Intensification of the inverted sugar syrup production using the rotor-pulsation processing

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Abstract

The aim of the work is to intensify the process of inverted sugar syrup production using the treatment of water-sugar solution in a rotor-pulsation apparatus.

Materials and methods. The aqueous solutions of chemically pure sucrose were used. The studies were conducted using a rotor-pulsation apparatus in the range of flow shear rates from 20×10^3 $s^{-1}$to 50×10^3 s^{-1}. Determination of carbohydrate content was carried out by high-performance liquid chromatography (HPLC).

Results and discussion. The increase in temperature, processing time, and decrease in pH value at a constant flow shear rate led to an increase of the amount of inverted sugar formed. Complete inversion of sucrose occured at a treatment of sugar solution at temperature of 80 °C, pH 3.0, and a flow shear rate of 20×10^3 s^{-1} for 30 minutes. At a flow rate of 50×10^3 s^{-1} almost all sucrose was hydrolyzed at pH 3.5, and the duration of the process was just 5 minutes under conditions of five-time processing in the rotor-pulsation apparatus in the circulating mode.

In the syrup prepared by the proposed technology at a temperature of 70 °C and the duration of treatment 5 minutes, all sucrose in the solution was inverted, and no traces of hydroxymethylfurfural were detected.

It is assumed that critical stresses occur at the site of the sucrose chain, and the chemical covalent bonds are broken. The break of these bonds during the process of mechano-chemical destruction occurs on the weakest in terms of energy bonds. As a result of mechano-chemical influence on the section of the sucrose chain (C – O – C), there are critical stresses and the connection is broken. This leads to the formation of free radicals. One radical attaches to the OH^- ion and another to the H^+ ion forming glucose and fructose.

Conclusions. The use of treatment of water-sugar solution in a rotor-pulsation apparatus by the proposed technology allows to intensify process of sugar inversion, namely, reduces the duration of the inversion from 120 to 5 minutes, and ensures almost complete inversion of sucrose excluding the formation of hydroxymethylfurfural.

Introduction

Inverted sugar syrups, product of hydrolyzed sucrose, which contains instead of sucrose the mixture of glucose and fructose in ratio 1: 1, become a major sweetener and additive used extensively in the production of a wide variety of foods and beverages. Inverted sugar syrups have many advantages compared to sucrose that makes them attractive to food manufacturers. These include its sweetness, solubility, acidity, and its relative cheapness (Parker et al., 2010). Starch produced from corn, wheat, or barley due to enzymatic hydrolysis is converted into glucose, and then due to isomerization into inverted sugar syrup (Ermolaeva et al, 2012).

There are several ways to obtain inverted sugar syrup from sucrose, namely enzymatic hydrolysis (Mouelhi et al., 2014); hydrolysis by ion exchange (Khan et al., 1996), but the simplest and most common is the method of inverting sucrose by the treatment with citric acid. This method, however, has a number of disadvantages, namely the high process temperature (80–100 ºC), long process duration (up to 2 hours), and most importantly, that this method allows to invert no more than 55% of sucrose due to increased accumulation of hydroxymethylfurfural (Vasilishina et al., 1986).

The task is to increase the yield of inverted sugar by using additional methods of the inversion process.

In the food industry, rotor-pulsation apparatus (high-shear mixers) are used to create emulsions with both high and low viscosity such as salad dressings, sauces, cottage cheese, fruit, vegetable and sour milk desserts, purees, and cream. Rotor-pulsation apparatus are also used for dispersing artificial sweeteners, and cloud agents in carbonated soft drinks, for blending miscible liquids of very different viscosities, to deagglomerate and uniformly disperse nanoparticles in liquids, and also to suspend fine air bubbles (e.g., ice cream) (Rodgers et al., 2016).

The physical effects of rotor-pulsation apparatus have been studied by many authors. For example, in (Avdeeva et al., 2011) the influence of hydrodynamic cavitation on the production of phospholipid nanostructures was studied.

The influence of alternating impulses of pressure occurring in the rotor-pulsation apparatus on liquid binary systems such as water systems and water-ethanol mixtures was studied in (Dubovkina, 2017).

Under the influence of alternating impulses of pressure, the quantity of the dissolved oxygen in the water and water-ethanol mixtures in comparison with the initial maintenance decreased practically by 50–55%.

Studies (Dubovkina, 2017a) of hydrodynamic oscillations generated to activate the hydrated lime suspension for beet juice treatment have shown an increase in the hydrogen potential of the hydrated lime suspension by 15%.

