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Partial nitritation of nitrogen-rich refinery wastewater (sour water) with different C_i/N molar ratios

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ABSTRACT

In this study, a SHARON reactor was used to treat synthetic and real ammonium-rich refinery wastewater (sour water) with different inorganic carbon to nitrogen (C_i/N) molar ratios, in order to evaluate its possible implementation downstream of a steam stripping unit in a double-stage SHARON–ANAMMOX or SHARON–heterotrophic denitritation process. A synthetic influent containing NH_4^+-N (2,000 mg/L) was initially fed to promote biomass acclimation, and then real sour water containing also organic substrate, cyanides, sulphides and phenols was supplied. With both synthetic and real wastewater, the applied C_i/N molar ratio was progressively increased from 1 to 2 and the SHARON reactor produced an effluent suitable for further treatment by autotrophic ANAMMOX or heterotrophic denitritation, respectively. Acute toxicity assessments based on the specific measurement of nitritation activity confirmed that biomass acclimation to the toxic substances contained in the real wastewater occurred successfully. Moreover, high removal of organic matter (73 ± 12%) suggested the absence of any competition between heterotrophic and autotrophic microorganisms. Controlling influent C_i/N molar ratio was shown to represent a key operating strategy to properly regulate SHARON performance, depending on the chosen downstream treatment, proving its actual feasibility under harsh operating conditions and providing useful indications for its implementation at full scale.

Keywords: Alkalinity; Ammonium; Industrial wastewater; Nitrogen removal; Partial nitritation; SHARON

1. Introduction

Since high nitrogen levels in water may cause the degradation of freshwater, estuarine and coastal marine ecosystems, as well as negative effects on human health [1], reducing these levels to acceptable values represents, from an environmental, sanitary and economic perspective, a fundamental challenge to face.

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Conventional biological nitrogen removal processes based on nitrification–denitrification are not suitable to treat ammonium-rich wastewaters, since they usually lack in terms of efficiency (high concentration of ammonium is toxic even for those microorganisms which are able to remove it) and cost-effectiveness (more energy is required to convert higher amounts of ammonium into nitrate by oxidation and a larger amount of external carbon source is needed for the heterotrophic denitrification step, as reported by Ahn [2], Khin and Annachhatre [3] and Van Hulle et al. [4]). On the other hand, chemical–physical processes like steam stripping are characterized by high removal efficiencies, but high operating costs too.

For such streams, advanced biological double-stage systems like partial nitritation SHARON (Single reactor for High activity Ammonium Removal Over Nitrite)—ANAMMOX (ANaerobic AMMonium OXidation) or SHARON-heterotrophic denitritation are more efficient and cost-effective. In the nitritation step, influent ammonium is oxidized to nitrite by ammonium oxidizing bacteria (AOB), according to the following reaction (Eq. (1)):

$$NH_4^+ + 1.5O_2 \rightarrow NO_2^- + 2H^+ + H_2O \quad (1)$$

At high temperature (optimal range is 35–45˚C) AOB have higher growth rates than nitrite oxidizing bacteria (NOB), therefore nitrite conversion into nitrate can be prevented in a continuous flow stirred tank reactor (CFSTR) if a sufficiently short solids retention time (SRT) is maintained (1–1.5 d): this would lead to the progressive wash-out of NOB from the system [4].

Since 2 mol of inorganic carbon (C_i) are required for the conversion of 1 mol of ammonium into nitrite, an influent inorganic carbon to nitrogen (C_i/N, as $HCO_3^-/NH_4^+NH_4^+ - N$) molar ratio of 1 allows the conversion of 50% influent ammonium into nitrite. In this case, the SHARON process may be coupled with the ANAMMOX process in a completely autotrophic double-stage treatment: the residual ammonium in SHARON outlet is converted into gaseous N_2 by ANAMMOX bacteria, with nitrite as the electron acceptor (Eq. (2)):

$$\begin{aligned} &NH_4^+ + 1.32NO_2{}^- + 0.066HCO_3^- + 0.13H^+ \\ &\quad \rightarrow 1.02N_2 + 0.26NO_3{}^- + 2.03H_2O \\ &\qquad + 0.066CH_2O_{0.5}N_{0.15} \end{aligned} \quad (2)$$

Compared to conventional treatments, the SHARON–ANAMMOX process requires less energy for oxygen supply (−60%), there is no need for external carbon sources and surplus sludge production is limited due to the extremely slow growth rate of ANAMMOX bacteria, resulting in lower costs for sludge treatment and disposal.

