

On the Utilization of Still Slop from Brandy Making

Recovery of Tartrates

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Previously, one of the authors¹⁾ had used the ion-exchange resins, "Diaion A" and "K" supplied by the MITSUBISHI KASEI Company, Tokyo, Japan, in the recovery of tartrates from pomace extracts.

The present paper presents preliminary results of an investigation of tartaric acid recovery from still slop. The process takes advantage of the remarkable efficiency of the available synthetic ion-exchange materials. The ionexchangers employed in this work are Amberlite IR-120 and Amberlite IRA-410.

MATERIALS AND METHODS

The general procedure followed has been reported previously¹⁾; it consisted of passing the solution to be treated downflow through the columns of the ion-exchange resins, Amberlite IR-120 and Amberlite IRA-410, in the glass tube.

1. *Experiment with a pure solution*

The column characteristics and charges used were as follows:

The experiment was carried out in 35 ml. glass tube containing 20 ml. anion-exchange resin, Amberlite IRA-410 (5.1 g. dry weight) in a column 1.8 cm. diameter and 9.5 cm. deep, with the resin resting on a pad of glass-wool.

The bed was regenerated downflow with 50 ml. of 1 *N* NaOH at an introduction rate of 1 ml. per minute.

The regeneration was followed by a slow rinse downflow with 60 ml. of distilled water at 1 ml. per minute, then a fast rinse at normal downflow of 5 ml. per minute until the effluent alkalinity was such that 10 ml. of effluent showed an acid reaction to methylorange on the addition of 1 drop of 0.1 *N* HCl.

Tartaric acid solution, 0.019 *M* (2.92 g/l.), was employed in preliminary studies: this approximate tartar concentration is frequently found in still slop. Approximately 1.5 l. of the influent solution were passed into the exchanger at the rate of 13 volumes of solution per volume of resin per hour (VVH).

The effluent was collected in 100 ml. samples and quantitatively analyzed for the tartaric acid in the solution.

2. Experiment on a still slop

The over-all efficiency of the tartar recovery process was tested on a laboratory setup consisted of three 11.5 g (dry weight) beds. The three-step ion exchange process which consists of removing the bases with a cation exchanger after adsorption of free acids with first anion exchanger and then adsorbing the free acids on an anion exchanger has been employed. In the case of the anion exchanger, three preliminary cycles (loading with 1% HCl and regenerating with 4% NaOH) were carried out before the still slop has been treated. The regeneration of cation exchanger has already been described²⁾.

A typical sample of slop produced during last year operation at this laboratory was furnished for this work.

A total of 7.5 l. of still slop (tartar content, 0.25 g/100 ml. as tartaric acid) were passed through each column. The influent flow rate was maintained at approximately 10 VVH. The columns were next wash with distilled water until the filtrate was neutral.

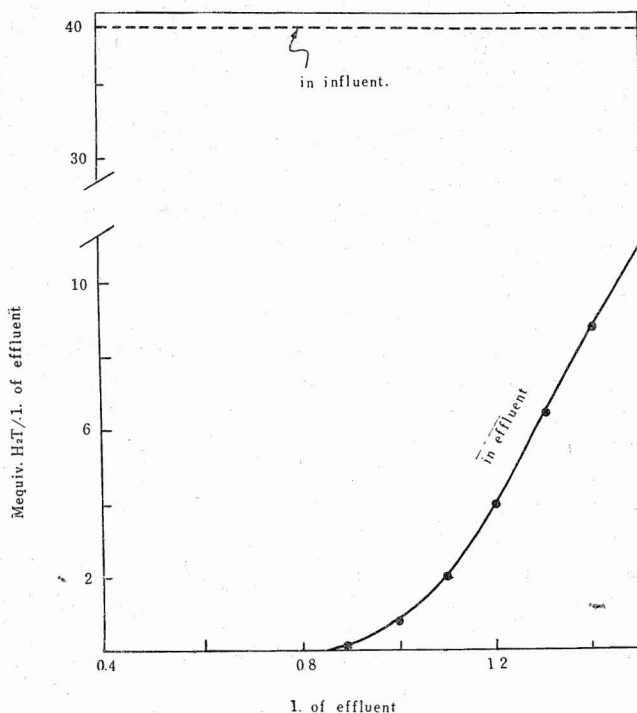


Fig. 1. Adsorption of Tartarate Ion from a Solution of Pure Acid. Dry weight of Bed 5.1 g., Volume about 20 ml., Flow Rate 13 VVH.

