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## OPTIMIZATION OF THE ISOLATION OF BIOLOGICALLY ACTIVE COMPLEXES FROM BUDS AND SHOOTS OF BIRCH AND POPLAR BUDS FOR THEIR FURTHER USE IN COSMECEUTICAL COMPOSITIONS

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*The aim is to optimize the isolation of biologically active birch and poplar complexes based on woodworking industry waste to study their biological activity to create cosmeceutical compositions that have cosmetic and pharmaceutical effects.*

**Materials and methods.** *The object of research is the vegetative organs of birch (buds and shoots) and poplar (buds). In the process of work, experimental studies were carried out on the extraction and separation of natural compounds, development of biological complexes of vegetative organs of birch and poplar; study of the chemical composition and standardization of biologically active complexes of birch and preparations based on them.*

**Results.** *The optimal methods for extracting natural complexes were determined. Created as a semi-industrial installation for producing biologically active complexes by barothermal method, combining two processes – high pressure and hydrodistillation. The optimal operating cycle and plant parameters were determined. The optimal solvents for the extraction of biologically active complexes from the vegetative organs of birch and poplar have been determined. Petroleum ether (33.60 %) extracted the most compounds from birch buds, in birch shoots – ethanol (8.34 %). Qualitative and quantitative indicators of biologically active complexes are determined, and projects for examining the obtained biologically active complexes are proposed. We determined the yield of essential oil depending on the time of collection of raw materials and determined that the largest amount of essential oil is contained in spring buds (0.21 % of dry raw materials) and shoots (0.08 %).*

**Conclusions.** *Biological complexes created from timber processing waste with high activity are the basis for the creation of cosmetic products. The created semi-industrial plant will provide the basis for creating new original products in industrial volumes*

**Keywords.** *Birch, poplar, essential oils, biologically active complexes, extraction, barothermal method, hydrodistillation, cosmeceuticals*

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### 1. Introduction

Despite the rapid development of chemistry and the growth in the number of new highly effective drugs of biotechnological and synthetic origin, plants continue to occupy a significant place in the arsenal of medicines. Medicinal raw materials are used for the industrial production of pharmacologically active substances in an individual state, especially those whose synthesis is not yet feasible or economically inefficient, and also for their possible use as a substance in developing drugs with an even more pronounced therapeutic effect. Traditional medicine has always actively used medicinal plants for many millennia, accumulating and transferring practical experience in the preparation of various potions and healing with the help of natural remedies. One of the ways of the directed search for new highly effective remedies among herbal substances is the systematic study of the experience of traditional medicine, the composition and pharmacological activity of the medicinal herbal preparations used by it, and the isolation and study of the components that make up their com-

position. The advantage of preparations based on biologically active substances of plants in many cases is obvious since, on the one hand, there are practically no complications and undesirable side effects when using them, and on the other hand, there is a wide scope for manoeuvring, which is provided by a rich selection of plants with the same species.

Birch, represented on the territory of Kazakhstan mainly by two species – drooping (*B. pendula* Roth.) and fluffy (*B. pubescens* Ehrh.), is the main forest-forming species of the North Kazakhstan region. Of these, the most common is the downy birch (*B. pubescens*). The total area of land covered with birch forest occupies 327015 hectares, which is 92 % of the entire forest area. Compared to other tree species, birch has the main share: for example, aspen makes up 7.5 %; poplar, 0.37 % [1].

Birch forests are well renewed. The average increase is 831.1 thousand m<sup>3</sup> or 2.5 % (poplar – 0.3 %). Annual planned felling yields 223 thousand m<sup>3</sup> of wood, which is used for processing. In the North Kazakhstan

region, mature and overmature, that is, economically valuable forests, make up 19084 hectares, which is 5.9 % of all birch forests; in addition, a low yield of commercial wood of  $\approx 7$  % gives a very large amount of waste that is simply burned.

In addition, annually, there is a planned maintenance felling, which is 100 thousand m<sup>3</sup>, and this wood from it is not processed at all [1].

Thus, planned deforestation, which results in a large amount of waste, provides regular supplies of plant materials for organizing the production of biologically active complexes and individual native substances, including betulin [2].

