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DESIGN FOR NITROGEN REMOVAL FROM TANNERY UNHAIRING WASTE

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BACKGROUND AND METHODS

The production of tanned leather from cattle hide requires a complex series of wet chemical processing steps which yield an equally complex series of high load effluents (Table I). In the Engineering and Development Laboratory, ERRC, we believe that the

Table I. Chrome Tannery Effluent Stream Loads [4].

Effluent Stream	Flow (%)	BOD (%)
Soaking	11.8	17.7
Unhairing	11.8	58.9
Liming	23.5	11.8
Bating	14.1	1.0
Chrome Tanning	3.5	4.7
Retan, Coloring and Fat Liquoring	23.5	2.4
Finishing	11.8	3.5
Total	100.0	100.0

best prospects for effective treatment of these effluents lie in the following steps: (a) physical-chemical pretreatment of segregated high load waste streams followed by (b) the pooling and biological treatment of the chrome-free streams not suitable for reuse. We have, therefore, developed a physical-chemical (P/C) pretreatment for lime-sulfide unhairing effluent—clearly the most concentrated of the effluent streams [1]. This P/C-treated effluent has served as the substrate in our investigations into denitrification, the subject of this chapter.

Table II shows the proximate composition of the unhairing waste as received from Ocean Leather Company (Newark, NJ) and the effluent from our physical/chemical pretreatment. Pretreatment removed about 80% of the chemical oxygen demand (COD) and total Kjeldahl nitrogen (TKN), and 95% of the sulfide was recovered. The remaining sulfide was oxidized. In spite of this substantial reduction, the pretreated effluent was still relatively strong waste and was diluted to about 200 mg/l TKN for much of the experimental work.

The nitrogen removal method selected for study was a single-stage denitrification system with the influent COD used to drive the nitrogen removal (see Figure 1 combined nitrification/denitrification). This system constitutes the first three zones of Bardenpho denitrification [2]. Here the waste is nitrified in the aerobic zone. The majority of this nitrified waste is then recycled to the first anoxic zone, where it is denitrified at an exogenous rate. Further denitrification is carried out in the second anoxic zone at a reduced rate. The first two zones of this system are, in principle, identical to oxidation ditch-type denitrification systems (e.g., Orbal, Lakeside and Carrousel systems) [3].

	Unhaired Effluent (mg/l)	Physical/Chemical Pretreated Unhaired Effluent (mg/l)
BOD ₅	14,000	2,000
COD	31,600	4,700
Sulfide (as S ⁼ Au)	2,000	< 1
TKN (as N)	3,000	420
Ammonia (as N)	200	60
Nitrite (as N)	—	< 1
Nitrate (as N)	—	< 1
Alkalinity (as CaCO ₃)	18,000	250

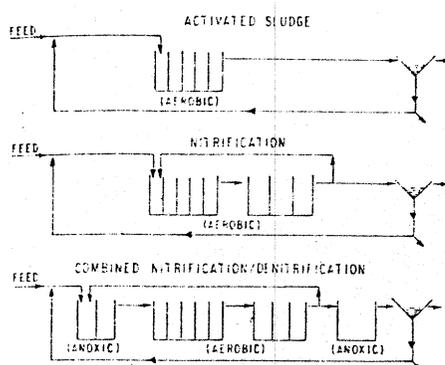


Figure 1. Biological treatments studied.

Operationally, the only major difference is the ratio of the internal recycle rate to the feed rate.

Combined nitrification/denitrification systems such as the one under investigation, possess a number of potential advantages. Because the waste water is the source of organic carbon for denitrification, no methanol is required. Further nitrate recycled to the front of the system serves as a source of alkalinity and oxygen, thus reducing the requirement for each. The major disadvantage of combined systems is the low rate of denitrification and the consequently large anoxic volume required [5]. The focus of attention in this presentation will be the factors that affect the rate of denitrification. The approach taken was to treat nitrate simply as an alternative to oxygen in combusting COD. Factors that affect the rate of utilization of the appropriate oxidant were observed as a laboratory treatment system was expanded from simple activated sludge to nitrification and finally to combined nitrification/denitrification.

