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Biodegradation of tannery wastewater using sequencing batch reactor—Respirometric assessment

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Abstract

This investigation proved that respirometry combined with sequencing batch reactor (SBR) could be an effective way for the removal of COD in tannery wastewater. Measurement of oxygen uptake rates (OUR) and corresponding COD uptake rates showed that a 12-h operating cycle was optimum for tannery wastewater. The removal of COD by degradation was stoichiometric with oxygen usage. A plot of OUR values provided a good indication of the biological activity in the reactor. A high OUR value corresponded to the feed period; at the end of the cycle, when the substrate was depleted, the OUR value was low. At a 12-h SBR cycle with a loading rate of 1.9–2.1 $kg\,m^{-3}\,d^{-1}$, removal of 80–82% COD, 78–80% TKN and 83–99% NH_3-N were achieved. These removal efficiencies were much higher than the conventional aerobic systems. A simple method of COD fractionation was performed from the OUR and COD uptake rate data of the SBR cycle. About 66–70% of the influent COD was found to be readily biodegradable, 10–14% was slowly degradable and 17–21% was non-biodegradable. The oxygen mass transfer coefficient, K_La ($19 \pm 1.7\,h^{-1}$) was derived from respirometry. It was observed that with the exception of high organic load at the initial feed the oxygen transfer capacity was in excess of the OUR, and aerobic condition was generally maintained. Simultaneous nitrification–denitrification was observed in the SBR during the feed period as proved by mass balance.

Keywords: Tannery wastewater; SBR; Respirometry; COD fractionation; Oxygen mass transfer coefficient; Nitrification–denitrification

1. Introduction

Leather tannery effluents are a source of severe environmental impacts. Tannery productive cycle includes a series of chemical treatments using a large number of chemicals such as surfactants, acid and metalorganic dyes, natural or synthetic tanning agents, sulphonated oils, salts, etc. to transform animal skin into an unalterable and imputrescible product (Di Iaconi et al., 2002). The inclusion of such a wide range of chemicals has rendered the biological treatment of tannery wastewater difficult and more complicated due to their low biodegradability. The conventional biological treatment systems in use for tannery wastewater are either inadequate or less cost-effective due to the large variations in tanning practices and the kinds of chemicals used in the process. It has been well documented that sequencing batch reactor (SBR) system as an attractive option due to its greater flexibility and cost effectiveness (Ketchum, 1997). SBR can obtain higher nitrogen and organic carbon removal efficiencies than conventional systems. Cyclic concentration gradients, to which biomass is exposed in a SBR reactor (Artan et al., 1996) permit selection and enrichment of particular microbial species more capable of carrying out biological processes such as nitrification and denitrification in the presence of inhibiting substances.

In an aerobic system, respirometry effectively links the concepts of oxygen utilisation with metabolic energetics. It allows the oxygen uptake in a microbial environment to be used as a surrogate parameter for cell growth or substrate removal. Respirometric techniques have been intensively used for the determination of BOD, toxicity and biokinetic parameters of toxic and non-toxic wastewaters (Spanjers et al., 1993; Vanrollegham et al., 1994). The OUR can also be

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used to determine the specific growth rate and other kinetic coefficients (Henze et al., 1987; Vanrollegham et al., 1999). Knowledge of the respiration rate and oxygen transfer function is of interest in both process control and process diagnosis (Lindberg and Carlsson, 1996). In situ respirometry has been proved to be a useful tool in managing the removal of phenol in a SBR (Yoong et al., 2000; Yoong and Lant, 2001).

Various options are available to optimise the react phase of the process. ORP was found to be a very sensitive parameter to follow-up the stages of a SBR treating piggery wastewater (Obaja et al., 2003). The distinct phases of the cycle (anaerobic, aerobic and anoxic) were well distinguished by means of the ORP profile. This paper addresses the role of respirometry in minimising the hydraulic retention time (HRT) required in the SBR to achieve effective COD removal in tannery wastewater. Carbon and nitrogen removal efficiencies were examined at two HRT values. Respirometric measurements in the reactor were used as an indicator of metabolic activity. Further, COD fractionation of influent tannery wastewater was performed from COD and OUR data.

