

AMMONIA REMOVAL FROM DELIMING EFFLUENT BY STRIPPING-REABSORPTION**

Abstract

The removal of ammonia nitrogen from tannery deliming effluent was studied in a pilot scale stripping-reabsorption system with a recirculating air stream. The stripping and reabsorption operations were carried out in columns packed with one-inch Tellerettes. Ammonia removal efficiency in the stripping column was a strong function of the liquid/gas flow rate ratio. At air loading rates of 630-830 cfm/ft² column cross-sectional area, high ammonia removal efficiencies (>95%) could only be achieved at liquid loading rates of 1.5 gpm/ft² column cross-sectional area or less. The effects of process variables are discussed and design data are presented.

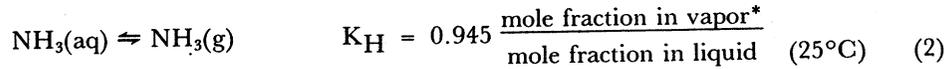
Introduction

The need for control of ammonia nitrogen in wastewaters of tanneries discharging directly to receiving streams remains a distinct possibility in the United States because of its toxicity to aquatic life. The mechanism by which ammonia may become a regulated pollutant is not the final E.P.A. effluent limitation guidelines for the leather tanning and finishing industry as published in 1982 (1) but rather the National Permit Discharge and Elimination System (NPDES) which regulates discharges to receiving waters and is largely administered by the states. Existing tannery wastewater treatment processes do not consistently achieve ammonia reductions required for projected effluent standards. Many biological processes treating tannery wastewaters are not designed for nitrification and in those cases where nitrification does occur, performance deteriorates badly in cold weather. Tannery waste treatment research conducted by the Engineering Science Laboratory of the Eastern Regional Research Center has been guided by the philosophy of water conservation, physicochemical pretreatment of segregated high strength streams, and recovery and reuse of chemicals.

The delime/bate stream is the predominant source of ammonia in tanyard wastewater and contributes a significant portion of the Total Kjeldahl Nitrogen (TKN) loading as well. In Constantin's study of a hide processor line (2), the ammonia from the delime/bate step represented 100 percent of the ammonia nitrogen in the tanyard waste and 33 percent of the TKN. Thus, a logical approach for control of ammonia in tannery wastewater would begin with segregation of this stream followed by treatment for ammonia removal. This paper will describe the results of an investigation of a stripping-reabsorption system for ammonia removal from tannery deliming effluent.

Theoretical. Ammonia is very soluble in aqueous solution in which it acts as a weak base distributing between two forms, the ammonia molecule, NH_3 , and the ammonium ion, NH_4^+ . Two equilibria govern the behavior of ammonia in stripping and absorption opera-

tions; the acid base equilibrium (eq. (1) below) and the Henry's Law relationship governing the equilibrium between the dissolved ammonia species and the ammonia content of the vapor phase in contact with the liquid (eq. (2) below).



The rates of ammonia stripping and absorption operations are strongly dependent upon the equilibrium mole fraction of ammonia in the vapor phase, $y^* \text{NH}_3$, which is often expressed as the partial vapor pressure, $p^* \text{NH}_3 = y^* \text{NH}_3 P_T$, where P_T is the total pressure. The partial vapor pressure of an ammonia solution at constant temperature is in turn a strong function of the solution pH, because of the effect of pH on the concentration of the molecular ammonia species. Table I gives an example of this functionality for a contemplated ammonia stripping operation. In the design of ammonia mass transfer processes, stripping should be carried out at a pH greater than 11 and absorption at a pH lower than 7.