If influent C_i/N molar ratio is 2, the combination of SHARON process and heterotrophic denitritation may also offer substantial savings compared to conventional systems: in the preliminary nitritation step, oxygen consumption is reduced by 25% compared to nitrification (ammonium is converted into nitrite and not nitrate), while in the subsequent denitritation process, the reduction of nitrite is approximately 1.8 times faster [5] and requires less external organic carbon to be supplied, compared to nitrate reduction.

When dealing with industrial streams containing highly toxic substances, the evaluation of the feasibility, efficiency and robustness of the SHARON process is fundamental in order to hypothesize the subsequent implementation of either autotrophic ANAMMOX or heterotrophic denitritation: although partial nitritation has been intensively investigated for the treatment of anaerobic digester supernatant, landfill leachates and manure effluents [6–10], studies focusing on the treatment of ammonium-rich industrial wastewater which have been carried out so far [11,12] did not concern the possibility to integrate the SHARON process in an existing chemical–physical treatment system and are mostly focused on its possible coupling with ANAMMOX, rather than heterotrophic denitritation. Within this framework, the main aim of the present study was to investigate the possibility to apply the SHARON process downstream of a steam stripping unit, in combination with both autotrophic ANAMMOX and heterotrophic denitritation processes, for the treatment of ammonium-rich refinery wastewater (sour water) produced by the desulphurization process at SARAS SpA refinery plant located near Cagliari (Italy). The sour water is characterized by high concentrations of ammonium (up to 4,000 mgN/L) and organic matter, as well as by the presence of toxic substances like cyanides, phenols and sulphides. It is currently treated at full scale by steam stripping with high removal efficiency (NH_4^+-N threshold level is set at 25 mg/L), but also high operating costs; raising the threshold value of the stripper up to 2,000 mgN/L and integrating the chemical–physical treatment with an advanced biological system would lead to considerable reduction in energy requirements and operating costs.

In order to provide a comprehensive overview of process behaviour, as well as to bring useful indications for its possible implementation at full scale in combination with the steam stripping treatment unit and with autotrophic ANAMMOX or heterotrophic denitritation processes, the SHARON reactor was operated in this study with increasing influent C_i/N

molar ratios (from 1 to 2) under ideal (synthetic influent) and harsh (real sour water) operating conditions. Process performance was extensively evaluated in terms of, among the others, NH_4^+-N removal efficiency, NO_2^--N and NO_3^--N build-up, effluent NO_2^--N/NH_4^+-N molar ratio, removal of organic substrates (the latter only for real wastewater) and effluent characteristics were compared to those required for the subsequent treatment by ANAMMOX or heterotrophic denitritation. Moreover, useful information about AOB acclimation to the toxic substances contained in the real wastewater was gathered via acute toxicity assessments based on the specific measurement of nitritation activity.

2. Materials and methods

2.1. Reactor set-up

A glass, water-jacketed CFSTR with a working volume of 2 L was used to carry out the experiments and operated as a chemostat (no biomass recirculation) at controlled temperature (35 ± 0.5°C). A pH-control system was implemented to avoid unexpected pH variations beyond the optimal range (7.0 ± 0.5) during reactor operation. Air was supplied by a membrane pump and introduced via a fine bubble aerator at the bottom of the reactor, in order to keep the dissolved oxygen concentration above 2 mg/L. Since the reactor was operated as a chemostat, the hydraulic retention time (HRT) and SRT coincided and were maintained at 1.25 d. Activated sludge drawn from the municipal wastewater treatment plant (WWTP) of Cagliari (Italy) was used as inoculum.