2. Methods

2.1. Sequencing batch reactor

A bench-scale SBR made of plexiglass and of working volume 8 l was used for the study. The reactor consisted of two exits: one for sludge withdrawal and the other for cleaning and emptying the reactor. Sludge was withdrawn directly from mixed liquor at the end of the aerobic phase. Influent feed and purified effluent decantation were performed from the top, by means of peristaltic pumps. Aeration was provided from a diffuser at the base of the reactor through two air stones at opposite ends. The reactor contents were mixed by means of a stirrer. The different phases of the SBR such as fill, mix, aeration, decantation and wastage were controlled by time switches.

2.2. Raw wastewater

Wastewater samples were obtained from the equalisation tank of a treatment plant designed to treat 2500 m^3/d of tannery wastewater generated from about 128 tanneries. The raw tannery wastewater was pre-treated with conventional coagulants for the amendment of suspended solids and precipitation of chromium. The pre-treated wastewater was used as feed to the SBR. The average chemical composition of raw and pre-treated tannery wastewater is shown in Table 1. The parameters were measured according to Standard Methods (1995).

2.3. Reactor biomass

The inoculum for the SBR was obtained from the aeration tank of the treatment plant, which employed a continuous flow suspended growth configuration. The biomass was acclimated to the SBR conditions. After an acclimation period of about 45 days, the biomass exhibited stable nitrification apart from good settling characteristics, which was an important requirement for the SBR process.

Table 1
Investigated tannery wastewater composition

Parameters	Raw effluent	Pre-treated effluent
pH	7.08 ± 0.28	7.69 ± 0.20
Total solids, TS	10,265 ± 1460	6810 ± 110
Suspended solids, SS	2820 ± 140	325 ± 105
Volatile suspended solids, VSS	1505 ± 90	140 ± 35
Total COD	4800 ± 350	1910 ± 174
Soluble COD	1950 ± 205	1890 ± 160
Total Kjeldhal nitrogen, TKN	225 ± 18	203 ± 23
Ammoniacal nitrogen, NH_3-N	128 ± 20	120 ± 15
Total chromium	95 ± 55	0.55 ± 0.11

Note: All parameters except pH are expressed in mg/l.

2.4. Oxygen uptake rate measurement

Oxygen uptake rate (OUR) was measured using a closed respirometric unit of a BOD bottle, in which the dissolved oxygen was measured with an YSI oxymeter model 58 equipped with a YSI probe model 5750. Mixed liquor samples from the SBR were taken at regular intervals in the BOD bottle and the DO probe was dipped into the bottle. The displaced liquid was allowed to overflow and thus air bubble accumulation inside the bottle was prevented. The sample was continuously stirred during measurement, and DO values were obtained for every 5 s. Oxygen uptake rate was determined as the slope obtained from the plot of DO concentration against time. DO measurements were recorded at short intervals in order to obtain high OUR values.

3. Results and discussion

3.1. Performance of SBR during start-up

The SBR inoculated with activated sludge was fed with the pre-treated tannery wastewater at 4 l/day and a hydraulic retention time (HRT) of 2 days. The effluent COD was initially high (Fig. 1), which was due to the slow acclimatization and poor settling property exhibited by the sludge. After 4 weeks of acclimation, the sludge showed marked

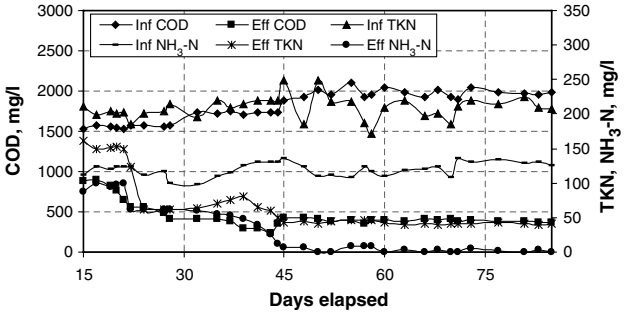

Fig. 1. Influent and effluent COD, TKN and NH_3-N time profiles throughout the experimental study.

removal of COD with a residual COD of less than 400 mg/l, improved settling characteristics (SVI = 50–60 ml/g) and stable nitrification with near zero values of effluent NH_3-N (Fig. 1). The high effluent COD (350–400 mg/l) was indicative of the presence of bio-recalcitrant chemicals in tannery wastewater (Carucci et al., 1999; Di Iaconi et al., 2002) such as the melamine and phenol–naphthalene based synthetic tanning agents (Arun kumar et al., 2004).