TABLE I
EFFECT OF pH ON AMMONIA VAPOR-LIQUID EQUILIBRIUM AT 25°C
Basis: 5000 mg/l TOTAL AMMONIA

pH	x_{NH_3} = mole fraction NH ₃ in Liquid	Equilibrium NH ₃ Concentration in Vapor		
		$p^* \text{NH}_3$ (Partial Pressure) mm Hg	$y^* \text{NH}_3$	
			Mole Fraction	ppm
6	2.98×10^{-6}	2.14×10^{-3}	2.81×10^{-6}	1.65
12	5.3×10^{-3}	3.8	5.0×10^{-3}	2930

An ammonia transfer process would occur in a gas-liquid contactor in which equilibrium considerations determine the limiting (maximum) degree of transfer but the actual performance of the stripper or absorber is dictated by the rate of mass transfer which is a function of process flow rates and gas-liquid contacting equipment. The relationships between process flow rates and vapor-liquid equilibrium data and their impact on design of stripping towers are schematically presented in Figure 1. (Absorption can be characterized in similar fashion).

An overall mass balance on ammonia for the stripper in Figure 1 yields:

$$Lx_2 + Gy_1 = Gy_2 + Lx_1 \quad (3)$$

where L, G are the liquid and gas molar flow rates and x, y are the mole fractions of ammonia in the liquid and gas streams, respectively, at the indicated tower locations. In Figure 1 a typical design procedure is indicated. In this particular case G, x_1 , x_2 , and y_1 are set by equipment specifications or process requirements. The slope of the line RP yields the maximum value of the flow rate ratio of L/G, since at point P the driving force for mass transfer is zero. With the aid of eq. (3), the maximum liquid flow rate can be calculated. Selection of an operating liquid flow rate such as that represented by line RQ requires the

*Calculated from experimental data in Perry, R. H., and Chilton, C. H., "Chemical Engineer's Handbook," 5th Edition, pp. 3-96, McGraw Hill, New York, 1983. The relationship is linear for the range of mole fractions in this study.

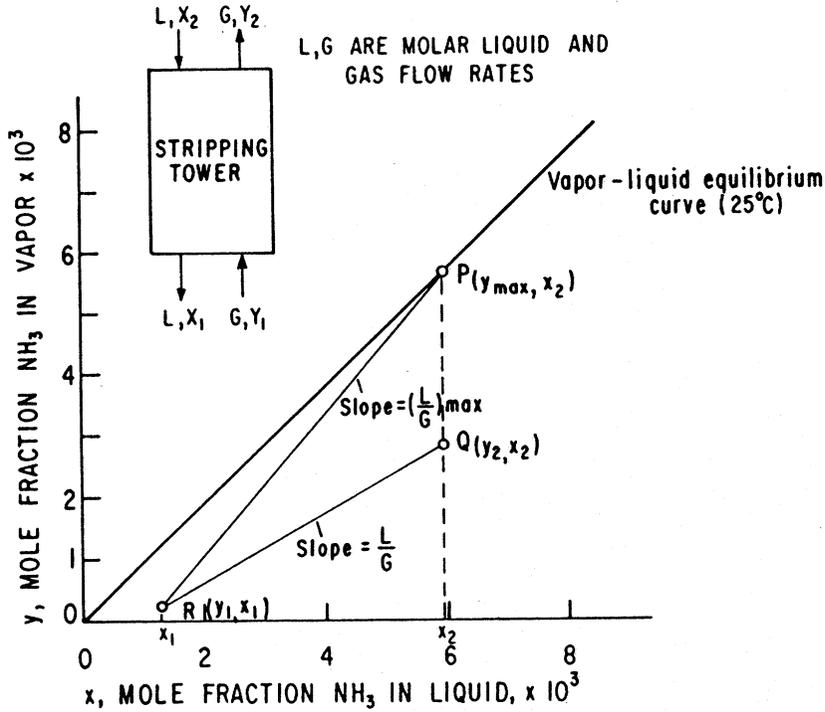


FIGURE 1. — Equilibrium and operating relationships in NH₃ stripping towers.

knowledge of the rate of mass transfer as a function of operating conditions. Rate information will allow calculation of tower height and diameter, from which an economic assessment of the process can be made.

