas the removal efficiency for SCN^- was very low at 5.5 hours HRT (~37%). Nevertheless, at the end of nitrification (reactor D), SCN^- concentrations were lower than 11 mg/L for all conditions. The low concentrations of SCN^- were due to the oxidation of SCN^- to SO_4^{2-}, thus increasing SO_4^{2-} concentrations up to a maximum of 3 250 mg/L. Hence, in reactor B, the microorganisms used SCN^- as an energy source to transform SCN^- into SO_4^{2-}, which was confirmed by the functional groups of nitrifying bacteria detected in the MBBRs, in agreement with previous findings (Villemur et al. 2015). In contrast, the NH_3-N oxidation in reactor C was adversely affected by the short reaction time, with an HRT of 5.5 hours, and low dilution factors. For example, the removal efficiencies for NH_3-N were lower at 5.5 hours HRT than at ≥ 8 hours HRT (< 15% vs. > 60%). In fact, at low HRT, the slow oxidation kinetics for the NH_3-N oxidation resulted in the accumulation of this contaminant, with NH_3-N concentrations exceeding 83 mg/L. These high NH_3-N concentrations could have caused an inhibitory effect, as previously demonstrated by Lay-Son et al. (2008). However, the NO_2^- concentration was 1.2 times higher in reactor C compared to reactor B, confirming that NH_3-N was transformed into NO_2^- in the presence of aerobic conditions. In reactor C, the microorganisms' activity was confirmed by the presence of ammonia monooxygenase genes. Afterwards, in reactor D, NO_2^- was oxidized into NO_3^-. The NO_3^- concentrations were 1.5 times higher in reactor D than in reactor C, when NO_2^- decreased (Tab. 3), except at a low HRT (5.5 hours) and dilution factor (1.5) in which case NO_2^- increased. Despite these differences, the results clearly indicated that nitration was achieved, because the NO_2^- was efficiently oxidized into NO_3^- (> 69%). In addition, the presence of nitrite reductase genes explains the NO_2^- oxidation.

Finally, in the post-denitrification step, NO_3^- was satisfactorily removed (> 80%) under all dilution factors and HRTs. Thus, the denitrification process was successful, as confirmed by the presence of N_2O reductase (*nosZ*) in the MBBRs. Although the reaction time was longer in the nit-denit process than in the Fe(VI) process (8 hours vs. 1 hour), NH_3-N and NO_3^- were efficiently removed.

Conclusion

For the simultaneous treatment of SCN^- and NH_3-N, the performances of the Fe(VI) and nit-denit processes were assessed and compared. Results showed that the coupling of both processes almost completely removed the SCN^- (> 97%). Nevertheless, in effluents treated with Fe(VI), the NO_3^- and NH_3-N concentrations increased (from 67 to 3 323 mg/L and from 85 to 117 mg/L, respectively). The nit-denit process successfully transformed the NH_3-N and

Table 3 *Variation of SCN-, NH_3-N, NO_2-, and NO_3- concentrations (mg/L) with respect to dilution factor and HRT (hours)*

Reactor	Dilution factor – HRT	SCN^-	NH_3-N	NO_2^-	NO_3^-	Efficiency (%)			
						SCN^-	NH_3-N	NO_2^-	NO_3^-
A	1.4 – 10	197±114	58±9	5±2	40±23	53	-	-	20
	1.5 – 5.5	302±71	76±26	4±1	9±7	37	-	-	79
	1.6 – 5.5	210±86	83±14	4±2	38±22	54	-	28	22
	1.8 – 8	104±77	63±24	5±2	63±38	77	-	28	-
B	1.4 – 10	45±24	21±17	264±75	18±14	77	64	-	54
	1.5 – 5.5	149±110	6±2	2±1	16±11	51	-	59	-
	1.6 – 5.5	102±60	94±20	11±22	31±22	51	-	-	15
	1.8 – 8	3±1	51±14	89±34	24±12	98	20	-	10
C	1.4 – 10	10±5	0.7±0.4	9±7	424±62	77	97	97	
	1.5 – 5.5	21±14	83±53	27±5	136±104	86	15	-	-
	1.6 – 5.5	2±1	88±36	52±39	46±28	98	6	-	-
	1.8 – 8	0.3±0.2	4±1	92±73	256±137	88	91	-	-
D	1.4 – 10	10±5	0.3±0.3	0.5±1.2	449±71	-	55	95	-
	1.5 – 5.5	1.4±0.9	63±49	32±16	153±109	93	25	-	-
	1.6 – 5.5	0.7±0.1	81±35	16±12	131±55	69	9	69	-
	1.8 – 8	0.3±0.5	2±1	24±48	315±92	0	60	74	-
E	1.4 – 13	12±7	2±1	3±2	39±26	-	-	-	91
	1.5 – 8	1.5±0.6	78±65	9±12	16±12	-	-	72	90
	1.6 – 10	0.4±0.2	85±31	2±1	30±20	37	-	87	80
	1.8 – 14	6±2	4±3	2±3	12±5	-	-	92	96
Total removal efficiency (feed to E)	1.4		99			97	-	97	95
	1.5		-			-			
	1.6		99			-			
	1.8		99			93			
			-						
			99						
			99						
			93						
			99						

Mean ± standard deviation

NO_3^-, as confirmed by the detection of ammonia-oxidizing bacteria. While a 1-hour reaction time was required for almost complete SCN^- removal using Fe(VI), optimal removal with the nit-denit process was achieved in 8 hours. NH_3-N and NO_3^- were efficiently removed using the nit-denit process, although the process required a lengthy reaction time. Finally, in the presence of Fe(VI), NH_3-N oxidation was incomplete and NO_3^- could not be removed, while effluents treated with Fe(VI) needed additional pH adjustment.

Acknowledgements

This study was funded by the Natural Sciences and Engineering Research Council of Canada (NSERC), the industrial partners of RIME UQAT-Polytechnique (Agnico Eagle, Canadian Malartic Mine, Iamgold Corporation, Raglan Mine-Glencore, and Rio Tinto), and Mabarex. The authors gratefully acknowledge the assistance of Marc Paquin during the experimental procedures.

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