Показано можливість отримання розчинних залізовмісних комплексів на основі полісахаридів печериці двоспорової (Agaricus bisporus). Максимальні виходи зразків з високим вмістом заліза можна отримати при масовому співвідношенні складових неорганічної та органічної природи 1:1,0 при pH=12,0 і 1:2,5 при pH=8,5. Методами ІЧ- і УФ-спектроскопії, гель-хроматографії підтверджено, що отримані продукти являють собою нанорозмірні комплекси поліциклічного гідроксиду тривалентного заліза і полісахаридів печериці

Ключові слова: комплекс, залізо, полісахарид, глюкан, печериця двоспорова, залізодефіцитні стани, анемія, імуномодулятор

Показана возможность получения растворимых железосодержащих комплексов на основе полисахаридов шампиньона двуспорового (Agaricus bis-porus). Максимальные выходы образцов с высоким содержанием железа можно получить при массовом соотношении составляющих неорганической и органической природы 1:1,0 при pH=12,0 и 1:2,5 при pH=8,5. Методами ИК- и УФ-спектроскопии, гель-хроматографии подтверждено, что полученные продукты представляют собой наноразмерные комплексы полициклического гидроксида трехвалентного железа и полисахаридов шампиньона

Ключевые слова: комплекс, железо, полисахарид, глюкан, шампиньон двуспоровый, железодефицитные состояния, анемия, иммуномодулятор

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PREPARATION AND CHARACTERIZATION OF IRON COMPLEXES BASED ON POLYSACCHARIDES FROM AGARICUS BISPORUS

N. Cherno
Doctor of technical sciences, professor, head of the department*
E-mail: onaft_foodchem@mail.ru
S. Ozolina
Candidate of chemical science, associate professor*
E-mail: ossofol@mail.ru
O. Nikitina
Junior scientific researcher
The problem research laboratory**
E-mail: ossofol@mail.ru
*Department of food chemistry**
Odessa National Academy of Food Technologies
Kanatnya str., 112, Odessa, Ukraine, 65039

1. Introduction

Minerals are necessary for implementing biochemical and physiological processes, maintaining the constancy of the internal environment, building structures of living tissues [1, 2]. Among them, iron is particularly important for the normal functioning of the human body. This essential microelement is part of proteins and heme enzymes, providing oxygen transport in the respiratory chain and tissue respiration. As a cofactor for nonhemin enzymes, iron is involved in energy metabolism, biosynthetic processes and the metabolism of many biologically active compounds. It promotes cell proliferation and differentiation and is also necessary for the functioning of the cellular, humoral immunity and nonspecific protection factors [3–5].

Imbalance of this microelement towards the predominance of its expenditure over supply leads to iron deficiency. There are three stages of iron deficiency. When prelatent iron deficiency, decline in microelement reserves in the body tissues without reducing the serum iron concentration is observed. Latent deficiency is characterized by the depletion of iron stores in depot while maintaining the hemoglobin concentration in the serum blood above the lower limit of normal. A decrease in hemoglobin concentration below the physiological values results in the third stage of iron deficiency – iron deficiency anemia. It is a circulatory system disease, which is accompanied by changes in iron metabolism parameters, quantitative and qualitative changes of red blood cells, clinical manifestations of anemic hypoxia, sideropenia and metabolic intoxication [3, 6].

Correction and prevention of iron deficiency are a global problem of mankind since, according to the WHO, latent iron deficiency is found in about 3.6 billion people around the world, and iron deficiency anemia is found in 1.8 billion [7, 8]. Because of the low absorption of iron, contained in food in the gastrointestinal tract, the solution of this problem using only dietotherapy is impracticable [9]. This necessitates the development of new effective drugs and dietary supplements with antianaemia properties that can restore a positive balance of iron in the human body.

2. Analysis of literature data and formulation of the problem

Depending on the method of administration to the body, iron preparations are classified as parenteral and oral [10]. The latter are the most often used in the iron deficiency treatment due to the proximity of their absorption to the physiological process of iron absorption, as well as the absence of side effects, inherent in parenteral drugs, simplicity and ease of use [11, 12]. In turn, oral preparations are divided into ionic and nonionic. They differ in structure and properties of the main active substance, and as a result, the metabolism in the conditions of the gastrointestinal tract [11].