The rate of mass transfer in absorption and stripping towers is generally expressed as a function of either a liquid or gas phase concentration difference. Based on the gas phase driving force, the following expression serves to define an overall mass transfer coefficient, K_y .

$$\frac{1 - y_e}{K_y} \frac{dW}{dA} = y_e - y \tag{4}$$

- where: K_y = overall mass transfer coefficient based on the gas phase, kg-mole/m²-sec
- A = total area for mass transfer in tower, m²
- W = molar mass transfer rate, kg-mole/sec
- y = mole fraction of ammonia in bulk gas phase
- y_e = mole fraction of ammonia which would be in equilibrium with liquid at that point in column.

With the following substitutions:

- $dW = G dy$
- $dA = a S dz$
- G = molar gas flow rate, kg-mole/sec
- a = interfacial area/volume of tower, m²/m³
- s = tower cross-sectional area, m²
- z = tower height, m

Assuming dilute gas compositions ($1 - y_e \cong 1$), rearrangement and integration yields:

$$K_y a = \frac{y_2 - y_1}{(y_e - y) \ln} \frac{G}{S_z} \quad (5)$$

where

$$(y_e - y) \ln = \frac{(y_e - y)_2 - (y_e - y)_1}{\ln \frac{(y_e - y)_2}{(y_e - y)_1}}$$

Equation (5) was used to calculate the values of the overall mass transfer coefficient, $K_y a$, from experimentally determined gas phase compositions in a pilot plant ammonia stripping-reabsorption system.

Literature review. Ammonia stripping has been applied to the renovation of domestic wastewaters, primarily as a unit operation for tertiary treatment of (3-8) or wastewater reuse (9). This research has been conducted on wastewaters containing only 20-80 mg/l total ammonia and has identified the two major problems associated with conventionally operated ammonia stripping towers; poor cold weather performance and buildup of calcium carbonate scale on the mass transfer surfaces. The removal and recovery of ammonia from a high strength industrial waste (2000 mg/l NH_3) was achieved via a desorption-absorption system employing a recirculating air stream (10). Since it is closed to its surroundings, such a system effectively eliminates the major problems associated with previous ammonia stripping towers.

Purpose and scope. Deliming with ammonium salts generally produces a deliming effluent containing approximately 5000 mg $\text{NH}_3 - \text{N/l}$, which is the major, if not sole, source of ammonia nitrogen in tanyard wastewater. The purpose of this study was to investigate the removal of ammonia from deliming effluent in a pilot scale stripping-reabsorption system. Actual tannery deliming effluent was used as the feed and the effects of process variables were examined.

Experimental

A schematic diagram of the pilot plant stripping-reabsorption system is presented in Figure 2. Lime is added to deliming effluent to raise the pH to 12-12.5, solids are allowed to settle, and the clarified effluent is fed to a feed tank prior to being pumped into the stripping column. Ammonia is first stripped from the aqueous feed by a countercurrent recirculating air stream and then reabsorbed from the air stream into a sulfuric acid solution in the absorbing column. The columns, connecting air lines, and absorbing solution holding tank were constructed in Fiberglass* by Croll-Reynolds Company, Inc., Westfield, NJ. The stripping column (8 in. I.D.) and absorbing column (10 in. I.D.) were packed to heights of 12 ft and 11.5 ft, respectively, with one inch toroidal shaped polyethylene Tellerette packing materials. Liquid flow to the columns was distributed by spray nozzles and both columns were equipped with mesh-type mist eliminators. The system is fully instrumented for measurement of temperature and static pressure at various locations and has sampling ports for withdrawal of air samples. The blower assembly consists of a high speed fan (Model No. CMHP6, Ceilcote Co., Inc., Berea, OH), variable speed drive, and a 2-HP motor.

The air flow rate in the unit was determined by the following procedure: ammonia gas was metered into the system at the suction side of the blower at a known rate as measured by a

*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.