In the first series of continuous experiments, a simple activated sludge treatment scheme was used (see Figure 1). The 10-liter plexiglas aeration basin was divided into five equal stages, approximating plug flow. The hydraulic retention time was 10 hr based on the nominal feed rate of 1 liter/hr. Each stage was aerated at a rate of 2-2.5 liter/min per liter of basin volume through coarse-fritted glass diffusers. The high aeration rate was necessary to insure complete suspension of the basin solids. The solids return was 25-45% of the feed rate. The solids retention time for the system (SRT_g) was maintained at two days based on the total mixed liquor volume. The clarifier was a 9-liter plexiglas cylinder with V-notch overflow weirs. The cylinder rested on a magnetic stirring table used to agitate the settled solids 2-4 sec every minute. Excess solids were wasted from the clarifier.

In the second series of continuous experiments, the SRT_s was increased to 20 days to promote stable nitrification. In anticipation of the resulting larger aeration solids inventory, we added a second 10-liter basin, doubling the total volume. The two basins were considered to comprise a single aerobic zone. The second basin was partitioned into three equal stages. Excess solids were wasted from the last aeration stage. Feed and solids return rates remained essentially the same. An internal mixed-liquor recycle stream was added to approximate the flow pattern of the combined nitrification/denitrification system. The internal recycle rate was varied (0-3 times the feed rate) in an attempt to broaden the range of substrate concentrations encountered across the aeration basins.

In the last series of continuous experiments, two anoxic basins were added to the treatment scheme, approximating the first three zones of a Bardenpho scheme. The anoxic basins were covered and sparged with nitrogen to exclude oxygen. The first basin was three liters and was partitioned into two equal stages. The second basin was four liters and single-staged. The SRT_s was maintained at 20 days as before, and again an internal recycle stream was employed to return the nitrified mixed liquor to the front of the processing line.

Batch fill-and-draw experiments were conducted concurrent with the two-day SRT_s continuous experiments (Figure 2). The continuous reactor supplied the source of

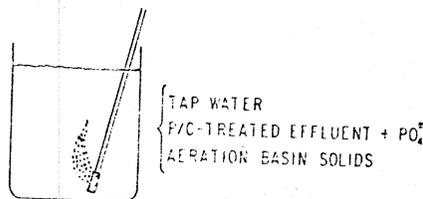


Figure 2. Schematic for batch fill-and-draw experiments.

biomass (volatile suspended solids) for the batch work. The aeration basin was rectangular and four liters in working volume. A fritted glass diffuser was used for aeration and mixing as in the continuous work. At the start of each experiment, tap water, P/C-treated effluent, potassium phosphate and settled solids from the last aeration stage of the continuous system were added to the batch basin. This yielded a mixed liquor having about 500 mg/l volatile suspended solids (VSS), a COD ranging from 500-4000 mg/l and a COD to phosphorus ratio of 150:1. After equilibration for 30 minutes, VSS increase was measured as a function of time over a 6-hr period. The average of the CODs taken at the time of the first and last VSS sampling was considered to be the COD during each experiment.

All of the results reported were obtained at 18-20 C.

THEORY

Substrate Utilization

Substrate utilized and net growth of biomass can be related as:

$$(ds/dt) = 1/Y_g (dx/dt) + mx \quad (1)$$

where

- (ds/dt) = rate of substrate utilization by the biomass (mg/l·hr)
- Y_g = yield of biomass on substrate used for growth (mg/mg)
- (dx/dt) = net growth rate of biomass (mg/l·hr)
- m = maintenance constant (mg/mg·hr)
- x = concentration of biomass (mg/l).

The constant m may be seen as a lumped parameter related to nongrowth energy consumption.

The specific growth rate of biomass (μ) is defined as:

$$\mu \equiv (1/x) (dx/dt) \quad (2)$$

so that in terms of a specific rate of utilization, Equation 1 becomes

$$(1/x) (ds/dt) = (1/Y_p) \mu + m \quad (3)$$

Specific growth rate is usually expressed as a function of the limiting substrate concentration. Common expressions for low, moderate and high substrate concentration, respectively, include a first-order model, a Monod model and an inhibition model—Equations 4, 5 and 6 [8].

$$\mu = Ks \quad (4)$$

$$\mu = \mu_{max} s/(K + s) \quad (5)$$

$$\mu = \mu_{max}/(1 + K_s/s + s/K_I) \quad (6)$$

where K = rate constant (mg/mg·hr)/mg
 s = substrate concentration (mg/l)
 μ_{max} = maximum growth rate (mg/mg·hr)
 K_s = saturation constant (mg/l)
 K_I = inhibition constant (mg/l)

At substrate concentrations encountered in the waste treatment field, the first-order model has often been found satisfactory [9]. Using the first-order model and chemical oxygen demand (COD) as the measure of growth-limiting substrate, we obtain:

$$(1/x) (ds/dt) = (K/Y_p) (COD) + m \quad (7)$$

Thus, the specific rate of utilization can be expressed as a linear function of COD. If stage-to-stage hysteresis can be neglected, Equation 7 can be used to relate the specific substrate utilization rate to the COD at each stage in the basin. Substrates to be evaluated are oxygen and nitrate.

