Recovery of Granular Fertilizer from Ion Exchange Spent Regenerants

J. R. JOHNSON[1]

Ion exchange operation in the sugar industry has shown itself to present three major effects.

1. Increased yield of low ash, low sulfite granulated sugar per unit of beets over conventional methods.
2. Economic effects varied by existing costs of equipment, regenerants, and the selling price or demand for final molasses.
3. Broader scope of possible by-product recovery from the beets and spent regenerants.

This paper is a study of the spent regenerant phase in the production of a granular complex ammonium sulfate fertilizer when sulfuric acid and aqueous ammonia are used as the regenerating mediums.

Preliminary experiments were conducted to develop methods of control analysis, to establish maximum pH levels, to investigate preferable boiling procedures and to determine drying temperatures and finished product characteristics (1)[2].

This work set up the following criteria:

1. A pH of 4.5 was found to be maximum to prevent nitrogen loss during concentration and crystallization. Lower pH's are undesirable from a corrosion standpoint.
2. Crystallization in a vacuum pan is best achieved by starting with a footing from the preceding pan.
3. Centrifugal work is best accomplished by drying in the centrifugal to about 8-9 percent moisture before discharging the product to the dryer.
4. The dryer should be operated at not less than 120° C. and should have adequate capacity to handle a fairly large recirculating load. The crystals are very hydroscopic when dried below 120° C. but when properly dried or calcined the mother liquor is charred or baked on the crystal surface and results in a product easily stored in paper bags and easily applied to the soil from drills, etc.

Pilot plant studies consisted of dropping 25 consecutive pans or batches by the in-boiling method described below the measuring yields, etc., throughout the entire operation (2) .

The schematic diagram (Figure 1) of the process flow will aid in following the steps in production as taken up.

Spent regenerants were taken directly from the spent acid and spent ammonia tanks and mixed in a volume ratio of 1 volume of ammonia to 1.04 volumes of acid. This yielded a feed liquor with a pH of 4.5 on the average. Each batch was adjusted to 4.5 by varying either acid or ammonia when necessary.

The mixed solution is then pumped to the evaporator feed tanks where measurements and samples are taken for control data.

[1] Chief Chemist Amalgamated Sugar Company.
[2] Numbers in parentheses refer to acknowledgment of work by others Clisted at the end of this article).

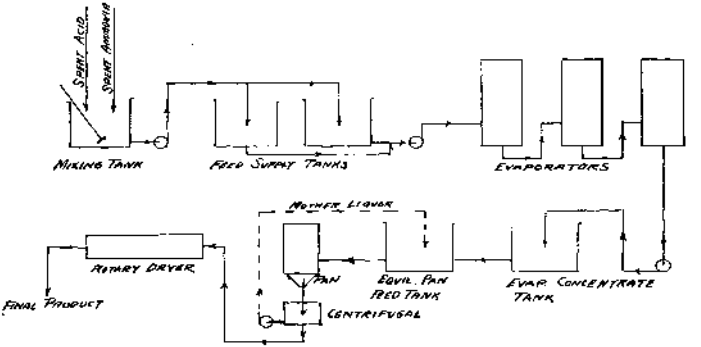

Figure 1. Schematic diagram ammonium sulphate fertilizer pilot plant. Amalgamated Sugar Company, Twin Falls, Idaho.

The evaporators consisted of three effects, the first operating at about 20 p.s.i. and the third under about 15 in. vacuum. Vacuum was provided by a barometric leg condenser. The thin feed enters the evaporators at about 4-5 percent solids and is discharged at about 30 percent solids.

The evaporated liquor was pumped into a primary pan storage tank from which it was measured into the equilibrium pan feed tank.

The equilibrium pan feed tank was operated by first admitting the green liquor spun from the fillmass produced in the preceding pan. The amount of this material is regulated somewhat by allowing the proper amount of mother liquor to remain on the crystals. Next evaporated liquor from the primary pan storage tank is pumped into the equilibrium tank to make up a definite volume of feed necessary for the next pan.

The pan used in this work was a 12 cu. ft. experimental sugar pan constructed from mild steel and equipped with four sets of internal steam coils. Vacuum was provided from a barometric leg condenser assisted by a steam-air-ejector. The pan also was equipped with a mechanical circulator.

The steam pressure on the coils was held at 100 p.s.i. so as to not cause scaling or burning on the coil surface.

Operating conditions were held as uniform as possible but water pressure fluctuations beyond control caused a little difficulty at times. A boiling temperature of 70 to 75° C. is desirable and the fillmass should be fairly loose for best circulation.