Drooping birch (*Betula pendula* Roth.) is a medium-sized deciduous tree that gets its common name from the white peeling bark on the trunk. This species is native to Europe and parts of Asia. Its range extends to Siberia, China, and southwestern Asia in the mountains of northern Turkey, the Caucasus, and northern Iran [3, 4].

Downy birch (*Betula pubescens* Ehrh) is a medium-sized broad-leaved tree species that occur naturally throughout Europe and Central Asia [5]. Silver birch and downy birch are similar to each other in general appearance, both are white-stemmed, reaching a height of 20–30 m. However, they differ in the morphology of leaves, branches, bark, seeds and catkin scales, as well as cell size and wood anatomy [4].

In traditional Eastern European medicine, silver birch is used as a diuretic, especially for cystitis, and to treat rheumatism and arthritis. However, recent studies have shown that its diuretic potential may be due to its endopeptidase inhibitory properties. In contrast, xanthine oxidase inhibitory properties may be responsible for its use in gout treatment [6–8]. Also, drooping birch is used for lung diseases, skin diseases, infertility, stomach diseases, kidney stones, jaundice, pneumonia, and cholera [9, 10].

Downy birch is used for heart failure and hypertension [11]. A decoction of downy birch inflorescences is used in the treatment of gout [12]. Flowers, leaves, bark and resin of downy birch, harvested in spring, are used as a choleric, diuretic, wound healing, hypnotic and decongestant, with gout and tartar [13]. The leaves are used to obtain a cream for epidermal application [9, 14].

Birch buds are pharmacopoeial raw materials, and therefore their use in cosmetic compositions is justified. However, annual shoots of birch, which contain a wide class of terpene compounds hyberrillins, are also of interest, and their use is also becoming relevant.

Also of great interest for study are the buds of a woody plant of the Willow family – balsam poplar (*Populus balsamifera* L.), widely growing in Kazakhstan.

Some scientific sources note that the use of poplar buds was first described in a book by John Gerard in 1597 as an ointment for inflammation [15]. Balsam poplar buds contain flavonoids, minerals, resins, glycosides, organic acids and waxes. The main ones are phenolic compounds, which determine the antioxidant, anti-inflammatory and antimicrobial properties of the raw material of this plant [15–17].

Studies have shown the antifungal activity of poplar buds against the fungus *Penicillium italicum* [18]. Castaldo and Capasso (2002) showed that balsamic poplar bud propolis extracts exhibited antimicrobial activity, mainly against Gram-positive bacteria (*Staphylococcus* spp. and *Streptococcus* spp.) [19]. Turkish scientists determined that poplar buds suppress clinically significant microorganisms, including yeast, but do not affect gram-negative bacteria [20, 21].

The anti-inflammatory and antioxidant effects of poplar buds were studied by Chinese researchers using DPPH and ABTS free radical scavenging methods [22].

Simard et al. reported the isolation of 12 new flavan derivatives from the buds of *P. balsamifera*, which were isolated as six pairs of enantiomers. Other scientists have studied these compounds' antibacterial activity and cytotoxicity against *S. aureus* and human skin fibroblast cells [23].

Therefore, the study of birch buds and shoots, as well as poplar buds growing on the territory of Kazakhstan, is of great scientific and practical importance.

**The aim** of the work is to optimize the isolation of biologically active complexes of birch and poplar and study their biological activity in order to create cosmetic compositions that exhibit both cosmetic and pharmaceutical effects.

## 2. Planning (methodology) of the research

Research stages:

1. Collection of a number of raw materials, species identification, and drying.

2. Collect the essential oil of birch buds and shoots with different collection times by hydrodistillation. Calculate the yield of essential oils of birch buds and shoots, depending on the time of collection of raw materials.

3. Extract buds and shoots with solvents with an increasing polarity gradient. As a result of the extraction of raw materials with organic solvents at each stage, an extractive solution is obtained, and some extractive substances precipitate out.

4. Barothermic method to obtain the essential oil of poplar buds. Then, determine the optimal technological parameters for obtaining biologically active substances from poplar buds.

## 3. Material and methods

### 3. 1. Plant material

This study was carried out at the University of Kozybaev in the organic synthesis laboratory in Petropavlovsk, North Kazakhstan region, Kazakhstan, in 2021–2022. The study included buds and shoots of birch and poplar buds.