The main component of ionic preparations is various ferrous salts. They are absorbed by means of passive diffusion. This mechanism works even after saturation of the transport system, so in the case of drug overdose or its application by healthy person there is a risk of intoxication because of free penetration of iron in the blood flow [11, 12]. Furthermore, in conditions of the gastrointestinal tract, the degree of its oxidation varies from +2 to +3. This process is accompanied by the activation of free radical reactions that damage cellular membranes [13, 14]. These drugs may also cause local irritation of the mucous membrane of the alimentary canal, which leads to dyspeptic disorders. To increase the portability, they are recommended to be taken during meals. However, this leads to a decrease in the iron bioavailability since a number of food ingredients inhibit its absorption [11, 12].

Nonionic preparations are complexes of soluble polysaccharides with ferric hydroxide. Although they differ in the carbohydrate component (pullulan, inulin, arabinogalactan), a mandatory requirement for them is the solubility, since only such complexes are active [15–17].

Dextran (polyisomaltosate) and dextrin (polymaltosate) are the most commonly used. Herewith, preference is given to the latter since the drug on its basis has a low allergenic potential [17]. This complex consists of polycyclic ferric hydroxide $(FeOOH)_n$, coated with non–covalently bound polymaltose molecules. Due to high molecular mass, it is not diffused through the intestinal mucosa membrane. It is believed that iron is absorbed by means of active transport, based on competitor ligand exchange among biopolymers, which determines its absorption rate. Therefore, when the body is saturated with iron, its absorption it terminated according to the principle of feedback, which eliminates toxicity [11, 18]. Furthermore, the complex does not possess prooxidant properties, does not cause dyspeptic disorders and does not interreact with the food components and drugs [12, 14, 19]. At almost the same efficiency of correction and prevention of iron deficiency states, this drug has significant advantages over ion due to the good tolerance and high safety profile [12, 18–20].

Duration of treatment by antianemic drugs ranges from 3 to 6 months [18, 20]. During this time, the immune system activity is reduced since iron deficiency causes the immune response dysfunction [4, 6]. In order to avoid the development of immunodeficient states, allergic and autoimmune diseases with the general anemic syndrome, for the prevention and correction of iron deficiency it is advisable to use drugs and dietary supplements that combine antianaemia and immunomodulatory properties. This can be accomplished using the carbohydrate component, exhibiting this activity. In this case, to reduce the risk of allergic reactions it is preferable to prepare complexes based on the non–microbial polysaccharides.

As such carbohydrates, soluble β–(1→3)/(1→6)–D–fungal glucans, which belong to the group of the most active immunomodulators can be considered. The source of these polysaccharides is cultured mushrooms, in particular, *Agaricus bisporus* [21, 22].

In the literature, there are no not only information about the physiological activity of soluble iron complexes based on polysaccharides of mushrooms, but also mentions of the possibility of preparing them. In this regard, preparation, characterization of their properties in comparison with the known carbohydrate-based iron complexes for further research in the *in vivo* conditions are relevant.

3. The purpose and objectives of research

The purpose of the work was the preparation and primary characterization of soluble iron complexes based on polysaccharides of *Agaricus bisporus*.

To achieve this goal, key objectives of the study were identified:

– to examine the impact of the preparation process parameters of iron complexes (ratio of components, concentrations of their solutions, pH of the reaction medium) on the yield and composition of the resulting product;

– to characterize the iron complex sample.

4. Methods of preparation and study of iron complexes based on polysaccharides of Agaricus bisporus

4. 1. Isolation of polysaccharides from Agaricus bisporus

Dried mushrooms were crushed, treated with 70 % alcohol at 60 °C for 40 min. The solid residue was separated by centrifugation and treated with 3 % boiling sodium hydroxide solution for 4 hours. The liquid phase was separated, acidified with HCl to pH=2, the precipitate formed was separated by centrifugation, the alcohol in a volume ratio 1:4 was added to the liquid phase, the precipitate was separated by centrifugation, dissolved in water and analyzed. The carbohydrate content of the solution was determined by the anthrone method [23], protein – by the Lowry method [24]. The sample was hydrolyzed with 2 % HCl solution for 4 hours, and then monosaccharide composition of the hydrolyzate was determined by gas-liquid chromatography on Hewlett–Packard 5890 A gas chromatograph [25].

