Comparative analysis of extraction methods in distilled drinks production

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Abstract. The paper presents the results of studying the extraction process in the technology of distilled drinks production at the stage of their aging with the use of oak chips, which reduces the duration of the technological process and reduces technological losses. At the engineering center “FOOD ENGINEERING” of Kemerovo State University, two extraction methods were selected for studying: 1) using a batch extractor for the “solid - liquid” system with exposure to oxygen, and 2) extraction based on the effects of microwave radiation. A comparative analysis of these processing methods has shown that the method of exposure to oxygen is more effective, taking into account the increase in optical density. The degree of oak chip charring was found to affect the color of the product significantly. Microwave radiation contributes to a more intensive accumulation of polyphenols, especially while using the chips with strong charring. When exposed to oxygen, the degree of charring had little influence on the content of polyphenols; still, exposure to oxygen intensified the process of phenolic compound extraction. The content of extractive solid compounds is higher in the samples exposed to oxygen and microwave radiation compared to the control sample, with little difference between different varieties of oak chips. In this regard, it has been planned to conduct further studies of the joint effect of these intensification methods on the extraction process.

1. Introduction

In the preparation of distilled drinks different countries have different historically established technological and tasting traditions. For example, the classic technology of whiskey production includes several stages: preparation and drying of malt, wort production, fermentation, distillation, aging in burnt oak barrels, blending, and bottling of finished products [1].

The most important stage in the production of distilled drinks is the aging of alcohols. It significantly affects the final organoleptic properties of the product, because in the process of aging in an oak barrel, phenolic compounds are extracted into alcohol, and complex processes of interaction between them and the initial compounds of raw alcohol occur. Thus, as the beverage ages, its taste and aroma improve. In the classical production technology this technological stage requires a long time period (at least three years), as well as the use of expensive oak barrels.

This explains the practicability of using the intensive technology to reduce the period of distillate aging with wood [2]. Currently, heat-treated oak chips are used in the production of such beverages. Oak chips have proven to be an affordable raw material component, and have a number of advantages over oak barrels [1-3]. The use of oak chips reduces the duration of the process as well as technological losses. At the same time, studies related to the formation of consumer properties and the choice of optimal technological methods for extracting and intensifying this process are relevant.
2. Problem statement

The purpose of the study was to determine the main indicators of the extract quality by means of comparative analysis of the extraction process with the use of different methods to create new effective ways for intensifying the process of extracting the target compounds from oak chips into a water-alcohol solution.

The study allowed solving the following research tasks:

1. Investigating the process of target compound extraction from oak chips with water-alcohol solution under exposure to oxygen.
2. Investigating the process of target compound extraction from oak chips with water-alcohol solution under exposure to microwave radiation.
3. Determining the main indicators of extract quality by comparative analysis of the extraction process with the use of different methods.

3. Materials and methods

All the studies were conducted at the «FOOD ENGINEERING» center of the Institute of Engineering Technologies of Kemerovo State University.

A model water-alcohol solution was used as an extractant for the comparative analysis of the intensification of target compound extraction from the solid phase by the alcohol-containing liquid phase. The solution consisted of premium-class rectified grain ethyl alcohol, meeting the standards of GOST 5962-2013, and distilled water, meeting the standards of GOST 6709-72. Heat-treated French oak chips of «Sweet Vanilla» brand (medium charring) and «Sweet Coffee» brand (strong charring) were selected as the solid phase. The chips had the same size of about 15×10×3 mm and did not contain bark particles (Figure 1).

![Figure 1. Heat-treated oak chips: (a) - «Sweet Vanilla», (b) - «Sweet Coffee»](image)

Two extraction methods were chosen: the method using a batch extractor for the solid-liquid system by exposure to oxygen, and the method based on exposure to microwave radiation (Figure 2).

![Figure 2. Extraction methods: (a) exposure to oxygen: 1- extractor, 2- oxygen concentrator ARMED 7F-3A, 3- photoelectric chromatometer; (b) - exposure to microwave radiation: 1- microwave oven TATUNG TMO 6610, 2- photoelectric chromatometer](image)
Samples of water-alcohol solutions were prepared in the «FOOD ENGINEERING» center. The alcohol concentration in the samples was 50% according to the classic whiskey preparation technology (GOST 33281). The ratio of oak chips was 1 g per 500 ml of the solution. Samples were stored and matured in glass tanks with tightly closed lids.

To conduct the comparative analysis and determine the dependence of the extraction process on the solid phase, the samples were divided into two groups: 1) the water-alcohol solution with the medium-charred «Sweet Vanilla» chips, and 2) the water-alcohol solution with the strong-charred «Sweet Vanilla» chips.

The control sample was aged in the classical way, while the model samples were subjected to intensification of the extraction process using two different methods. The first method consisted in applying oxygen to the water-alcohol solution with oak chips in the batch extractor for the solid-liquid system; the extractor was designed and developed by the team of authors at Kemerovo State University [4]. The second method consisted in subjecting the water-alcohol solution with chips to ultra-high-frequency radiation.

During the research, measuring methods were used to evaluate the extract samples. The samples were analyzed by optical density using spectrophotometry, the content of polyphenol compounds was measured using the Erumanis method, and the content of solid compounds was measured by drying [4-6].