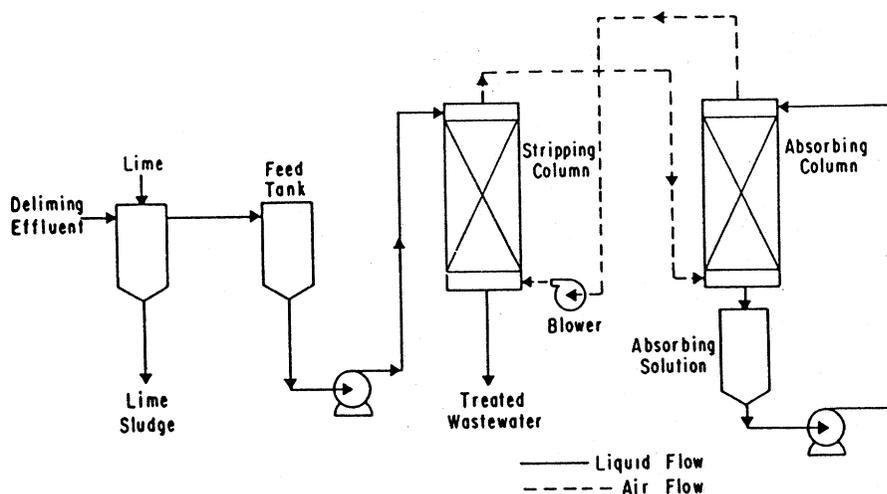


FIGURE 2. — Schematic diagram of stripping-reabsorption pilot plant apparatus.

calibrated rotameter. The absorbing column was operated normally but there was no liquid feed to the stripping column. Measurement of the ammonia content of the air stream allowed calculation of the air flow rate at a given fan shaft speed via a mass balance.

Air samples for measurement of their ammonia concentration were withdrawn by a laboratory Gast pressure-vacuum piston pump. The air sample passed through a rotameter and bubbled through 80 ml of a 25 g/l boric acid absorbing solution containing 1 ml of a methylene blue-methyl red indicating reagent. Upon adjustment of the volume to 100 ml, the boric acid absorbing solution was analyzed for ammonia by a standard titration procedure (11) with the stripping step omitted. The ammonia concentration of the air stream was calculated from the air sampling volume and the total amount of ammonia absorbed in the boric acid reagent.

Chemical analyses and the methods of analysis used in this study were as follows: TKN (12), fat (13), calcium (14), total solids (15), chemical oxygen demand (16), and ammonia nitrogen (11).

Deliming effluent wastewater was obtained from a local tannery; its composition is reported in Table II. The data are presented as averages since the waste was obtained in

TABLE II
COMPOSITION OF DELIMING EFFLUENT

Parameter	Concentration ^a	
	Deliming Effluent	Deliming Effluent After pH Adjustment and Settling
Total solids, %	2.66	1.59
TKN, mg/l as N	4340	4280
NH ₃ , mg/l as N	4230	4470
Calcium, mg/l	1600	2600
Fat, mg/l	1400	1400
COD ^b , mg/l	9700	7900
pH	9-9.5	12.0-12.2

^a Average for all runs

100-300 gal quantities throughout the course of the study. Prior to treatment in the pilot plant stripping-reabsorption system, sufficient lime was added to raise the pH to 12.0-12.5 and solids were allowed to settle for 2 hr. The lime addition and settling had no effect on the TKN or ammonia content of the deliming effluent. The clarified deliming effluent was then pumped into the feed tank and served as the feed to the stripping column.

The experimental conditions for the pilot plant runs are summarized in Table III. Static pressure was measured manometrically via taps placed at the entrance and exit of each column. Liquid flow rate was measured directly. Aqueous samples were preserved prior to analysis by acidification to pH 2 with concentrated H_2SO_4 . Thirty-one pilot plant runs were made during the course of the study.

