

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 dextrans

conversion 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 dextrins [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), “Rusky 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 not more than 8.0% was reached and then crushed manually until a sufficiently uniform groat size was obtained in accordance with the requirements of GOST 28402–89 [24].

To characterize the saccharified wort composition obtained using returnable baking waste, indicators were used, adopted for assessing the quality of wort from grain raw materials: solids mass concentration, reducing sugars mass fraction, total fermentable carbohydrates mass concentration [2]. Additionally, the individual sugars mass concentration was determined using high performance liquid chromatography on an Agilent Tech-

nologies 1200 Series device (Agilent, USA) using a standardized method [25].

When studying processes at the fermentation stage of saccharified wort, the dynamics of carbon dioxide evolution by the gravimetric method has been determined [26], the ethyl alcohol mass concentration and the actual extract content [27], the volatile components composition and content by gas chromatography on a Thermo Trace GC Ultra device (Thermo, USA) with a flame ionization detector. Chromatographic column — HP FFAP: length 50 m, internal diameter 0.32 mm with a film thickness of the stationary phase 0.5 μm. Sample volume — 1 mm³. [28]. When analyzing samples of fermented wort, they were previously freed from extractive components by distillation and the volatile components composition in the distillate was determined. In order to conduct a comparative analysis of the studied samples, the volatile components concentration was expressed in 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 1:3.5, introducing a diluting enzyme preparation with mesophilic alpha-amylase with a dosage of 0.5 units of AA/g (amilase 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 17.0–21.3%. 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

Table 1

Comparative characteristics of wort samples from various returnable baking waste types

Samples	The feedstock composition	The concentration in wort, %			Purity, %
		Solids	Total fermentable carbohydrates	Reducing sugars	
1	Hearth 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	75.7
6	Sliced long loaf III	19.8	14.8	2.7	74.6
7	Hearth wheat-rye simple bread, piece	19.8	14.3	2.6	72.3
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

sample 10 (11.0%), characterized by a reduced starch content (47.7%). A clear dependence on the content of reducing sugars, depending on the type of returnable waste, has not been identified. Purity, by contrast, depends on the composition of the raw materials used to make bakery products. Processing of rye-wheat bread is characterized by a decrease in the quality indicator against samples obtained from wheat flour, i. e. as soluble components, the former contains an increased content of non-fermentable carbohydrates.

It was found that samples 1–6, obtained from wheat bread and sliced long loafs, contained more glucose and maltose, compared with samples that used rye flour (Table 2). The noted difference can be related both to the increased starch content in the samples from wheat flour, and to its greater tolerance to enzymatic hydrolysis.

Table 2

The influence of returnable waste type on the individual sugars content

Sample	Mass concentration, %				
	Fructose	Glucose	Sucrose	Maltose	Maltotriosis
1	0.38	2.22	0.13	1.11	1.76
2	0.45	2.31	0.09	1.33	1.61
3	0.54	2.97	0.12	1.15	1.36
4	0.73	2.97	0.11	1.25	1.32
5	0.65	2.98	0.12	1.31	1.41
6	0.44	2.42	—	1.12	1.46
7	0.20	1.43	—	0.95	1.45
8	0.40	1.97	—	0.68	1.18
9	0.41	1.79	0.11	1.01	1.53
10	0.38	1.57	—	0.58	1.25
11	0.39	1.45	—	0.69	1.46
12	0.47	1.68	0.20	0.80	1.34

In addition, the proportion of solids and starch transition into the liquid phase, i. e. in a soluble state. The liquid phase was an extract obtained by adding excess water to the wort (dilution 5 times) with its further filtration. The obtained result made it possible not to take into account the structure of bread, which is characterized by porosity and capillarity in comparison with traditional starch-containing raw materials (grain), and to establish the maximum transition of solids and starch into a soluble state.

The data presented in Table 3, led to the following conclusions:
 □ 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.

Table 3

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

Sample	Concentration in the extract, %		Transition to a soluble state, % of the feedstock	
	Solids	Total reducing substances	Solids	Starch
1	4.1	3.1	87.6	99.2
2	4.2	3.0	88.6	98.1
3	4.2	3.3	88.1	99.5
4	4.3	3.2	90.7	98.1
5	4.2	3.2	88.9	97.9
6	4.2	3.1	88.4	98.6
7	4.3	2.9	90.0	96.3
8	4.2	2.3	88.9	88.1
9	4.2	2.5	88.5	89.4
10	4.3	2.1	89.9	88.0
11	4.2	2.2	88.6	87.1
12	4.2	2.1	89.1	85.0

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 cy-tases (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).

