Color and Ash – Is there a relationship between them?

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Many questions surround color and ash and a possible relationship between these two components of sugar. Color and ash content of beet and cane sugars including beet, juice, and extract campaign beet sugars as well as raw and refined cane sugars were studied. The appropriate ICUMSA method of determination for color and ash was used for each type of sugar. For beet sugars, ash ranged from 0.003% to 0.015% while color ranged from 20 IU to 57 IU. For the refined cane sugars tested, color ranged from 18 IU to 58 IU while ash ranged from 0.007% to 0.011%. Raw cane sugars ranged from 800 IU to 3335 IU with ash values of 0.173% to 0.317%. These sugars were washed using a high brix white sugar solution to remove the syrup surface layer similar to the affination step in the cane refinery. The samples were dried and then tested for ash and color again after washing. Color and ash removed by washing is believed to be contained in the syrup surface layer surrounding the crystal. These components in the syrup layer are believed to be involved in reactions that promote color increase during storage of sugar. This presentation will show the differences in ash and color in the whole sugar sample as well as in the syrup layer for the various types of sugars studied.

INTRODUCTION

One of the goals of sugar production is to produce sugar that is low in color and ash whether from sugar beets or sugarcane. Pure sucrose is colorless while non-sucrose components can transfer into the sugar crystal giving sugar color. Sugar that is not white may be presumed by the consumer or customer to be of lower quality. This perception that sugar higher in color is lower in quality makes color an important quality indicator in sugar processing.

Color is a general term used to describe the many components that contribute to the visual color of granulated sugar. The two main sources of sugar color are plant derived colorants and colorants formed during processing. Colorants are materials made up of various molecular weight, 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. Sugarbeet colorants are generally produced during processing. These colorants are mostly alkaline degradation products of fructose and glucose; and also melanoidin, colorants that are formed by the reaction of sucrose or invert with amino acids. [(1)] Other colorants, called melanins, are very dark colorants formed from the enzymatic reaction of amino-phenols released from sugarbeets on processing. In comparison, in sugarcane processing, the major colorants tend to be plant pigments, especially phenolics, that are associated with hemicellulosic polysaccharides found in the sugarcane plant. Sugarbeet color is generally lower than sugarcane color. At the same time, white beet sugar can be crystallized from a much darker solution than cane sugar, highlighting the different nature of the colorant types in beet and cane processing. Therefore, the raw sugar produced from sugarcane must be further refined to remove more color to produce refined white sugar. White sugar color is typically in the range of 20 IU. Cane sugar processing products range from over 14,000 IU for the raw juice to 1000 IU for raw sugar, and down to white sugar levels after refining. Beet sugar processing products range from over 3400 IU for raw juice down to 1600 IU for syrup and finally 20 IU for sugar. The most effective color removal step for the sugar is crystallization. In sugar processing, more color is transferred to the crystal in sugarcane than in sugarbeet.

Ash is another important parameter in sugar quality. Ash is made up of soluble and insoluble salts of organic and inorganic compounds – mostly oxides and sulfates of potassium, sodium, calcium and magnesium cations. Some ash comes from the sugar beets and is not removed during processing, some ash is added during the processing of the sugar beets and some can come from processing equipment erosion. Ash content of refined granulated sugar must not exceed 0.015% by most standards. Ash content can be reduced by maintaining proper filtration, sufficient washing during centrifugation of the sugar, and proper handling of the sugar during drying and screening. As sugarbeet quality decrease, ash content can increase because a large part of the ash comes from soluble components in the sugarbeet that are not removed during processing. Ash components such as calcium and iron have been shown to play a part color formation by catalyzing or impeding various colorant formation reactions. [2] The role of the ash components on color formation can depend on the colorant reaction taking place, pH, and temperature.

EXPERIMENTAL

In this study beet and cane sugars were analyzed for color and ash content. Beet sugars consisted of nine thick juice campaign samples, seven sugars produced from the molasses desugarization extract, and seven beet campaign sugars for a total of 23 beet sugar samples. Cane sugars consisted of four refined sugars and eight raw sugar samples for a total of 12 cane sugar samples. A separate set of 58 raw sugars were also analyzed for color and ash.