TABLE III
EXPERIMENTAL CONDITIONS

Parameter	Stripping	Absorption
Liquid rate, $Kg/m^2 - sec$ (gal/min-ft ²)	.42-2.74 (0.6-4.04)	4.08-5.44 (5.50-7.34)
Gas rate $Kg/m^2 - sec$ (cfm/ft ²)	3.73, 4.95 (628, 834)	2.39, 3.17 (402, 534)
Temperature, °C	22-29	22-29
Pressure drop, in H_2O/ft column	.12-.98	.10-.36

Results and Discussion

Absorption. The absorbing solution used throughout the study was a 14.6 percent (w/w) H_2SO_4 solution, pH approximately zero. Under these conditions, equilibrium considerations favor ammonia absorption and the rate of absorption is expected to be high. This was confirmed in actual pilot plant runs in which column absorption efficiency averaged 99 percent. Since absorption under the conditions of the pilot plant experiments was essentially equilibrium controlled, the effects of absorption column process variables were not investigated. The absorbing solution loading rate was maintained in the range 4.1-5.4 $kg/m^2 - sec$ (5.5-7.3 gpm/ft^2) in all runs.

In continuous operation the management of the absorbing solution becomes a processing consideration. Two parameters need to be specified for design purposes; the total amount of absorbing solution and its acid strength. Replacement of the absorbing solution would be determined by these parameters as well as the ammonia concentration and flow rate of the feed solution to the stripping column. Replacement must be done before the pH of the absorbing solution increases to a level where absorption efficiency deteriorates. Figure 3 demonstrates the dependence of absorbing solution pH on absorbed NH_3 concentration for an initial 3.5 N H_2SO_4 solution (approx. 15 percent). This relationship was calculated from the acid base equilibria of the $NH_3 - H_2SO_4$ system. At the stated initial H_2SO_4 concentration the stoichiometric $(NH_4)_2SO_4$ ratio occurs at pH 4.5. The absorbing solution pH should not be allowed to exceed pH 7; thus replacement at the stoichiometric ratio would both maintain absorption efficiency and allow convenient reuse of the $(NH_4)_2SO_4$ for deliming.

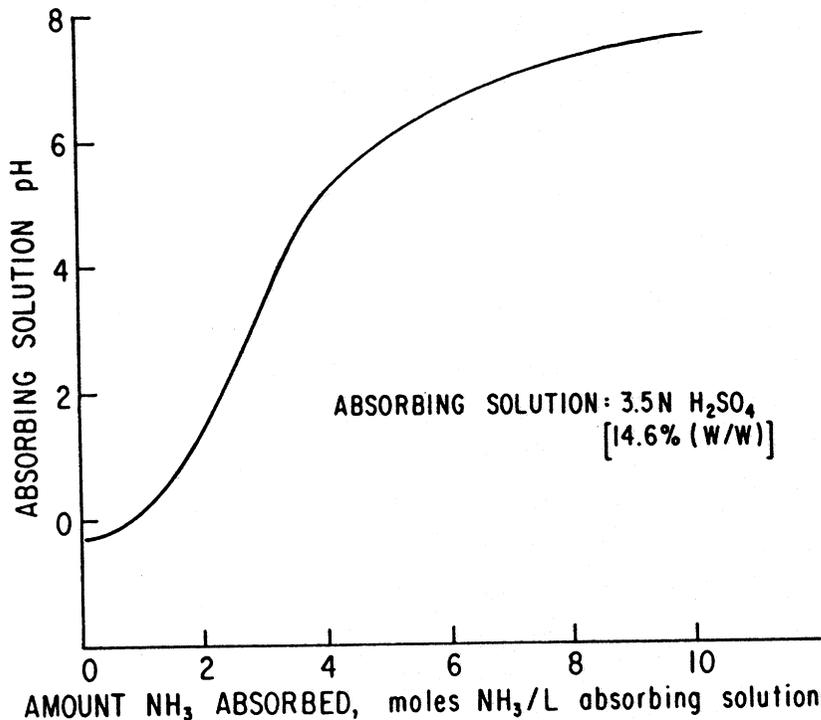


FIGURE 3. — Absorbing solution pH vs. amount NH₃ absorbed for absorption of NH₃ in dilute H₂SO₄ solution.