The collection of plant raw materials of birch and poplar was carried out from the beginning of March to mid-May 2021–2022. Raw materials collection area Meshchansky forest of the North Kazakhstan region, distance from the highway is more than 1 km.

Buds of poplar and birch were collected in early and mid-April in the phase of swelling before their blooming (before the divergence of the covering scales at the top of the bud).

Birch shoots were harvested from early March to mid-May. Shoots are cut, tied in bunches, and dried for 3–4 weeks in the open air or a well-ventilated area. Raw materials are stored in paper bags with labels indicating the place and date of collection.

### 3.2. Obtaining the essential oil of buds and annual birch shoots by hydrodistillation. Calculation of the yield of essential oils of birch buds and shoots, depending on the time of collection of raw materials

Hydrodistillation is when raw materials are mixed with water before distillation. Most often, this method is used in the laboratory and at home.

In the process of water distillation, the prepared material is immersed in water and heated to a boil, stirring to avoid overheating the lower layers. The water vapour is condensed in a condenser cooled by running water and collected in a flask. Phyto essences are lighter than water, so they float on their surface. Water is continuously drained from the flask, and the essential oil is collected in a special container.

The installation scheme includes a flask, direct refrigerator and receiving device. Hoses for running cold water are connected to the refrigerator. Ginsberg's receiver is used as a receiving device [24].

100 grams of crushed raw materials were placed in a flask with a capacity of 1 litre and filled with water so that the surface of the water covered the plant material. The flask was placed on an electric stove.

The Ginsberg receiver was fixed in the flask: it was carefully lowered 2–3 cm below the upper edge of the flask, and the refrigerator was inserted into the flask with the other hand so that the tip of the refrigerator entered the receiver with a gap of 2–3 mm.

The resulting steam carried particles of phytoessence into the refrigerator, where the steam turned into a liquid consisting of water and small drops of essential oil. This mixture was collected in a Ginsberg receiver.

After the end of the process, the flask was cooled, the refrigerator was removed, and then the Ginsberg receiver was removed, which was transferred to a beaker. The resulting essential oil contained a small amount of water and looked cloudy, so anhydrous sodium sulfate was poured into the test tube at the tip of a knife. The turbidity disappeared, and the essential oil became transparent.

The content of essential oil in volume-weight ( $X$ ) in terms of absolutely dry raw material was calculated by the formula (1)

$$X = \frac{V \cdot 100}{m \cdot (100 - W)}, \quad (1)$$

where  $V$  – the volume of essential oil, ml;

$m$  – the mass of raw materials, g;

$W$  – weight loss during drying of raw materials, %.

### 3.3. Extraction of buds and shoots with solvents with an increasing polarity gradient

According to the literature data, birch buds and shoots contain many compounds belonging to various

classes of biologically active compounds, which requires further study and identification.

To do this, we sequentially extract the raw materials with solvents with an increasing polarity gradient (petroleum ether, hexane, toluene, chloroform, ethyl acetate, ethanol) in a Soxhlet apparatus (Fig. 1) in order to separate biologically active complexes by polarity.

The raw material was crushed, and then a cartridge made of filter paper was filled. Then the cartridge was placed in a Soxhlet apparatus and successively extracted with petroleum ether, hexane, toluene, chloroform, ethyl acetate, and ethanol until complete extraction of all extractives that are soluble in these solvents.

The extraction was stopped when the Soxhlet solvent stopped staining and remained colourless. As a result of the extraction of raw materials with organic solvents, an extractive solution was obtained at each stage and precipitation of some extractive substances was observed.



Fig. 1. Soxhlet apparatus

### 3.4. Barothermic method for obtaining essential oil of poplar buds. Determination of optimal technological parameters for obtaining BAS from poplar buds

This method consists of extracting essential oil from poplar buds opened under high temperature and high pressure with superheated water vapour, which is then cooled, condensed and collected in a receiver.

The barothermal method of extracting essential oil involves using steam and pressure to extract the oil from the buds.

Simplified, the installation consists of a steam generator, a reactor and a refrigerator.

Accordingly, in the steam generator, there is a process of steam generation in the reactor – the process of extracting organic substances from raw materials, in the refrigerator – cooling and condensation of steam with essential oil, which begins to condense at 120 °C.

