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VARIATIONS IN THE COMPOSITION OF STEFFENS WASTE II

EDWARD BARTOW, GEORGE H. COLEMAN AND H. E. REDMAN

Steffens waste is the filtrate from the precipitation of sugar from beet molasses by the addition of lime. The molasses, as it comes from the beet sugar extraction process, contains about 50 per cent sugar, so it is too valuable to be fermented to produce alcohol in this country, although in Europe, much of the beet molasses goes directly to production of alcohol by fermentation.

Of the processes which have been developed for the recovery of the sugar from beet molasses, the Steffens process is the best and most used in this country. In the Steffens process the beet molasses is diluted to a 5 per cent sugar solution; powdered lime is then added to the cold solution (15°C.) which precipitates the sugar as the tricalcium saccharate, the precipitate is filtered off, the filtrate is then heated to 80°C., where more tricalcium saccharate precipitates, the precipitate is filtered off and the liquid from this last filtration is known as Steffens waste and contains only about 0.3 per cent sugar.

There are numerous substances known to be present in Steffens waste, such as malic acid, succinic acid, aconitic acid, citric acid, asparagine, glutamic acid, betaine, sodium and potassium, but of these substances only three, glutamic acid, betaine and potassium are present in relatively large quantities. Potassium is listed as such, because the ratio of organic potassium salts to inorganic potassium salts has not been determined, although it has been indicated that both do exist in the waste.

In 1931 Bartow and Benninghoff (4) obtained samples of Steffens waste from 13 beet sugar factories scattered from Michigan to California. The composition of the different samples varied. The variations were most noticeable in the amounts of glutamic acid, which ranged from 0.0 g. in Colorado to 22 g. per 500 g. of the concentrated waste from Mason City, Iowa.

The wide variations in the amounts of glutamic acid found in the various wastes was very surprising, and because of these surprising variations the present work was undertaken with the thought of improving and standardizing the method of obtaining

glutamic acid from the waste and of verifying the variations found by Benninghoff.

Samples of the Steffens waste were obtained during the 1936 campaign from eleven of the same places from which samples were obtained in 1931 and additional samples from three other plants. All samples of the dilute waste, when received, were treated with carbon dioxide to a phenolphthalein end point in order to precipitate the calcium as the carbonate. The supernatant liquid was then drawn off into a vacuum evaporator where the waste was concentrated to a specific gravity of approximately 1.40. The concentrate was then placed in closed jars and stored in the ice box at 4°C. This concentration was necessary before efficient recovery of any of the glutamic acid, potassium or betaine could be effected.

Several methods have been developed for obtaining glutamic acid, betaine and potassium from Steffens waste.

Andrlik (2) was the first to develop a really successful method of recovering glutamic acid from the concentrated waste. His best method was to add tartaric acid to precipitate potassium acid tartrate. The filtrate from this precipitation was seeded with crystals of glutamic acid and the glutamic acid crystallized out on standing. The yield was fairly good; 60 g. glutamic acid from 1 Kg. of concentrated waste or a 6.0 per cent yield. His two other methods were to precipitate out the glutamic acid and inorganic salts by use of phosphoric acid and sulphuric acid and effect a separation of the glutamic acid by fractional crystallization. The yields of glutamic acid by these methods were not so good, being 30 g. or 3 per cent in both cases.

Barta (3) applied the procedure used by Ehrlich on beet molasses to the concentrated Steffens waste. He extracted the betaine with ethyl alcohol, precipitated the potassium by addition of concentrated HCl, filtered off the KCl and concentrated the filtrate to obtain betaine hydrochloride. He used Andrlik's method for the recovery of glutamic acid from the insoluble residue.

Tressler (6) obtained three patents on methods for separation of glutamic acid from Steffens waste. (1) Betaine hydrochloride and potassium chloride were precipitated by saturating the waste with gaseous hydrogen chloride. The hydrochloride of the betaine was separated by hot alcoholic extraction from the potassium chloride. The glutamic acid hydrochloride remaining in solution was precipitated by neutralizing with sodium carbonate. (2) Calcium chloride and sulphuric acid were added to the waste to generate

hydrochloric acid in solution. The excess calcium ions were removed as sulphate leaving the glutamic acid in solution. (3) Calcium chloride was added to salt out inorganic salts after the addition of hydrochloric acid to the waste. After heating the waste for one hour at 80°-90°C. and neutralizing the solution with sodium carbonate, the glutamic acid precipitated.

