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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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GAS CHROMATOGRAPHIC ANALYSIS OF THE CHEMICAL COMPOSITION OF ETHYL ALCOHOL OBTAINED FROM PLANTS OF THE GENUS ASTERACEAE

Abstract. In recent years, serious attention has been paid around the world to the chemical and biotechnological processing of biomass of easily renewable plant raw materials. One of the most mass-produced aliphatic alcohols is ethyl alcohol.

The aim of this work was gas chromatographic analysis of the chemical composition of ethyl alcohol obtained from plants of the genus Asteraceae (dahlia and jerusalem artichoke tubers, chicory and big burdock roots) growing on the territory of Kazakhstan.

The article discusses the methods of obtaining and application of ethyl alcohol. The results of the analysis of the chemical composition of ethyl alcohol obtained from plants of the genus Asteraceae are presented. As a result of the study, alcohols were obtained in different concentrations. The chemical composition of ethyl alcohol was determined by gas chromatography. The impurity composition of ethyl alcohol obtained from various types of raw materials is also shown.

The composition of impurities in various samples of ethanol and alcohol-containing products is considered in detail. The results of the study were discussed, and the peculiarities of using the gas chromatography method were shown.

Key words: Ethyl alcohol, plants of the genus Asteraceae, gas chromatographic method, chromatogram, chemical composition.

Introduction. Ethyl alcohol is one of the most important raw materials in the modern organic synthesis industry, light and food industry. In terms of production, ethyl alcohol is one of the first organic products. An important consumer of ethyl alcohol is the food industry, where ethyl alcohol is used for the production of alcoholic beverages. In the production of alcoholic food products, only ethyl alcohol obtained by fermentation of food raw materials-various cereals, potatoes, grapes, sugar cane, etc. can be used [1].

The quality of ethyl alcohol used for the manufacture of alcoholic beverages largely determines the quality of the final product. There is a possibility of substitution of food ethyl alcohol with cheaper synthetic alcohol in the manufacture of alcoholic products. The impurities in ethyl alcohol regulated by state standards do not provide complete information about the method of alcohol production [2].

Ethyl alcohol refers to substances that are characterized in as much detail as possible by a variety of analytical data, which is due to its wide application in the chemical and food industries [3].

The purity of ethyl alcohol is very important in the production of wine and vodka products. According to the impurity composition of ethyl alcohol, it is possible to determine the source of its origin. The limits of impurity detection reached in gas chromatography with a flame ionization detector are at the level of 0.1 mg and allow reliable monitoring of methanol, acetaldehyde and isopropanol in rectified alcohol [4].

Alcohol is a natural antiseptic, has a high rate of destruction of bacteria and is able to quickly evaporate. In addition, alcohol has a bactericidal effect against most gram-positive, gram-negative and tuberculosis bacteria, and also works well against some types of fungi and many viruses, including RS viruses, hepatitis virus [5].

The number of scientific papers devoted to the gas chromatographic identification of impurities is so large that it is advisable to limit the number of them to specifying only a few works of the authors.

The review [6] presents new experimental data on the chemical equilibrium in the propionic acid – ethyl alcohol – ethyl propionate – water system at 293.15 K and atmospheric pressure. Gas chromatographic analysis has been used to determine the compositions of chemically equilibrium phases corresponding to the liquid-liquid phase equilibrium. Using the 1H- NMR method, homogeneous chemical equilibrium compositions were determined, based on which the concentration equilibrium constant was calculated. It is experimentally established that the region of these compositions decreases with increasing temperature, while the chemical equilibrium surface does not change its shape and position in the concentration space in the temperature range 293.15–313.15K and atmospheric pressure. Compositions corresponding to the liquid-liquid phase equilibrium were also obtained by gas chromatographic analysis for the pseudotroic system ethyl alcohol and ethylpropionate with the participation of deep eutectic solvents (DES) based on choline chloride and glycerol/urea in the entire concentration range.