Intensification of the process of inverted sugar syrup production can be achieved by including of the rotor-pulsation apparatus in the appropriate technological scheme (Myronchuk et al., 2019).

Therefore, there is a need to study the influence of physicochemical effects that occur during the processing of glucose-fructose syrups in a rotor-pulsation apparatus on the intensity of sucrose inversion.

The main technological parameters of the sucrose inversion process are temperature, pH of the solution, and the process duration. Given the fact that the proposed technological scheme includes the processing of syrup in a rotor-pulsation apparatus, it is advisable to enter another parameter influenced on the intensity of the hydrolysis process. This parameter is the shear flow rate γ, s^{-1}, in the gap between the rotor and the stator of the rotor-pulsation apparatus (Dolinskij et al., 1998; 1999; 1999a).

The aim of this work was to intensify the technology of inverted sugar syrup production from sucrose using the treatment of water-sugar solution in a rotor-pulsation apparatus.

Materials and methods

The object of study was the process of inverted sugar syrup production. The subject of research was determination of the effect of flow rate in the rotary pulsation apparatus on the inversion of sucrose in aqueous solutions of sucrose.

Experimental instalation

Experimental studies of this process were conducted on a specially created pilot plant, the scheme of which is presented in Figure 1. The volume of the sugar syrup vessel was 100 liters. The productivity of the rotor-pulsation apparatus (**RPA**) was 6 m^3/h.

The rotor-pulsation apparatus consists of two coaxial cylinders namely a fixed stator and a movable rotor with radial channels in the side walls of different cross- sections (Fig. 2) (Zhang et al., 2012). The substance to be treated is fed into the rotor cavity, through the channels passes into the inter-cylinder gap, and subsequently through the stator channels.

The shear rate of the flow was regulated by changing the speed of the rotor shaft using a frequency converter.

The range of flow shear rates was from 20×10^3 s^{-1} to 50×10^3 s^{-1}.

During the studies, the dependence of sucrose inversion on temperature, pH and process duration was determined at a constant flow shear rate, which was 20×10^3 s^{-1}. The amount of inverted sugar formation was determined depending on the flow shear rate, pH and process duration at a temperature of 70 °C.

Technological operations were performed in the following order. In the sugar syrup vessel 3, the sugar was mixed with water to a dry matter concentration of 65%. In citric acid solution preparation vessel 1, a 25% solution of citric acid was prepared and added to the sugar syrup vessel 3 in an amount of 0.75 kg of citric acid per 100 kg of sugar.

This solution was treated in a rotor-pulsation apparatus in a recirculation mode until complete hydrolysis of sucrose was occurred, and then sent for cooling and storage. The dry matter content in inverted glucose-fructose syrup was about 65%.

Determination of carbohydrates

Determination of carbohydrates content was carried out by high-performance liquid chromatography (HPLC) (Costa et al., 2015).

An Agilent 1100 chromatograph with a diode array detector was used in this study. UV detection was performed at 195 nm at a column temperature of 30 °C and a column with the aminopropyl stationary phase (Zorbax Carbohydrate 250x4.6 mm, 5 μm, manufactured by Agilent). The ratio of acetonitrile used and deionized water was from 82 to 18% vol.

Determination of pH value

To determine the degree of activity of hydrogen ions in the liquid used a pH meter Ezodo 5011.

