Studies on the Continuous Inversion of Sucrose: I, Research Data

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The gradual increase in total sugar consumption, resulting from population growth and other factors, has resulted in an increasing market for liquid sugars. In addition to the growth in the liquid sugar market, there has been a change in the use pattern for certain types of liquid sugars. In the canning industry, the use of 50% invert syrup has been increasing at the expense of sucrose syrup. These market changes have created problems in remaining in a competitive position to supply liquid sugars, and in particular, 50% invert syrup.

A review of our present liquid sugar installations led to the consideration of installing new facilities at the Manteca factory of the Spreckels Sugar Division. The review also indicated a need to study methods of invert syrup production to aid in the design of an efficient process to produce a high quality syrup.

Such a system was developed and installed. A discussion of the research program and the transition to full scale production is beyond the scope of a single paper and, therefore, is presented as a series of three papers. This first paper deals with the conception of the method and the laboratory work on the effect of process variables on inversion rates and syrup quality. The second paper discusses the use of the research data for sizing equipment and selection of control limits. The third paper describes the physical installation of the new continuous inversion system at the Manteca factory.

Production Methods

In the beet-sugar industry, liquid sugars have usually been made on a batch basis. Granulated sugar is dissolved in water to make a sucrose syrup, which may be inverted to produce invert syrup. In preparing invert syrups, hot acid inversion using hydrochloric acid has been the basic method used. In the cane-sugar industry a continuous procedure using acid inversion for invert syrup production has been used. The batch methods for invert syrup production, in general, are relatively inefficient and present problems in maintaining product quality.

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Considerable experience had been gained within the company on batch systems. A procedure of blending fully inverted syrup with granulated sugar or sucrose syrup had been used to produce 50% invert syrup. The relatively high temperature used and the prolonged time for production resulted in color formation, and a decolorizing step was required to improve syrup quality. In other systems of blending, lower temperatures were used and reagent usage reduced which resulted in improved syrup quality and reduced the necessity for decolorization of finished syrup. However, considerable time was still required to produce a finished syrup.

A batch system was developed in which a high density sucrose syrup was partially inverted to the desired degree. The rate of inversion is affected by sugar and water quality, and this association was used to control the process. By determining the conductivity ash of the sugar and alkalinity of the water, the time required to invert 50% of the sucrose present could be determined from previously prepared charts. The analyses, however, were inconvenient and considerable care was still required to produce a good quality product.

Because of the exothermic nature of the inversion reaction, the temperature of the batch would increase, with the actual increase being dependent on the rate of heat loss of the installation. This fact was utilized to fully automate the batch process for producing 50% invert syrup (2)[2]. In this system a computer was used to monitor the temperature change, and when the proper temperature increase was recorded, the batch was neutralized by automatic addition of calcium hydroxide.

In addition to production by methods using acid inversion, invert syrup has been manufactured commercially by use of enzymes and of ion exchange. Enzyme systems using yeasts have been used to invert sucrose syrup, and syrups of excellent quality are produced by ion exchange, since both ash and color are removed in this system (5). These methods, particularly ion exchange, operate most efficiently with dilute syrups and, as in the continuous acid inversion process mentioned earlier, a concentration step is required to produce a syrup of high density. Because of more readily available high-purity sucrose syrups, these other methods have been utilized more in the cane-sugar industry.

Economic comparisons were made of installations, based on the different methods, for producing 50% invert syrup. For equivalent production, high operating costs appeared to be associated with an enzyme system, and high investment costs with an ion exchange system, as compared to a multibatch acid inversion system. Because of favorable economic considerations the batch acid inversion method was more closely studied.

[2]Numbers in parentheses refer to literature cited.

Experience had shown that prolonged holding of finished invert syrup resulted in color formation. In the batch system the pumpout and cooling facilities must be designed for high flow rates to gain capacity and to permit rapid processing to minimize color formation. These disadvantages were overcome in a continuous system. Color formation is minimized because of less syrup holdup in the system. The process is rapid, and the design may be more economical because of lower flow rates for equivalent production. In addition, by inversion of high density syrup as done in the batch system, there would be no need for a concentration step to achieve the required product density.

Previously collected data did not appear adequate for the design of a continuous system. The literature indicated that inversion temperatures ranging from 36° to 85°C have been used commercially with corresponding inversion times of 23 hours to 30 minutes (6). The effect of process variables on inversion rates and syrup quality did not appear clearly established for the process conditions anticipated for a new installation. Brief laboratory tests had indicated that 50% invert syrup of good quality could be rapidly made at relatively high temperatures. Additional tests were necesary to determine the effect of process variables when inverting a high density syrup at high temperatures, since most of the reported data on inversion rates had been obtained at relatively low temperatures and syrup densities (8).

