Utilization of Concentrated Ion Exchange Nonsugar Waste on Dried Beet Pulp

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Received for publication May 26, 1970

Introduction

The profitable operation of any ion exchange plant necessarily involves the judicious disposal of organic and inorganic waste streams from the regeneration of the anion resin and subsequent salt stripping of cation resins (2). The biological oxygen demand (BOD) of the organic material present in the combined regeneration waste stream requires an alternative disposal approach other than digestive ponding, if BOD loading is to be maintained within normal pond capacity.

Determinations by other investigators (1) have shown that for each gram of nonsugar sorbed by an ion exchange facility 0.5 gram of BOD is eluted on regeneration. Assuming a nonsugar elimination of 20 tons per day, 10 tons per day of BOD will be eluted by the Hamilton City, California ion exchange plant. Analysis of these reconstituted nonsugars (RNS) indicates essentially the same chemical composition as Steffen hot waste (3). It would therefore be advantageous to utilize this potentially valuable by-product, and turn a probable waste disposal liability into an asset. In answer to this disposal problem Holly Sugar Corporation constructed a three effect evaporator station which concentrates these nonsugars and adds this heretofore waste to pressed pulp for drying. The result of this addition is a cattle feed of higher nutrient value and the elimination of 10 tons of BOD per day.

Test Equipment and Procedures

All tests for metal content were carried out with a Beckman Model 979 Atomic Absorption unit equipped with an Aztec Model AB 41 burner which in turn was coupled to a Beckman DB-G Spectrophotometer. Construction and cleaning of test coupons used in the corrosion study were according to Uhlig (6).

Percent protein figures were obtained by analysis of dried pulp for total nitrogen, as described by Harris (4), and the result multiplied by 6.25. Procedures used in obtaining the re-

1 Research Chemist, Supervisor of Analytical Section, and Director of Process Research respectively, Holly Sugar Corporation, Colorado Springs, Colorado.
2 Numbers in parentheses refer to literature cited.
remaining figures on Table 2 are described in Section 22, Grain and Stock Feeds, of the A.O.A.C. Methods of Analysis (5).

Discussion

As previously stated RNS is the resultant effluent of the anion regeneration cycle and the subsequent salt stripping (softening) prior to acid regeneration of a cation column. A solution containing a high concentration of ammonium salts is reclaimed from the anion ammonium hydroxide regeneration cycle for use in the first step of cation regeneration. This ammonia salt stripping solution is rinsed from the cation column and contains recombined cationic and anionic nonsugars (RNS) removed from the ion exchange treated No. 2 green plus thin juice (Figure 1).

Figure 1.—Cation nonsugar strip profile.

Preliminary analysis of Tracy, California Steffen hot waste and Hamilton City, California ion exchange reconstituted nonsugars has shown RNS to have excellent potential as a concentrated Steffen filtrate-(CSF) type concentrate for addition to cattle feed. The analysis indicated that the composition of this waste stream is very similar to the Steffen hot waste with the RNS having two distinct advantages over CSF (3).

1—The average total solids are higher by approximately 43%. Initial RNS-RDS is 5.0, CSF is 3.5. Clearly less water to evaporate.

2—Total nitrogen as expressed on RNS dissolved solids is approximately 30% greater than CSF. This difference is by and large due to the higher ammonia salt concentration of the RNS.

Due to the different type of processes used some differences do occur in the chemical composition of CSF and RNS waste systems. Organic acids, for example, are subject to precipitation as calcium salts in the saccharate cake in the Steffen house, consequently there are less organic acids in the Steffen hot waste than in the ion exchange effluent. RNS is consistently higher
in nitrogenous compounds, sulfates, chlorides and magnesium. Many of the differences are not excessive and can be attributed to the variation in beet quality at the two locations.

The RNS concentrator station consists of two mild steel long tube effects and a concentrator (formerly the Steffen waste concentrators at Alvarado, California factory), capable of concentrating 66.1 GPM of 5 Brix RNS to 60 Brix (Figure 2). Operating conditions of each effect include: 14 PSI pressure at 237° F in the first effect, 10 PSI pressure at 224° F for the second effect and 20 inches of vacuum at 170° F in the concentrator. Output delivery to the concentrated waste tank is 4.3 GPM of 60 Brix RNS concentrate.

Preliminary experimental work indicated that a very serious drop in pH through the evaporators could be expected because of the temperatures involved and the concentration of ammonium salts contained in the waste streams. This pH drop is probably the result of the decomposition of ammonium salts causing the liberation of ammonia which was driven off in the evaporator vapors. During the first week of operation the pH consistently dropped from 9.5 at the evaporator supply tank to approximately 5.2 in the concentrator. As a possible solution to this potentially corrosive pH drop, 43 Brix milk of lime was added to the second effect for a period of 24 hours. No appreciable change in pH was noted in the third effect concentrator. However, a definite increase in ammonia vapors was noted in the second effect vapors. Milk of lime was then pumped in the concentrator at a maximum pump capacity of 800 ml/min. This action resulted in a concentrator pH increase from 5.3 to 6.0. Although the pH was increased slightly, undesirable precipitation of calcium salts, promoting scaling, coupled with extremely high third vapor ammonia levels pointed out the fallacy of pH control with

![Diagram of RNS evaporator station.](image-url)
lime. The CaO was merely converting ammonium salts to calcium salts and releasing free ammonia. Under these circumstances the pH change due to lime addition is understandably small.