To create this installation, a single-circuit scheme was used to combine the heating of the steam generator

and maintain the temperature inside the working container (to avoid steam condensation on the raw material), which makes it possible to exclude additional heating of the container.

The pipe for transferring steam from the steam generator to the working reactor ends with a distributor (atomizer), which contributes to the formation of a uniform steam flow through all the raw materials and avoids the ingress of condensate into the steam generator.

Due to the shape of the reactor, condensate will collect at the bottom, where a drain cock is provided. Through the same tap, a thick substance is drained.

The feed container is located at some distance from the walls of the reactor in order to avoid overpressure. The bypass valve located on the cover of the steam generator and designed for a steam pressure of 1.9 atmospheres serves the same purpose. The reactor cover is removable, and loading is made through the top.

Steam and essential oil are cooled in a coil immersed in a coolant (cold water) container.

After cooling, the emulsion of water and essential oil is collected in a receiver and sent for separation.

The steam transfer pipe from the steam generator to the reactor ends with a distributor (atomizer), contributing to the formation of a uniform steam flow through all the raw materials and preventing poplar oil from entering the steam generator.

Determination of optimal technological parameters for obtaining BAS from poplar buds.

In order to determine the optimal conditions for achieving the maximum yield of the preparation «Topolin» and essential oil, a series of experiments was carried out.

The research was carried out on several technological parameters:

- 1) pressure effect;
- 2) dependence of drug yield on layer height;
- 3) length of extraction time;
- 4) the dependence on the yield of essential oil and the drug «Topolin» at the time of the collection of poplar buds.

## 4. Research results

### 4.1. Yield of essential oils of birch buds and shoots depending on the time of collection of raw materials

The essential oil of birch buds and shoots with different collection times has been produced.

Essential oils of buds and shoots differ in physical constants. The essential oil of the kidneys is yellowish, and the oil of the twigs is a light brown liquid, thickening and darkening during storage under the influence of light, air and temperature.

The yield of essential oils of birch buds and shoots was calculated depending on the time of collection of raw materials. The results are shown in Fig. 2.

As a result of the study, we found that the yield of essential oil depends on the time of collection of raw materials and the highest content of essential oil was found in spring buds, which amounted to 0.21 % of dry raw materials.

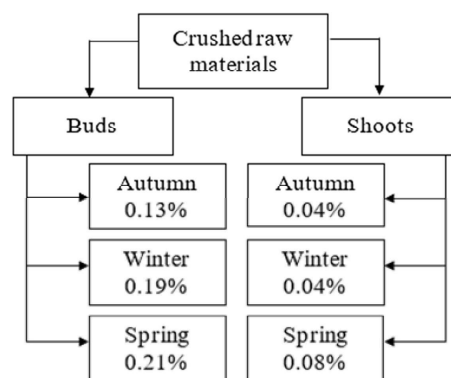


Fig. 2. Comparative characteristics of the yield of essential oils of birch buds and shoots, depending on the time of collection of raw materials

The yield was 0.13 % in autumn buds, and in winter buds, 0.19 %. In the shoots, the essential oil is found in much smaller quantities. Its highest content also fell in the spring period (0.08 %). The yield of essential oil of autumn and winter shoots did not change depending on the time of collection and amounted to 0.04 % of the mass of dry raw materials.

### 4.2. Determination of the content of extractive substances in various fractions of birch buds during extraction with solvents with an increasing polarity gradient

Birch buds. For fractionation, dried birch buds were used, previously subjected to steam distillation, i.e., without essential oil. The mass of raw materials was 55 g.

As a result of the extraction of raw materials with organic solvents, an extractive solution was obtained at each stage and precipitation of some extractive substances was observed.

The experiment was carried out 5 times. The results have convergence. The average results are shown in Table 1.

Table 1  
The content of extractive substances in various fractions of birch buds

Extractant	Sediment, g/%	Smolka, g/%	The total amount of extractives
Petroleum ether	2.75	15.73	18.48
	5.00	28.60	33.60
Hexane	0.25	0.14	0.39
	0.47	0.26	0.73
Toluene	0.02	1.76	1.78
	0.04	3.20	3.24
Chloroform	0.45	0.06	0.51
	0.82	0.11	0.93
Ethyl acetate	0.10	1.19	1.29
	0.18	2.16	2.34
Ethanol	0.01	3.02	3.03
	0.02	5.49	5.51

Note: above the line – the mass of sediment and tar (g), below the line – % of the absolutely dry sample; resins were obtained as a result of the complete evaporation of extracts

Based on the results of the analysis of Table 1, we can conclude that the largest amount of extractives was extracted with petroleum ether (33.6 %), ethyl alcohol (5.51 %) and toluene (3.24 %).