2.2. Influent composition

The composition of the synthetic influent was NH_4^+-N 2,000 mg/L (dosed as NH_4HCO_3), KH_2PO_4 1,000 mg/L, $MgSO_4$ 100 mg/L and trace elements solution 10 mL/L [13].

Real wastewater taken from the outlet of the stripping unit was collected monthly at the SARAS SpA refinery plant and stored at 4°C before feeding. Sour water was characterized by high pH (9.5 ± 0.3) and alkalinity (up to 1,970 mg_{CaCO3}/L), and contained also dissolved organic carbon (DOC) (293 ± 20 mg/L) and toxic compounds such as cyanides (up to 9 mg/L), sulphides (up to 31 mg/L) and phenols (up to 90 mg/L), which were not present in the synthetic influent. NH_4Cl was added in order to reach the target level of 2,000 mgN/L.

For both synthetic and real wastewater, the influent flow-rate was kept at 1.1 mL/min, resulting in a volumetric nitrogen loading rate of 1.6 gN/L d. Influent C_i/N molar ratio was set at 1.0, 1.5, 1.75 and 2.0 by adding proper amounts of $NaHCO_3$, and the effects of each influent C_i/N molar ratio on process performance were explored for at least 7 times the SRT; the operating conditions were modified only when overall process performance remained stable for at least thrice the SRT.

2.3. Analytical methods

Ammonium (as $NH_4^+ - N, NO_2^- - N, NO_3^- - N$) and DOC concentrations in the real wastewater, as well as in reactor influent and effluent, were determined as previously described in Milia et al. [13], and analyses were performed in triplicate. The composition of real wastewater was assessed at each supply also in terms of cyanides, phenols and alkalinity according to standard methods [14]. Sulphides were determined by Hach–Lange spectrophotometer (DR-2800, cuvette test kit LCK-653). Total suspended solids and volatile suspended solids (VSS) were determined by standard methods [14].

A titrimetric biosensor (ANITA, Ammonium NITrification Analyser) was used in order to evaluate the acute inhibiting effects of real wastewater on AOB, as described in Milia et al. [13]. Tests were performed in triplicate using unacclimated and acclimated biomass drawn from the lab-scale SHARON reactor, as well as using unacclimated activated sludge drawn from both the municipal WWTP of Cagliari and the industrial WWTP at SARAS SpA facilities.

3. Results and discussion

In order to promote the selection of AOB, the SHARON reactor was initially fed with a synthetic influent containing NH_4^+-N asthe only available substrate and characterized by an influent C_i/N molar ratio of 1; biomass acclimation to high nitrogen content in the wastewater was achieved by gradually increasing NH_4^+-N influent concentration from 100 mg/L up to the target concentration (2,000 mg/L, results not shown). The experimental results observed with a NH_4^+-N concentration in the influent of 2,000 mg/L and with a C_i/N molar ratio ranging from 1.0 to 2.0 in the synthetic influent (Run I) and in the real wastewater (Run II) will be described and discussed in this paper.

3.1. Effects of the C_i/N ratio on the SHARON process treating synthetic influent

When the reactor was fed with the synthetic influent (Run I) and the C_i/N molar ratio was set to 1.0, the observed NH_4^+-N removal efficiency and NO_2^--N concentration in the effluent were 48.9 ± 2.4% and 932.9 ± 59.4 mg/L, respectively, with a corresponding NO_2^--N/NH_4^+-N ratio in the effluent of 0.91 ± 0.08. Nitrate build-up was negligible and NO_3^--N concentration was 42.5 ± 7.6 mg/L on an average, indicating almost complete wash-out of NOB from the system due to the combined effects of high temperature and short HRT, as previously reported [4]. Such results were in line with the expected stoichiometric values, and effluent characteristics were comparable to those reported in the literature and considered suitable for subsequent treatment by ANAMMOX [4,15,16].