4. 2. Preparation and chemical composition of iron complexes

Concentrated alkali solution in an amount, necessary to produce the predetermined level of the pH medium was added to aqueous solution of polysaccharides, heated to 90 °C, and then, when mixing, an equal volume of iron chloride (III) solution with concentration of 0.075 % was added. The process was carried out at this temperature for 10 min. To vary the mass ratio of iron: polysaccharides from 1:0.25 to 1:3.00, the polysaccharide solution concentration was changed from 0.019 to 0.230 %. For more concentrated solutions of complexes, 0.45 % polysaccharide solution and 0.30 % iron chloride (III) solution were used. The process temperature in all experiments was constant – 90 °C. The resulting solutions were separated from the precipitate by filtering and the content of carbohydrate was determined by the anthrone method, and iron – by the thiocyanate method [26].

Ferric hydroxide (III) was prepared in a similar way, using water instead of the polysaccharide solution. The precipitate was separated by centrifugation and dried.

4. 3. Characterization of the iron complex

The complex, as well as source polysaccharides, were characterized using gel chromatography on Sephadex

G–150 (h=38 cm; d=3,1 cm; Pharmacia, Sweden). The column was calibrated by markers – dextrans (Sigma) with known molecular masses, eluent – water. The content of carbohydrates and iron was determined in fractions [27].

IR spectra were recorded on FT–IR spectrometer FTIR–8400S in the range of 400...4000 cm^{-1}. UV spectra were recorded on spectrophotometer SP–102. Electron microscopy was performed on a transmission electron microscope TEM 100–01.

5. Results of studying the effect of the preparation conditions of iron complexes on their yield, composition and their discussion

The polysaccharide component, used to produce the complexes was a product, containing 88.5 % of carbohydrates and 3.8% of protein. Glucose (85.0 %), the presence of which was mainly caused by the cleavage of β–(1→3)/(1→6)–D–glucans prevailed in polysaccharide hydrolysate composition.

Most authors use mass ratio of iron:carbohydrates in the range from 1:30 to 1:19 at a pH of the reaction medium of 12.0–12.5 [15, 16, 28, 29] for the preparation of complexes. To study the possibility of increasing the iron content in the composition of the desired product, the complex formation process, reducing the share of the carbohydrate component was investigated. Thus, Fig. 1 shows that reducing the mass fraction of polysaccharides from 75.0 to 50.0 % of the reaction mixture composition does not influence the yield of water soluble target product. Its value is at the level of 99,1–96,0 %, i.e. starting components are almost completely included in the complex. The maximum iron content in the formulation reaches 47,9 %.

Decrease in the mass fraction of polysaccharides in the reaction mixture (ratio of iron:carbohydrates 1:0.75) is accompanied by a sharp drop in yield of target soluble complex with a slight decrease in the iron content in it. Further reduction in the amount of polysaccharides, added to the inorganic component leads to the fact that the liquid phase contains only 11,2–15,6 % of the complex. It is noteworthy that the amount of iron therein is very little and makes up 0,8–7,4 %. In these conditions, an insoluble product, in which metal accounts for more than three quarters is mainly formed.

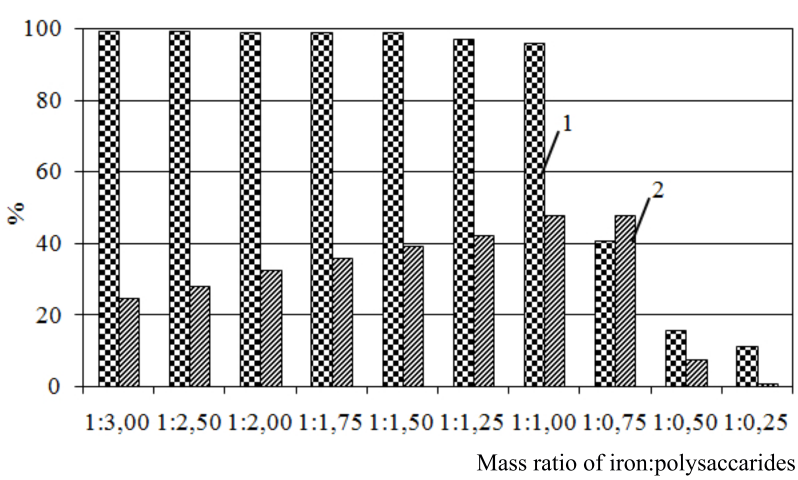

Fig. 1. Dependence of the yield of iron complexes and the iron content in them on the mass ratio of inorganic and organic components at pH = 12.0: 1 — yield of the complex; 2 — iron mass fraction in the complex