The scientific work "Scientific support of control of biotechnological processes of ethyl alcohol production" presents the results of testing in production conditions, developed by the Institute of modern instrumental Express analysis methods, which are necessary for the functioning of a comprehensive control system for all stages of technological processes of yeast generation and alcoholic fermentation. The system implements an integrated approach to the control of alcohol production processes and provides not only control of finished products, but also control of products and intermediates formed in the production process, based on the use of innovative electrophoretic, gas chromatographic, chromato-mass spectrometric methods in a comprehensive control system [7].

Gas chromatographic determination of impurities in organic solvents, which is most often performed when evaluating the purity of the latter and analyzing the organic extract after concentration, is complicated by the presence of a wide solvent peak on the chromatogram, which may overlap the peaks of the detected impurities (analytes). At the same time, the lower the concentration of the analyte, the more difficult it is to determine[8,9].It is particularly difficult to determine analytes that are held in the gas chromatographic column stronger than the main component, since the peak of the latter, as a rule, has an elongated tail. One of the solutions to this problem when determining impurities in ethanol can be a modification of the sorbent in the packing column with cobalt (II) chloride, which selectively retains high-polar oxygen-containing compounds, including low-molecular-weight aliphatic alcohols [10,11].

Gas chromatography is the most popular method for separating mixtures of various substances that evaporate without decomposition. In this case, the components of the separated mixture move along the chromatographic column with the flow of the carrier gas.As it moves, the separated mixture is repeatedly distributed between the carrier gas (mobile phase) and the non-volatile stationary liquid phase deposited on the inert material (solid carrier) that fills the column [12]. The components of the mixture are selectively retained by the latter, since their solubility in this phase is different, and thus they are separated (components with a higher solubility take longer to exit the liquid phase than components with a lower solubility).The substances then exit the column and are detected by the detector. The detector signal is recorded as a chromatogram and automatically recorded by the computer. The advantages of this method are: high sensitivity, stability of the experimental conditions, short duration of the experiment, measurement accuracy, and the absence of special requirements for the preparation of the test sample [13].

Experimental part. Dahlia and Jerusalem artichoke tubers, chicory and big burdock roots collected in 2018-2019 were used as raw materials.

There are two ways to produce alcohol: biochemical and chemical or synthetic. The biochemical method is fermentation with sugar, the synthetic method is the interaction of ethylene with water in the presence of a catalyst.

We used a biochemical method to produce ethyl alcohol.

The technology for producing ethyl alcohol includes the following stages: 1) boiling the grain with water 2) cooling of the boiled mass and saccharification of starch with enzymes 3) the fermentation of sugars by yeast in biosport 4) distilling alcohol and its rectification [14].

0.5 kg of Raw materials are washed and crushed.Besides 1:2, 1:3, 1:4 water is poured, then (Ty BY 100104781.010-2005 produced in Belarus, dried alcoholic yeast wort Saccharomyces cerevistal) is filled with pre-prepared yeast, mixed and put on for 2-4 weeks at room temperature 20-22^{0}C. After two or four weeks, they filtered it out. After weighing the amount of wort, distill. The resulting wort was poured into a heat-resistant flask, and an electric stove was used to heat it. The temperature of the first distilled alcohol is 89^{0}C. Because the concentration of the first distilled alcohol is low. To increase the concentration, alcohol was re-distilled. Distilled alcohol contains impurities. To determine their quantity and purify them from alcohol-containing impurities, it is necessary to adsorb and distill them with activated carbon, zeolite, and calcium oxide. The density of the resulting wort, refractive index, and boiling point were determined [15,16] .

Gas chromatographic analysis was performed using an «Agilent 5973N» gas chromatograph.

The figures and tables below show the overall results of the study.

