Comparative evaluation of sugar beet processing intermediates color using various treatment methods

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Abstract. The article presents the results of a study of the influence of raw juice obtaining methods, namely, using an extractant, acidified with sulfuric acid with the addition of a suspension of freshly prepared gypsum, and using an extractant, treated with sulfur dioxide, as well as subsequent treatment of obtained thin juices with sulfur dioxide before evaporation on the color of sugar beet processing intermediates. A brief description of methods to reduce intermediates color used in sugar industry – sulfitation, ion-exchange and active carbon treatment is given. The advantage of intermediates treatment with sulfur dioxide compared with treatment with ion exchange resins and activated carbon is theoretically justified. The methodology for setting up laboratory tests is given and substantiated. The color indicators of raw juices, thin juices and obtained thick juices are given. It was established that a single treatment of intermediates with sulfur dioxide at the stage of raw juice obtaining provides a decrease in thick juice color by 7.11 %, a single treatment before evaporation – by 29.70 %, and a double treatment at the stage of raw juice obtaining and before the evaporation station – by 38.37 %.

1. Introduction

The color of sugar production intermediates is an important technological indicator, since it determines the crystalline sugar color. According to the current GOST 33222-2015, highly colored crystalline sugar belongs to lower quality categories and, as a result, has a low wholesale price. Given this, establishing a technological regime in production aimed at the highest yield of finished product, and not focusing on its consumer properties, ultimately, increases the risk of low quality categories sugar production – TS2 and TS3, and, as a result, the factory revenue will be lower than it would have been if it was produced high quality category sugar – Extra and TS1.

In the studies aimed at identifying coloring substances embedded in the yellow sugars crystal lattices, colleagues from the Voronezh State University of Engineering Technologies found that the predominant coloring substances are reducing sugars decomposition products [1]. It is noted that the content of reducing sugars decomposition products in yellow sugars is 10 times higher than caramels and melanoidins.

The increased content of reducing sugars in thick juice fed to crystallization is due to improperly selected technological modes of raw juice extraction and subsequent lime-carbon dioxide juice purification, as well as the time of its evaporation, which can be solved by adjusting the technological modes and imparting thermal stability to thin juice. Another factor is the initially high content of...
reducing sugars in the raw material. This case is more critical, since the adjustment of technological modes does not allow to completely eliminate it, which may be due to insufficient production capacity of technological equipment.

The most common causes of increased reducing sugars content in sugar beet are the conditions of its vegetation [2, 3]. Vegetation conditions may be independent of human factors, for example, lack of rainfall, spring cooling, early autumn frosts, as well as dependent factors, such as failure to comply with the optimal timing of fertilizer application, the timing of sowing and harvesting, also the level of agricultural machinery used [4].

Given this, in order to obtain crystalline sugar of Extra and TS1 quality categories, it is necessary to minimize the influence of coloring substances coming from raw material, as well as those that are formed under industrial conditions.

The most effective technological method to reduce the intermediates color is their treatment with sulfur dioxide or, so-called, sulfitation [5]. Sulfuric acid formed as a result of sulfur dioxide with water reaction blocks the aldehyde and ketone groups of monosaccharides, making their reaction with amino acids impossible.

When sulfide anhydride is exposed on unsaturated chromophore groups of coloring substances, hydrogen integrates double bonds, and coloring substance turn into colorless compounds [6]. In this case, the light absorption maximum shifts to the ultraviolet spectrum, the molecule becomes discolored in the visible spectrum due to a decrease in unsaturated double bonds. Sulphurous anhydride more intensely decolorizes the products of reducing sugars alkaline decomposition and substances with low molecular mass (caramelan), than with high molecular mass (caramelen, caramelin and melanoidins).

Less common is the method of decolorization by ion exchange resins, which is mainly used for concentrated intermediates – thick juice, liquor and raw sugar solutions (remelts) [7]. Strongly basic anionic resins are most prevalent, since their use requires less reagents to restore ion-exchange properties compared to acidic cationic resins, as a result less environmentally hazardous waste are formed. Nevertheless, even for strong base anionic resins regeneration up to 200 g of sodium chloride per 1 liter of resins and 10-15 times the volume of water relative to ion-exchange columns bed volume are required.