Graphically, the dependence of the total amount of extractive substances in birch buds (%) on the extractant used is shown in Fig. 3.

Consequently, the largest amount of biologically active substances in birch buds are lipid compounds: fatty acids, sterols and terpene compounds. Their mass was 18.48 g.

Phenolic compounds, which are extracted by polar organic solvents, are present in the raw material in a much smaller amount.

The highest yield of these substances (5.51 %) was found in the alcoholic extract, which amounted to 3.03 g. The mass fraction of birch bud extractives was 46.32 % of the raw material's mass.

**Birch shoots.** Annual shoots of birch were subjected to sequential solvent extraction with an increasing polarity gradient (similar to birch buds). The experiment was carried out 5 times. The results have convergence.

Data from studies of buds compared with birch shoots are given in Table 2.

Analyzing the obtained results, it can be stated that the mass fraction of extractive substances in the shoots was 7 g (13.09 %), which is 3.5 times lower than the content of extracted substances in the buds (46.32 %) (Table 2).

Based on the table, it should be noted that the shoots, compared with the buds, contain a relatively small amount of compounds extracted by organic solvents.

Petroleum ether extracted the largest amount of substances from the buds (33.60 %), while in the shoots, the maximum amount of substances were extracted by ethanol (8.34 %).

Graphically, the dependence of the total amount of extractive substances in birch buds (%) on the extractant used is shown in Fig. 4.

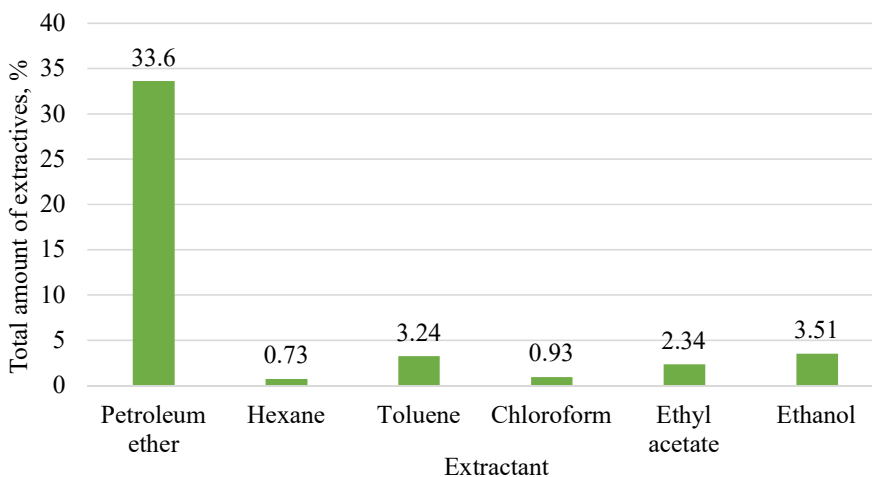


Fig. 3. Graphic dependence of the total amount of extractives in birch buds on the extractant used

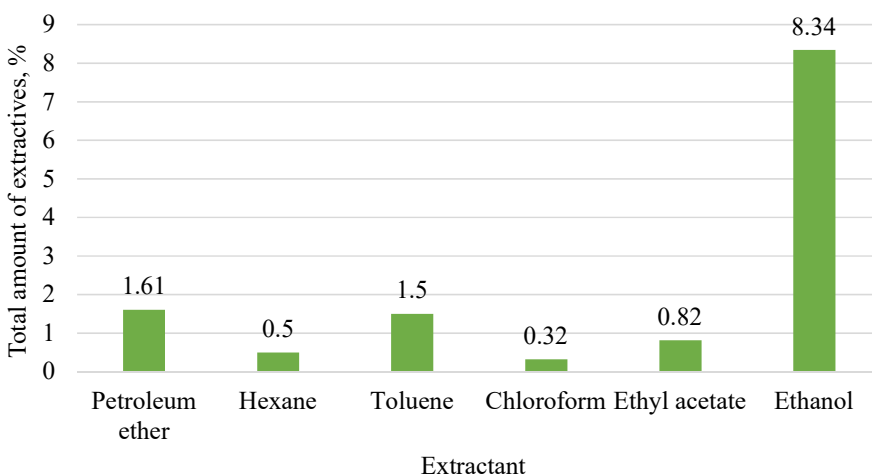


Fig. 4. Graphic dependence of the total amount of extractives in birch shoots on the extractant used

