Recovery of Waste Ammonia in Ion Exchange Operation

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In the commercial application of ion exchange to the purification of beet sugar juices as practiced at the Hardin, Montana, plant, ammonia is used to regenerate the anion column. The amount of ammonia used per cycle ranges from 400 pounds to 600 pounds. Of this amount approximately 250 to 300 pounds is fresh make-up and the remainder is recycled regenerant. There are 412 cubic feet of Duolite A-2 resin in each anion column. The fresh ammonia requirements are approximately 5 to 7 pounds per ton of beets. This figure rises as the purity of the beets drops. Ammonia at Hardin

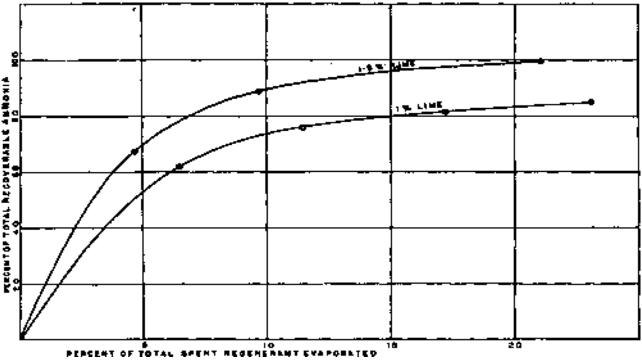

Figure 1.

is very expensive in part because of freight. A study was conducted there with the objective of reducing ammonia requirements in ion exchange by recovery of ammonia through distillation of spent anion regenerant.

In the present system of regeneration at Hardin, after backwashing, a solution of ammonia from 2 to 4% is passed through the column. The effluent from this column goes first to the spent regenerant tank. The flow continues until a large proportion of the impurities held by the anion column has been displaced. At this time the concentration of ammonia in the spent anion regenerant has become relatively high and the flow is diverted to the ammonia make-up tank. This provides a solution relatively rich in ammonia and fairly low in impurities into which is later added fresh

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ammonia for regeneration of the next anion column. Once these requirements have been met the flow is again diverted to the spent anion tank. The spent anion tank and the spent cation tank, which contains acid from regenerating the cation column, are dumped simultaneously to the sewer *in* order that much of the sulfuric acid in the spent cation regenerant can be neutralized by the ammonia.

Preliminary studies were made to determine the approximate concentration and amount of ammonia which could be freed by distillation from the spent anion regenerant. Several results were obtained:

(1) A relatively small proportion of the total volume of spent anion regenerant had to be distilled in order to free a large proportion of the distillable ammonia.

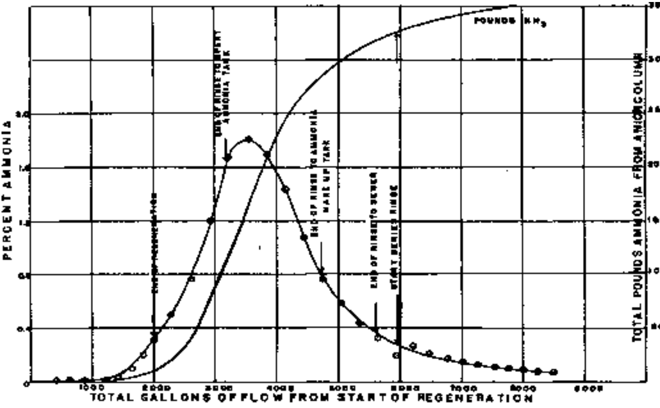

Figure 2.

(2) It was found that if one distilled ammonia from the spent anion regenerant there would be frequently, but not always, a white precipitate which would foul the surface of the condenser.

(3) It was next determined that the use of lime added to the spent anion regenerant allowed one to recover more ammonia than if no lime was added; it allowed one to recover ammonia at a faster rate; it prevented the formation of the white scale on the ammonia condenser.

The amount of ammonia in the spent regenerant was found to vary between 0.5 to 1.0% distillable ammonia. Figure 1 presents results for experiments in which two levels of lime were added to the spent regenerant followed by distillation. It will be seen that if 1% lime is added to spent regenerant that by evaporating 10% of the total volume of liquid one can recover approximately 75% of the total distillable ammonia, and by distilling 20% of the total volume one can recover approximately 82% of the total distillable ammonia. When one adds 1.5% lime, he can recover 90% of the total distillable ammonia by distilling only 10% of the spent re-