3.2. SBR and respirometry

The evaluation of SBR with the aid of respirometry was performed after the acclimation period. The SBR was initially operated at one cycle per day with an aerated, mixed reaction phase of 23 h, which included a 1-h fill at commencement. This was followed by a 1-h settling phase, which included decantation during the last 15 min of the settling phase. The SBR loading rate was 0.9–1 kg COD m^{-3} d^{-1} with a sludge retention time (SRT) of 20 days and HRT of 2 days. The average biomass as mixed liquor volatile suspended solids was 1900 mg/l.

Typical OUR, COD utilisation rate (COD-UR) and DO profiles in a 24-h SBR process are shown in Fig. 2. The continuous plot of the DO concentration and OUR provided a good indication of the biological activity in the reactor. As DO uptake and aerobic microbial respiration in the system are closely linked, it was observed that the DO and OUR values were particularly sensitive to changes in microbial activity. This phenomenon was possibly the result of chemical signalling between cells inducing rapid oxygen uptake in the presence of high organic loading (Brock and Madigan, 1991; Yoong et al., 2000). Corresponding OUR and COD-UR profiles showed similar parallel effects during the course of the SBR cycle (Fig. 2). The OUR and COD-UR profiles indicated that a 12-h SBR operating cycle was possible, which reduced the HRT and increased the organic loading of the reactor.

The closeness of OUR and COD-UR was only coincidental as nitrification took place simultaneously with COD removal, and hence OUR due to nitrification was deducted in order to draw a mechanistic understanding of OUR and COD-UR data. The linear regression analysis of the OUR (after nitrification inhibition) and COD-UR values (Fig. 3) returned a coefficient of determination of 0.92 and slope of

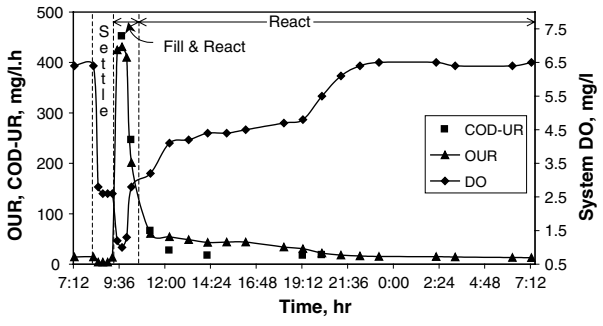

Fig. 2. Typical OUR, DO and COD profile of a 24-h SBR cycle.

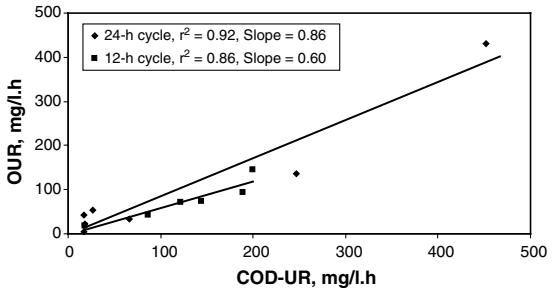

Fig. 3. Linear regression of COD-UR and OUR.

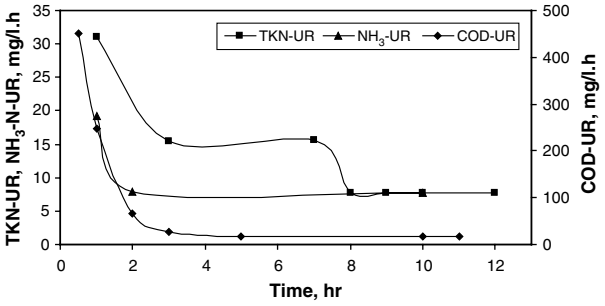

Fig. 4. Substrate (COD, TKN and NH_3-N) uptake rate during 24-h SBR cycle.