The adsorbed materials in the anion exchanger, including tartaric acid, were then recovered with 10 % NaOH at a flow rate of 6 VVH.

Tartaric acid was analyzed by the A. O. A. C. volumetric titration procedure³⁾.

RESULTS AND DISCUSSION

The experiments with pure solution were undertaken to learn the action of anion-exchange resin, Amberlite IR-410, on simple solution of the tartaric acid found in still slop.

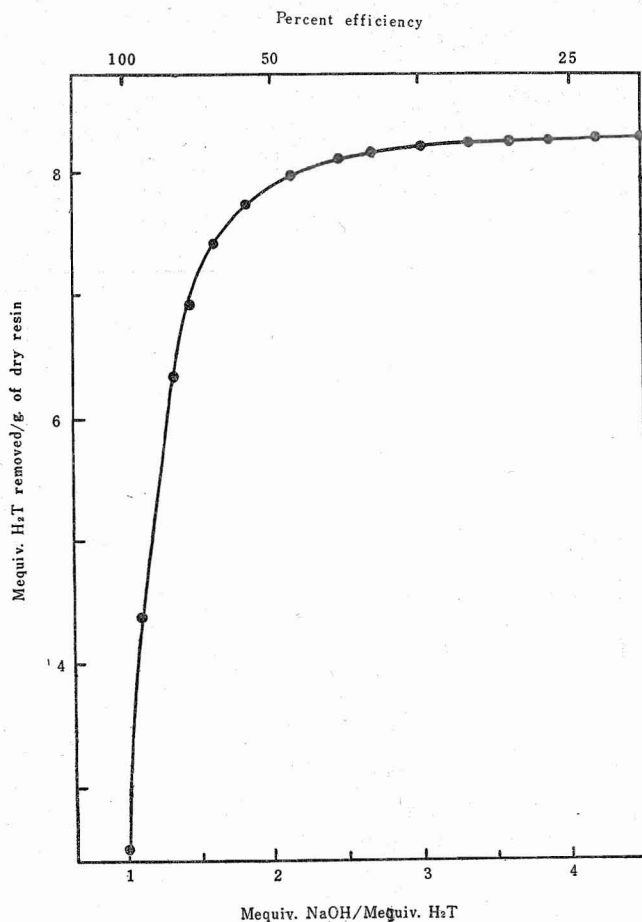


Fig. 2. Efficiency of Sodium Hydroxide Utilization in Regeneration and Concurrent Tartar Recovery, Calculated from Milliequivalents (Mequiv.) of Reagent required per Mequiv. of Tartaric Acid (H₂T) removed from Bed loaded from Solution of Pure Acid.

The break-through point was designated as that point at which the concentration of the tartaric acid in the effluent reached a value which was 5 % of the initial concentration and 95 % was still being adsorbed by the exchanger. The volume of solution passed through the bed to the break-through point times the normality of the solution equals the milliequivalent (Mequiv.) adsorbed to the break-through point. Figure 1 presents data for the adsorption of acid by Amberlite IRA-410 from single solution containing tartaric acid.

When 0.039 *N* (3.9 Mequiv. per 100 ml.) solution of the acid was passed through the exchanger, 38.9 Mequiv. of tartaric acid were adsorbed to the break-through point. The resin effected a nearly adsorption of the acid to the break-through point after which the acid concentration in the effluent increased sharply.

All the tartaric acid was adsorbed from the first 800 ml. of a solution containing 3.9 Mequiv. of tartaric acid per 100 ml. of influent. A 99.8 % adsorption of the tartaric acid was effected from the first one liter of effluent. The adsorption rapidly decreased to about 1 % adsorption of the acid after 1.4 l. of the solution were passed through the Amberlite IRA-410-OH bed,