Thus, it can be assumed that at low polysaccharide content in the system, there is an interaction of inorganic and organic components in two directions. In one case, the iron compounds react with carbohydrates with the formation of soluble iron complex, characterized by a low metal content. At the same time, the process of interaction among the inorganic component and alkali soluble polysaccharides with the formation of an aggregatively unstable system that, as a consequence, leads to the occurrence of insoluble iron associates. Further, these compounds have not been investigated since insoluble forms of iron do not exhibit physiological activity [11].

The preparation process and composition of complexes are significantly affected by the pH of the reaction medium. When the pH drops from 11.5 to 8.5 there is a tendency of reducing the yield of the desired product (Fig. 2). Thus, with a minimum content of polysaccharides in the reaction mixture, it falls to 72.1 %. When approaching a neutral pH medium value, a sharp decrease in this indicator is observed, which is 9.8–75.7 %, depending on the component ratio. Herewith, there is a direct correlation between the mass fraction of the organic component in the reaction mixture and the yield of preparations. It is interesting to note that pH almost does not affect the system, containing the maximum number of polysaccharides.

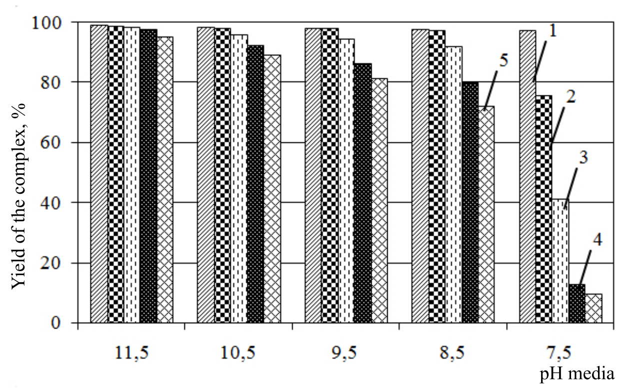

Fig. 2. Dependence of the iron complex yield on the pH medium and the mass ratio of inorganic and organic components: 1 — 1:3.0; 2 — 1:2.5; 3 — 1:2.0; 4 — 1:1.5; 5 — 1:1.0

Similar patterns are observed in changing the mass fraction of iron in samples (Fig. 3).

At a high mass fraction of the carbohydrate component (71.4–75.0 %) in the system in the pH range 8.5–11.5, this figure remains practically unchanged, while for samples with fewer polysaccharides in the reaction mixture there is a tendency to its decrease. Under conditions, close to neutral medium, the iron content in all samples except the system with a maximum of carbohydrates, greatly reduces. When using mass ratio of inorganic and organic components of 1:1.5 and 1:1.0, it is only 9.9 % and 4.8 % respectively of the theoretical value.

To obtain drugs with greater mass fraction of solids, the possibility of using more concentrated solutions of components was considered. It was found that at increasing the concentration of polysaccharides in the solution to 0.45 %, and iron – 0.30 % at pH=12.0 and mass ratio of 1:1.5 there is a decrease in the yield of the desired product by 2 times. In this case, the mass fraction of iron in it is 23.0 %. This indicates the feasibility of using dilute solutions of reactants.

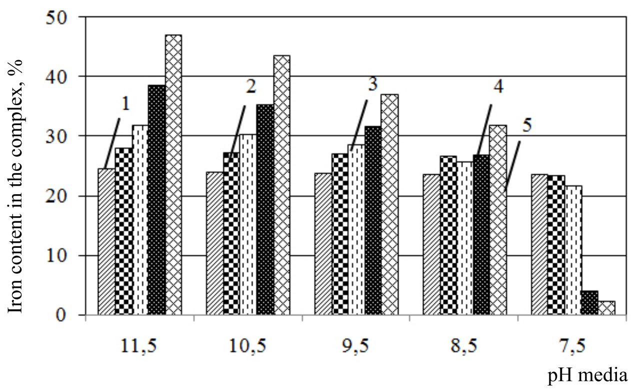

Fig. 3. Dependence of the iron content in complexes on the pH medium and the mass ratio of inorganic and organic components:
1 — 1:3.0; 2 — 1:2.5; 3 — 1:2.0; 4 — 1:1.5; 5 — 1:1.0

Thus, the possibility of preparation of soluble metal complex based on polysaccharides of mushroom was shown. Its yield and composition depend on the ratio of iron: polysaccharides, concentration of reactants, pH medium.