Stripping. Ammonia is a highly soluble gas and high air/liquid ratios are required for ammonia stripping operations. Results of pilot plant stripping experiments are summarized in Figure 4. Removals of greater than 90 percent are only achieved at rather low values of L/G, 0.4 or less, which corresponds to a minimum air/liquid ratio of 420 ft³/gal. Previous research with ammonia removal from municipal wastewaters via stripping is in quantitative agreement with these results. An Orange County, CA, water reclamation facility (9) utilized a design air-liquid ratio of 400 ft³/gal; a similar facility in South Africa (4) operates at a nominal 616 ft³/gal; in a pilot plant research facility, O'Farrell (8) reported that 90 percent removal at 80°F was achieved at 500 ft³ air/gal of wastewater.

Overall mass transfer coefficients for design calculations were computed from measured gas phase ammonia concentrations and eq. (5). Results of all experiments at low air flow rate are given in Figure 5. These results are in close agreement with the work of Trulsson (10) which reported values of K_{ya} of .03-.05 kg - mole/m³ - sec in a similar system treating a food waste containing 2000 mg NH₂/l. The effect of liquid rate is slight over the range of liquid rates investigated. At high air flow rates the reproducibility of the data was poor and reliable values of the overall mass transfer coefficient could not be obtained.

The factors that must be considered in the design of stripping columns include the liquid wetting of the packing, the gas handling capability of the packing, and equilibrium considerations outlined previously and in Figure 1. The packing used in this study has a high void volume fraction of 83 percent and has been found to be superior to other conventional packings such as Raschig rings because of its ability to promote increased liquid phase turbulence and interstitial holdup (17). The good gas handling ability and performance at low liquid

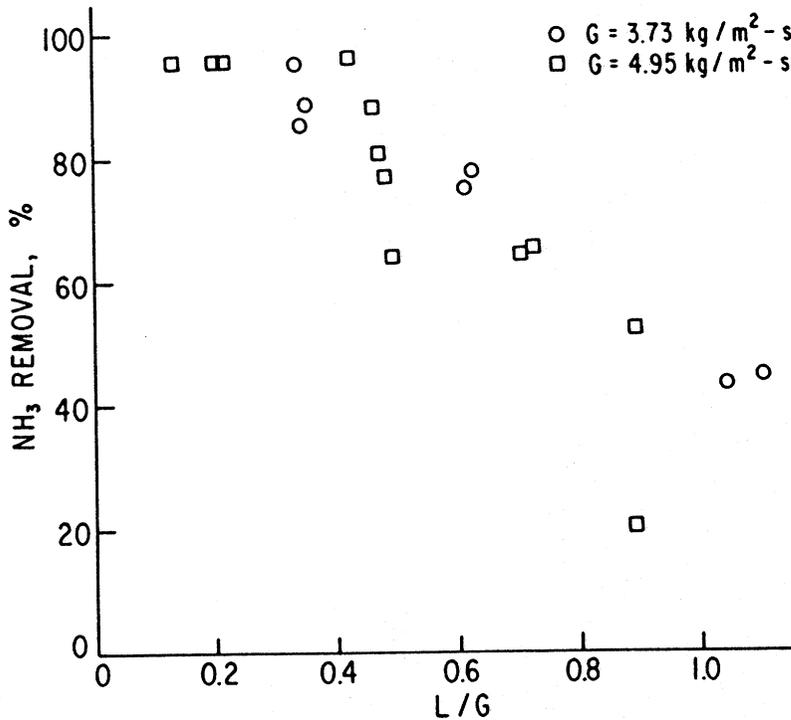


FIGURE 4. — Removal of NH₃ in stripping column. L/G represents flow rate ratio (molar basis).

