RETURNABLE BAKING WASTE — A NEW TYPE OF RAW MATERIALS FOR DISTILLATES PRODUCTION
(PART II. STAGE OF RAW MATERIALS PREPARATION FOR DISTILLATION)

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ABSTRACT

The processes that take place upon saccharified wort obtaining from the returnable baking waste and its fermentation was the research subject of this work. The development of operational parameters at the stage of returnable baking waste preparation for distillation, which provides a high-quality product is the purpose of the work. The samples of saccharified and fermented wort obtained from various bread and bakery products types produced by large enterprises in Moscow were the objects of the study. To characterize the composition of saccharified and fermented wort, the indicators to assess the quality of the wort from grain raw materials were used. The mass concentration of individual sugars in the wort was determined using high performance liquid chromatography on an Agilent Technologies 1200 Series device. The qualitative composition and volatile components concentration in the fermented wort was determined using gas chromatography on a Thermo Trace GC Ultra device. It was established that the percentage of solids transition to a soluble state does not depend on a returnable waste type and is in the range from 87.6% to 90.7%, and the starch transition to a soluble state, on the contrary, is determined by the processed raw materials type. It is shown that the use of rye-wheat bread after its preliminary enzymatic treatment with thinning and cytolytic drugs in a mixture with wheat bread in a ratio of 1:1 to 1:2 can improve the wort rheological characteristics and transfer from 98.1% to 99.3% starch of raw materials in a soluble state. It has been shown that for the efficient process of saccharified wort fermentation from returnable baking waste, the Fermiol and Turbo-24 alcohol yeast races are most suitable, which ensure a high yield of ethanol and an optimal composition of volatile components in the fermented wort.

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1. Introduction

As you know, the quality of distillates depends not only on the feedstock biochemical composition, but also is formed at all technological stages of their production. When distillates are produced from traditional types of starch-containing raw materials (wheat, rye, barley, corn, etc.), the stage of its distillation preparation involves several successive technological processes: raw materials preparation for cooking (grinding); grinded grain cooking to destroy the cellular structure and starch transfer into a soluble state; starch saccharification with malt enzymes or with the complex enzyme preparations use; sugars fermentation by yeast [1].

To transfer native raw materials starch to accessible state for saccharification and fermentation, various modes of water-heat treatment are used [2,3,4]. Current trends in improving the process of saccharified wort obtaining are aimed at switching to mild conditions for raw materials starch preparing for fermentation without excessive pressure and temperature above 100 °C use [5,6]. In the case of mechanical-enzymatic method of raw materials processing in alcohol production with the enzyme's participation of microbial origin, at each process stage, the optimal conditions are created for the action of α- and glucoamylases necessary for the starch hydrolysis. Moreover, this technology allows to take into account both the specificity of the processed raw materials and the spectrum of enzyme preparations that can be used [7,8,9].

For starch raw materials saccharification and plant cell biopolymers destruction in order to prepare it for fermentation, enzyme preparations of various spectrum of action are used [10,11]. Thus, preparations containing cytolytic enzymes, while ensuring optimal conditions for their action, allow grain non-starch polysaccharides hydrolysis, which are potential sources of fermentable carbohydrates [12,13]. This technique allows to increase the alcohol yield by 3–5%, as well as to facilitate the process by reducing the processed media viscosity.

It should be noted the development of enzyme preparations use containing proteases of fungal and bacterial origin [14,15]. The main purpose of their introduction into the technological process of alcohol production is the accumulation in the wort of low molecular weight nitrogen-containing compounds necessary for yeast feeding, improving their vital activity, and, as a result, intensifying alcohol fermentation [16,17].

Wort fermentation is one of the most important stages in the fermentation technology. At this production stage due to the enzymatic yeast cells apparatus, qualitative indicators of the final product are formed, which include ethanol and aromatizing secondary fermentation products [18].Saccharified wort from starch-containing raw materials is a complex system that includes, in addition to fermentable sugars (maltose and glucose), intermediate starch hydrolysis products that are not involved in the yeast metabolism [1,19,12,14].

Saccharomyces cerevisiae yeasts of various races are used in fermentation industry, and their living conditions are determined by the composition and technological parameters of the wort fermentation. It is known that in many respects the efficiency of the fermentation process depends on the dextrins
concentration contained in saccharified wort to ethanol, since most yeast races are not able to ferment them. For this reason, the use of special yeast races with glucoamylase activity (S. cerevisiae Y – 717) is required [20,21].