0.86. Such a high correlation indicated that the removal of COD by degradation was stoichiometric with oxygen usage, which validated the importance of respirometry for the evaluation of SBR systems treating tannery wastewater. COD was estimated to be a more useful parameter as it enabled one to make appropriate correlations among substrate, biomass and dissolved oxygen in terms of electron equivalence (Orhon and Ubay Cokgör, 1997).

TKN and NH_3-N removals were evenly distributed during the 24-h cycle (average TKN-UR = 7.8 mg/l h and average NH_3-UR = 2.9 mg/l h) as indicated by their utilisation rates (Fig. 4), but removal of these components was complete by the 12th hour of the cycle, similar to COD removal. The substrate removal efficiency in terms of COD, TKN and NH_3-N for a typical 24-h SBR cycle was 81.2%, 78.6% and 92.8%, respectively (Table 2).

3.3. Reduced hydraulic retention time

The SBR operating cycle was reduced to 12 h, with a feed period of 4 h (fill to time ratio, FTR = 0.33), a react phase of 7- and 1-h quiescence, including 15 min of decantation. The SBR loading rate was 1.9–2.1 kg COD m^{-3} d^{-1} with a SRT of 13–15 days and HRT of 1 day. The average DO and OUR profiles showed similar trends (Fig. 5) as that of the 24-h cycle. The linear correlation between the OUR (after nitrification inhibition) and COD values for the 12-h SBR (Fig. 3) returned a correlation coefficient of 0.86. Although, only 60% of the variability of the COD was explained by the OUR values, the coefficient of determination was statistically significant at 95% confidence level.

Table 2
SBR performance during typical 24- and 12-h time cycles

Parameter	Phase/sample	24-h time cycle	12-h time cycle
COD, mg/l	Influent	2077	1908
	End of feed[a]	1234	1138
	End of feed[b]	536	473
	Effluent	391	368
TKN, mg/l	Influent	217.7	207.3
	End of feed[a]	140.0	134.9
	End of feed[b]	108.8	41.5
	Effluent	46.6	41.5
NH_3-N, mg/l	Influent	108.8	124.4
	End of feed[a]	58.3	72.6
	End of feed[b]	39.0	20.7
	Effluent	7.8	20.7
NO_x-N, mg/l	Influent	–	1.88
	End of feed[b]	–	52.8
	Effluent	–	54.5
COD removal, %		81.2	80.7
TKN removal, %		78.6	80.0
NH_3-N, %		92.8	83.4

[a] Theoretical value calculated after mixing the influent with the mixed liquor remaining in the SBR at the end of the previous cycle.
[b] Measured value.

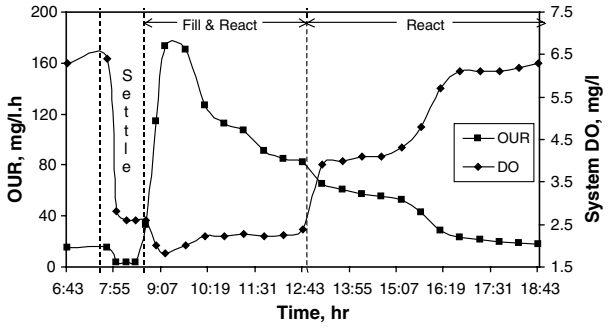

Fig. 5. Typical OUR and DO profile of a 12-h SBR cycle.

The measured average biomass was 1890 mg/l. The COD, TKN and NH_3-N removed in a typical 12-h cycle were 80.7%, 80% and 83.4% respectively (Table 2), and $81 \pm 2\%$, $80 \pm 3\%$ and $85 \pm 2\%$ respectively during the entire study period.

SBR technology was used successfully for the treatment of tannery wastewater and other high strength wastewaters. Obaja et al. (2003) found that SBR was a very flexible tool and particularly suitable for the treatment of piggery wastewater, characterized by high nutrient content and by frequent changes in composition and therefore affecting process conditions. Lefebre et al. (2005) studied the treatment of the segregated wastewater stream of tannery soaking operation (tannery soak liquor) in a SBR seeded with halophilic bacteria, and have concluded that high removal efficiencies of COD, TKN and suspended solids were achieved at a HRT of 5 days and OLR of 0.6 kg COD m^{-3} d^{-1}. Although, SBR technology was found to be an adequate solution for tannery soak liquor, the requirement of such a high HRT will have implications during large-scale designing of SBR system and contradicts the very fact that SBR systems as cost-effective treatment systems. Our investigation on the treatment of composite tannery wastewater using SBR had proved to be an advantage over conventional systems, as the required HRT and OLR were 1 day and 1.9–2.1 kg COD m^{-3} d^{-1} respectively with high removal efficiencies.