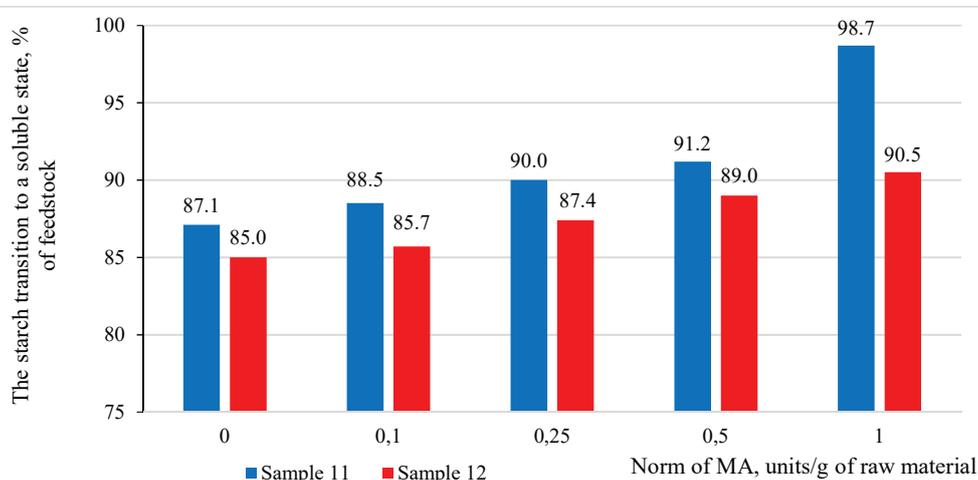


Figure 1. The influence of the MA carrying norm when receiving wort from rye-wheat bread on the transition of starch to a soluble state

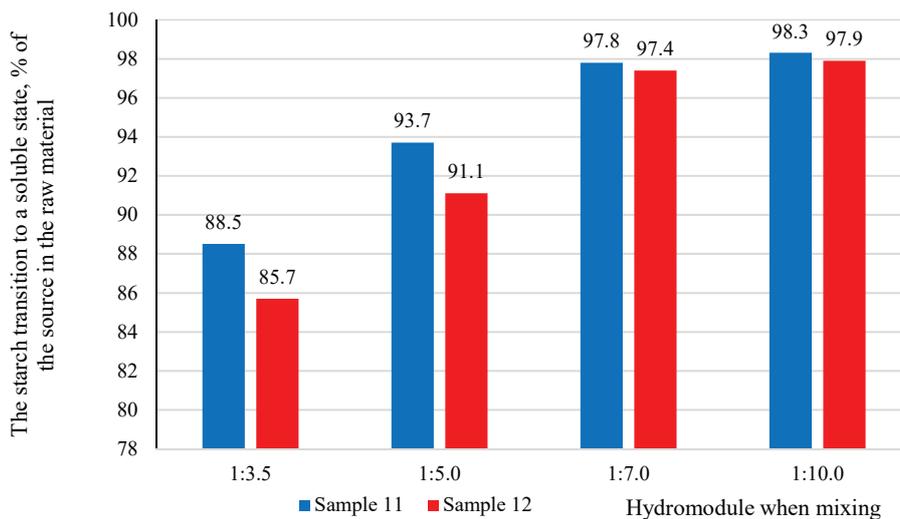


Figure 2. The hydromodule influence upon receipt of the wort from rye-wheat bread on the starch transition a soluble state

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).

Table 4

The influence of regime parameters for the wort from the returnable baking waste production on the starch transition to a soluble state

Name	Control	Test 1	Test 2
<i>Raw material type</i>	<i>Rye-wheat bread</i>	<i>Mixture 1:1</i>	<i>Mixture 1:2</i>
Hydromodule on mixing stage	1:3.5	1:7.0	1:10.0
Total Hydromodule	1:3.5	1:3.5	1:3.5
The starch transition to a soluble state, % of the source in the raw material	88.5	98.1	99.3

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).