System and Local SRT

In a completely mixed treatment system, solids retention time (SRT) is often defined on the basis of total basin volume

$$SRT_s \equiv (x')/(\Delta x'/\Delta t) \quad (8)$$

where x' = inventory of basin volatile suspended solids (mg)
 $\Delta x'/\Delta t$ = volatile suspended solids wasting rate (mg/hr)

At steady state, the rate of solids wasting will be equal to the rate of solids production in the treatment basin. Thus,

$$(1/x) (dx/dt) \doteq (1/x') (\Delta x'/\Delta t) \quad (9)$$

and

$$1/SRT_s \doteq \mu [6,8] \quad (10)$$

In a staged system, such as the one studied here, the same calculation can be used to determine an overall system SRT (SRT_s) and an overall growth rate (μ_s). However, in a staged system, each completely mixed stage is apt to possess a distinct COD and hence a distinct local growth rate (μ_L). A local SRT (SRT_L) can thus be defined as:

$$1/SRT_L \equiv \mu_L$$

As will be shown, both SRT_s and SRT_L play an important role in determining the specific substrate utilization rate.

RESULTS AND DISCUSSION

Batch Fill-and-Draw

The results of the batch fill-and-draw experiments are shown in Figure 3. Each point represents an individual experiment wherein \ln VSS was plotted as a function of time. The slope of the linear portion of the curve obtained is the specific growth rate shown. The COD is the average soluble COD during the experiment. Over the range of COD studied, there is a region where each of the growth rate models is suitable. Below 750 mg/l COD the first-order model appears satisfactory. First-order kinetics was therefore assumed in the continuous work.

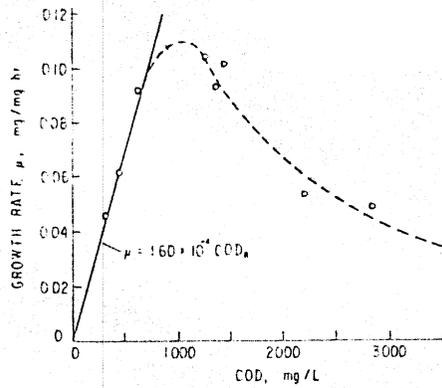


Figure 3. Growth rate as a function of COD in batch fill-and-draw experiments.

The specific growth rate is expressed as a function of COD_R —removable COD—rather than soluble COD. In tannery waste, not all of the soluble COD is removable through biological means [10]. In the continuous experiments conducted concurrent with the batch study, extrapolations indicated that about 90% of the soluble COD was removable. This factor is incorporated into the correlation shown.

Activated Sludge at $SRT_s = 2$ Days

Figure 4 shows the local specific oxygen uptake rate ($SOUR_L$) as a function of the

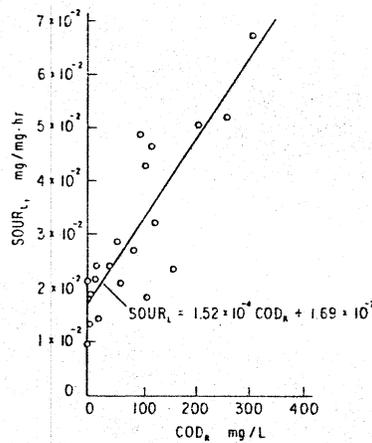


Figure 4. Local specific oxygen uptake rate ($SOUR_L$) as a function of removable COD (COD_R).

removable soluble COD. By use of a linear model:

$$SOUR_L = 1.52 \times 10^{-4} COD_R + 1.69 \times 10^{-2} \text{ mg/mg}\cdot\text{hr} \quad (11)$$

$1.69 \times 10^{-2} \text{ mg/mg}\cdot\text{hr}$, the maintenance constant, is the rate at which oxygen is consumed by the VSS at no net growth. The high value obtained at $SRT_s = 2$ days indicates that a large fraction of the VSS is active biomass [11].