Features of the structural-mechanical and biochemical composition of the returnable baking waste, consisting in an increased starch content, its better accessibility to hydrolysis, fewer non-starch polysaccharides, in a higher sugars concentration and high molecular weight dextrans [22,23] compared to traditional raw materials, allow grinding and cooking operations to be excluded. Thus, the use of returnable baking waste in the distillates production will reduce the cost of the product and increase the efficiency of the whole production process.

The use of a new raw material type in any production requires a thorough study of the processes occurring at each stage. This approach is also fully justified in the wort from returnable baking waste production and fermentation.

The purpose of this work was to develop regime parameters at the stage of preparing returnable baking waste for distillation, providing a high-quality product.

2. Materials and methods

The objects of the study were samples of saccharified and fermented wort, obtained from various feedstock types. As a raw material, industrial samples of returnable baking waste were used, obtained both from wheat flour and from a mixture of rye and wheat flour in different proportions, in addition, sugar, vegetable oil and complex additives were included in the recipe of individual products. Returnable waste samples were obtained from bakeries in Moscow (bakery No. 24, "Peko" bakery, "Cheryomushki" bakery, Moscow bakery and confectionery "Kolomenskoye", "Nizhegorodsky khleb OOO" (Limited Liability Company), "Russky khleb OOO" (Limited Liability Company), "Cherkizovo OAO" (Open Joint-stock Company)). Samples were prepared as follows: the product was manually cut into cubes 1cm x 1cm in size, the cubes were dried under mild conditions (temperature not more than 100 °C) until a moisture content of 1 cm of absolute alcohol (mg/dm³ of absolute alcohol (mg/dm³ of a. a.).

Research results processing was carried out using statistical methods. Illustrative material presents the average values of three dimensions.

3. Results and discussion

As a method for saccharified wort production, in this work, at the first stage, the previously developed regime parameters for the wheat bread processing provided for the ethyl alcohol technology were used [22]. They included mixing the raw material with warm water (at a temperature 70 °C) at a hydromodule of 1:5.5, introducing a diluting enzyme preparation with mesophilic alpha-amylase with a dosage of 0.5 units of AA/g (amylase activity/g) of conventional starch of raw materials, holding the mixture at the specified temperature for 90 minutes, raising the temperature to 95–98 °C and mixture processing for 30 minutes, cooling the resulting mass to a temperature of 56–58 °C, introducing the saccharifying enzyme preparation with dosage of 6.0 units of GLA/g (glucoamylase activity/g) of conventional starch raw materials, saccharification for 30 minutes.

It was established (Table 1) that the returnable baking waste processing at the accepted operating parameters makes it possible to obtain a fairly concentrated wort, this indicator varies between 17.0–21.5%. A tendency to increase the wort concentration was revealed when using samples obtained from wheat flour as raw materials against samples from rye-wheat flour.

Analysis of total fermentable carbohydrates (TFC) showed that this indicator correlates with the starch content in raw materials. The maximum TFC value corresponds to sample 3 (15.7%), in which the starch content exceeds its value for other samples. The minimum concentration of TFC was detected in

| Samples | The feedstock composition | The concentration in wort, % | Purity, % |
|---------|--------------------------|-----------------------------|-----------|
| 1       | Hearths wheat bread, piece | 21.0 | 14.9 | 3.7 | 71.1 |
| 2       | Wheat panloaf, peace     | 18.9 | 13.8 | 3.9 | 72.9 |
| 3       | White bread              | 21.3 | 15.7 | 4.5 | 73.8 |
| 4       | Sliced long loaf I        | 20.5 | 15.0 | 4.8 | 73.0 |
| 5       | Sliced long loaf II       | 20.1 | 15.2 | 4.8 | 73.7 |
| 6       | Sliced long loaf III      | 19.8 | 14.8 | 2.7 | 74.6 |
| 7       | Hearths wheat-rye simple bread, piece | 19.8 | 14.3 | 2.6 | 72.5 |
| 8       | Rye-wheat hearth simple bread, piece | 18.5 | 12.2 | 3.1 | 66.1 |
| 9       | Rye-wheat simple panloaf, peace I | 19.5 | 12.8 | 3.3 | 65.9 |
| 10      | Rye-wheat simple panloaf, peace II | 17.0 | 11.0 | 2.7 | 64.5 |
| 11      | Rye-wheat simple panloaf, peace III | 19.2 | 12.8 | 2.7 | 66.6 |
| 12      | Rye-wheat scalded panloaf, peace | 20.1 | 11.8 | 3.0 | 58.5 |

Table 1

Comparative characteristics of wort samples from various returnable baking waste types
The transition of solids percentage in the soluble state does not depend on the type of returnable waste and is in the range of 87.6–90.7%.