A method patented by Ikeda (5) was to add sulphuric acid to the waste to precipitate potassium as the sulphate, then add more acid and digest at 80-180°C. to decompose organic nitrogen compounds to glutamic acid. Slaked lime removed excess sulphate. Glutamic acid precipitated after standing several days. He used oxalic acid to precipitate betaine as the oxalate.

Albrook (1) obtained glutamic acid from Steffens waste by extracting the waste with alcohol and concentrating the insoluble portion and then adding an equal weight of hydrochloric acid and refluxing for 4-6 hours. Glutamic acid separated out as the hydrochloride.

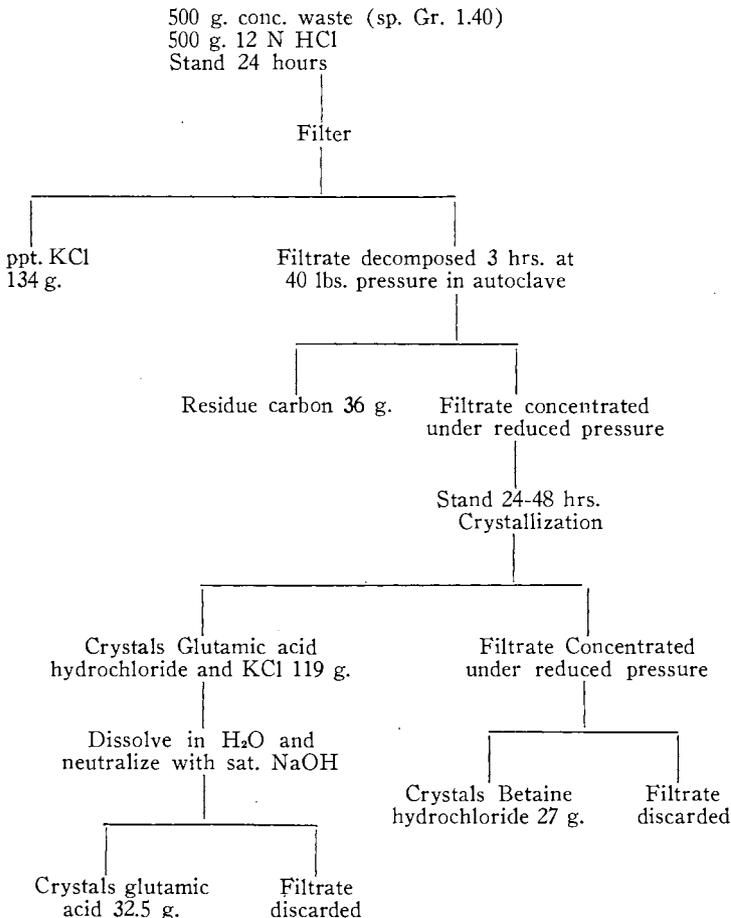
Benninghoff (4) later modified Albrook's method by heating the insoluble portion of the waste with hydrochloric acid in an autoclave for three hours at 40 pounds pressure. Benninghoff developed a method for obtaining glutamic acid and betaine from Steffens waste by treating the waste with concentrated hydrochloric acid to precipitate potassium chloride. The filtrate was then hydrolyzed in an autoclave for three hours at 40 pounds pressure. The solution after hydrolysis was filtered to remove carbon and was then concentrated to crystallize out glutamic acid hydrochloride; the solution was then filtered and the filtrate was further concentrated to crystallize out betaine hydrochloride. The glutamic acid hydrochloride was dissolved in water and the acid precipitated by neutralizing with saturated sodium hydroxide solution.

The following chart shows the flow sheet for Benninghoff's method and the results obtained in the present work for the Tracy, California Sample.