Also, activated carbon was previously used for sugar beet processing intermediates decolorization. However, this method is not currently used, since it requires high reagent consumption and also increases the load on the filter equipment, which significantly increases finished product cost.

The main difference between these three methods is that sulfitation, along with bleaching, also has a preventive effect, since sulfite ions block the aldehyde and ketone groups of glucose and fructose, preventing the Maillard reactions. The greatest effect is observed in technological stages with high temperature: thin juice evaporation stage and massecuite boiling stage. Ion exchange and activated carbon, in turn, solve the problem of high color reducing only in cases where this problem already exists due to the Maillard reactions.

Given this, the sulfitation process is more preferable in comparison with the ion-exchange process and activated carbon treatment, since it is less equipment-intensive, material- and energy-consuming, and characterized by the absence of significant amount of reagents usage and environmentally harmful waste. In addition, sulfitation to a greater extent prevents the cause of coloring substances formation problem, but not eliminates its consequences.

Considering that intermediates sulfitation is mainly carried out at three technological stages, namely, extractant preparation, thin juice processing and thick juice and/or liquor processing, the setting of experiments was aimed at studying the impact of sulfitation treatment on the quality of intermediates, when it is carried out at one or several stages of production, as well as in its complete absence.
2. Materials and methods

For experimental verification of sulfitation treatment effectiveness in laboratory conditions it was carried out a series of studies of the influence of various modes of extractant preparation for obtaining raw juice and subsequent lime-carbon dioxide purification according to the method of prof. P.M. Silin on the color of sugar beet processing intermediates.

Preliminary, there were conducted experiments with various raw juice purification methods, namely, according to the method of prof. P.M. Silin and to the method of lime-carbon dioxide purification, the most commonly used in industry, including progressive preliming with carbonation sediments suspensions return, cold and hot main liming, first carbonation, post liming and second carbonation. As a result, the purifying method of prof P.M. Silin was chosen as more affordable in time and reagents usage in laboratory studies. In addition, the juices purified by this method produce somewhat deteriorated quality, which, with their subsequent sulfite treatment, gives more visual results. The objects of research were raw juices, obtained from them thin juices, purified by the method of prof. P.M. Silin, as well as thick juices obtained from thin juices.

At the first stage of studies, raw juices were obtained using two extractant preparation methods: acidification with sulfuric acid with the addition of calcium hydroxide suspension to achieve the resulting freshly prepared gypsum content of 3 % by cossettes weight (Experiment 1) and treatment with sulfur dioxide (Experiment 2). The pH of the extractant in the experiments was 5.50 ± 0.10, the extraction time was 60 minutes. The color of obtained raw juices was determined.

At the second stage of studies, obtained raw juices were purified according to the method of prof. P.M. Silin, described in the «Instruction for chemical-technical control and accounting of sugar production», to an average alkalinity of 0.017 % CaO [8]. Next, the carbonated juices of each experiment were divided into two equal parts. One part was left untreated, and the second part was treated with sulfur dioxide to a pH of 8.80 ± 0.10, filtered and the color of four samples of thin juices was determined.

At the third stage of the studies, the obtained four samples of thin juices were evaporated in order to identify the influence of the method of extractant preparation and thin juice treatment with sulfur dioxide on the color of obtained thick juices. For this purpose, thin juices were evaporated on glycerol bath at 120 °C for 90 minutes to a dry-substance content of 55-60%. The color of obtained thick juices was determined.

The color of sugar beet processing intermediates was determined by the photometric method according to the generally accepted method (ICUMSA-420) [8].

3. Results and Discussion

Table 1 presents the averaged data of five series of studies on the influence of the extractant preparation method on the color of raw juice.

| Table 1. The influence of extractant preparation method on the color of obtaining raw juice |
|---------------------------------------------------------------|
| Experiment 1                                                                 | Experiment 2                                                                 |
| Raw juice, obtained using an extractant, acidified with sulfuric acid with the addition of calcium hydroxide suspension | Raw juice, obtained using an extractant, treated with sulfur dioxide |
| Juice color, ICUMSA unit                                      | 466,54                                                                 | 349,65                                                                 |
| Color reduction, ICUMSA unit                                  | –                                                                       | 116,89                                                                 |
| Color reduction, %                                            | –                                                                       | 25,05                                                                 |
From the presented data it follows that, ceteris paribus, the method of extractant treatment with sulfur dioxide provides the obtaining of raw juices with coloring substances content lower than when the extractant is acidified with sulfuric acid with the addition of a suspension of freshly prepared gypsum in an amount of 3 % by weight of chips.