3.4. COD fractionation

The simulation models of activated sludge systems, such as the IAWQ model (Henze et al., 1987), is structured on the basis of the division of the wastewater into various COD fractions. All significant methods proposed for the determination of COD fractions rely on respirometric measurements conducted under aerobic or anoxic conditions, using continuous or batch reactors (Ekama et al., 1986; Vanrollegham et al., 1999). Procedures involving other techniques, such as that involving physical separation (Mamais et al., 1993) are also in vogue.

The OUR profile obtained in the present investigation was typical of a batch respirogram. The COD removed during the feed period alone was $68 \pm 2\%$ of the influent COD [i.e., from Table 2, $S_{S1}/C_{T1} = 2 \times$ (end of feed COD[a] − end of feed COD[b])/COD_{inf}, where 2 represents the number of cycles]. COD removal was similar during the feed period of the 24- and 12-h time cycles, irrespective of the feed duration (1 or 4 h). Such rapid uptake was possible of the RBCOD (S_{S1}), which presumably comprised of simple and soluble compounds such as volatile fatty acids, simple sugars, alcohols, amino acids etc. (Henze et al., 1987; Orhon and Ubay Cokgör, 1997). Thus, this fraction corresponding to the feed period was taken as the RBCOD. About $12 \pm 2\%$ of the influent COD was removed after the feed period and before the end of the cycle [i.e., 2 × (end of feed

Table 3
Comparison of experimental COD fractions of tannery wastewater with reported results

Reference	C_{T1}, mg/l	S_{T1}, mg/l	S_{S1}, mg/l	S_{S1}/S_{T1}, %	S_{H1}, mg/l	S_{H1}/S_{T1}, %	aX_{S1}, mg/l	aX_{S1}/S_{T1}, %	X_{S1}, mg/l	X_{S1}/C_{T1}, %	S_{I1}, mg/l	S_{I1}/S_{T1}, %
SBR (this study)	2077	2077	1396	67.2	–	–	290	14.0	–	–	391	18.8
Batch respirometry (this study)	1912	1912	346	18.1	800	41.8	303	15.8	–	–	462	24.2
Orhon et al. (1998)	2300	1300	435	19.0	650	28.5	–	–	730	31.5	215	9.5
Orhon et al. (1998)	1100	1100	385	35.0	540	49.0	–	–	–	–	175	16.0
Ubay Cokgör et al. (1998)	2500	1295	460	35.5	–	–	–	–	–	–	–	–
Ubay Cokgör et al. (1998)	2410	1205	300	24.9	–	–	–	–	–	–	–	–
Ubay Cokgör et al. (1998)	2175	1300	340	26.2	–	–	–	–	–	–	–	–
Orhon and Ubay Cokgör (1997)	1080	1080	340	31.5	540	50.0	–	–	–	–	200	18.5
Orhon and Ubay Cokgör (1997)	775	775	230	29.8	425	54.8	–	–	–	–	120	15.5
Orhon and Ubay Cokgör (1997)	2410	1210	300	24.8	770	63.6	–	–	935	38.8	140	11.6
Kabdash et al. (1994)	1500	1500	–	–	–	–	–	–	–	–	323	21.5
Kabdash et al. (1994)	1075	1075	–	–	–	–	–	–	–	–	262	24.4
Kabdash et al. (1994)	1870	1870	–	–	–	–	–	–	–	–	464	24.8

COD^b – effluent COD)/COD_{inf}]. This fraction accounted for the slowly biodegradable COD part of the influent. The residual 19 ± 2% COD formed the non-biodegradable fraction. Thus, the continuous measurement of OUR and estimation of the COD at the time of precipitous change in OUR in a SBR, yielded useful information regarding the influent COD fractions.