In the concentration of the filtrate after removal of carbon, it was found that it was best to concentrate the liquid until crystallization begins and then continue the concentration until the liquid becomes quite viscous. The liquid should then be allowed to stand 24-48 hours to allow as much glutamic acid hydrochloride to crystallize out as possible. In this way a maximum amount of the glutamic acid hydrochloride is obtained.

It was found that it was more convenient, gave a purer initial

FLOW SHEET OF METHOD FOR OBTAINING GLUTAMIC ACID FROM STEFFENS WASTE TRACY, CALIFORNIA SAMPLE



product and increased the yield of glutamic acid if the water solution of the hydrochloride was completely decolorized before precipitation of the acid rather than decolorizing during the recrystallization of the glutamic acid from hot water.

COMPARISON OF AMOUNTS OF GLUTAMIC ACID

The variations in the amounts of glutamic acid obtained from the different samples were even larger than the variations found by Benninghoff. The yield for each sample has been checked and is on a basis of 500 g. of the concentrated waste to which has been added 500 g. of 12 N HCl.

Table II—Amounts of Glutamic Acid from Steffens Waste

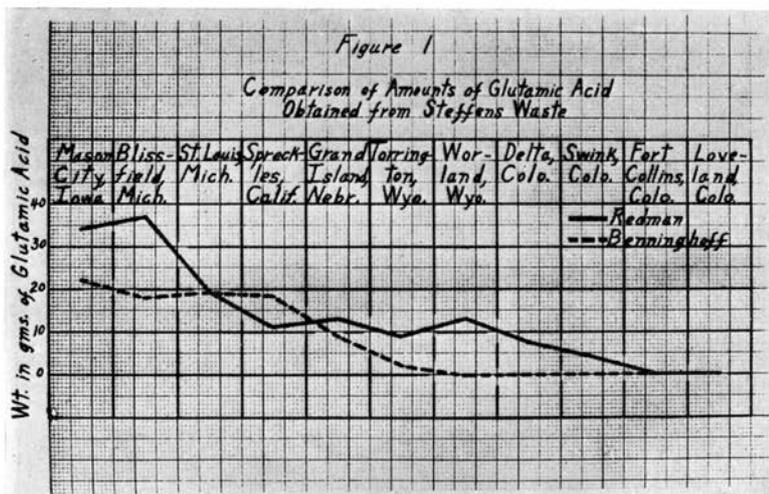
Sample	Benninghoff Grams Glutamic Acid	Redman Grams Glutamic Acid
Blissfield, Michigan	18	37
St. Louis, Michigan	19	19.5
Swink, Colorado	0	4.5
Delta, Colorado	0	7.5
Fort Collins, Colorado	0	0
Rocky Ford, Colorado	—	0
Loveland, Colorado	0	0
Torrington, Wyoming	2	9
Worland, Wyoming	0	13
Spreckles, California	18	11
Tracy, California	—	32.5
Mason City, Iowa	22	34
Grand Island, Nebraska	9	13
Garland, Utah	—	16

The maximum yields of glutamic acid were obtained from the Michigan, Iowa and California samples, as was the case in Benninghoff's results, whereas little or no glutamic acid was obtained from the Colorado samples.

In general the amounts of glutamic acid from the different samples were found to vary almost uniformly with the results found by Benninghoff. There was only one case in which a smaller amount of glutamic acid was found.

A comparison of the variations in the amounts of glutamic acid found by Benninghoff and the results found in the present work are shown in Figure 1.

The explanation for the wide variation in the different samples



has been a question for some time. Several theories have been suggested, such as a difference in the beets, a difference in the processing of the beets, variation in the soils and variations in climatic conditions. Apparently the same kind of beets and the same methods are used, so that the variations in the glutamic acid may be caused by the difference in soils and variations in the climatic conditions under which the beets are grown.

SUMMARY

1. The amounts of glutamic acid vary almost uniformly with the results found by Bartow and Benninghoff.

2. In general, larger yields of glutamic acid were obtained than those recorded by Benninghoff.

3. It is more convenient and better yields are obtained if the solution is decolorized before precipitation of the glutamic acid.

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