Color is measured by the absorbance of light (usually at 420nm) by the sugar solution. This single measurement accounts for all the various types of colorants present in the sugar solution. This may not be the ideal way to measure for all colorants but with the complex chemistries of the colorants, it gives the best method of analysis for the sugar laboratory technician. The color of each sugar was determined using the appropriate ICUMSA color method. For sugars less than 50 IU in color, ICUMSA method GS2/3-10 The Determination of White Sugar Solution Color while method GS1/3-7 Determination of the Solution Colour of Raw Sugars, Brown Sugars, and Coloured Syrup at pH 7.0 was used for samples with colors greater than 50 IU.

Ash content is measured in a number of ways including conductivity and a gravimetric method referred to as sulfated ash method. Conductivity ash uses a conductance meter to measure the soluble inorganic compounds in a sugar solution. The sulfated ash method requires concentrated sulfuric acid added to the sample to replace chloride, nitrate, and carbonate. The sample is then heated in a muffle furnace until all volatile components and carbon are removed from the sample. Conductivity is the preferred method for measuring ash because it is faster and more environmentally friendly. The results of the two methods have been shown to be comparable. ICUMSA conductivity methods for ash determination were used in this study. For the raw sugars ICUMSA Method GS1/3/4/7/8-13 (1994) The Determination of Conductivity Ash in Raw Sugar, Brown Sugar, Juice, Syrup and Molasses was used. For the white sugar samples ICUMSA Method GS2/3/9-17 (2011) The Determination of Conductivity Ash in Refined Sugar Products and in Plantation White Sugar was used for ash determination.

After initial analysis, the samples were washed with a high brix white sugar solution, centrifuged to remove the solution and air dried overnight. The color and ash measurement were then repeated on the washed sugar samples to determine the amount of color and ash that remained in the sample. The difference between the color and ash of the washed and original sample is the amount of material believed to be contained in the surface layer of material surrounding the sugar crystal.

RESULTS AND DISCUSSION

Results of the color analysis of beet sugars produced during thick juice campaign, from the molasses desugarization extract, and beet campaign sugars are shown below in Figure 1. Samples TJ1-TJ9 in the figure below represent the color of the nine thick juice campaign sugars before and after washing. The colors of the sugars ranged from 24-28 IU. After washing the colors ranged from 12-19 IU. Washing the sugars removed 32.1% to 53.6% of the color from the samples. An average 44.6% of color was removed from the thick juice campaign sugar by washing indicating 44.6% of the color for these samples can be found in the surface layer on the crystal.

Another campaign by beet sugar manufacturers is the extract campaign. This is the process which crystallizes white beet sugar from the sugar rich extract of the molasses desugarization process. Seven white sugar samples from this campaign were analyzed indicated by E1-E7 in Figure 1. Color