If the pH drop cannot be feasibly controlled, the other alternative is to compensate for the corrosiveness of ammonium salts by use of corrosion resistant evaporator materials. An investigation was conducted to determine the extent of corrosion to mild steel evaporator tubes, tube sheets and liquid exposed shell. Samples of feed and RNS from the three effects were collected for a period of 24 hours and analyzed for iron content. Assuming that potassium remains unchanged and is concentrated along with other solids throughout the system, potassium concentration was first determined on each sample set and each of the evaporator samples were diluted to the exact potassium concentration of its feed sample. The iron concentration of each evaporator sample could then be determined, corrected for feed iron and expressed as percent on potassium.

Total weight units of potassium eliminated by the ion exchange plant per day and subsequently discharged in RNS were calculated. For example, the ion exchange plant during the time of testing removed approximately 14 tons of nonsugars/day. Examination of ion exchange nonsugar data indicates that 8.6% of the total nonsugars are potassium and essentially 100% of the potassium is removed during ion exchange. Then 0.086 \times 14 \text{ tons} \times 2000 \text{ lbs/tons} = 2,408 \text{ lbs. potassium/day going through the evaporators.}

Using average percent iron values on potassium the lbs of iron corroded/day may be calculated. In this manner relative corrosion in each effect could be compared directly. Iron corrosion proved to be excessive throughout the concentrator system with 19, 40 and 19 pounds of iron per day, respectively, corroded from the first, second and third effects. Elevating the concentrator pH with lime had little effect on arresting corrosion with 15, 30 and 18 pounds of iron per day corroded from each of the effects.

An additional investigation aimed primarily at finding a suitable material which would resist RNS corrosion confirmed the mild steel corrosion capabilities of the RNS waste streams. Three spool-type specimen holders, each containing mild steel and stainless steel 304 coupons were placed in each of the three effects below the bottom tube sheets. After a 73 day exposure period to RNS concentrate their removal revealed considerable corrosion. Approximately 63% by weight, occurred with the mild steel coupons (Table 1). Stainless steel 304 was totally uneffected by the RNS concentrate and with minimum cleaning each was returned to its original state. An inspection of the mild steel
evaporator tubes revealed considerable pitting and holes. Each effect has necessarily been retubed with stainless steel 304. Evaporator results during the 1969 fall campaign did not indicate any corrosion of the evaporators retubed with stainless steel 304.

Table 1.—Evaporator station corrosion study results.

<table>
<thead>
<tr>
<th></th>
<th>Coupon thickness after exposure</th>
<th>Pitting</th>
<th>% Weight loss</th>
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<tbody>
<tr>
<td>1st Effect</td>
<td></td>
<td></td>
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<tr>
<td>Mild Steel</td>
<td>.014</td>
<td>yes</td>
<td>60</td>
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<tr>
<td>SS-304</td>
<td>.028</td>
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<td>0</td>
</tr>
<tr>
<td>2nd Effect</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Mild Steel</td>
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<td>65</td>
</tr>
<tr>
<td>SS-304</td>
<td>.028</td>
<td>no</td>
<td>0</td>
</tr>
<tr>
<td>Concentrator</td>
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<tr>
<td>Mild Steel</td>
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<td>yes</td>
<td>63</td>
</tr>
<tr>
<td>SS-304</td>
<td>.028</td>
<td>no</td>
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</tr>
</tbody>
</table>

*Original Coupon Thickness—Mild Steel .030
—SS-304 .028

A composite analysis of Tracy dried pulp with CSF and Hamilton City pulp with RNS added, further substantiates the previously stated advantages of RNS addition to dried pulp (Table 2). Total protein available in Hamilton City pulp averaged 13.44% on dry substance, a 24% increase over Tracy pulp with CSF addition. Clearly, there is an increase in the nitrogen content of RNS-enriched pulp. This increase must be attributed to the ammonia nitrogen recovered from the ion exchange regeneration process. All other differences are insignificant and are a function of beet quality or process.

Table 2.—CSF vs. RNS dried pulp analysis.

<table>
<thead>
<tr>
<th></th>
<th>CSF*</th>
<th>RNS**</th>
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<tr>
<td>% Moisture</td>
<td>11.97</td>
<td>9.07</td>
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<tr>
<td>% Crude Fiber</td>
<td>16.03</td>
<td>18.00</td>
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<tr>
<td>% Ash</td>
<td>4.75</td>
<td>3.40</td>
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<tr>
<td>% Protein</td>
<td>10.81</td>
<td>15.44</td>
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<tr>
<td>% Crude Fat</td>
<td>0.31</td>
<td>0.33</td>
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</table>

* CSF Pulp
  20.70% Cane Molasses
  10.20% CSF
** RNS Pulp
  18.97% Cane Molasses
  10.35% RNS

Summary

The concentration of ion exchange RNS waste for addition on pressed pulp before drying is feasible if corrosion resistant materials are employed in evaporators. The use of RNS on pulp has significantly increased the nutritional value of pulp due to higher protein content. The use of RNS on pulp has eliminated a costly waste disposal problem. In retrospect a potential liability has been converted into a definite asset.
Literature Cited