Results on experimental work carried out on COD fractionation of tannery wastewater by various authors and their comparison with the present study are given in Table 3. RBCOD varied between 19% and 35% of the influent soluble COD of tannery wastewater (Orhon and Ubay Cokgör, 1997; Orhon et al., 1998; Ubay Cokgör et al., 1998). COD fractionation of tannery wastewater by batch respirometry method (Ekama et al., 1986; Boursier et al., 2005) yielded a RBCOD fraction of 18% (Table 3). The consistency of the batch respirometry method was tested at various S_0/X_0 ratios between 0.25 and 0.53 (results not shown), and the S_{S1} ranged between 15.6% and 18.1%. This value was found closer to some of the reported results (Orhon and Ubay Cokgör, 1997; Orhon et al., 1998; Ubay Cokgör et al., 1998).

The S_{S1} obtained with the proposed method (SBR–COD method) was much higher than the batch respirometry method. The readily hydrolysable COD (S_{H1}) fraction (using batch respirometry method) was 41.2%, which was in agreement with the literature values. The combined values of S_{S1} and S_{H1} from the batch method equalled the S_{S1} of SBR–COD method. Thus, proper differentiation was not possible between the readily degradable and the rapidly hydrolysable COD fractions by the SBR–COD approach. Perhaps, this differentiation was not required as both these fractions were utilised simultaneously during the feed time of the SBR process. The importance of these fractions in the SBR process needs to be worked out by tracking the biomass growth and the related kinetics parameters.

The soluble slowly biodegradable COD fraction designated as aX_S (Boursier et al., 2005) was found comparable between the batch and SBR method (14% and 15.8% respectively). There is no data available in literature for this particular fraction for tannery wastewater. Although, SBCOD posed to be the rate-determining component, its concentration was far less compared to the RBCOD. Hence, RBCOD concentration was conceived as the rate-limiting component for heterotrophic growth, which is in line with the methodology adopted in current mathematical models (Henze et al., 1987, 1995).

Although, the soluble inert COD fraction of tannery wastewater was within the reported values (Orhon and Ubay Cokgör, 1997; Orhon et al., 1998; Ubay Cokgör et al., 1998; Kabdash et al., 1994), the non-biodegradable COD formed due to residual microbial products (Orhon et al., 1986, 1999) was not accounted for. Formation of residual microbial products in the SBR needs to be evaluated for accurate estimation of influent inert COD.

3.5. Oxygen mass transfer coefficient, K_La

In the SBR, the oxygen mass transfer coefficient was determined by measurement of the OUR and DO changes in the reactor (Yoong and Lant, 2001). In Figs. 2 and 5, typically at a point where the substrate was close to depletion, the gradual increase in DO and the corresponding average OUR were measured. The average K_La in tannery wastewater evaluated from the SBR test was 19 ± 1.7 h^{-1}. With the measured K_La value, the calculated oxygen transfer capacity was shown to be adequate (i.e., greater than the OUR) during the react period. However, at the initial feed period the OUR exceeded the oxygen transfer capacity. This was evident from the very low DO values (Figs. 2 and 5). It was observed that after the feed period, OUR value declined and DO was in excess of 2 mg/l. However, in the 12-h SBR cycle, with an extended feed period of 4 h, the DO concentration was above 2 mg/l, the threshold concentration normally considered for aerobic systems.

3.6. Simultaneous nitrification–denitrification (SND) process

Simultaneous nitrification–denitrification occurred in the SBR as proved by nitrogen mass balance. SND occurs

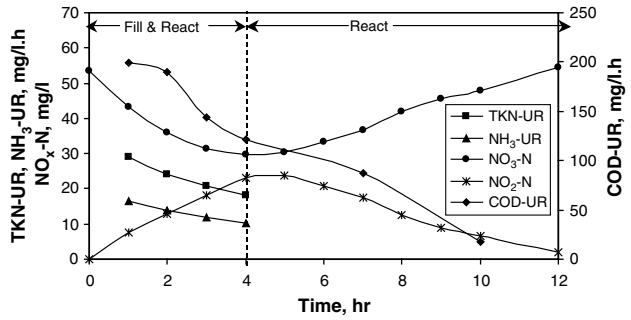

Fig. 6. Substrate (COD, TKN, NH_3-N) uptake rate and NO_x-N profile during 12-h SBR cycle.

as a consequence of DO concentration gradients within microbial flocs or biofilms due to diffusional limitations. That is, the nitrifiers exist in regions with high DO concentrations, whereas the denitrifiers will preferentially be active in zones with low DO concentrations (Münch et al., 1996).