With this constant, it is possible to use $SOUR_L$ data and μ_L data to obtain $1/Y_g$, the growth yield constant. The substrate utilization equation thus obtained is:

$$SOUR_L = (1/81)\mu_L + 1.69 \times 10^{-2} \text{ mg/mg}\cdot\text{hr} \quad (12)$$

Based on Equations 11 and 12,

$$\mu_L = 1.25 \times 10^{-4} \text{ COD}_R \text{ mg/mg}\cdot\text{hr}$$

Note that in Equation 12 Y_p is the yield of VSS per unit weight of oxygen used for growth. The yield of sludge on a COD basis was approximately 0.37 mg/mg which compares quite favorably with literature values [7].

When COD_R is known, Equations 11 and 13 can be used to calculate the oxygen consumed and biomass grown in any stage. However, the COD_R is not generally known and its prediction is made difficult because of the adsorptive properties of the basin solids. In Figure 5, the curve labeled CODR is the measured rate at which COD was removed from

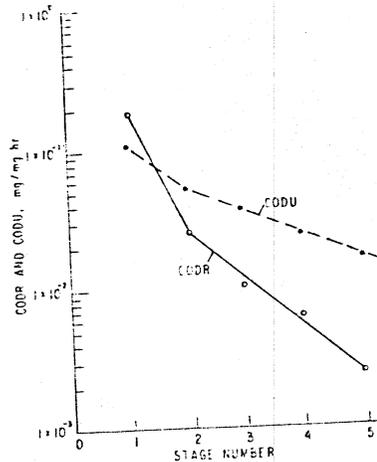


Figure 5. Specific COD removal and utilization rate (CODR and CODU) as a function of basin stage at $\text{SRT}_s = 2$ days.

solution at each stage in the basin. The CODU curve is the rate at which COD was utilized. Points on the CODU curve are calculated and are the sum of the COD burned (SOUR_L) and the COD converted to biomass (Equation 13 and 1.48 mg COD/mg biomass).

In the first stage COD is removed from solution at about twice the rate it is utilized. It therefore appears that a sizeable portion of the unutilized COD is adsorbed by the basin solids in the first stage. A complete model for the design of a multistage process must therefore account for adsorptive effects. Such adsorption models are being evaluated.

Equally significant to the direct effect adsorption has on design is the indirect effect of adsorption on the substrate utilization rate. According to the model presented, the substrate utilization rate (e.g., SOUR_L) is a function of the soluble COD and is consequently unaffected by the concentration of adsorbed COD. If so, the adsorption process, which lowers the concentration of COD in solution, indirectly reduces the rate at which substrate will be utilized.

Nitrification at $\text{SRT}_s = 20$ Days

Before discussing the nitrification results, it is appropriate here to mention two changes made in the composition of the P/C-treated effluent in order to obtain complete stable nitrification.

Prior to the nitrification experiments, ferric chloride was added to the P/C-treated effluent to oxidize residual sulfide. As a consequence, the effluent contained 200-300 mg/l iron. Examination of the data revealed that nitrification was observed only during periods when ferric chloride addition was relatively low. The use of this oxidant was therefore discontinued in favor of hydrogen peroxide.

The second change made in composition of the P/C-treated effluent was to increase the alkalinity by adding sodium bicarbonate (NaHCO_3). The alkalinity of P/C-treated effluent was found sufficient to support only partial nitrification (i.e., a 50% removal of TKN.) In order to achieve complete nitrification and provide a residual of 100 mg/l alkalinity as

required for good clarification of solids, it was necessary to add 2.0 g of NaHCO_3 per liter of effluent. Denitrification reduced this requirement to 0.6 g.

The results of the 20-day SRT_s experiments are shown in the lower half of Figure 6.

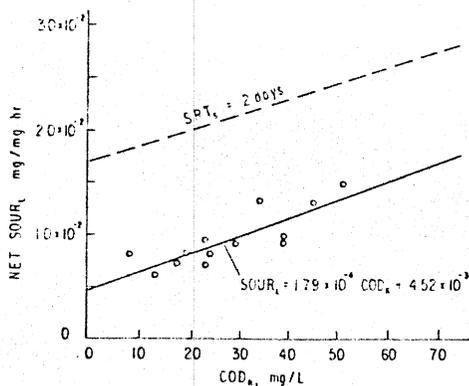


Figure 6. Net specific oxygen uptake rate (SOUR_L) as a function of removable COD (COD_R) at $\text{SRT}_s = 20$ days and $\text{SRT}_s = 2$ days.