The progressive increase of influent C_i/N molar ratio was accompanied by a corresponding rise of NH_4^+-N removal efficiency, due to the increased availability of inorganic carbon in accordance with nitritation stoichiometry: as the C_i/N ratio was set to 2, 96.8 ± 1.8% of influent NH_4^+-N was mostly converted into NO_2^--N, as indicated by the high effluent NO_2^--N/NH_4^+-N ratio (36.3 ± 13.5) and NO_2^--N concentration (1,886 ± 48 mg/L). Average NO_3^--N concentration in the effluent was 32.4 ± 9.2 mg/L (very low if compared to removed NH_4^+-N), again confirming that most of NOB had been washed out from the system and that stable nitritation occurred (Fig. 1).

In agreement with nitritation stoichiometry, effluent from the SHARON reactor was suitable for the subsequent treatment by heterotrophic denitritation, which may be implemented at full scale without investment costs since a denitrification step already exists in the WWTP at SARAS SpA facilities and methanol is available as a waste product.

For all the C_i/N ratios tested, pH self-stabilized within the control range, than no dosage of acid or base solution was required throughout Run I. Moreover, mass balances indicated a negligible loss of nitrogen (always below 5% of influent NH_4^+-N) which was likely due to ammonia stripping, N_2O and NO productions.

3.2. Effects of the C_i/N ratio on the SHARON process treating real sour water

Differently from the synthetic influent, real wastewater was characterized by high acute toxicity due to the presence of cyanides, sulphides and phenols, which may inhibit AOB activity and compromise reactor performance irreversibly. Compared to unacclimated activated sludge drawn from the municipal WWTP of Cagliari and from the industrial WWTP at SARAS SpA facilities, unacclimated biomass in the SHARON reactor showed a higher or similar tolerance against the toxic substances contained in the real wastewater, as indicated by the inhibitory concentrations (IC) reported in Table 1.

To test the feasibility of partial nitritation as the preliminary treatment of real sour water in view of its coupling with the ANAMMOX process, influent C_i/N molar ratio was set to 1 and increasing amounts of real wastewater were progressively fed to the SHARON reactor, until the synthetic influent was completely replaced: despite its acute toxicity, gradual addition of real sour water did not cause any significant worsening of SHARON performance, thus proving process robustness and reliability (Fig. 2).

Such behaviour suggests the progressive acclimation of biomass to the toxic compounds in the real wastewater, as confirmed by the results achieved by acute toxicity assessments carried out on unacclimated and acclimated biomasses on days 110 (Run I) and 295 (Run II), respectively (Fig. 3): IC10 and IC50 increased from 3.6 to 23.3 and from 8.7 to 67.3 mL/L, respectively, while IC90 could not be determined with acclimated biomass (it was 21.05 mL/L with unacclimated AOB).

During Run II, only real sour water was fed to the reactor (NH_4^+-N influent concentration was 2,000 mg/L). Positive results obtained with the synthetic influent were confirmed with real sour water (C_i/N of 1) and the effluent from the SHARON

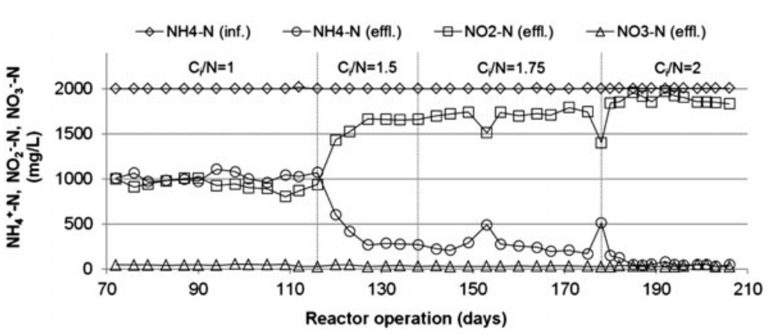

Fig. 1. Performance of SHARON process fed with synthetic influent (Run I).