Inversion Time

The effect of temperature on inversion time and product quality had been studied in beaker type tests. With formulations as used in the factory, the sucrose syrup to be inverted was about 79 rds. Tests showed 46 hours were required to produce a 50% invert syrup by partial inversion of the sucrose syrup at room temperature and 3.3 minutes at 90°C. Color development varied from about 20 units at the longer time to two units at the shorter time[3]. A comparison of results when using partial inversion and blending procedures showed less color development during partial inversion. Other tests showed that rapid cooling of the finished melt minimized color formation caused by holding the syrup. Based on the preliminary data, further tests were made using a small tubular reactor to continuously invert the sucrose syrup.

A preheated sucrose syrup of about 80 rds and diluted hydrochloric acid were continuously mixed and pumped through a small stainless steel coil submerged in a temperature-controlled bath. The effluent from the reactor was immediately neutralized with calcium hydroxide and cooled by passing through a small cooling coil. The concentrations of sucrose syrup, acid, and hydroxide were adjusted

[3]Reported color values were determined with the Bernhardt-Phoenix Sphere Photometer and are expressed as 1000 (−Log T/bc). Samples were adjusted to 50 rds and 5.0 pH.

so the finished syrup would be the density required by product specifications. By starting with a high density syrup of 80 rds, the extra expense of evaporation to adjust product density was avoided, and processing costs reduced accordingly. With the small flow rates of 97 ml/min of syrup and 3 ml/min of acid, close control of variables such as flow rates and syrup density was necessary to obtain repeatable results. Comparisons of reaction rates with those obtained by batch inversion showed reaction rates obtained by either method were the same. In some cases, it was necessary to use batch test data because of difficulties in maintaining uniform conditions in the laboratory continuous apparatus.

In these tests, data indicated that for practical purposes the rate of reaction for inversion of sucrose was similar to that for a first-order reaction. Reaction rate constants for continuous inversion, assuming constant syrup density and dissolved solids content, were calculated according to the following equation:

$$k = \frac{\text{feed rate (ml/sec)}}{\text{reactor volume (ml)}} \ln \left(\frac{1}{1 - \frac{\text{g sucrose inverted}}{\text{g sucrose in feed}}} \right)$$

The reaction rates obtained at various temperatures and acid additions are given in Table 1.

Table 1.—Effect of temperature and acid addition on rate of sucrose inversion.

		Reaction rate constant	
Temp °C	Acid addition (g HCl/g sucrose) x 10⁴	Continuous Sec^{-1}	Batch Sec^{-1}
85	0.81		0.000619
90	0.81	0.00109	0.000976
100	0.81	0.00306	0.00285
85	1.06	-	0.000973
90	1.06	0.00153	0.00157
100	1.06	0.00432	0.00436
85	1.29	-	0.00127
90	1.29	0.00192	0.00206
100	1.29	-	0.00551

The acid additions shown are the amounts actually available for catalyzing inversion. Acid required for neutralizing water alkalinity has not been included. The effect of temperature on reaction rate is shown graphically in Figure 1. The data show that the effect of temperature on reaction rate closely followed the Arrhenius relationship; that is, the rate constant increases in an exponential manner with temperature. The empirical relationship is shown by the Arrhenius equation, $K = Ae^{-Ea/RT}$.

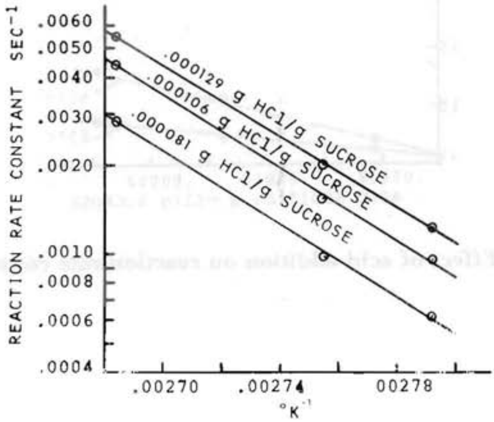

Figure 1.—Effect of temperature on reaction rate constant.

Although reaction rates for inversion have been reported in the literature, the conditions under which they were obtained were considerably different than the current tests indicated could be used and still obtain acceptable product quality. Activation energy values calculated from reaction rates obtained at somewhat lower syrup densities and temperatures have been reported that range from 25 to 30 kcal/mole (3,4,7,8). Activation energy calculated from data shown in Table 1 was 27 kcal/mole. By increasing the inversion temperature from 85° to 100°C, the reaction rate was increased by a factor of 4.5.

The effect of acid addition on the reaction rate constant is shown in Figure 2. The addition of 1.06×10^{-4}g HCl/g sucrose corresponds to formulations normally used in the factory batch procedure for partially inverting a high density sucrose syrup. An increase in acid concentration by a factor of 1.6 resulted in an increase of the reaction rate constant by a factor of about two. The time required to produce a 50% invert syrup with normal acid addition varied from 11 minutes at 85°C to 2.5 minutes at 100°C.