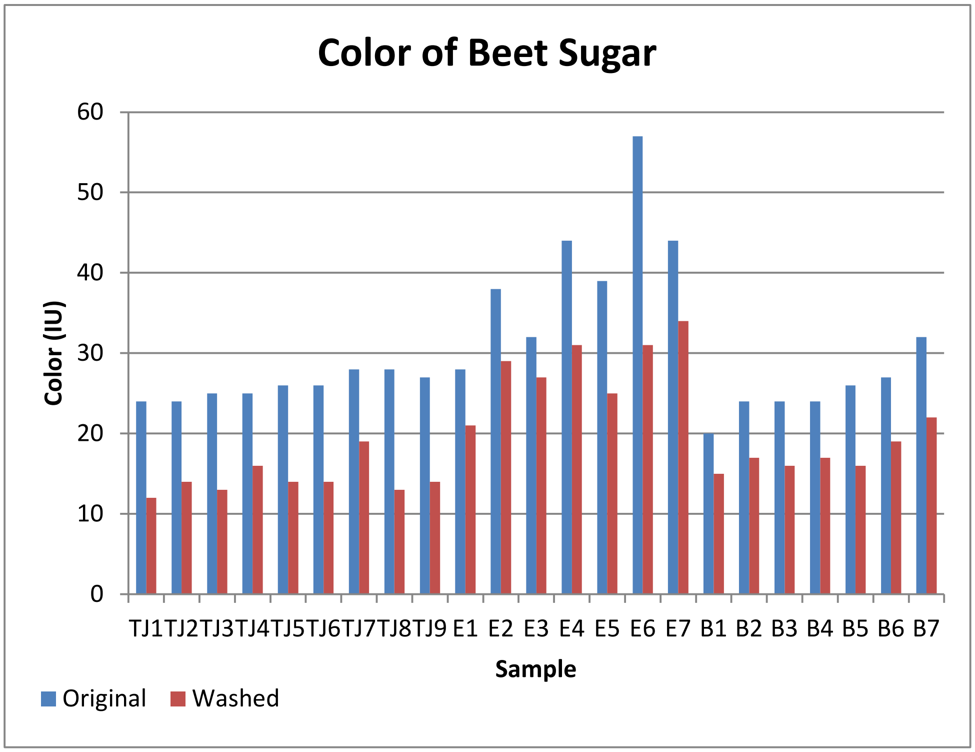


Figure 1. Color of beet sugar before and after washing

before and after washing is shown in Figure 1. Color for the extract samples ranged from 28 IU to 57 IU before washing. After washing, the color dropped to 21 IU to 34 IU with color removal of 15.6% to 45.6% for the extract campaign samples.

The third type of campaign for beet sugar production is directly processing sugarbeets from harvest or frozen storage for white sugar production. Seven sugars from this campaign were analyzed in this project. The colors of these sugars ranged from 20 IU to 32 IU before washing and 15 IU to 22 IU with 25% to 33% of the color being removed or contained in the surface layer.

After color analysis the samples were analyzed for ash content. Result of this analysis is shown in Figure 2. From looking at the figure it becomes clear that the extract campaign sugars (samples E1-E7) have the lowest ash content before and after washing. Ash ranged from 0.003% to 0.009% in the original samples and 0.002% to 0.004% after washing with an average of 30.9% removed.

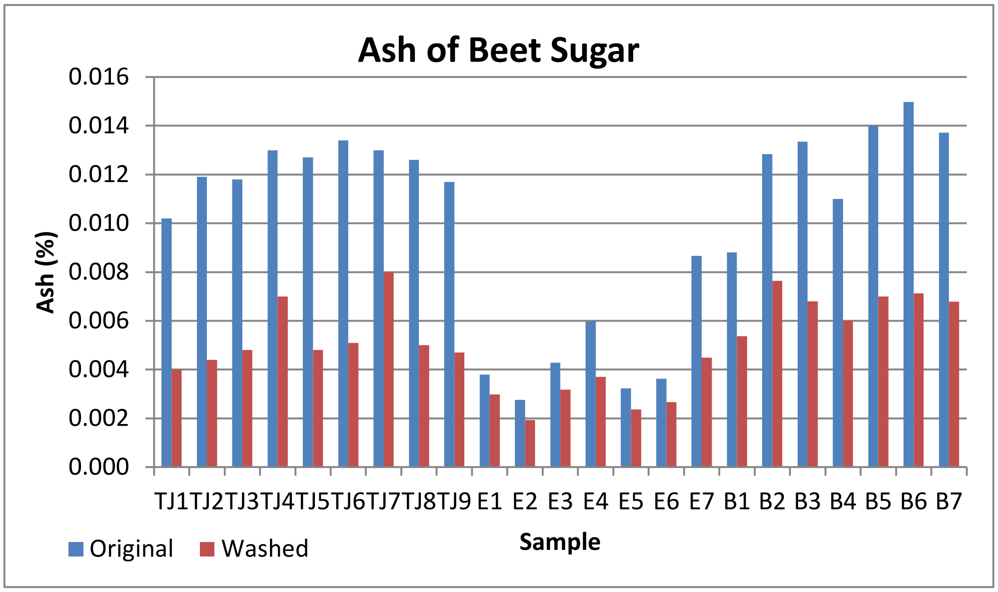


Figure 2. Ash content of beet sugar samples

The ash of the thick juice campaign sugars ranged from 0.010% to 0.013% before washing and 0.004% to 0.008% after washing. Approximately 38.5% to 63.0% with an average of 56.9% decrease in ash is observed with washing. This indicates that over half other ash for these sugars is found in the surface layer surrounding the crystal. For the beet campaign, original ash for the seven samples ranged from 0.009% to 0.015% with 39% to 52% of the ash being removed by washing. Ash values for the washed samples ranged from 0.005% to 0.008%.