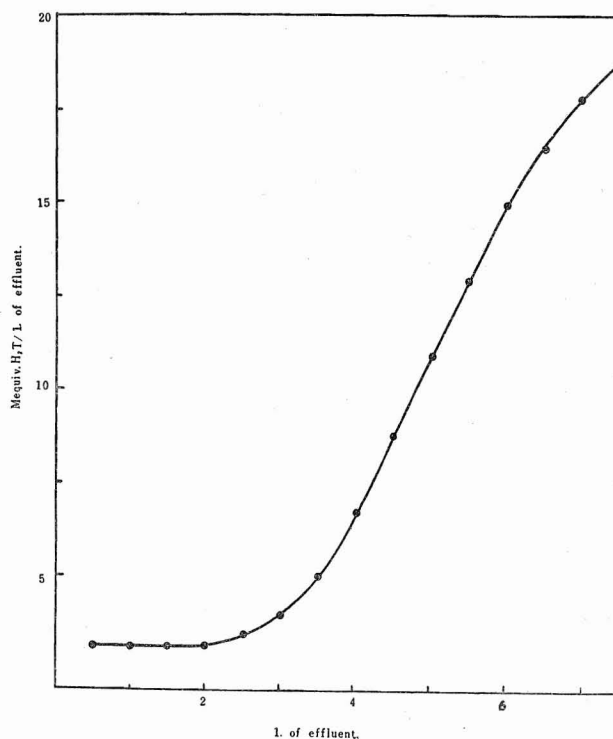


Fig. 3. Adsorption of Tartrate Ion from Still Slop. Dry Weight of Bed 22.4 g., Volume 100 ml., Flow Rate 10 vvh.

and the adsorption leveled off at this value.

Regeneration of the anion-exchange resin serves the purpose of preparing the bed for the recovering the tartaric acid in a concentration suitable for precipitating a calcium salt of high purity in maximum yield¹⁾. The tartaric acid from a bed loaded with the pure acid could be recovered in about 4% concentration when 10% NaOH is used as regenerant (Figure 2).

In case of the experiment on still slop, a 97.6% adsorption of tartaric acid was effected from the first 31. effluent, after which the concentration of the acid in the effluent increased rapidly (Figure 3).

The adsorption of the acid was 1% when 61. of still slop were passed through the columns, and the effluent cut off at this value (Figure 3).

Figure 4 indicates the results with regards to the recovery of tartaric acid from anion-exchanger. Regeneration of the anion exchanger serves the dual purpose of preparing the bed for the succeeding cycle and recovering the tartar in a concentration suitable for precipitating a calcium salt of high purity in maximum yield. We must get a regeneration effluent of 5~6% concentration (calculate as tartaric acid) for the purpose described above. From this viewpoint, it may be observed from Figure 4 that we must carry into effect the four-pass process in regeneration of anion exchanger as the preceding paper¹⁾.

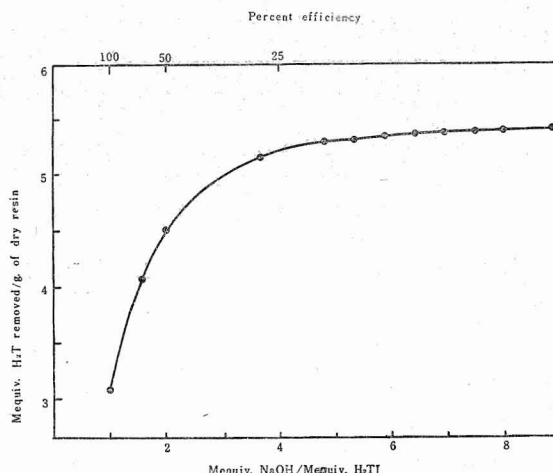


Fig. 4. Efficiency of Sodium Hydroxide Utilization in Regeneration and Concurrent Tartar Recovery, Calculated from Mequiv. of Reagent required per Mequiv. of Tartaric Acid removed from Bed loaded from Still Slop.

SUMMARY

In addition to the work on the recovery of tartaric acid discussed in the preceding paper¹⁾, we have carried out on the recovery of tartrate from still slop.

The still slop (obtained from the distillation of wine in this laboratory) was introduced first into an acid removal unit. Here the anion exchanger base removes the free acid producing potassium tartrate. This was passed through a hydrogen exchanger where the potassium ions are exchange for hydrogen ions. The resulting tartaric acid was removed by the third unit, which contains anion exchanger base.

For recovery, the two anion exchange columns were treated with 10 % NaOH, and we have suggested the four-pass process is essential to this work as the preceding report ¹⁾.

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LITERATURE CITED

- 1) KAGAMI, M.: Studies on the Application of Ion Exchange Process to the Fermentation Industries. Part 1. The Recovery of Tartrates from Winery Wastes. *Bull. Res. Inst. Ferm. Yamanashi Univ.*, **1**, 75 (1954)
- 2) KAGAMI, M.: Studies on the Application of Ion Exchange Process to the the Fermentation Industries. Part 3. Ion-exchange Treatment of Grape Must in Wine Making (I). *J. Fermentation Tech.*, Japan, **33**, 263 (1955)
- 3) *Official and Tentative Methods of Analysis* of the A.O.A.C. 7th Ed. Washington (1950)