

COLOR INCREASE DURING LABORATORY STORAGE OF SUGARBEET PROCESSING STREAMS

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Color increase of beet thick juice and molasses desugarization extract during storage can lead to difficulties in processing when products are returned to the beet sugar factory for crystallization. Recently Sugar Processing Research Institute has studied the effect of storage under laboratory conditions on beet thick juice and extract. Samples of thick juice and extract were placed on the bench top and aliquots removed at specific intervals for analysis. The analyses performed included pH, brix, color, turbidity, and organic acids such as lactic acid and acetic acid. Based on the samples analyzed, pH and brix remain constant over time and a very slight upward trend in turbidity was observed. A strong correlation between color and storage time was found. For thick juice, a 5.21 ICUMSA color unit increase per day was noted and for extract, the color increase per day was found to be 6.98 ICUMSA units per day. These results along with the organic acids analysis will be discussed.

Introduction:

Color is a generic term used to describe a wide range of components or colorants that contribute to the visual appearance of sugar. Colorants are materials made up of various molecular weights, pH sensitivity, ionic charge, chemical composition, and affinity for the sugar crystal. The major component of beet raw juice is sucrose. Several minor components of beet raw juice include organic acids, anions, cations, oligosaccharides, fatty acids, nitrogenous compounds, reducing sugars, enzymes, polyphenolics, and polysaccharides. Many of the minor components are removed during processing, but a few remain and can contribute to color formation. Organic non-sugars such as organic acids, amino acids, or reducing sugars may act as catalysts for color formation in sugarbeet solutions. The presence of this mixture of compounds in contact with moisture, air and light seem to present the opportunity for color formation in sugarbeet processing.

Color is important for many reasons. First of all, colorants can transfer into the sugar crystals creating sugar that is not white and therefore presumed lower quality by consumers and customers. This perception that high color sugar is lower in quality makes color an important quality indicator in sugar processing. Also higher color products such as thick juice within the sugar factory, can cause decreased yields of sugar for the factory. By causing a lower yield of sugar, higher color may be linked to a loss of money for the sugar factory. Higher color may also be linked to filterability issues leading to another problem for sugar factories.

Beet sugar color is created during the carbonation step of the process at high temperature and pH.¹ The color that is formed during the processing of sugar beets tends to be reactive in nature and subject to increasing over time. Beet sugar color is considered to be autocatalytic due to the increase of color over time without the addition of any other components to the sugarbeet system.

SPRI has long had an interest in studying the changes in color during storage of various sugar products. In a study of soft sugars, it was found that high moisture content was the best predictor of color increase over time. Organic acids and phenolic acids were also found to contribute to the formation of color over time.² Another study by SPRI examined components of the surface film of white beet sugars and how these components can contribute to color formation over time.^{3,4} Pyroglutamic acid and lactic acid were the two components identified in the study that changed over time as color increased. Lactic acid is a primary product of alkaline decomposition of sucrose and a major microbiological product that may have several sources. Pyroglutamic acid is a major nitrogenous compound found in sugar beet processing from the glutamine in the sugarbeet plant. Under the alkaline or heated conditions used in sugarbeet processing, glutamine will lose ammonia to form pyroglutamic acid.

The objective of this work is to determine the effect of storage on the color of thick juice and chromatographic extract products from sugarbeet processing. Changes in pH, brix, color, turbidity, invert sugar, organic acids, dextran, and total polysaccharides over time will be examined.

Experimental:

Sugarbeet thick juice and chromatographic extract were provided by SPRI sponsors. Three thick juice samples and one extract sample were stored on the bench top under SPRI laboratory conditions in plastic containers until detectable signs of deterioration were observed. The length of study for the samples ranged from 68 to 112 days. The samples were mixed well and aliquots were removed at specific intervals for analysis. Removed aliquots were immediately frozen until ready for analysis. Analysis of the samples included pH, brix, color, turbidity, invert sugars, organic acids, dextran and total polysaccharides. Color was measured by ICUMSA Method GS1/3-7, and turbidity determined by difference using the same method. Invert sugars and organic acids were determined by ion chromatography.