Figure 3. Ash vs color for beet sugars

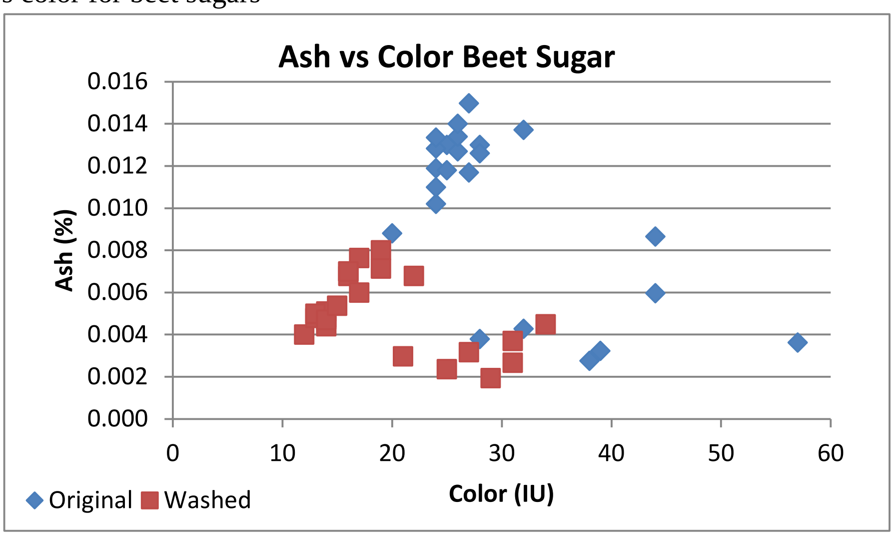


Figure 3 shows a relationship between ash and color for original and washed beet sugars produced during various campaigns. The original data shows much higher color and ash values for all samples studied. After washing a significant reduction in color and ash is observed. Sugars produced during the extract campaign are separated from the thick juice and beet campaigns due to the low ash content of the samples. The extract sugars also show some of the highest color values recorded.

Correlation between ash and color for the various campaign beet sugars was examined and shown in Table 1. For the original samples the correlations between ash and color range from 0.35 for the extract campaign sugars to 0.76 for the beet campaign sugars with correlation values of 0.44 for thick juice campaign sugars and 0.66 for all beet sugars. The strongest correlation between ash and color appears to be for the beet campaign sugars based on the samples studied here. For the washed samples, the correlations range from 0.36 for the beet campaign sugars to 0.92 for the thick juice campaign sugars with 0.78 correlation for all beet sugars and 0.52 for the extract campaign sugars. Color and ash do not seem to correlate well for the extract campaign beet sugars either original samples or washed samples. This could be a result of the molasses desugarization process removing most of the ash components as shown earlier while the extract sugars were on average the highest color sugars studied. There is a stronger correlation for ash and color in sugars produced during the beet campaign in the original samples when compared to the washed samples. This could be showing that the ash and colorant materials contained in the syrup surface layer removed by washing are more closely related that those occluded in the sugar crystal. In thick juice campaign sugars, correlation for ash and color was 0.92 in the washed samples compared to 0.44 for the original samples. In the case of samples, it appears the ash and colorant material occluded in the sugar crystal correlate stronger. When looking at the beet sugar produced from various campaigns over the harvest season, the correlations between ash and color are slightly higher for the washed samples at 0.78 compared to 0.66 for the original samples.

Table 1. Correlation between ash and color for beet sugars

Sample	Original	Washed
Beet Campaign	0.76	0.36
Thick Juice Campaign	0.44	0.92
Extract Campaign	0.35	0.52
All beet sugars	0.66	0.78

Cane sugar samples

The next group of samples studied was refined white cane sugar. Four samples of refined cane sugar samples were studied with origins from all over the world. These samples were treated in the same manner as the beet sugar samples. The original color and ash was measured for each sample followed by washing and then determination of color and ash on the washed samples.