6. Results of the study of iron complex with maximum iron content and their discussion

Next, the characteristic of the sample with the maximum content of iron, prepared with a mass ratio of organic and inorganic components of 1:1.0 and pH=12.0 using UV and IR spectroscopy, gel chromatography and electron microscopy was given.

The IR spectrum of the studied drug is similar to spectra of iron complexes based on dextran and pullulan [15, 29, 30]. In the IR spectrum of both ferric hydroxide (III), and starting polysaccharides, absorption bands at a wavelength of about 3400 cm^{-1} that corresponds to vibrations of hydroxyl groups were found. In the IR spectrum of the complex it is shifted towards lower values of the wave number, which indicates the presence of strong hydrogen bonds. A simultaneous absorption band shift from 690 cm^{-1}, caused by the presence of hydroxyl groups in the IR spectrum of the ferric hydroxide (III) to 700 cm^{-1} in the complex confirms the above. It should be noted that these bonds are stronger compared with those in the starting substances. The presence of the absorption band of about 1625 cm^{-1} indicates the presence of the crystallisation water in the structure of the complex. The IR spectrum of ferric (III) hydroxide, absorption at 430–450 cm^{-1}, caused by fluctuations of Fe–O···Fe was revealed. The same band is present in the IR spectrum of the studied complex that confirms the presence of this metal in its composition.

The absorption spectrum of the resulting complex in the visible and ultraviolet areas is identical to the spectra of metal complexes, described in the literature [29, 31]. Characteristic absorption of around 300 and 500 nm indicates that the iron is present in the octahedral form, surrounded by oxygen atoms and there are hydrogen bonds among polysaccharides and the inorganic component.

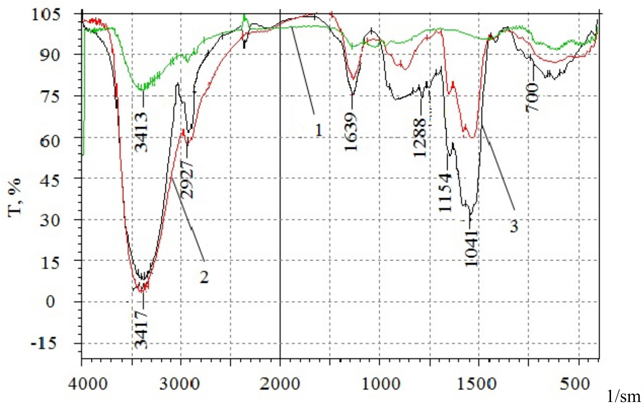

Fig. 4. IR–spectra of samples: 1 —ferric hydroxide (III); 2 — polysaccharides; 3 — iron complex

The data obtained suggest that the sample under investigation is structurally similar to complexes, described in the literature. It is believed that their core is polycyclic ferric hydroxide $(FeOOH)_n$, covered by the carbohydrate component, which is linked to it by hydrogen bonds [15, 28–31].

Results of the study of metal complex based on polysaccharides of mushroom by gel chromatography on Sephadex G–150 have revealed that elution curves of organic and inorganic components are combined. This also confirms the formation of the complex. Moreover, the carbohydrate component at a given ratio of the components is fully included in the complex. Its molecular mass exceeds that of the starting polysaccharides and is in the range of 100–120 kDa.