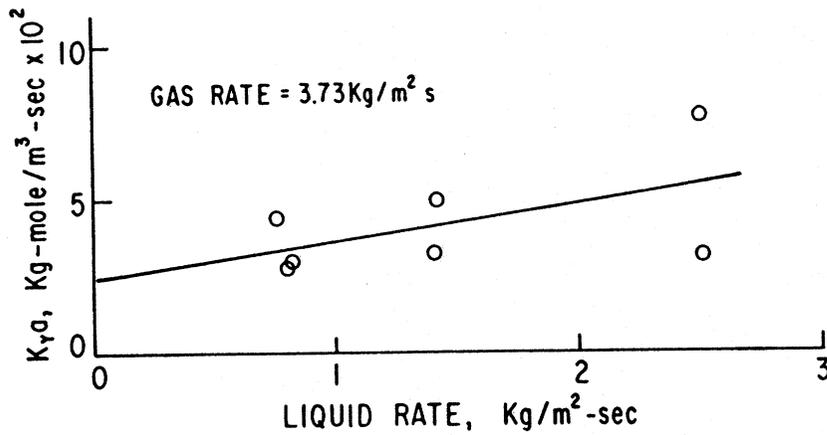


FIGURE 5. — Effect of liquid flow rate on overall mass transfer coefficient for stripping.

loadings make Tellerette packing materials a good choice for this application. This is not to imply that other types of packing would not be suitable but the results and conclusions presented apply only to the Tellerette packing material.

Operational considerations. Previous attempts at ammonia stripping, especially in tertiary wastewater treatment, have identified two serious operational problems; CaCO₃ scale formation and poor performance at low temperature (due to the decrease in K_H with tempera-

ture). A system such as the one utilized in this study eliminates these problems because of the recirculation of the air stream. Recirculated air system costs are more sensitive to pressure drops in columns and piping, however. In the pilot plant apparatus, stripping column pressure drop averaged 0.36 in H₂O/ft column packing and 0.72 in H₂O/ft column packing at air rates of 3.73 kg/m² - sec (628 cfm/ft²) and 4.95 kg/m² - sec (833 cfm/ft²), respectively.

Foaming was not observed during any pilot plant runs at the low air flow rate (3.73 kg/m²-sec). However, foaming did occur at the higher air flow rate (4.95 kg/m² - sec) and a chemical antifoaming agent was required.

It must be emphasized that removal of ammonia nitrogen from the segregated deliming stream does not remove all of the ammonia generating potential of tanyard wastewater. Protein breakdown in downstream biological treatment in conjunction with incomplete nitrification can generate high levels of ammonia in wastewaters of direct dischargers. Tanners confronting the issue of nitrogen control must consider the type and operating efficiency of their biological treatment system if they wish to assess the impact of physicochemical pretreatment for ammonia removal on effluent ammonia concentrations.

Although ammonia recovery as (NH₄)₂SO₄ and its reuse in the tannery is one aspect of this process, this factor cannot be used as an economic justification for the process because of the low cost of ammonium sulfate. A preliminary cost analysis was performed using design data as given below.

Basis 2000 hides/day
 1.35 lb deliming effluent/lb raw hide weight
 24 hr/day operation
 Ammonia content of deliming effluent = 5000 mg/l

Design calculations (symbols are as defined in Figure 1 or text)

Stripping column design:

G = 0.171 kg moles/m²-sec, based upon satisfactory air loading rate for packing
 L/G = 0.2 (experimentally determined, based upon 96% removal)

Therefore:

$$L = 0.2(G) = 0.2(0.171 \text{ kg moles/m}^2\text{-sec}) \\ = 0.0342 \text{ kg moles/m}^2\text{-sec (0.9 gpm/ft}^2\text{)}$$

The total daily flow is:

$$Q = 2000 \frac{\text{hides}}{\text{day}} \times \frac{55 \text{ lbs}}{\text{hide}} \times \frac{1.35 \text{ lbs deliming effluent}}{\text{lb hide}} \times \frac{\text{gal}}{8.33 \text{ lbs}}$$

$$Q = 17,800 \text{ gal/day} \\ = 0.0434 \text{ kg mole/sec}$$

The required column area, A, is thus:

$$A = \frac{Q}{L} = \frac{0.0434 \text{ kg mole/sec}}{0.0342 \text{ kg mole/m}^2\text{-sec}} = 1.276 \text{ m}^2$$

$$A = 13.74 \text{ ft}^2$$

$$\text{and column diameter} = 4.2 \text{ ft}$$

The entrance and exit liquid and gas compositions expressed as mole fractions are as follows:

$$\begin{aligned} X_2 &= 0.005 & Y_1 &= 0 \\ X_1 &= 0.004(1 - .96) = 0.00016 & Y_2 &= ? \end{aligned}$$