The starch transition into a soluble state is determined by the processed raw materials type. The use of samples from wheat flour with the accepted regime parameters of raw material processing is characterized by a high value of this indicator (97.9–99.5%); from a mixture of rye and wheat flour — lower values (85.0–89.4%).

In general, summarizing the obtained data, it can be noted that the use of returnable baking waste makes it possible to obtain saccharified wort with a high dry matter content, including total reducing substances.

The influence of the returnable waste type on the transition of solids and starch into a soluble state

| Sample  | Solids Starch |
|---------|--------------|
| 1       | 4.1 3.1      |
| 2       | 4.2 3.0      |
| 3       | 4.2 3.5      |
| 4       | 4.3 3.2      |
| 5       | 4.2 3.2      |
| 6       | 4.2 3.1      |
| 7       | 4.3 2.9      |
| 8       | 4.2 2.3      |
| 9       | 4.2 2.5      |
| 10      | 4.3 2.1      |
| 11      | 4.2 2.2      |
| 12      | 4.2 2.1      |

The influence of the MA carrying norm when receiving wort from rye-wheat bread on the transition of starch to a soluble state

| Norm of MA, units/g of raw material | Sample 11 | Sample 12 |
|------------------------------------|-----------|-----------|
| 0                                  | 87.1      | 88.5      |
| 0.1                                | 88.5      | 85.7      |
| 0.25                               | 90.0      | 87.4      |
| 0.5                                | 91.2      | 89.0      |
| 1                                  | 98.7      | 90.5      |

At the same time, it was found that when using samples obtained from rye-wheat bread, the wort is characterized by a lower value of purity and the starch transition to a soluble state, which necessitates the adjustment of the regime parameters of its production. To increase the content of reducing substances and the wort purity index, a variant of using a mixture of wheat and rye-wheat bread as a raw material for distillation can be considered. At the next research stage, saccharification processes using enzyme preparations of cytolytic action were studied. It was found that obtaining wort from rye-wheat bread with the adopted hydromodule 1:3.5 with the additional use of microbial cytolases (enzyme preparation Cellulase Cl. 14) even at an increased dosage (0.1–0.2 units of the main activity (MA) of raw materials, recommended by the manufacturer) does not allow to completely starch transition into a soluble state (Figure 1).
It was established that the hydromodule affects the saccharification process to a greater extent than the dosage of the enzyme preparation (Figure 2).

The hydromodule increase to 1:7.0 ÷ 1:10.0 made it possible to almost completely transit starch of rye-wheat bread to a soluble state. However, such a strong dilution of the technological environment is not economically justified, as it will significantly reduce the total wort concentration and the content of total reducing carbohydrates, as a result, the fermented wort strength.

In connection with the identified factor, it is proposed to use rye-wheat bread as a mixture with wheat bread in a ratio of 1:1 to 1:2, after preliminary processing of the first (hydromodule from 1:7.0 to 1:10.0, the introduction of enzyme diluent and cytolytic drugs, holding the mixture at a temperature of 50–55 °C for 30 minutes). At the same time, wheat bread was introduced into the mixture until the total hydromodule reached 1:3.5. It was established that such a new technical solution allows almost completely transit raw materials starch to a soluble state (Table 4).

| Name                  | Control | Test 1 | Test 2 |
|-----------------------|---------|--------|--------|
| Raw material type     |         |        |        |
| Rye-wheat bread       |         |        |        |
| Mixture 1:1           | 88.5    | 98.1   | 99.3   |
| Mixture 1:2           |         |        |        |
| Strength, % vol.      | 8.11    | 7.74   | 8.15   | 8.07   | 7.63 |
| Valid extract, % wt.  | 5.5     | 6.0    | 5.9    | 6.2    | 7.0  |
| The yield of alcohol, dal/t of conventional starch of raw materials | 65.25 | 60.36 | 63.56 | 62.94 | 59.51 |

The use of the hydromodule 1:7.0 ÷ 1:10.0 instead of the hydromodule 1:3.5 at the first stage of rye-wheat bread preparation and the carrying of the entire estimated amount of enzyme preparations of a diluting and hemicellulase action contributes, firstly, to improving the rheological characteristics of the mix for due to a deeper hydrolysis of the hemicellulose raw materials, secondly, an increase in the enzymatic starch attackability due to the destruction of its complexes with hemicelluloses and proteins, thirdly, it increases the percentage of protein water-soluble fractions.