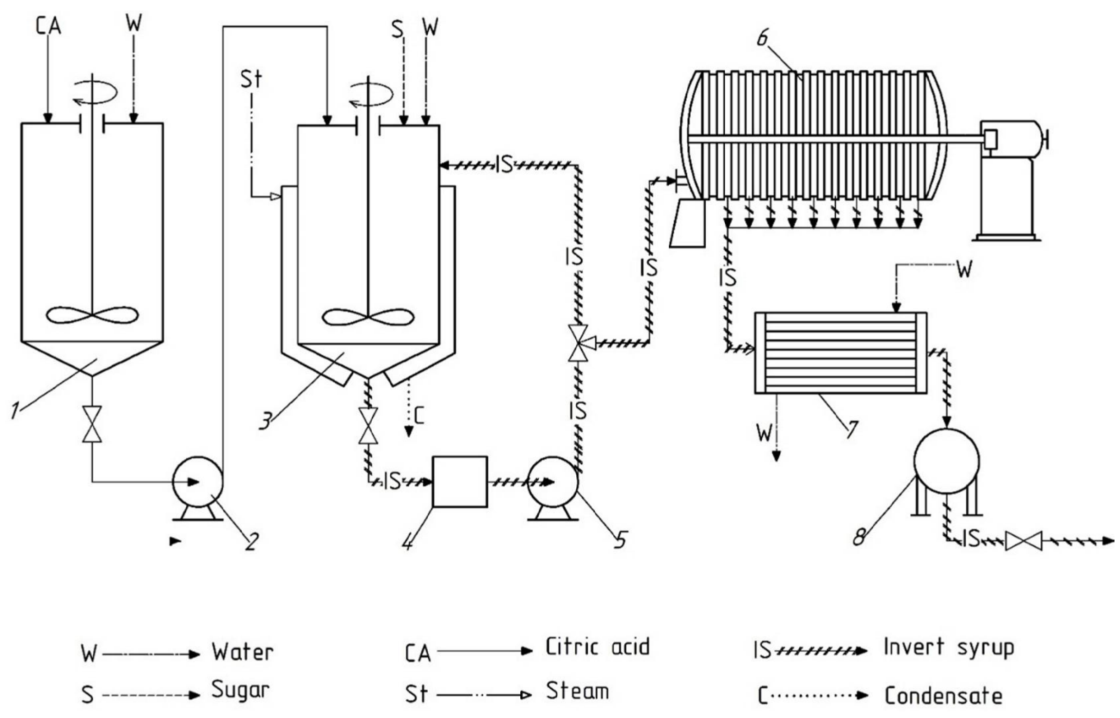

Figure 1. Technological scheme of the inverted sugar syrups preparation using a rotor-pulsation apparatus:
1 – citric acid solution preparation vessel: 2 – pump;
3 – sugar syrup vessel; 4 – filter; 5 – rotor-pulsation apparatus;
6 – filter; 7 – heat exchanger; 8 – storage vessel

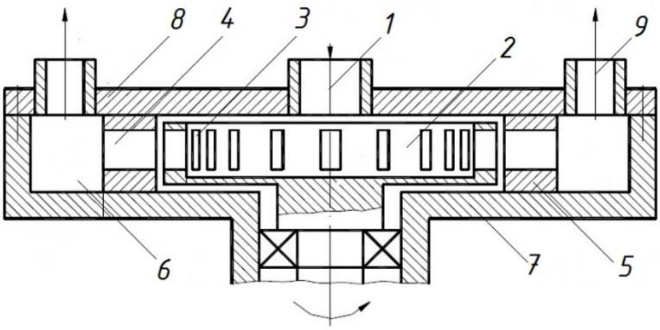

Figure 2. The design of rotor-pulsation apparatus:
1 – outlet; 2 – rotor; 3 – channels in the rotor; 4 – channels in the stator;
5 – stator; 6 – sound camera; 7 – housing; 8 – cover; 9 – inlet pipe.

Results and discussion

Dependence of the formation of inverted sugar on the temperature, pH, and duration of the process at a shear rate of 20×10^3 s^{-1}

The dependence of the inverted sugar formation on the temperature, pH, and the process duration at a flow shear rate of 20×10^3 s^{-1} is shown in Table 1. It was determined that with the process duration increasing from 30 to 120 minutes at a flow shear rate of 20×10^3 s^{-1}, the amount of the formed inverted sugar increased from 43 to 46% at pH = 4.0; from 51 to 57% at pH = 3.5; from 61 to 66% at pH = 3.0; from 69 to 71% at pH = 2.5 at a temperature of 50 °C. Increasing the temperature to 80 °C led to the fact that with increasing process duration from 30 to 120 minutes at a flow shear rate of 20×10^3 s^{-1} the amount of the formed inverted sugar increased from 53 to 58% at pH = 4.0; from 72 to 75% at pH = 3.5; from 98 to 100% at pH = 3.0. Increasing the temperature to 90 °C led to the fact that with increasing process duration from 30 to 120 minutes at a flow shear rate of 20×10^3 s^{-1} the amount of the formed inverted sugar increased from 98 to 100% at pH 4.0.

Dependence of the inverted sugar formation on the flow shear rate, pH, and the process duration at a constant temperature of 70 °C

The dependence of the inverted sugar formation on the flow shear rate, pH, and the process duration at a constant temperature of 70 °C is given in Table 2.