Table 1
IC* determined via acute toxicity assessments carried out with real wastewater on unacclimated biomass drawn from the lab-scale SHARON reactor and from full-scale activated sludge municipal and industrial WWTPs

Type of biomass	IC10 (mL/L)	IC50 (mL/L)	IC90 (mL/L)
SHARON (unaccl., day 110)	3.6	8.7	21.05
Municipal WWTP (unaccl.)	3.45	5.35	8.25
SARAS WWTP (unaccl.)	5.0	8.5	14.45

*Defined as the amount of real wastewater per litre of reactor that causes 10 (IC10), 50 (IC50) and 90% (IC90) acute inhibition on AOB activity.

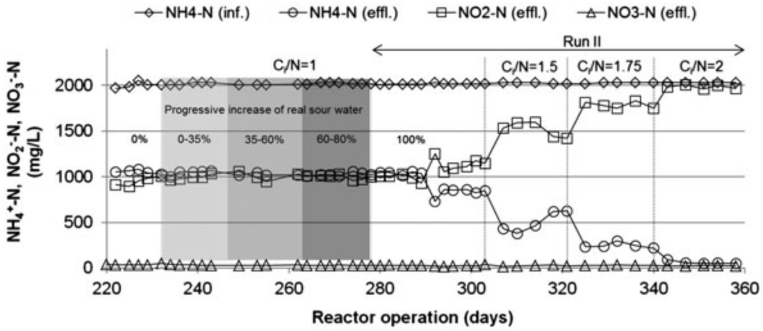

Fig. 2. Performance of SHARON process fed with real sour water and different C_i/N molar ratios.

reactor was suitable for subsequent treatment by ANAMMOX: under steady-state conditions half of the influent NH_4^+-N was oxidized (54.0 ± 5.8%) and nitrite build-up was observed (NO_2^--N concentration and NO_2^--N/NH_4^+-N ratio in the effluent were 1,073 ± 94 mg/L and 1.19 ± 0.26, respectively) with low nitrate production (Fig. 2).

The possibility to couple partial nitritation with heterotrophic denitritation was also assessed using real sour water. Influent C_i/N molar ratio was progressively increased from 1.0 to 2.0 and process performance was evaluated: the increase of C_i/N molar ratio was accompanied by the increase of NH_4^+-N removal efficiency from 54.0 ± 5.8% ($C_i/N = 1$) to 96.9 ± 0.9% ($C_i/N = 2$), in line with previous observations made with the synthetic influent. As shown in Fig. 2, the same positive correlation was observed for NO_2^--N effluent concentration which increased from 1,073 ± 94 mg/L ($C_i/N=1$) up to 1,983 ± 21 mg/L ($C_i/N = 2$), while no effect on nitrate build-up was observed (NO_3^--N effluent concentration remained below 40 mg/L throughout Run II, confirming that nitritation was maximized, with negligible NOB activity). Consequently, an increase of effluent NO_2^--N /NH_4^+-N ratio from 1.19 ± 0.26 ($C_i/N = 1$) to 32.6 ± 6.5 ($C_i/N = 2$) was observed. At the highest C_i/N ratio tested, effluent from the SHARON reactor was suitable for subsequent treatment by heterotrophic denitritation. Similar results were reported by Torà et al. [17], who achieved 95% conversion of influent ammonia into nitrite by applying an automatic control loop to an activated sludge nitrifying reactor treating real reject water, and by Kulikowska and Bernat [18], who achieved a high conversion of ammonium into nitrite (up to 99%) with negligible nitrate production in a sequencing batch reactor (SBR) treating landfill leachate.