Results and Discussion

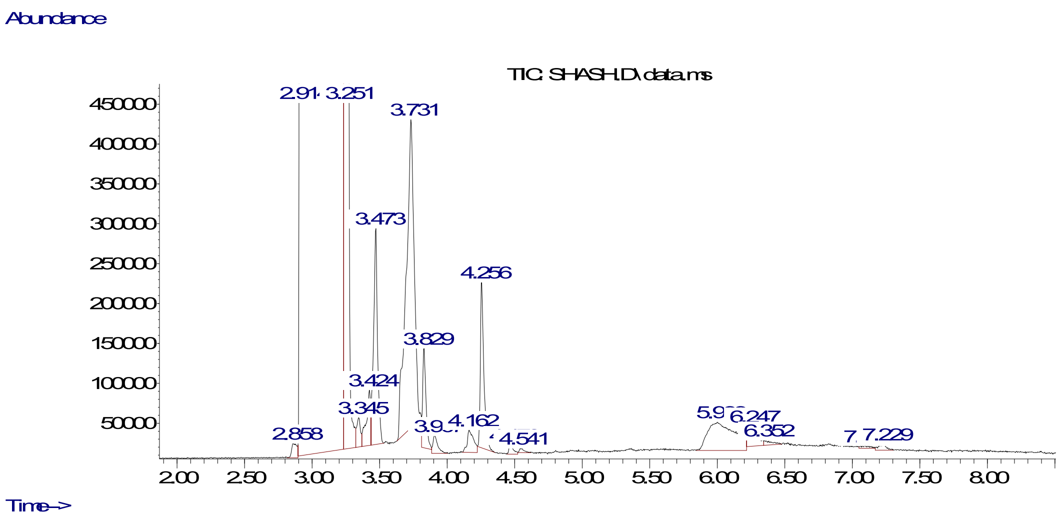

Figure 1 - Chromatogram of 87% ethyl alcohol obtained from chicory roots

In the composition of ethyl alcohol obtained from chicory roots, 26 compounds were determined. They are combined four at 13 points of the spectrum: 1(2.914) -ethanol; 2(3.345)-1-propanol; 3(3.473)-acetic acid; 4(3.731)-2-butanone, 3-hydroxy-; 5 (3.829)-1-butanol, 3-methyl-; 6(4.256)- isopropyl alcohol; 7(5.999)- glycerin; 8(9.024)- N-Isopropyl-3-phenyl propane amide.

Table 1 - Chemical composition of 87% ethyl alcohol obtained from chicory roots

Name of components	Retention times,tR min	Content,%
Ethanol	2.914	95.05
1-Propanol	3.345	0.03
Acetic acid	3.473	0.84
2-Butanone 3-hydroxy-	3.731	2.17
1-Butanol, 3-methyl-	3.829	0.28
Isopropyl alcohol	4.256	0.64
Glycerin	5.999	0.43
N-Isopropyl-3-phenylpropanamide	9.024	0.03

The obtained results, which are presented in table 1, clearly show that the composition of the studied object from the mixture contains in greater quantities: carboxylic acid, aliphatic and amyl alcohols, esters, aldehydes. The content of impurities is 4.42%, and ethyl alcohol is 95.05%.

The content of carboxylic acids, aliphatic and amyl alcohols decreases, but the content of esters and aldehydes increases slightly [17].