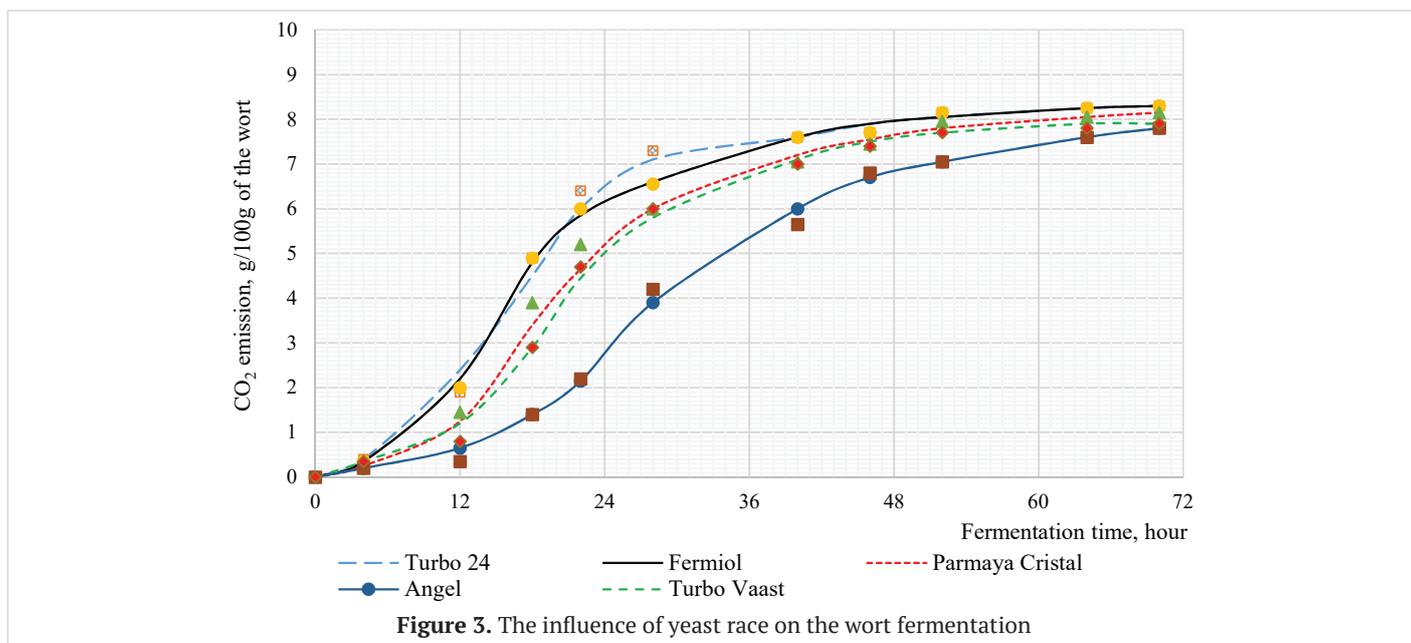
Table 5

The influence of yeast race on the quality indicators of fermented wort and alcohol yield

Indicators	Yeast race				
	Fermiol	Turbo Vaast	Turbo-24	Parmaya Cristal	Angel
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	63.25	60.36	63.56	62.94	59.51

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

Table 6

Name of volatile components	The volatile components content, mg/dm ³ of A.A.				
	Fermiol	Turbo Vaast	Turbo-24	Parmaya Cristal	Angel
Acetaldehyde	275	382	295	667	1034
Ethyl acetate	112	101	150	177	107
Methanol	8	10	13	25	12
Σ of higher alcohols, including:	4619	4315	5200	5575	3567
– 1-propanol	446	438	477	486	380
– isobutanol	1244	1081	1311	1372	1060
– isoamylol	2929	2796	3412	3717	2127
Enanthic ethers	19	14	23	16	19
Phenylethyl 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

wort from recyclable baking waste in a much higher concentration than in wort from grain raw materials. Usually, during distillation, phenylethyl alcohol is isolated with a tail fraction and remains in the distillers' spent grains, but only a small part of it transit directly to the distillate. The highest concentration of ethers, including enanthic ethers, which are the most valuable aroma-forming components, is noted in the sample obtained using the Turbo-24 race.

In general, the analysis of the obtained data gave reason to recommend the Fermiol and Turbo No. 24 races for fermentation of wort from returnable baking waste products, the use of which allows one to obtain fermented wort with maximum strength and high rates of valuable volatile components.

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 cyto-lytic complex.

For the efficient process of saccharified wort from the return-able 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.

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