Fig. 6 shows the NO_2-N and NO_3-N profiles during the 12-h SBR cycle. Accumulation of nitrite was observed during the feed period, after which conversion of nitrite to nitrate was predominant. Nitrite accumulated as long as low DO levels prevailed (during the feed period) and was no longer built up when the DO concentration during the react period was substantially higher than the oxygen half-saturation coefficient of Nitrobacter (Münch et al., 1996).

The rate of nitrification during 12-h SBR cycle was obtained, as the average ammonium uptake rate (AUR), i.e., 13.2 mg/l h (Fig. 6), and the specific nitrification rate was 6.9 mg/g MLVSS d. The amount of TKN removed in each cycle ($TKN_{end\ feed} - TKN_{effluent}$) was always greater than the amount of oxidised nitrogen ($NO_x - N_{effluent}$). For example, as shown in Table 2, it resulted that the TKN removed in a typical cycle was 93.4 mg/l (134.9 − 41.5 = 93.4 mg/l) whereas the oxidised nitrogen was 24.8 mg/l (from Fig. 6). Out of the difference, 68.6 mg/l (93.4 − 24.8 = 68.6 mg/l), 21.9 mg/l (365 mg VSS/l × 0.06 mg N/mg VSS = 21.9 mg/l) was ascribed to the nitrogen necessary for the biomass growth (where, 365 mg/l is the excess sludge produced). The remaining nitrogen, 46.7 mg/l (68.6 − 21.9 = 46.7) was denitrified. The rate of denitrification was 11.7 mg N/l h and the specific rate of denitrification was 6.24 mg/g MLVSS h. These values are within the reported values (Henze, 1991; Kujawa and Klapwijk, 1999). However, in order to establish the exact rate of denitrification, a complete nitrogen balance including TKN and possibly nitrogen gas concentration measurements would have to be carried out.

Complete denitrification requires an easily biodegradable substance, which might be present in the wastewater or added externally as acetic acid, methanol or hydrolysed molasses (Quan et al., 2005). The rates of denitrification were studied with acetic acid as the external carbon source (Obaja et al., 2003) and non-digested pig manure as an internal carbon source (Obaja et al., 2005) in the SBR treating piggery wastewater. Higher mean removal rates were obtained with the internal carbon source and it proved to be an advantage in terms of the savings in chemicals.

The presence of high RBCOD fraction (66–70%) in tannery wastewater could be utilized for complete denitrification in the SBR, for which an additional anoxic fill phase needs to be worked out.

4. Conclusions

The study showed that respirometry combined with SBR was an effective tool for the removal of COD in tannery wastewater. Data collected enabled the determination of an optimum operating cycle for tannery wastewater. The OUR profile followed closely that of COD removal. About 74–88% of COD utilized was explained by OUR measurements, justifying the reason as to why COD should be used as model component for the design of biological treatment systems. Measurement of OUR and corresponding COD uptake rates showed that the 12-h operating cycle was able to achieve similar removal efficiencies as that of the 24-h cycle. These removal efficiencies were much higher than the continuous aerobic systems, which showed that SBR could also be used as a high-rate process. Such enhanced performance with SBR over conventional activated sludge process was perhaps due to the enforced short-term unsteady state conditions, which facilitated the required metabolic conditions for treatment of wastewater.

Although the proposed method of COD fractionation was quite simple, it was inadequate in the sense that proper differentiation between the rapidly and slowly hydrolysable COD was not drawn completely. However, this differentiation was not important as both these fractions were utilised simultaneously during the feed phase of the SBR. The inert COD fraction also contained residual microbial products, which was not estimated. But, the problem associated with the high effluent COD in tannery wastewater is global, and the solution lies in the development of more eco-friendly tanning chemicals. Once this could be realised, SBR coupled with respirometry could be a cost-effective and a clear alternative to the conventional biological system for the treatment of tannery wastewater.

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