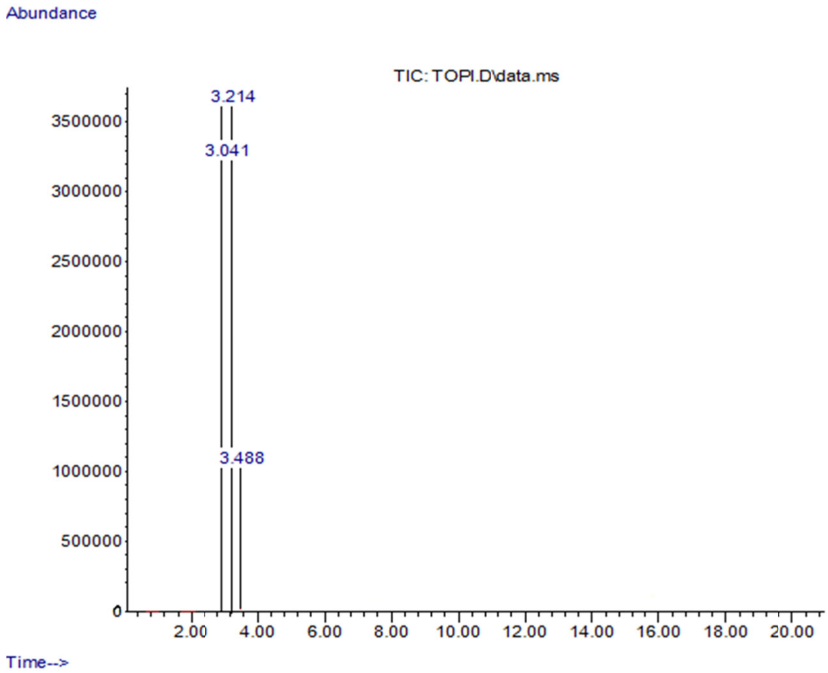

Figure 2 - Chromatogram of 90% ethyl alcohol obtained from Jerusalem artichoke tubers

90% ethyl alcohol was obtained from jerusalem artichoke tubers. They are located one at a time at 3 vertices of the spectrum. 1 (3.041) - ethanol; 2(3.214) - ethanol; 3 (3.488) - ethanol.

Table 2 - Chemical composition of 90% ethyl alcohol obtained from Jerusalem artichoke tubers [18]

Name of components	Retention times,tR min	Content, %
Ethanol	3.041	59.32
Ethanol	3.214	35.53
Ethanol	3.488	5.15

Table 2 shows the retention time and the content of 90 % ethyl alcohol obtained from Jerusalem artichoke tubers. From the table, you can see if the retention time increases, the alcohol content decreases.

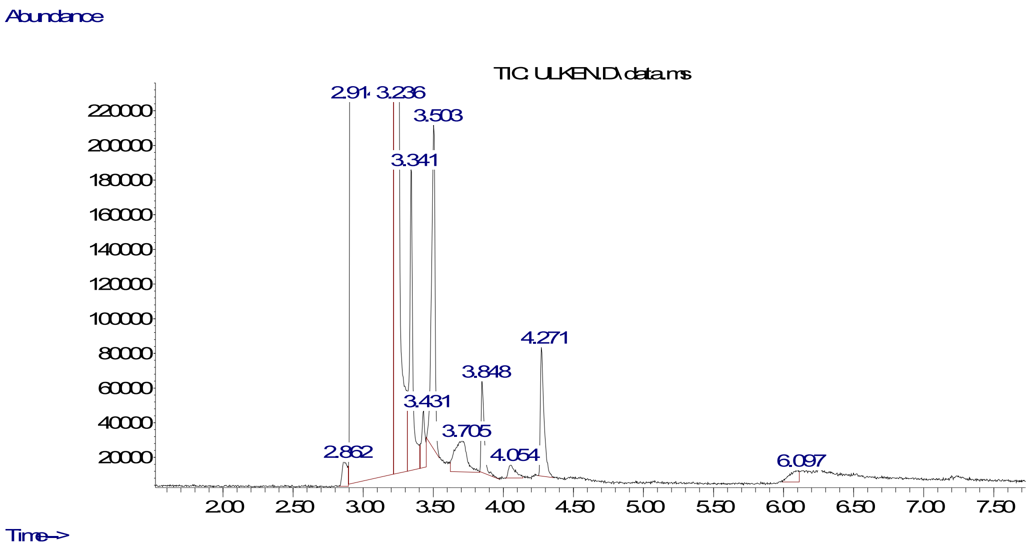

Figure 3 - Chromatogram of 85 % ethyl alcohol obtained from burdock root

57 compounds were identified in the composition of ethyl alcohol obtained from the burdock root. They are combined in four at the 14 vertices of the spectrum. Table 3 below shows the types of compounds that contain the largest number. In the composition of ethyl alcohol obtained from the root of burdock, 57 compounds were determined. They are combined by four in 14 vertices of the spectrum. 1(2.914)-ethanol; 2(3.038)-ethanol; 3(3.236)-ethanol; 4(3.341)-1-propanol; 5 (3.503)-acetic acid; 6(3.848)-1-butanol, 3-methyl-; 7(4.271)-isopropyl alcohol; 8(5.722)-benzaldehyde; 9(6.828)-acetaldehyde; 10(9.163)-acetic acid ; 11(9.845)-acetic acid.