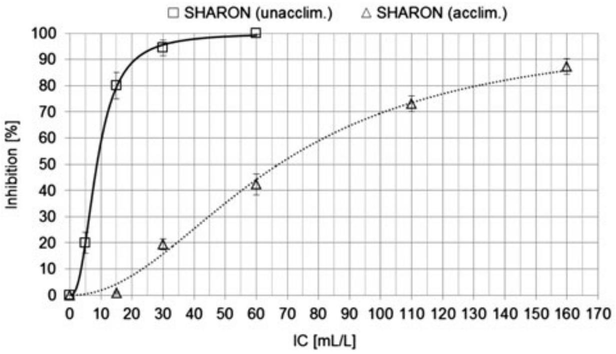

Fig. 3. Dose–Response curves determined via acute toxicity assessments with unacclimated (day 110) and acclimated (day 295) biomass drawn from lab-scale SHARON reactor.

A direct comparison of process performance with synthetic (Run I) and real sour water (Run II) is shown in Fig. 4.

For all the C_i/N molar ratios tested, no significant differences were observed between ideal (Run I, synthetic influent) and harsh working conditions (Run II, real sour water), indicating that the presence of toxic substances in the real sour water did not negatively affect the overall process performance and proving the actual SHARON feasibility as the preliminary treatment in a double-stage SHARON–ANAMMOX or SHARON–denitritation process.

Differently from the other process parameters, no clear correlation was observed between the C_i/N molar ratio and VSS concentration in the reactor, even though overall trends were very similar for Run I and II, as shown in Fig. 5.

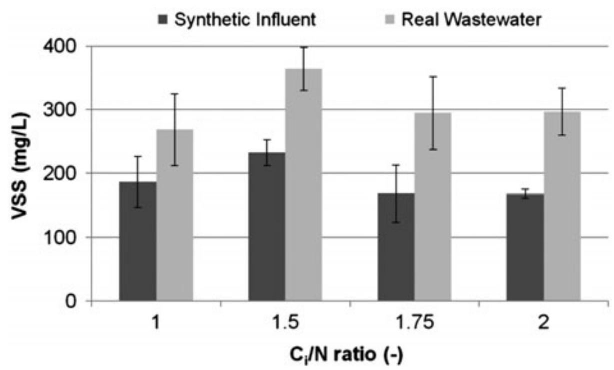

Fig. 5. Influence of C_i/N molar ratio on VSS concentration.

Given the same C_i/N ratio, average VSS concentration was higher when real sour water was used (Run II), likely due to the availability of organic substrate in the influent and the consequent development of heterotrophic biomass in the SHARON reactor, whose presence was confirmed by the considerable removal of influent DOC (removal efficiency throughout Run II was 73 ± 12%). Campos et al. [19] observed a 10% inhibition of AOB activity in the SHARON process when readily biodegradable acetate was fed in a 0.3 gC/gN ratio. Hanaki et al. [20] suggested that transport of ammonia from the bulk liquid phase to the cells of AOB may be hindered by the presence of the crowded cells of heterotrophs which assimilate the ammonia and consume the oxygen before it reaches AOB: such inhibitory effect on ammonia oxidation is enhanced by low dissolved oxygen concentration. In this study, the sour water had a resulting gC (as DOC)/gN ratio of approximately 0.15 and dissolved oxygen concentration in the system was always maintained above 2 mg/L, therefore nitritation was not hindered and no competition was observed between heterotrophic biomass and AOB. A similar behaviour was observed by Wang et al. [6] in a SBR for the treatment of landfill leachate by simultaneous partial nitrification, ANAMMOX and denitrification.

Again, pH self-stabilized within the control range throughout Run II (no need for active pH-control) and loss of nitrogen as stripped ammonia, N_2O or NO in the gas outlet was shown to be negligible by mass balances (below 4% of influent NH_4^+-N), in agreement with previous studies: gaseous NO and N_2O emissions from a full-scale SHARON reactor treating reject