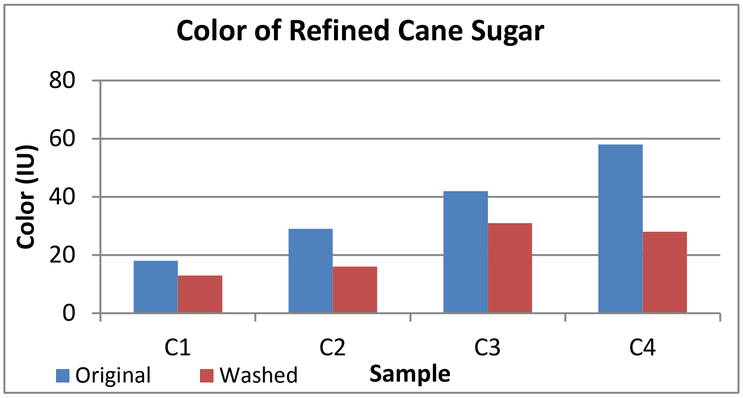


Figure 4. Color of refined cane sugar before and after washing

Figure 4 shows the color of four refined white cane sugars before and after washing. The colors of the sugars ranged from 18-58 IU. After washing the colors ranged from 13-31 IU. Washing the sugars removed 26.2% to 51.7% of the color from the samples. The color that is removed by washing is equivalent to the color found in the surface syrup layer surrounding the sugar crystal. The ash of the sugars ranged from 0.0067% to 0.0117% before washing and 0.0011% to 0.0021% after washing. Approximately 80% to 86.3% decrease in ash is observed with washing as shown in Figure 5.

Color and ash both decrease for each sample with washing illustrating the ash and color contained in the sugar crystal. For color an average of 37.6% of color was removed by washing indicating this amount of color is contained in the syrup layer on the surface of the sugar crystal. This also indicates that approximately 62% of the color in refined cane sugar is found inside the sugar crystal. Washing removed an average of 82.8% of the ash from these refined sugars samples indicating only about 17% of the ash is inside the sugar crystal.

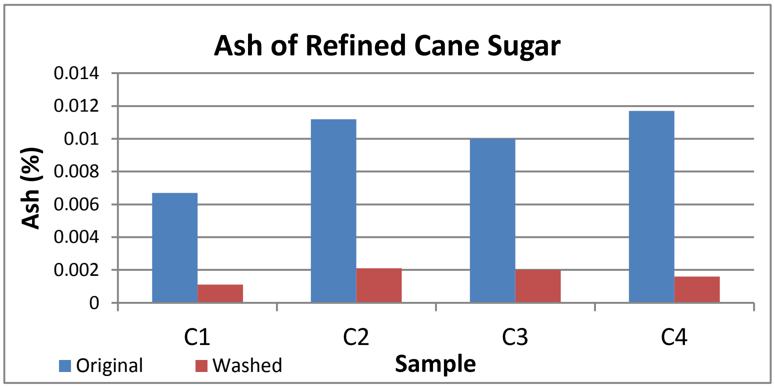
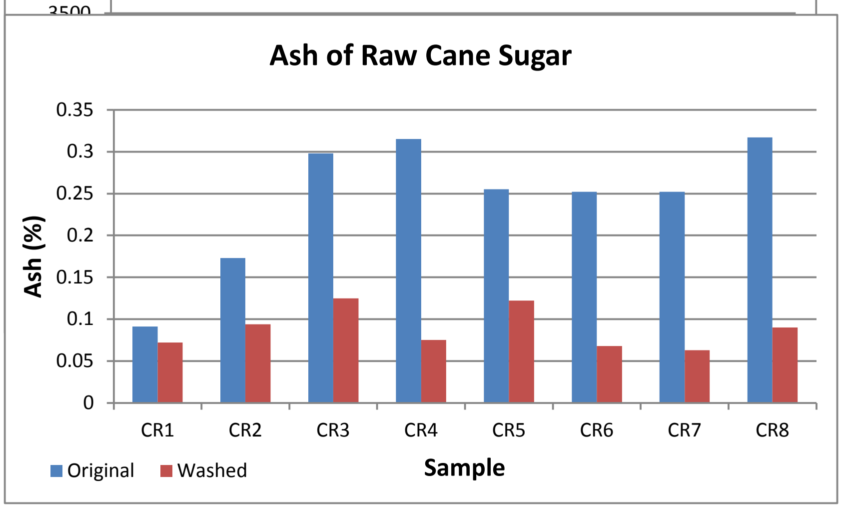