Table 3 - Chemical composition of 85 % ethyl alcohol obtained from burdock root [19]

Name of components	Retention times,tR min	Content,%
Ethanol	2.914	24.84
Ethanol	3.038	18.68
Ethanol	3.236	54.65
1-Propanol	3.341	0.54
Acetic acid	3.503	0.82
1-Butanol, 3-methyl-	3.848	0.19
Isopropyl alcohol	4.271	0.20
Benzaldehyde	5.722	0.01
Acetaldehyde	6.828	0.04
Acetic acid	9.163	0.01
Acetic acid	9.845	0.02

From the table data, it can be seen that the composition of ethyl alcohol from the mixture contains more: carboxylic acid, amyl alcohol, aldehydes. The content of impurities is-1.83%, and ethyl alcohol-98.17%.

In the experimental sample, compared with the control, the content of isobutanol, isopropanol and isoamylene decreases, but the content of aldehyde and carboxylic acids increases slightly.

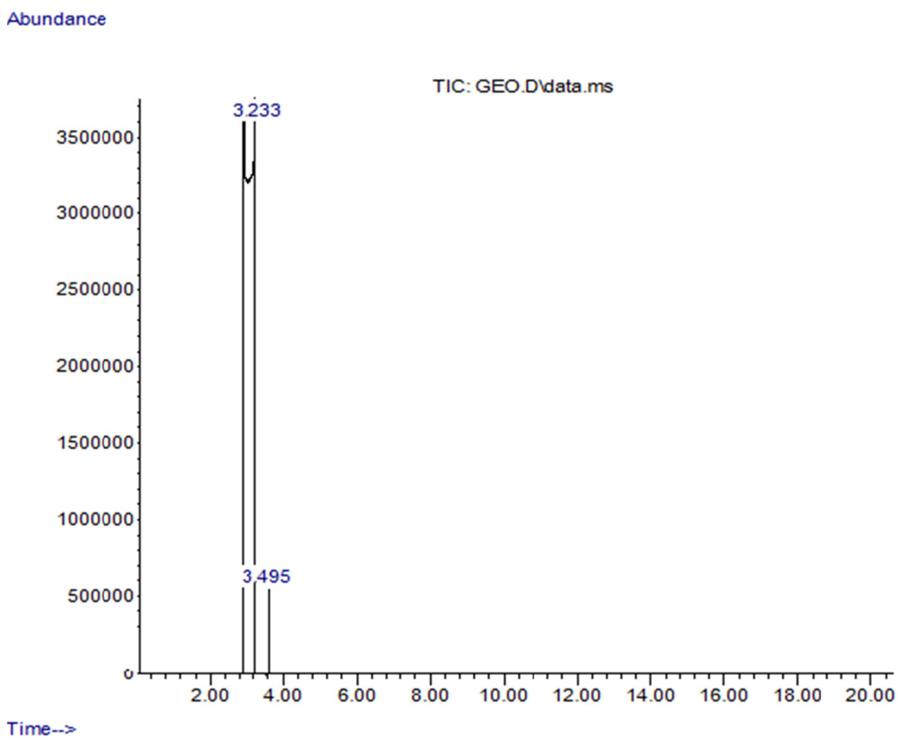

Figure 4 - Chromatogram of 87 % ethyl alcohol obtained from Dahlia tubers

In the composition of ethyl alcohol obtained from dahlia tubers, 2 compounds were determined. They are combined one at a time at 2 vertices of the spectrum. 1(3.495) - ethanol; 2 (3.233)- propanol-1.

Table 4 - Chemical composition of 87 % ethyl alcohol obtained from Dahlia tubers [20].

Name of components	Retention times,tR min	Content,%
Ethanol 1-Propanol	3.495 3.233	97.57 2.43

Table 4 shows the retention time in the knee and the content of 87 % ethyl alcohol obtained from Dahlia tubers.

Conclusions. The chemical composition of ethyl alcohol obtained from plants of the genus Asteraceae growing on the territory of Kazakhstan was determined by gas chromatographic method. As well as the impurity composition of ethyl alcohol obtained from various types of raw materials.