It was determined that with increasing of the process duration from 5 to 20 minutes at the flow shear rate of 30×10^3 s^{-1} the amount of inverted sugar increased from 65 to 73%; at a shear rate of the flow of 40×10^3 s^{-1} from 75 to 85%; at a flow shear rate of 50×10^3 s^{-1} from 87 to 93%. It was determined that with increasing the process duration from 5 to 20 minutes at the flow shear rate of 30×10^3 s^{-1} the amount of inverted sugar increased from 65 to 73%; at a flow shear rate of 40×10^3 s^{-1} from 75 to 85%; at a flow shear rate of 50×10^3 s^{-1} from 87 to 93%. Reducing the pH to 3.5, depending on the process duration from 5 to 20 minutes at a flow shear rate of 30×10^3 s^{-1}, amount of the inverted sugar increased from 74 to 81%; at a flow shear rate of 40×10^3 s^{-1} from 89 to 96%; at a flow shear rate of 50×10^3 s^{-1} almost all sucrose was hydrolyzed in 5 minutes of processing (Patent UA 9399. A method of preparing inverted sugar syrup).

Table 1

Formation of inverted sugar depending on the temperature, pH and the process duration at a flow shear rate of 20×10^3 s^{-1}

t, °C	pH	Process duration, min	Amount of inverted sugar, %
50	4.0	30	43
		60	45
		120	46
	3.5	30	51
		60	55
		120	57
	3.0	30	61
		60	65
		120	66
	2.5	30	69
		60	71
		120	71
80	4.0	30	53
		60	57
		120	58
	3.5	30	72
		60	74
		120	75
	3.0	30	98
		60	99
		120	100
90	4.0	30	98
		60	100
		120	100

Table 2

Formation of inverted sugar depending on the flow rate, pH and the process duration at a temperature of 70 °C

pH	Process duration, min	γ, $\times10^3$ s^{-1}	Amount of inverted sugar, %
40	5	30	65
	10		71
	20		73
	5	40	75
	10		83
	20		85
	.	50	97
			91
			92
3.5	5	30	74
	10		79
	20		81
	5	40	89
	10		94
	20		96
	5	50	100
	10		100
	20		100

The traditional technology of the acid inversion of sucrose is accompanied by the formation of a toxic by-product of the deep decomposition of sucrose hydroxymitylfurfural. This is due to the long duration and the high temperature of treatment. The use of rotor-pulsation apparatus in the process of inverted sugar syrup production eliminates the formation of hydroxymethylfurfural due to reducing the inversion process duration to 5 minutes.

Determination of the composition of inverted sugar syrup depending on the processing method

The characteristics of inverted sugar syrup prepared according to the existing and proposed technology are presented in Table 3.

Table 3

Characteristics of inverted sugar syrup obtained in the traditional way and using a rotor-pulsation apparatus

Method of treatment	Duration, min	Amount of inverted sugar, %	Content of hydroxymethylfurfural, mg/1000 g of syrup
90 ºC, without treatment in a potor-puslation apparatus	120	55	0.09
70 ºC, with treatment in a potor-puslation apparatus	5	100	-

The results indicate that application of proposed technology for production of inverted sugar syrup reduces the process duration from 120 min to 5 min, ensures almost complete hydrolysis of sucrose, and increases the amount of inverted sugar in solution from 55 to 100% without the formation of hydroxymethylfurfural.

Effect of the inversion duration on the composition of sugars at a temperature of 70ºC, pH 3.5 and the flow rate of 50×10^3 s^{-1}

Figure 3 shows the results of studies of the effect of the inversion duration on the composition of sugars at a temperature of 70 ºC, pH 3.5 and a shear flow rate of 50×10^3 s^{-1}.

Effect of the number of processing cycles in the rotary pulsation apparatus on the degree of sucrose inversion.

Results shown in Figure 4 indicate that complete inversion of sucrose was achieved by five times treatment in a rotor-pulsation apparatus in a circulating mode for 5 minutes at a temperature of 70 °C, pH 3.5 and shear flow rate of 50×10^3 s^{-1}.