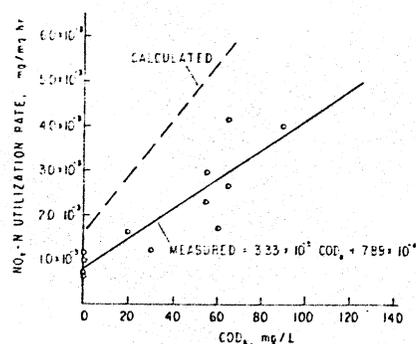


Figure 7. Measured and calculated nitrate and nitrite ($\text{NO}_x\text{-N}$) utilization rate at $\text{SRT}_s = 20$ days.

The regression line for the 2-day SRT_s work is shown at the top. The ordinate values are expressed as net SOUR_L s. These values were calculated as measured local oxygen uptake rates less the oxygen consumed in the production of nitrate from ammonia. Across the range of COD_R encountered, the SOUR_L for the longer SRT_s is significantly lower than that for the shorter SRT_s . This reduction in substrate utilization rate with increasing SRT_s is generally viewed as a reduction in the biological activity of the VSS in the basin solids—as SRT_s is increased the fraction of the VSS that is active biomass decreases. With this model one would expect the ratio of SOUR_L s for the two SRT_s to be constant across the COD spectrum. Consequently the slope for the 20-day SRT_s line should be smaller than that of the 2-day SRT_s . The slopes, however, are not significantly different at the 95% confidence level. No satisfactory explanation has yet been found for this discrepancy.

In spite of the discrepancy, two clear patterns emerge. First, high SRT_s , which is required for nitrification, markedly reduces the rate at which oxygen can be utilized. In particular, at or near maintenance conditions, this rate can be reduced as much as 75%. Second, low SRT_L , achievable at the front end of the treatment system through staging, can offset much of the loss in rate encountered by increasing SRT_s .

Denitrification at $\text{SRT}_s = 20$ Days

The results of the denitrification experiments are shown in the lower portion of Figure 7. These data are the denitrification rates or the rates of decrease in nitrate and nitrite measured as nitrogen ($\text{NO}_x\text{-N}$). In general the ratio of nitrate to nitrite was greater than 10:1 so that this curve is largely representative of the nitrate denitrification. The upper curve is the SOUR_L regression equation obtained in the nitrification experiments expressed here in terms of an equivalent denitrification rate. The conversion from oxygen to $\text{NO}_x\text{-N}$ is calculated on an electron equivalent basis, assuming that all the $\text{NO}_x\text{-N}$ is nitrate (i.e., 2.86 mg O_2 /mg N).

The upper curve provides a means of comparing the measured denitrification rates with those based on nitrification results. As shown at and near the maintenance (endogenous) level, the measured rate is about half the predicted value. This result agrees with similar comparisons found in the literature [12,13]. Thus, denitrification rate can be related to SOUR when compared at the same SRT_s . At higher COD_R the measured denitrification rate is about 40% of the predicted value.

The maintenance constant for denitrification at $\text{SRT}_s = 20$ days is 8.0×10^{-4} mg/mg·hr

of about 0.02 lb/lb-day. Although somewhat low, this figure falls within the observed endogenous rates summarized by the EPA [15]. As COD_R increases, the denitrification rate quickly rises beyond comparable observed values [14]. As was true with the $SOUR_L$ results, the elevated substrate utilization rates can be attributed to staging and resultant low SRT_L at the front end of the treatment system.

In the context of the Bardenpho process, these results demonstrate that although it is possible to improve the rate of nitrogen removal in the first anoxic basin, endogenous denitrification occurring in the second anoxic basin remains painfully slow. For this reason a significantly larger endogenous basin volume is required in order to achieve the same amount of nitrogen removal that can be achieved in the first anoxic basin. The results suggest that with a high load stream, such as the one studied here, it may be possible to reduce or eliminate the endogenous basin volume by use of appropriate front end staging.

CONCLUSIONS

The rate of utilization of oxygen and nitrate have been found to be a function of soluble COD_R . In a staged system, adsorptive effects must be accounted for in order to predict the COD_R profile. The rate of utilization of oxygen and nitrate are adversely affected by the high SRT_S required for combined nitrification/denitrification. Because the greatest portion of the SRT_S in combined systems is that required for nitrification, there is added importance in determining optimum conditions for nitrification. The rate of utilization of oxygen and nitrate can be enhanced by low SRT_L . In particular, the rate of denitrification in combined nitrification/denitrification systems can be enhanced by appropriate front end staging.

DISCLAIMER

Reference to brand or firm name does not constitute endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

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