4. Results and Discussion

Study of the effect of oxygen on water-alcohol solution with oak chips. Three prepared samples for each type of chips were exposed to oxygen in a batch extractor for different periods of time. During one year, the samples were saturated with oxygen every 48 hours: Sample 1 was exposed to oxygen for 5 minutes, Sample 2 – for 10 minutes, and Sample 3 – for 15 minutes. The control sample was not exposed to oxygen and was aged using the classical method [7].

The quality indicators of the samples were measured every 28 days.

Study of the effect of microwave radiation on water-alcohol solution with oak chips. Three prepared samples for each type of chips were exposed to ultra-high-frequency radiation in a microwave oven for different periods of time. During one year, the samples were exposed to microwaves for 1, 2, and 3 minutes, respectively, every 48 hours. The control sample was not exposed to microwaves and was aged using the classical method.

One of the important consumer properties of spirits is the colour, which is formed during aging and depends on the type of oak, barrel charring, and type of raw material; it varies from light yellow to brown. In these studies, the colour of the samples was determined visually and by optical density.

![Figure 3](image-url). Changes in the optical density of the samples with oak chips: (a) – exposed to oxygen, (b) – exposed to microwave radiation.
The comparative analysis of the changes in the optical density of the samples with oak chips «Sweet Vanilla» brand (medium charring) and «Sweet coffee» brand (strong charring) subjected to intensification of the extraction process in two different ways by various methods of influence is given in Figure 3.

Longer time of exposure to oxygen of Samples 1, 2, and 3 correlates with the higher level of their optical density compared to the control sample. The maximum value is reached in Sample 3 which was exposed to oxygen for 15 minutes (Figure3). As for «Sweet Vanilla» chips, the value of the optical density indicator in Sample 3 is 0.05 compared to 0.023 in the control sample, exceeding the latter by 60%. As for «Sweet Coffee» chips, the value of optical density indicator in Sample 3 exceeds that in the control sample by 48% [6]. This regularity is explained by the design of the developed extractor: rising from the bottom up, oxygen bubbles stir the oak chips, randomly mixing them in the entire volume of the extractant, which leads to a larger contact area of the chip surface with the liquid alcohol-containing phase and intensifies the process of the target compounds extraction [4].

Visual evaluation of the colour showed that the samples with the «Sweet Coffee» chips have a richer honey-amber colour compared to the light-straw colour of the samples with the «Sweet Vanilla» chips (Figure 4).

![Figure 4. Changes in the colour of the samples with oak chips: (a) – «Sweet Vanilla», (b) – «Sweet Coffee»](image)

Changes in the optical density of the samples exposed to microwave radiation show that the optical density increases proportionally with the increasing exposure time by an average of 20%. Comparative analysis of the optical density of samples with different chips confirmed the fact that the color is more affected by the degree of chip charring. The maximum values of the optical density of «Sweet Coffee» samples (strong charring) are 0.021 units higher than those of «Sweet Vanilla» samples (medium charring) [7].

Thus, taking into account the increase in optical density, the method of exposure to oxygen proved more effective.

Since oak chips contain a certain amount of phenolic antioxidants, we determined the content of polyphenols, which also have a significant impact on the formation of consumer properties of the finished drink. For example, flavones (belonging to flavonoids) are responsible for bitter taste, tannin is responsible for astringent properties, and anthocyanins provide color [8].

The comparative analysis of the content of polyphenols in samples with different types of oak chips after saturation with oxygen and exposure to microwaves is provided in Figure5.

The content of polyphenols in the control sample of the «Sweet Vanilla» brand was 121.8 mg/dm³. When treated with oxygen for 15 minutes (Sample 3), the number of polyphenols increased to 220.89 mg/dm³. As for «Sweet Coffee» chips, the values were 144.6 mg/dm³ and 234 mg/dm³ respectively. The exposure to microwaves led to an almost 2-time increase in the content of polyphenols amounting to 248.4 mg/dm³ («Sweet Vanilla») and 310 mg/dm³ («Sweet Coffee»).

It should be noted that microwave radiation contributed to a more intensive release of polyphenols from oak chips, especially in the strong charring variety («Sweet Coffee»). When exposed to oxygen,
the degree of charring had little influence on the content of polyphenols in samples; still, exposure to oxygen intensified the process of phenolic compound extraction.

Figure 5. The content of polyphenols in the samples with oak chips: (a) – exposed to oxygen, (b) – exposed to microwave radiation

The content of solid compounds in the samples was measured after one-year aging. The results are provided in Figure 6.

Figure 6. The content of solid compounds in the samples with oak chips: (a) – exposed to oxygen, (b) – exposed to microwave radiation

A direct ratio of the solid compounds content to the extraction time indicates a positive effect of exposure to oxygen and microwave radiation. At the same time, there are no significant differences related to the degree of oak chip charring.

5. Conclusion
A comparative analysis of the different methods of the extraction process intensification has shown that the method of exposure to oxygen is more effective, taking into account the increase in optical density. The degree of oak chip charring was found to affect the color of the product significantly: the stronger the charring of chips, the faster the colour is saturated. Microwave radiation contributes to a more intensive accumulation of polyphenols, especially while using the chips with strong charring
(«Sweet Coffee»). When exposed to oxygen, the degree of charring had little influence on the content of polyphenols in samples; still, exposure to oxygen intensified the process of phenolic compound extraction. The content of extractive solid compounds increased in the samples exposed to oxygen and microwave radiation compared to the control sample, with little difference between the studied chip brands. In this regard, it has been planned to conduct further studies of the joint effect of these intensification methods on the extraction process.

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