Table 2  
Comparative characteristics of the content of substances extracted by organic solvents in the buds and shoots of birch

Solvent	The yield of extractives, %	
	In the kidneys	On the run
Petroleum ether	33.60	1.61
Hexane	0.73	0.50
Toluene	3.24	1.50
Chloroform	0.93	0.32
Ethyl acetate	2.34	0.82
Ethanol	5.51	8.34
General output	46.32	13.09

The fractions obtained by extraction with ethyl alcohol were examined for the determination of the content of polyphenolic compounds by the Folin-Ciocalteu method.

In a test tube, 5.0 ml of an aqueous solution of the Folin-Ciocalteu reagent (diluted in a ratio of 1/10) were mixed, 1.0 ml of the extract and 4.0 ml of a 7.5 % sodium carbonate solution were added. After keeping the solutions for 60 min at room temperature, the optical density of the solutions was measured with a spectrophotometer at a wavelength of  $\lambda=765$  nm. A calibration curve was built for a standard substance (protocatechuic acid), and the polyphenols' content in the extracts was calculated from it. The results were expressed as percentages per 1 mg of the extractive.

According to the results of the experiment, the content of flavonoids in the B. Pendula bud extract was 7.1 %, and in the shoot extract, it was 5.3 %.

### 4.3. Optimization of the isolation of biologically active complexes from poplar buds

An installation was developed and created for obtaining the essential oil of poplar buds by balsamic barothermal method, combining two processes – high pressure and hydrodistillation (Fig. 5). A pre-patent of the Republic of Kazakhstan was received for the barothermal installation [25]. This installation has no similar analogues in producing essential oil, thereby representing a scientific novelty.

Influence of pressure on the yield of essential oil.

When extracting essential oil from balsamic poplar buds, the pressure in the apparatus was changed within the following limits – from 0.5 to 2.8 atm.

The upper limit (2.8 atm.) is the maximum allowable for this laboratory setup.

It is not advisable to use a higher pressure because of the high temperature, which negatively affects the quality of the essential oil obtained, and the increase in the cost of equipment, along with an increase in the risk of operation.

The experiment was carried out 5 times. The results have convergence. The results were taken as the arithmetic mean.

In the course of conducting studies with a change in pressure, the data displayed in Table 3 were obtained.

The optimal operating mode of the installation is a pressure of 1.5 atm. up to 1.8 atm., which gives the highest yield of essential oil from 0.50 to 0.57 %.

Graphically, the dependence of the yield of essential oil on pressure is shown in Fig. 6.