Laboratory studies, later verified by factory experience, showed that a temperature rise of 3.41°C in a batch of invert syrup indicated

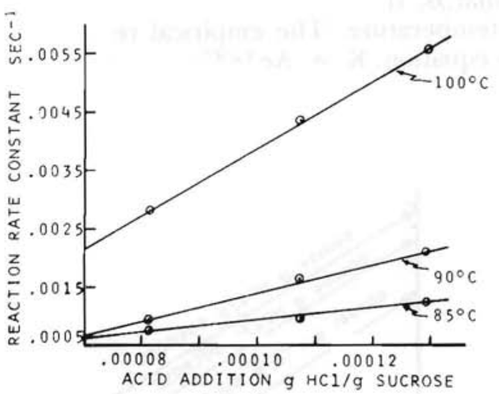

Figure 2.—Effect of acid addition on reaction rate constant.

that 50% of the sucrose present was inverted. Temperature measurements made at different locations along the laboratory tubular reactor showed that the change in syrup temperature could be used for process control.

Color Formation

The effect of prolonged process time at elevated temperature on color formation in invert syrup has probably been well known (1). However, very little published information was found on the effects of process variables on color and ash content of finished syrup.

Analysis of syrup samples representing syrup at different stages of the overall process showed there was not a significant change in syrup color during the inversion step at the conditions used. Color formation did occur during the dissolving of the dry granulated sugar to form the high density sucrose syrup and when the finished invert syrup was held at elevated temperatures. Dissolving sugar at 95°C to make about an 80 rds syrup required about 10 minutes in the laboratory. Color formation during this time and subsequent holding of the syrup for one hour was only two to three color units. Depending on the pH of the finished syrup, color formation varied with holding time as shown in Table 2.

When the neutralized syrup was held for an appreciable length of time at 90°C, color increase of up to 10 units occurred, depending on the pH of the syrup. These data show the need for rapid cooling of the neutralized syrup. In the batch procedure, the quality of the

Table 2.—Effect of pH and temperature on color increase of 50% invert syrup stored for short periods.

pH	Temperature, °C	Syrup color		
		Storage time		
		0.0 Hrs	0.5 Hrs	1.0 Hrs
3.6	90	24.7	26.8	29.8
	50	24.5	24.8	24.3
4.4	90	25.6	29.1	31.7
	50	26.4	26.3	26.9
5.3	90	25.4	30.1	35.1
	50	26.1	-	26.4

finished syrup was somewhat dependent on the cooling capacity of the installation. Additional tests, simulating storage of the finished syrup, showed that color formation was minimized by storing the syrup at relatively low pH and temperature. Over a period of 10 days, a color increase of 5 units occurred in 50% invert syrup stored at 3.5 pH and 35°C compared to 8 units in syrup of 5.0 pH. Greater color increases occurred in syrups held at higher temperatures.

Ash Content

The major portion of ash in 50% invert syrup comes from the reagents added. The amount of acid used affects the amount of neutralizing agent required and a reduction in acid usage may have a significant effect on the ash content of the syrup. Since increasing temperature decreases the inversion time, acid requirements may be reduced with the use of high temperature, depending on production requirements. The water used contributes to the ash of invert syrup and to aid in the design of the overall continuous inversion system, tests were made to determine the effect of different types of water on syrup quality.

Deionized water, cold soda-lime treated water, and well water were used to prepare batches of 50% invert syrup. The granulated sugar used had an ash content of 0.0057% conductivity ash. Results are shown in Table 3.

Table 3.—Effect of water quality on ash content of 50% invert syrup.

Water			50% Invert syrup
Type	Total solids ppm	M Alkalinity ppm $CaCO_3$	% Conductivity ash
Deionized	3	2	0.0211
Treated	195	111	0.0304
Well	457	280	0.0366

The syrups were analyzed for color but little, if any, effect of water quality on color was found. As expected, however, the best quality water resulted in invert syrup of lowest ash content. The type of water to be used would be dependent on product specifications. However, reagent usage and sugar quality affect the ash content of the invert syrup and all factors must be considered for a particular location.

Summary

The data have shown that at a relatively high temperature and with appropriate addition of hydrochloric acid, a 50% invert syrup may be produced in a few minutes by partial inversion of a high density sucrose syrup. Color formation during inversion at temperatures in the range of 85°C to 100°C was found to be negligible. Color formation occurred during the preparation of the high density sucrose syrup and in the neutralized invert syrup when held at elevated temperatures. Data indicated that with rapid processing from sucrose syrup preparation through cooling of the finished syrup, there would be little, if any, color formation. The ash content of finished syrup may be reduced by use of good quality sugar and water.

The validity of the data and process concepts have been verified by the successful operation of a commercial installation at the Manteca plant of the Spreckels Sugar Division.

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