Figure 5. Ash of refined cane sugar before and after washing

The third set of samples that were included in the study includes raw cane sugars. This group was treated as the first two in determining original color and ash followed by washing and then determination of color and ash after the syrup layer surrounding the crystal is removed. Results are shown in Figures 6 and 7.

Figure 6. Color of raw cane sugars before and after washing



For raw cane sugars, colors before washing ranged from 552 IU to 3335 IU. Washing the crystals removed 31-88% of the color from the samples with colors after washing ranging from 337 IU to 859 IU.

Figure 7. Ash of raw cane sugars before and after washing

Original ash content for the samples ranged from 0.091% to 0.317% for a raw cane sugar. Ash removal ranged from 20.9% to 76.2% with ash content after washing ranging from 0.063% to 0.125%. Ash removal with washing is shown in Figure 7 for these samples.

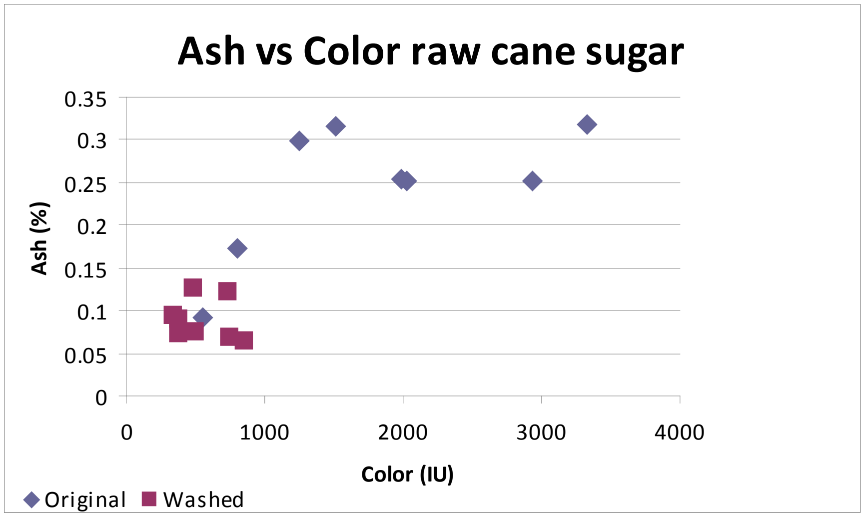
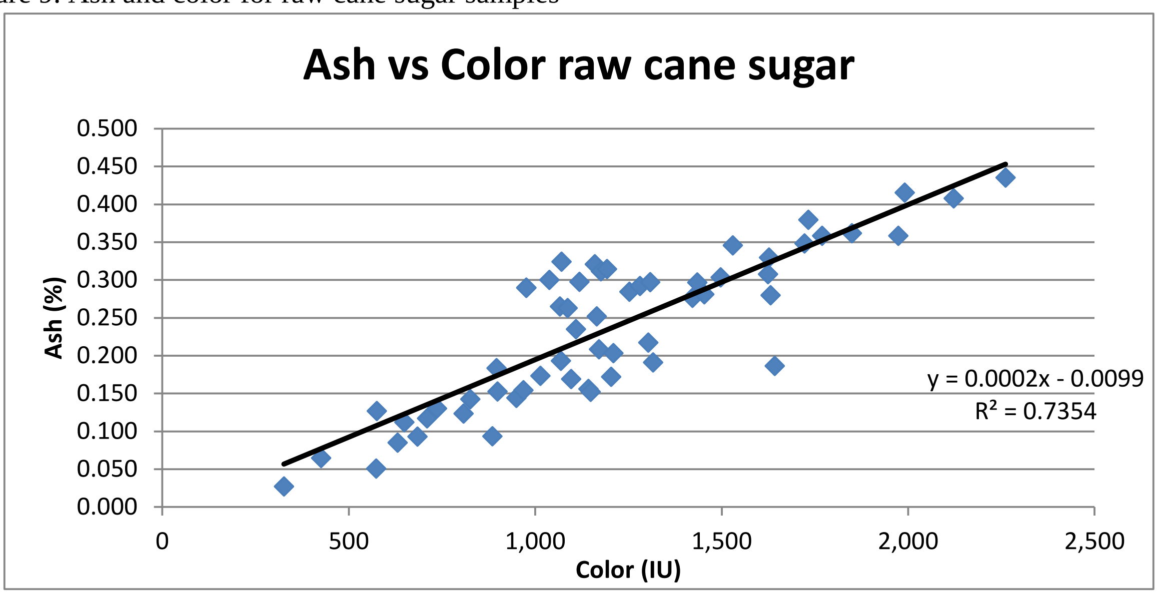