Results and Discussion:

Thick Juice Results

Three thick juice samples (TJA, TJB, and TJC) were analyzed during this study. TJA was studied for 68 days, TJB sampled for 73 days, and TJC for 112 days before deterioration was evident and the sampling was stopped. The first parameter analyzed was pH. The pH of TJC showed a dramatic decrease in the first 35 days then it was stable. TJB showed a slight downward trend after 45 days and TJA remained constant. TJA and TJB had an initial pH of around 9 while TJC had an initial pH of approximately 8. These results are shown in Figure 1 below.

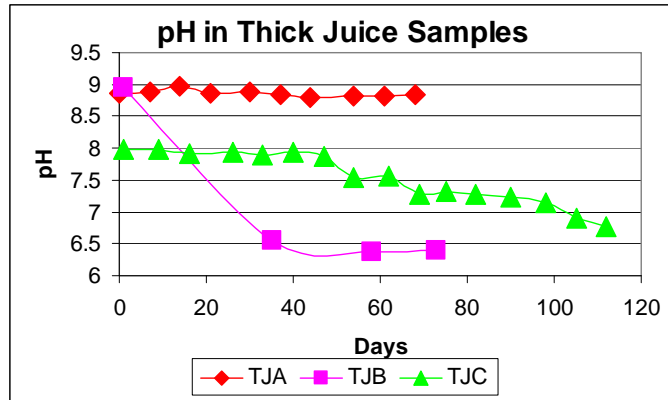


Figure 1. pH of thick juice samples over time.

The three thick juice samples were also analyzed for brix. For TJA, the brix remained relatively unchanged for the duration of the study. TJB showed a slow decline from 69.3 to 68.5. TJ C had an initial brix of 64.2 and gradually increased to 66.5 in the first 30 days and then remained constant. No significant changes were detected in the analysis of the brix over time.

The thick juice samples were also analyzed for total polysaccharides and dextran content. No trend was observed in either total polysaccharide or dextran content for the duration of the study only natural sample variation was recorded.

Color exhibited a linear increase in all three thick juice samples. Thick Juice A had the highest initial color at 4256 ICU which increased to 4687 ICU by day 68 when the sampling stopped. TJB with an initial color of 2873 ICU increased to 3054 ICU in 73 days and TJC initial color was 2691 which increased to 3236 ICU in 112 days. The linear relation of the color over time is shown in Figure 2 below.

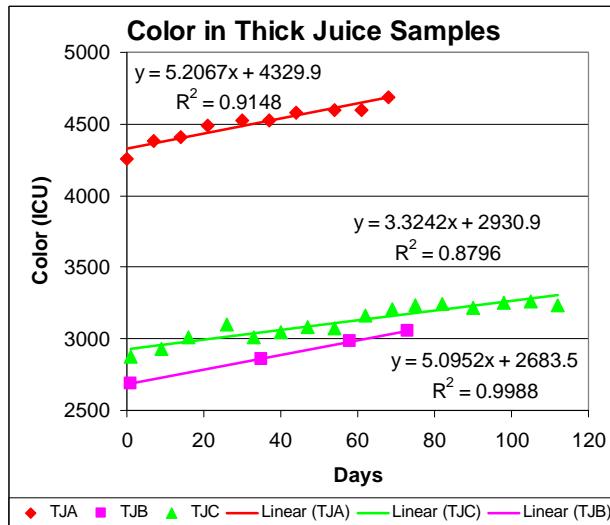


Figure 2. Linear increase in color over time for thick juice samples. Also shown are linear regression equations for each sample studied.

As shown in the figure above, there is a strong linear relationship between color increase and time for the thick juice samples studied here. The slope of the regression line represents the color increase per day for each sample and the y-intercept closely approximates the initial color for each thick juice sample. For TJA, the slope or color increase per day is 5.21 ICU/day and the y-intercept is 4330 ICU with an actual initial color of 4256 ICU. In the case of TJB, the slope is 3.32 ICU/day and the y-intercept is 2391 ICU with an actual initial color of 2873 ICU. TJC shows a color increase per day of 5.10 ICU/day and a y-intercept of 2684 ICU and an initial color of 2691 ICU.

The thick juice samples except for TJB were also analyzed for turbidity. For TJA, the initial turbidity was 310 ICU with a gradual upward trend over time. For TJC, initial turbidity was 139 ICU with a significant increase in the latter part of the study to a final turbidity of 1049 ICU.