To obtain alcohol, raw materials (plants of the genus Asteraceae) were fermented. Then the volume and chemical composition were determined. To purify the alcohol, it was adsorbed with activated carbon, zeolite, and calcium oxide.

The impurities contained in the resulting alcohol were determined by titrimetric, photocolorimetric and gas chromatographic methods.

During gas chromatographic analysis, 87% ethyl alcohol and 52 compounds were determined from chicory root. From Jerusalem artichoke tuber 90% ethyl alcohol. From the root of big burdock 85% ethyl alcohol and 57 compounds. Of Dahlia tubers, 87% is ethyl alcohol. The results of the study are discussed, and the application of the gas chromatography method is shown.

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КҮРДЕЛІГҮЛДІЛЕР ТҰҚЫМДАСЫ ӨКІЛДЕРІНЕН АЛЫНҒАН ЭТИЛ СПИРТІНІҢ ХИМИЯЛЫҚ ҚҰРАМЫН ГАЗДЫ ХРОМАТОГРАФИЯ ӘДІСІМЕН ТАЛДАУ

Аннотация. Соңғы жылдары бүкіл әлемдеқалпына келетін өсімдік шикізатының биомассасын химия-лық және биотехнологиялық қайта өңдеу мәселелеріне үлкен көңіл бөлінуде. Ең көп өндірілген алифатты спирттердің бірі-этил спирті.

Бұл жұмыстың мақсаты Қазақстан аумағындағы Asteraceae тұқымдасына жататын өсімдіктерден алын-ған этил спиртінің химиялық құрамын газды хроматографиялық әдісін қолданыпталдау болды.

Бұл мақалада Қазақстан аймағында өсетінкүрделігүлділер тұқымдасы өкілдерінен алынған этил спир-тінің химиялық құрамын газды хроматография әдісімен анықтау нәтижелері келтірілген. Зерттеу нәтиже-сінде әр түрлі концентрациядағы этил спирті алынды. Этил спиртінің химиялық құрамы газды хроматогра-фиялық әдіспен анықталған. Күрделі гүлділер тұқымдасы өкілдерінен алынған этил спиртінің құрамындағы қоспалар мөлшері де кең көлемде қарастырылған. Зерттеу нәтижелері талқыланып, газды хроматография әдісінің қолдану ерекшеліктері көрсетілді.

Түйін сөздер: этил спирті, күрделі гүлділер тұқымдасы өкілдеріне жататын өсімдіктер, газды хромато-графия әдісі, хроматограмма, химиялық құрам.

Г.Е. Азимбаева[1], А.К. Камысбаева[1], А. А. Бакибаев[2]

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ГАЗОХРОМАТОГРАФИЧЕСКИЙ АНАЛИЗ ХИМИЧЕСКОГО СОСТАВА ЭТИЛОВОГО СПИРТА, ПОЛУЧЕННОГО ИЗ РАСТЕНИЙ РОДА ASTERACEAE

Аннотация. В последние годы во всем мире уделяется серьезное внимание вопросам химической и биотехнологической переработки биомассы легковозобновляемого растительного сырья. Одним из наиболее массово производимых алифатических спиртов является этиловый спирт.

Целью настоящей работы являлся газохроматографический анализхимического состава этилового спирта, полученного из растений рода Asteraceae (клубни георгина и топинамбура, корницикория и большого лопуха), произрастающих на территории Казахстана.

Встатье рассмотреныметоды полученияи области применения этилового спирта.Приведены результаты анализа химического состава этилового спирта, полученного из растений рода Asteraceae. В результате исследования полученыспирты в разных концентрациях. Химический состав этилового спирта определен газохроматографическим методом. Также показан примесный состав этилового спирта, полученного из различных видов сырья.

Детально рассмотрен состав примесей в различных образцах этанола и спиртосодержащих продуктов. Были обсуждены результаты исследования, показаны особенности применения метода газовой хроматографии.

Ключевые слова: этиловый спирт, растенияpода Asteraceae, газохроматографический метод, хроматограмма, химический состав.

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