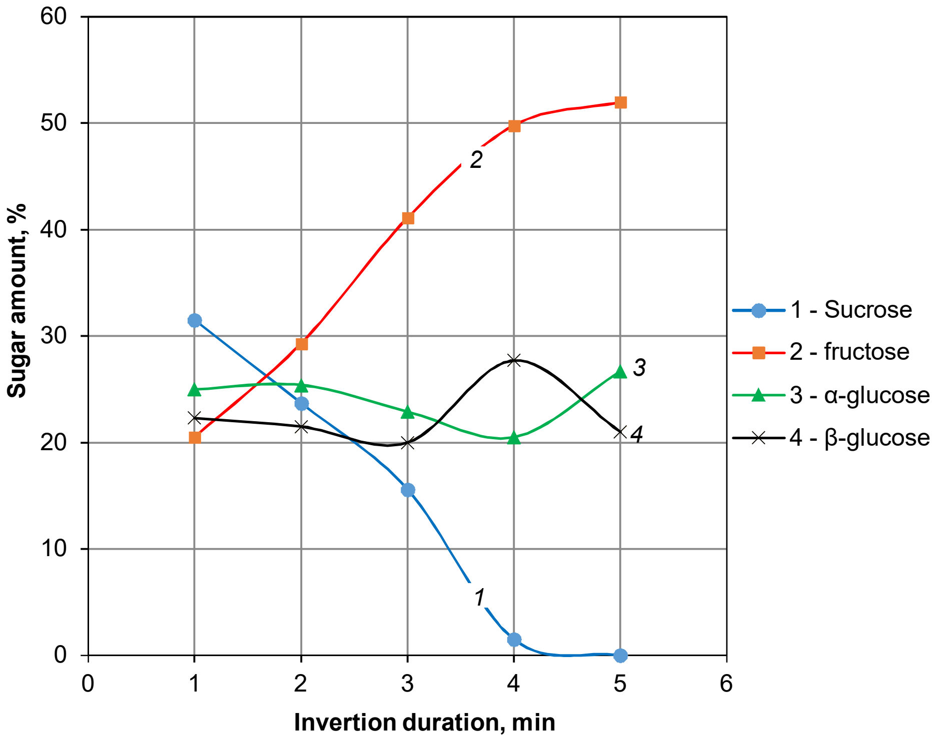

Figure 3. Production of sugars depending on the inversion duration at a temperature of 70 °C, pH 3.5 and a shear flow rate of 50×10^3 s^{-1}

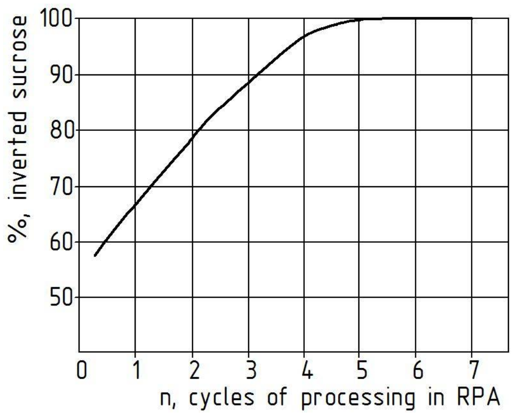

Figure 4. Amount of inverted sucrose depending on the number of cycles of glucose-fruit syrup processing in a rotor-pulsation apparatus

Therefore, the intensive inversion of sucrose using of rotor-pulsation treatment causes destruction of an acetal bond (an acetal oxygen bridge) (C – O – C) which joins glucose and fructose units in molecule of sucrose due to the simultaneous action of chemical and physical influences. The breaking energy of this bond is 1076 kJ/mol.

Critical stresses occur at the site of the sucrose chain and chemical covalent bonds are broken. The break of these bonds in the process of mechano-chemical influence occurs on the weakest in terms of energy bonds. In the case of mechanically activated hydrolysis, the destruction of the chains is localized at heterogeneous bonds, and in this respect, such processes do not differ from the corresponding purely chemical ones.

The breakdown of the sucrose chain usually follows the acetal bonds, but under certain conditions of the process, there is a breaking of carbon (C–C) bonds (Stick, 2001).

Action of citric acid weakens the (C–O) bond in sucrose and allows implementing a mechanically activated chemical process. Because of mechano-chemical influence on the section of the sucrose chain (C–O–C) there are critical stresses and the connection is broken. This leads to the formation of free radicals. One radical attaches the OH^- ion and another to H^+ ion As a result, the process of production of inverted sugar is intensified, i.e., the formation of a mixture of monosaccharides of glucose and fructose.

It is important that the proposed technology has significant advantages in terms of energy and resource saving characteristics (Dolinskiy et al., 2012).

Conclusion

The use of circulating five-time rotor-pulsation treatment of water-sugar solution in the circulating mode in the technology of inverted sugar syrups production at a temperature of 70 °C, pH 3.5 and a flow rate of 50×10^3 s^{-1} allows intensifying this process, namely:

- Reduce the duration of inversion to 5 minutes;
- Increase the amount of inverted sucrose from 55% to almost complete inversion;
- Exclude the formation of hydroxymethylfurfural.

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