TJB was further analyzed for sugars and organic acids. Invert sugars were studied using ion chromatography. Invert sugars were present at approximately 0.13% for the first 35 days of the study. The invert concentration increased to 0.628 % at the end of the study. The glucose increased from 0.040% to 0.327% while fructose increased from 0.088% to a final concentration of 0.301%.

Organic acids were also analyzed for using Ion chromatography. Several organic acids were detected in TJB. These include citric acid, lactic acid, formic acid, acetic acid and pyroglutamic acid. The organic acid profile over time is shown below in Figure 3.

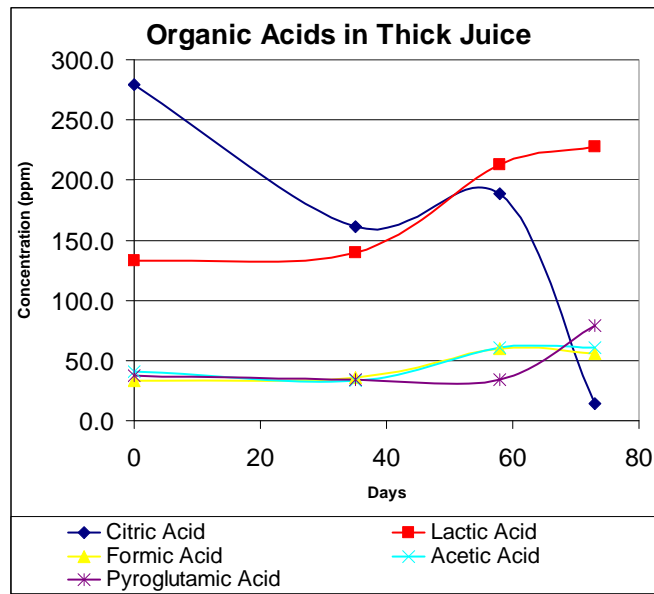


Figure 3. Organic acid profile of TJB over 73 day study.

Citric acid showed an initial concentration of 279 ppm and a final concentration of 13.9 ppm, a 95% decrease in concentration with a rapid decline in the last 8 days of the study. Lactic acid showed a 71% gradual increase over time with an initial concentration of 133ppm to 228ppm during the 73 day study. Formic acid and acetic acid showed little change in concentration during the first 35 days of the study. Formic acid initial concentration was 33.3ppm and slowly increased to 56.1 ppm. Acetic acid initial concentration was slightly higher

at 41.0ppm and the final concentration was 61.0 ppm. Pyroglutamic acid concentration of 33.7ppm to 38.6ppm except for the last 8 days of the study. During the last 8 days, the pyroglutamic acid concentration increased by 110% to a final concentration of 78.7ppm.

Previous work by SPRI has shown organic acids in soft sugars and in the surface film layer of white beet sugar can be contributing factors to autocatalytic color increasing over time. Large changes in lactic acid and pyroglutamic acid concentrations in the surface syrup layer of white beet sugars is related to color increase during accelerated storage tests. Products results from alkaline degradation of sugars such as invert and pyroglutamic acid could be contributing factors to color formation over time. A strong correlation of lactic acid concentration to color is observed for the TJB as shown in Figure 4.

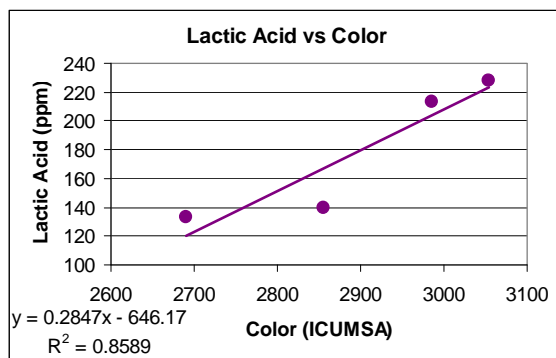


Figure 4. Lactic acid correlation with color of thick juice solution over time.