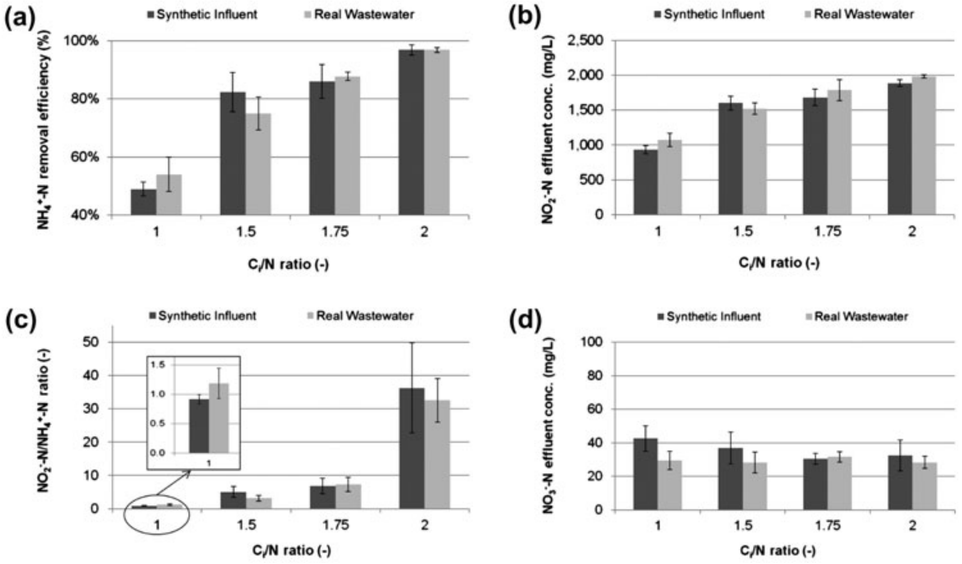

Fig. 4. Effects of C_i/N molar ratio on NH_4^+-N removal efficiency (a), NO_2^--N build-up (b), effluent NO_2^--N/NH_4^+-N ratio (c) and NO_3^--N concentration (d).

wastewater accounted for only 0.2 and 1.7% of the nitrogen load, respectively [21].

4. Conclusions

The possible application of the SHARON process for the treatment of refinery wastewater (sour water) characterized by high concentrations of NH_4^+-N (2,000 mg/L) and toxic substances like cyanides, phenols and sulphides was assessed in this study, in view of its coupling with either autotrophic ANAMMOX or heterotrophic denitritation (the latter in case sensitive ANAMMOX process is hindered by toxic compounds contained in the wastewater).

A preliminary evaluation of SHARON feasibility was assessed using a synthetic influent with different C_i/N molar ratios (Run I). Results proved that influent C_i/N molar ratio plays a key role in regulating SHARON performance: depending on the applied C_i/N ratio, the SHARON reactor produced an effluent suitable for further treatment by autotrophic ANAMMOX ($C_i/N=1$) or heterotrophic denitritation ($C_i/N=2$). Moreover, the chosen operating temperature (35˚C) would not require any additional cost at full scale, since the real wastewater has a temperature above 60˚C which could even allow excess heat recovering.

On the basis of the positive results achieved with the synthetic influent, sour water was fed to the reactor in order to test SHARON performance under real, harsher operating conditions (Run II). Acute toxicity assessments carried out on biomass drawn from the SHARON reactor proved that acclimation to toxic substances occurred successfully. Despite the complexity and variability of real influent characteristics, stable partial nitritation was achieved when the C_i/N molar ratio was set to 1 and effluent from the SHARON reactor was suitable for its subsequent treatment by ANAMMOX. When the C_i/N molar ratio was set to 2, $96.9 \pm 0.9\%$ of influent NH_4^+-N was oxidized and converted mostly into nitrite with low nitrate production, producing an effluent suitable for subsequent treatment by heterotrophic denitritation. Moreover, it was shown that development of heterotrophic biomass due to organic substrate availability did not cause any detrimental effect on AOB activity, since oxygen was not a limiting factor. For all the C_i/N ratios tested with real sour water, nitrate in the effluent was found to be low enough (<40 mgN/L), as well as nitrogen loss due to gaseous emissions as stripped ammonia, NO and N_2O, which was always below 4% of influent nitrogen.

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