Figure 8. Relation of color and ash before and after washing for raw cane sugars.

Figure 8 shows the relation of ash and color content of raw sugar samples before and after washing. Color and ash both decrease for each raw sugar sample with washing illustrating the ash and color contained in the sugar crystal. A large decrease in color and ash by washing show the amount of colorant and ash material contained in the surface syrup layer surrounding the sugar crystal that is easily removed by affining the sugar.

Figure 9. Ash and color for raw cane sugar samples



A second set of raw sugars studied consists of 57 samples and includes the first set along with more sugars of typical color and ash content. Color ranges from 326 IU to 2,261 IU with an average of 1,198 IU. Ash ranges from 0.027% to 0.435% with an average of 0.235%. Figure 9 shows the trend of ash as color increases in the samples. The general trend is that ash increases in the samples as color increases for the raw sugars.

Ash and color correlations for the raw and refined white cane sugars were studied with correlations of 0.64 for raw sugars and 0.76 for white sugars as shown in Table 2. The correlation was better at 0.86 for all raw and refined cane sugars studied. For the washed samples, the correlations were much lower with a correlation of -0.18 for raw cane sugars and 0.42 for refined sugars but when taking all cane sugars into account, the correlation for color and ash was 0.74. Based on the data presented it appears there is a strong correlation between ash and color for cane sugars. This could be due to the limited number of samples studied within each type of cane sugar. Increasing the number of samples may strengthen the correlations for each type of cane sugar.

Table 2. Correlation of ash and color for raw and refined cane sugars

Sample	Original	Washed
Raw cane sugars	0.64	-0.18
Refined cane sugars	0.76	0.42
All cane sugars	0.86	0.74

CONCLUSION

White beet sugar crystallized from thick juice campaign, extract campaign, and the beet campaign as well as cane sugars including raw and refined white sugars were studied for color and ash content. Determination of ash and color was done using ICUMSA methods before and after washing of the sugars. The difference in the color and ash values before and after washing indicates that amount of material contained in the syrup surface layer that surrounds each sugar crystal. For the beet sugar samples included in this study, extract sugars were found to have the lowest ash content but on average the highest color. Most of the ash content in the thick juice and beet campaign sugars is found in the surface layer and easily removed by washing. For refined cane sugars, washing removed 26-51% of the color and 80 to 86% of the ash indicating more ash in the surface layer than in the crystal. For raw cane sugar with higher levels of color and ash compared to the white sugars, there seems to be a trend that as color increases so does the ash content of the sugars with 31-88% color removed by washing and 20 -76% ash removed by washing which simulates affining which is the first step of the refining process. The colorants found in this surface layer can be involved in the autocatalytic color reactions that cause color to increase during storage. Through centrifuge optimization with regard to washing times the color and ash components found in this surface layer may be removed. Correlations between ash and color were observed for both beet and cane sugars. Ash components such as iron and calcium can be involved in colorant reactions in both beet and cane processing. Colorants and color formation reactions are different in sugarbeet and sugarcane systems due to differences in the plants as well as processing conditions.

REFERENCES

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