This strong correlation between lactic acid concentration and color in thick juice agrees with work previously done by SPRI showing a relation between lactic acid concentration and color in white beet sugar.⁴

Beet Extract Results

A sample of the chromatographic extract was obtained from a SPRI sponsor company and studied in the same manner as the thick juice. The extract was placed in a plastic container on the bench top under laboratory conditions for 68 days. The initial pH of the extract was 10.65 and the final pH was 10.57 with the average for the 68 days of 10.58. Brix also remained consistent during the study with an initial value of 71.4 and a final value of 71.5 and an average of 71.4 for the study. Turbidity showed a slight upward trend over the 68 day study period. There was some variation in turbidity measurements but the initial turbidity was 125 ICU and the final measurement was 189 ICU. The final parameter measured for the extract was color. As shown in Figure 5 below, a strong linear correlation exists between color and time for the extract as observed in the thick juice study. The initial color value for the extract was 11,529 ICU with a final value of 12,057 ICU. As in the thick juice study, the slope of the regression line represents the color increase per day. The color increase per day for the extract samples studied is 6.98 ICU/day. The y-intercept closely predicts the initial color value at 11,522 ICU, with an actual value of 11,529 ICU.

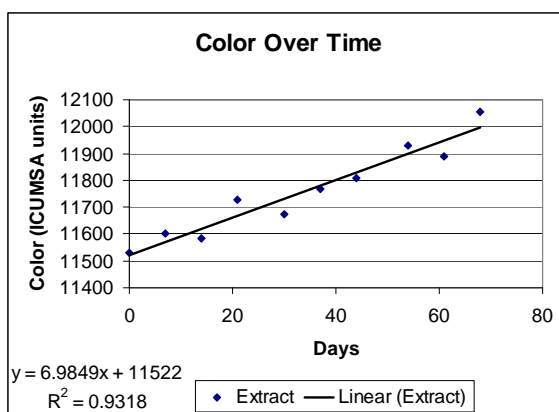


Figure 5. Linear increase in color over time for chromatographic extract sample. Also shown is the linear regression equation for extract sample studied.

According to the literature, onsite storage of chromatographic extract at beet sugar factories does not cause the color to increase as rapidly as observed in our laboratory studies.⁵ There may be several reasons for the differences in laboratory storage and onsite storage. One of the first reasons may be that ambient temperature is higher in laboratory storage conditions compared to onsite storage. A second difference may be the type of container in which the samples were stored. In laboratory storage a plastic bottled with a screw cap lid was utilized compared to the stainless steel tanks employed in onsite storage. Previous work at SPRI has indicated that autocatalytic color reactions may require adequate light, oxygen, and moisture to occur. The plastic containers used in the laboratory study combined with frequent mixing of the samples may have allowed more light and oxygen into the samples to promote the autocatalytic color reactions. Color increases are still observed in the stainless steel tanks used for onsite storage, but at a much lower rate than those in the laboratory. The stainless steel tanks may slow or prohibit the autocatalytic reactions by not allowing enough light and oxygen into the tank to cause as rapid color increases.

Summary and Conclusions:

The pH of the one thick juice sample showed a significant drop in the first 35 days of storage and then remained constant while one sample showed a downward trend after the 45 days. The third thick juice sample as well as the chromatographic extract sample pH remained steady over the course of the laboratory storage study. One thick juice sample showed an increase in brix initially but then remained constant. All other samples (thick juice and extract) remained constant over the storage period. The thick juice samples exhibited a color increase ranging from 3.32ICU/day to 5.20ICU/day while the extract sample showed 6.98ICU/day color increases. Turbidity in the case of thick juice and extract exhibited an upward trend especially toward the end of the study period for all samples. For the thick juice sample, invert sugars were constant for approximately the first 35 days then increased as degradation occurred. Total polysaccharides for the thick juice samples showed natural variation over the course of the storage study and dextran showed no significant changes. Several organic acids were detected in the thick juice sample. These include citric acid which decreased over time, formic and acetic

acids which slowly increased over time, lactic acid which steadily increased over time and pyroglutamic acid which dramatically increased at the end of the study period. Lactic acid showed a strong correlation with color over time indicating that it may be involved in reactions that increase color over time. Steel storage vessels combined with lower ambient temperatures at sugarbeet factories may slow autocatalytic color formation reactions compared to plastic containers used in the laboratory storage studies.

References:

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