Table 2 presents the averaged data of five series of studies on the influence of extractant preparation method and thin juice treatment with sulfur dioxide on the color of juices obtained due to the method of prof. P.M. Silin.

| Experiment 1 | Experiment 2 |
|--------------|--------------|
| Thin juice, not treated with sulfur dioxide | Thin juice, treated with sulfur dioxide |
| Thin juice, not treated with sulfur dioxide | Thin juice, treated with sulfur dioxide |
| Juice color, ICUMSA unit | 87,36 | 72,80 | 67,25 | 50,40 |
| Color reduction, ICUMSA unit | 14,56 | 20,11 | 36,96 |
| Color reduction, % | 16,67 | 23,02 | 42,31 |

From the presented data it follows that, single intermediates treatment with sulfur dioxide at the stage of raw juice purification provides thin juice color reduction by about 17 %, while single treatment at the stage of raw juice obtaining provides color reduction of 23 %. This is explained by the efficiency of blocking the aldehyde and ketone groups of reducing sugars in the early stages is higher, since during lime-carbon dioxide purification Maillard reactions occur to a lesser extent. Most effective for thin juice color reduction is twofold treatment with sulfur dioxide, i.e. at the stage of raw juice obtaining and at the stage lime-carbon dioxide purification before evaporation. In this case, the thin juice color is reduced by 42 % compared with thin juice, untreated with sulfur dioxide.

Table 3 presents the averaged data of five series of studies on the influence of extractant preparation method and thin juice treatment with sulfur dioxide on the color of obtaining thick juices.

| Experiment 1 | Experiment 2 |
|--------------|--------------|
| Thick juice, obtained from thin juice, not treated with sulfur dioxide | Thick juice, obtained from thin juice, treated with sulfur dioxide |
| Thick juice, obtained from thin juice, not treated with sulfur dioxide | Thick juice, obtained from thin juice, treated with sulfur dioxide |
| Juice color, ICUMSA unit | 186,80 | 131,32 | 173,51 | 115,13 |
| Color reduction, ICUMSA unit | 55,48 | 13,29 | 71,67 |
| Color reduction, % | 29,70 | 7,11 | 38,37 |

From the presented data it follows that residual content of sulfite ions in thin juice treated with sulfur dioxide at the stage of its obtaining was lower than in thin juice treated directly before evaporation, which led to a greater increase in the color of thick juice. So, the decrease in color of thick juice was 7 % in the first case and 29.7 % in the second case. This result is explained by the fact that sulfur acids formed as a result of sulfitation at the stage of extractant treatment are unstable when
heated. With the free sulfurous acid oxidation in juice, sulfonic acids are decayed in accordance with the active masses law, especially in evaporation conditions, which is associated with an inevitable increase in intermediates color [6]. Therefore, sulfitation should be carried out to the content of a certain free sulfur dioxide amount, which was shown by the experiment. In this way, the most effective is twofold intermediates treatment with sulfur dioxide, which provides thick juice with color of 115.13 ICUMSA units, that is, 38 % lower than the color of thick juice not treated with sulfur dioxide.

However, even single treatment of thin juice with sulfur dioxide directly before its evaporation under severe temperature conditions provides thick juice with color of 29.70 % lower compared to thick juice not treated with sulfur dioxide.

4. Conclusions
Based on the studies, it can be reasonably concluded that twofold treatment of sugar beet processing intermediates with sulfur dioxide, namely, extractant for raw juice obtaining treatment and subsequent thin juice treatment, provides the lowest color of thick juice fed to crystallization, which will subsequently allow get crystalline sugar of a high quality category.

However, single thin juice treatment with sulfur dioxide before its evaporation also allows a sufficient degree of aldehyde and ketone groups of glucose and fructose blocking, thereby preventing the formation of coloring substances as a result of thermochemical effects during the evaporation process. However, the resulting effect is lower than twofold treatment of sugar beet processing intermediates.

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