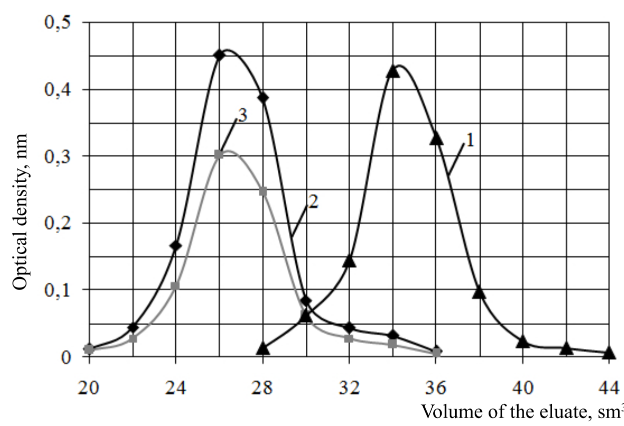

Fig. 5. Output curve of gel chromatography of polysaccharide and iron complex on Sephadex G–150:
1 — polysaccharides; 2 — polysaccharides in the complex;
3 — iron in the complex

The size of the complex, according to the results of its studies by electron–electron microscopy was 170–239 nm, which can be attributed to its nanostructures. The particles have an irregular ellipsoidal shape, the shape factor is 0.80. This approximates to the corresponding characteristics of iron complexes based on dextran and pullulan [15, 17, 28, 31].

7. Conclusions

Possibility to prepare soluble metal complexes based on polysaccharides of mushroom (*Agaricus bisporus*) was proved. Their yield and composition depend on the ratio of iron: polysaccharides, pH medium, concentration of reactants. The maximum yields of complexes with high iron content can be obtained at mass ratio of the inorganic and organic components of 1:1.0 at pH=12.0 and 1:2.5 at pH=8.5.

According to IR and UV spectroscopy, the products obtained are complexes of polycyclic ferric hydroxide, linked with polysaccharide molecules by hydrogen bonds. The complex, which includes 47.9 % share of iron, does not contain the nonmetal polysaccharide component and has a molecular mass of 100–120 kDa. The complex belongs to the nanoscale structures.

By varying the process conditions, it is possible to prepare iron complexes based on polysaccharides with different ratios of components. This allows to predict the effectiveness of their use in the prevention, correction of iron deficiency and immune response dysfunctions, caused by them due to the simultaneous presence of iron and β–(1→3)/(1→6)–D–glucans – powerful immunomodulators in complexes.

Currently in Ukraine there are no analogues of these drugs, therefore, their further comprehensive study is of interest.

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РАЗРАБОТКА МЕТОДИК КАПИЛЛЯРНОЙ ХРОМАТОГРАФИИ ТЕРПЕНОВЫХ УГЛЕВОДОРОДОВ И КИСЛОРОД-СОДЕРЖАЩИХ КОМПОНЕНТОВ ЭФИРНЫХ МАСЕЛ

Н. Э. Фролова
Кандидат технических наук, доцент
Кафедра технологии оздоровительных продуктов*
E-mail: nef1956@mail.ru
Е. М. Усатюк
Ассистент
Кафедра молекулярной и авангардной гастрономии*
E-mail: llenausatiuk@gmail.com
*Национальный университет пищевых технологий
ул. Владимирская, 68, г. Киев, Украина, 01601

У статті наведено дослідження умов проведення газохроматографічного аналізу ефірних олій на капілярних колонках з максимальним поділом терпенових вуглеводнів і кисневмісних компонентів. Під час розроблення методик підібрано нерухомі фази, обрано газ-носій і його швидкість, температури нагріву колонок, детектора, інжектора. Розроблені методики можуть бути використані для вивчення складу ефірних олій як ефіроолійних культур, так і пряно-ароматичних рослин нової селекції

Ключові слова: ефірна олія, нерухома фаза, терпенові вуглеводні, кисневмісні компоненти, газохроматографічний аналіз

В статье приведены исследования условий проведения газохроматографического анализа эфирных масел на капиллярных колонках с максимальным разделением терпеновых углеводородов и кислородсодержащих компонентов. При разработке методик подобраны неподвижные фазы, выбран газ-носитель и его скорость, температуры нагрева колонок, детектора, инжектора. Разработанные методики могут быть использованы для изучения состава эфирных масел как эфиромасличных культур, так и пряно-ароматических растений новой селекции

Ключевые слова: эфирное масло, неподвижная фаза, терпеновые углеводороды, кислородсодержащие компоненты, газохроматографический анализ

1. Введение

Эфирные масла (essential oils) – сложные смеси органических соединений, состоящие, в основном, из терпенов и их кислородсодержащих производных – терпеноидов [1].

Всестороннее развитие в современной методологии изучения состава сложных смесей органических