Fermentation is one of the main stages in the distillates production, during which, under the action of the yeast enzyme complex, the initial formation of product quality indicators occurs. In this regard, special attention should be paid to the selection of the yeast race, which provides high fermentation efficiency and the synthesis of valuable volatile components. The objective was to select the optimal yeast race for fermenting the wort from the returnable baking waste.

Five races of dry alcoholic yeast of foreign manufacture were tested: Fermiol (USA), Turbo-24 (Great Britain), Alcotec Whiskey Turbo (Great Britain), Parmaya Cristal (Turkey) and Angel (China). The yeast application rate was 100 mg/100 g of the wort. Saccharified wort from a mixture of wheat and rye-wheat bread in a ratio of 1:1 was used as a raw material. It was found that the most intensive fermentation process, characterized by the dynamics of carbon dioxide evolution, takes place using Fermiol and Turbo-24 races (Figure 3).

These races were also the most effective in the accumulation of ethyl alcohol, and, consequently, in the yield of alcohol from a unit of starch raw materials (Table 5).

At the final work stage, the qualitative and quantitative composition of volatile components in the samples of fermented wort was determined. Analysis of the obtained data showed that the use of yeast Fermiol and Turbo-24 allows to get fermented wort with a minimum concentration of acetaldehyde (Table 6). When using the yeast Parmaya Cristal and Angel, the content of this component, which adversely affects the distillates organoleptic characteristics, increases by 1.4–3.8 times.

It was also found that the higher alcohols content, which form the basis of the final product aroma, is determined by the race of used yeast.
The influence of yeast race on the composition of the fermented wort volatile components

| Name of volatile components | The volatile components content, mg/dm³ of A.A. |
|-----------------------------|-----------------------------------------------|
|                             | Fermiol | Turbo Vaast | Turbo-24 | Parmaya Cristal | Angel |
| Acetaldehyde                | 275     | 382         | 295      | 667            | 1054  |
| Ethyl acetate               | 112     | 101         | 150      | 177            | 107   |
| Methanol                    | 8       | 10          | 15       | 25             | 12    |
| Σ of higher alcohols,       | 4619    | 4315        | 5200     | 5575           | 3567  |
| including:                  |         |             |          |                |       |
| — 1-propanol                | 446     | 438         | 477      | 486            | 380   |
| — isobutanol                | 1244    | 1081        | 1311     | 1372           | 1060  |
| — isomylol                  | 2929    | 2796        | 3412     | 3717           | 2127  |
| Enanthic ethers             | 19      | 14          | 23       | 16             | 19    |
| Phenyethyl alcohol          | 334     | 456         | 400      | 390            | 702   |
| Σ of volatile components*  | 5417    | 5322        | 6081     | 6850           | 5497  |

* In the sum of volatile components, all identified substances were taken into account, some of them are not presented in this Table

The maximum concentration of higher alcohols was detected in samples of fermented wort obtained using Parmaya Cristal yeast, the minimum — Angel. When using the latter, the maximum accumulation of phenylethyl alcohol was also revealed. Phenylethyl alcohol is formed during the hydrolysis of the phenylalanine amino acid, which is present in saccharified raw materials, secondly, an increase in the enzymatic starch attackability due to the preliminary enzymatic hydrolysis of rye-wheat bread with a high hydromodule and the use of enzyme preparations of the cytolysis complex.

For the efficient process of saccharified wort from the returnable baking waste fermentation, the selection of the Fermiol and Turbo-24 yeast races is scientifically justified, allowing not only a high ethanol yield, but also an optimal volatile components composition.

4. Conclusion

Based on the results of studies, it is recommended to use a mixture consisting of wheat and rye-wheat bread as raw materials to obtain high-quality distillates from returnable baking waste. To obtain saccharified wort from this raw material type, a new technological method has been developed based on preliminary enzymatic hydrolysis of rye-wheat bread with a high hydromodule and the use of enzyme preparations of the cytolytic complex.

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