By varying the amount of fillmass left in the pan for a footing the desired grain size can be attained and an equilibrium is soon set up throughout the process. In boiling and dropping 25 consecutive pans little difficulty

was encountered in maintaining the proper ratios by in-boiling method. Xhis, we feel, indicates that this method can be carried out indefinitely.

Separation was accomplished in a basket-type centrifugal. Washing is not desired and cycle time regulates the mother liquor retention by the crystals. The green is sent back to the equilibrium pan feed tank as mentioned above.

The dryer consisted of an inclined cylindrical shell, 2.5 ft. in diameter by 12.0 ft. long, similar in characteristics to the conventional granulator. A recirculating scroll was used to feed a blend of partially dried material and wet material into the head end. Heat was applied by means of a kerosene burner which forced the hot air through the dryer and out the stack. Temperature was maintained from 120 to 135° C.

The dried product is black in color due to the film of mother liquor baked on the crystal surface. It has the general shape of commercial ammonium sulphate and is easy to handle.

A typical analysis of the fertilizer produced at Twin Falls is as follows, calculated to approximate combination as single salts:

Ammonium Sulfate	71.5 %
Potassium Sulfate	12.7
Sodium Sulfate	4.9
Calcium Sulfate	1.7
Magnesium Sulfate	0.6
Silica	.05
Phosphate	.02
Amino Acids by Difference	8.5
Total	99.97

The actual forms of combination are uncertain as ammonium and potassium sulfates can form in any proportion. There is also a possibility that glaserite ($3K_2SO_4 . Na_2SO_4$), glauberite ($CaSO_4 . NaSO_4$) or other double salts may form under some conditions. Hydrates seem improbable under the existing operating temperatures but may form.

Screen analysis reported in accumulative percentages:

+ 30 mesh	24.4%
+ 50 mesh	68.6
+ 100 mesh	97.7
+ 100 mesh	2.3

During the experimental run we produced a total of about 4 tons of dried crystaline fertilizer material.

The extraction figures show a loss of dry substance through the evaporators and pans of 11.9 percent, and a dryer loss of 6.85 percent, resulting in an actual yield of 85.1 percent. Due to the fact that on some batches we found that pH's were over 4.5 and at times we found entrainment to occur in boiling, we feel it is safe to estimate a yield of at least 90 percent in a full scale operation where a more uniform control can be attained. Amino nitrogen loss was found to be negligible but on the other hand total nitrogen

loss was high, which bears out the pH error mentioned above. We had some dust loss through the dryer which in a full scale operation could be minimized.

As an estimated figure for actual fertilizer production based on the Twin Falls ion exchange operation and on the facts found in the pilot operation we can realize at least 27.5 pounds of fertilizer per ton of beets sliced or about 275 one hundred pound bags per each 1,000 tons sliced.

This process has not been fully projected into a possible full scale operation as yet but we would like to point out a few possibilities and suggestions.

Successful pilot runs have been made employing continuous crystallization equipment such as manufactured by Swenson Evaporator Company and by Struthers Wells Corp.

In any type of operation particular emphasis must be placed on having a large drying capacity as this is one place where a bottleneck can develop if sufficient capacity is not planned for.

There is evidence that continuous centrifugals such as the type put out by the Bird Machine Co. or a drum type filter may replace the basket type centrifugal.

Mild steel probably would not withstand the low pH for any length of time; however, we found no bad conditions existing during our brief run. No. 316 stainless test plates indicated that this material would last indefinitely.

In actual practice if a pond area is available and if a C.S.F. plant is also contemplated the same boiler and evaporator installations could be used by making a C.S.F. run, then later pumping the spent regenerants from the pond for a fertilizer run. This would greatly reduce installation costs.

In reviewing the above procedure it is readily seen that, by in-boiling and by not washing at the centrifugal, we have eliminated an end product or molasses. The reason for this was that we could not see a ready market for this viscous material even though it is a good fertilizer base for liquid application.

By washing and producing an end liquor we can produce a light colored granular fertilizer which has suitable drying characteristics should a market develop for the end liquor.

After two years of ordinary storage the material we produced is still in first class condition.

Comparisons made with regular ammonium sulfate and spent regenerant fertilizers (3) applied to sugar beets and based on unit nitrogen content resulted in increased yield of tonage and sugar per acre (3) .

From the application tests we found spent regenerant to be equal on unit basis to other ammonium sulfate fertilizers.

ACKNOWLEDGMENT

(1) Ray T. Jacobs, The Amalgamated Sugar Co. 1948.
(2) H. G. Rounds, The Amalgamated Sugar Co. 1949.
(3) C. P. Parrish, The Amalgamated Sugar Co. 1949-50.