Y_2 is calculated from equation (3) text:

$$\begin{aligned} Y_2 &= \frac{L}{G} (X_2 - X_1) = 0.2(0.005 - 0.00016) \\ Y_2 &= 0.001 \end{aligned}$$

The value of K_{ya} used is 0.025 kg mole/m³ - sec (experimentally determined). The column height required is calculated from equation (5).

$$Z = \frac{G'}{K_{ya}} \frac{(Y_2 - Y_1)}{(Y_e - Y) \ln} ; \quad G' = \frac{G}{S}$$

from the equilibrium curve:

$$\begin{aligned} Y_{e2} &= 0.00475 \\ Y_{e1} &= 0.00019 \end{aligned}$$

Upon substitution, $Z = 18.9$ ft

In similar fashion the absorption column can be designed. The gas flow rate was the same as in the stripping column and an overall mass transfer coefficient for absorption was obtained from Trulsson (10). Results of those calculations were:

$$\begin{aligned} Z_{Abs} &= 10.1 \text{ ft} \\ \text{Column diameter} &= 5.2 \text{ ft} \\ \text{Absorbing solution flow rate} &= 5.5 \text{ gal/min-ft}^2 \end{aligned}$$

For design purposes the stripping column was sized at 20 ft high by 5 ft in diameter while the absorption tower was to be 12 ft high by 6 ft in diameter. Based on these sizes and appropriate volumns and sizes of other equipment the cost of a stripping-reabsorption system as depicted in Figure 2 was estimated. Equipment costs were calculated to be approximately \$120,000 (exclusive of land and buildings) while operation and maintenance costs (chemicals, labor, utilities) amounted to roughly \$5.0/1000 gal deliming effluent.

Conclusions

Ammonia nitrogen can be removed from tannery deliming effluent via a stripping-reabsorption system employing a closed recirculating air stream. Ninety percent removal of a 5000 mg NH₃/l feed to the stripping column required 420 ft³ air/gal of feed. Absorption was essentially complete in a 14.6 percent H₂SO₄ absorbing solution. Foaming in the stripping column occurred at high air flow rates but was absent at an air rate of 3.73 kg/m² - sec (628 cfm/ft²). This study has identified a process for possible adoption by tanners faced with control of nitrogen in tannery wastewaters.

Acknowledgment

The authors thank Ronald L. Stabile for the preliminary cost analysis.

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MR. MICHAEL KOMANOWSKY (Eastern Regional Research Center, Discussion Leader): Thank you for an interesting and well presented paper.

MR. STEPHEN SHIVAS (L.H. Lincoln): Did it do anything to decrease the BOD of the effluent?

DR. O'BRIEN: Not really, except for the removal of the ammonia which exerts a significant BOD. The BOD of ammonia is 4.6 mg of oxygen per mg of ammonia, so this is a significant reduction itself. The settling removed a little BOD but our effluent did not have a lot of settleable solids for some reason.

MR. KOMANOWSKY: You mentioned recycling of the air. Would you elaborate on why we recycled and also, is the cost of \$5/1000 gal due mostly to the recycling or to something else?

DR. O'BRIEN: The reason for going to the recirculating air system is that, if you have an open system, the CO₂ in the air, together with the calcium in the water, combine to produce a massive build up of calcium carbonate scale in the columns. Another reason is the effect of temperature on the equilibrium constants if cold outside air is used. The performance deteriorates badly if these open systems are situated outside in cold climates.

In this closed system, a large part of the operating cost is due to pumping the air. That is the most significant operating cost.

MR. KOMANOWSKY: Suppose that a tanner were to treat something other than a bating solution, which is high in ammonia (5000 ppm); would he have the same percentage removals if he started with a lower concentration of ammonia in solution?

DR. O'BRIEN: Using the same column, the lower the concentration the poorer the results. In other words the lower the concentration the lower the percentage removal.