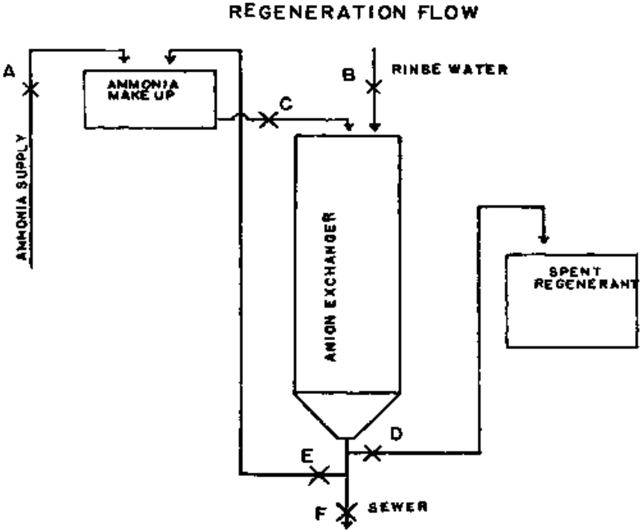

Figure 3.

generant, and finally if one distills 15% of the total volume he can recover 97% of the total distillable ammonia when 1.5% lime is added. In order to obtain the last remaining 3% of the total distillable ammonia one would have to distill another 714% °f tne water. Not shown in Figure 1 are data obtained where 2% and 3% lime on spent regenerant were added. The curves were very slightly steeper at 5% evaporation but practically identical at 10 and 15% evaporation with the curve obtained with 1.5% lime. It was interesting to note that the condensate obtained by distillation of spent anion regenerant had almost exactly the right concentration of ammonia for regenerating the anion column. It should also be pointed out that the results were substantially identical whether the distillation was carried out by heating the regenerant *in* a flask or by injecting live steam into the solution. The latter technique was studied because in commercial practice we would expect difficulty from scaling of heat transfer surfaces.

Figure 2 shows concentrations of distillable ammonia plotted against gallons of flow from the start of the regeneration cycle at the Hardin plant.

REGENERATION FLOW WITH AMMONIA RECOVERY

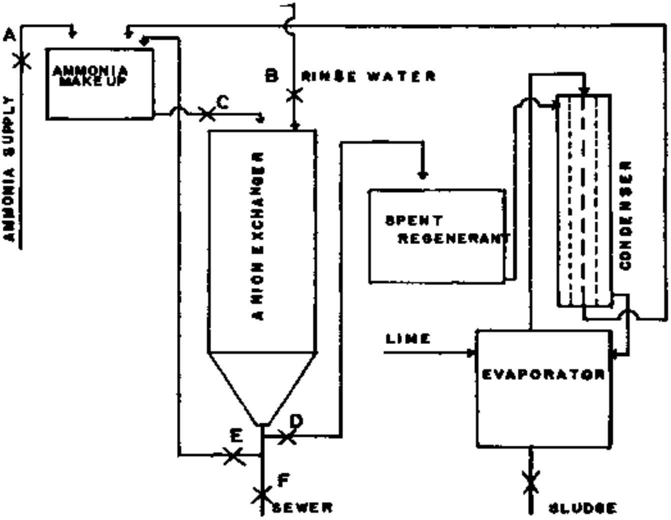

Figure 4.

The top curve shows the cumulative total pounds of ammonia from anion column from the start of the cycle. It shows the various operations in the regeneration cycle. From this graph one can determine what the amounts and concentrations of ammonia would be if one saved only spent regenerant having 0.2% ammonia or greater. This was done, neglecting that amount of ammonia which goes to the ammonia make-up tank since the present anion exchange system already reuses this ammonia profitably. This cut, between 3,240 and 4,740 gallons, is used to dissolve fresh ammonia for regeneration. In the first study made, it was found that 260 pounds of fresh ammonia were used on each cycle and that one could recover by distillation 138 pounds of ammonia from a selected cut of the spent regenerant obtained by the flow volume 1,440 to 3,240 and 4,740 to 5,640. This represents a reduction of approximately 50% of the fresh make-up ammonia requirements. From Figure 2 it was calculated that the percentage ammonia should be 0.75%. A subsequent series of determinations gave a calculated average concentration of 0.81% recoverable ammonia for the same portions of the cycle. A large composite sample (45 gallons), representing these portions and made from several cycles, showed an actual concentration of 0.83% recoverable ammonia. The regenerant solution in these cases was stronger than had been used previously. The recovery would be effected by the use of 1.5% lime and 15% distillation. Economic analyses indicate that this would be a very profitable operation on a plant scale. One of the principal advantages of the system is simplicity. For example, in contrast to many ammonia recovery systems no complicated equipment, compressors, etc., are needed. One simply distills the ammonia, the vapors are condensed on a water cooled condenser and are reused. The water cooled condenser can operate as a preheater for the feed. Figures 3 and 4 show the old system of regeneration and the new system modified for recovery of ammonia. The process is covered by a patent application.