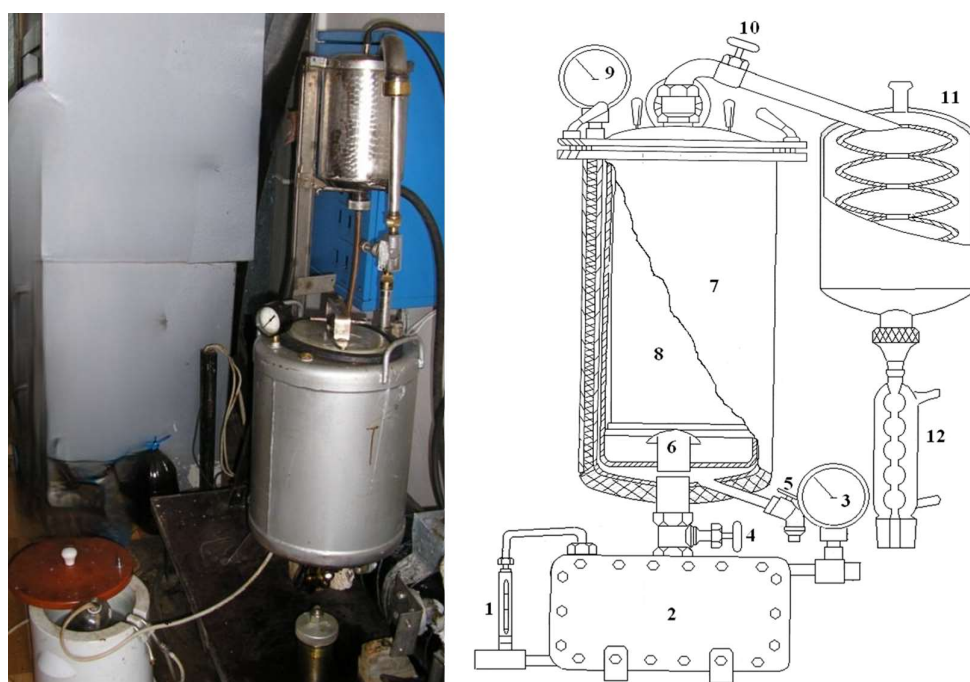


Fig. 5. Photo and Scheme of the barothermal installation: 1 – water level indicator; 2 – steam generator; 3 – steam generator pressure indicator; 4 – steam admission valve to the reactor; 5 – bottom release valve; 6 – steam distributor; 7 – reactor; 8 – container; 9 – reactor pressure indicator; 10 – top outlet valve; 11 – water cooler; 12 – reverse water cooler

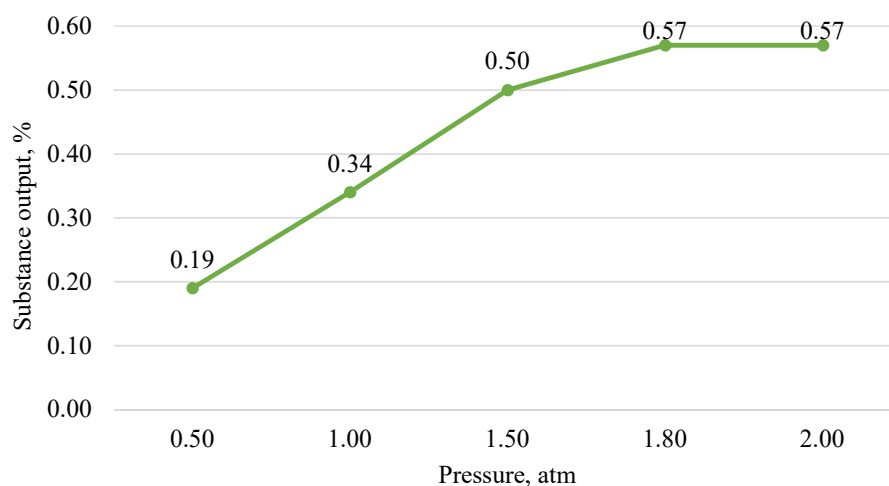


Fig. 6. Graph of dependence of essential oil yield on the pressure value

Table 3

The effect of pressure changes on the yield of essential oil

Pressure, atm.	0.50	1.00	1.50	1.80	2.00
Substance output, %	0.19± ±0.01	0.34± ±0.03	0.50± ±0.02	0.57± ±0.07	0.57± ±0.01

*Dependence of drug yield on layer height.*

When conducting experiments on a laboratory setup, the height of the raw material layer was changed (from 10 to 100 %), and the dependence of the yield of essential oil on this indicator was studied. The yield increases as the bed height increases due to the greater contact of the steam with the feedstock and hence the greater saturation. But at a layer height of more than 50 %, a decrease in yield is observed, which is a consequence of the compaction of the raw material, which is caked in lumps and makes it difficult for steam to contact the raw material. The experiment was carried out 5 times, and practically equal results were obtained in each case. During the experiment, data were obtained (Table 4).

The optimal height of the raw material is from 40 to 50 %.

Based on the data in Table 4, a graph was built (Fig. 7).

The effect of changing the height of the raw material layer on the yield of essential oil

Table 4

Height layer raw materials, %	10	20	30	40	50	60	70	80	90	100
Substance output, %	0.21± ±0.03	0.36± ±0.01	0.52± ±0.03	0.58± ±0.05	0.60± ±0.07	0.50± ±0.02	0.43± ±0.02	0.37± ±0.04	0.37± ±0.03	0.30± ±0.01

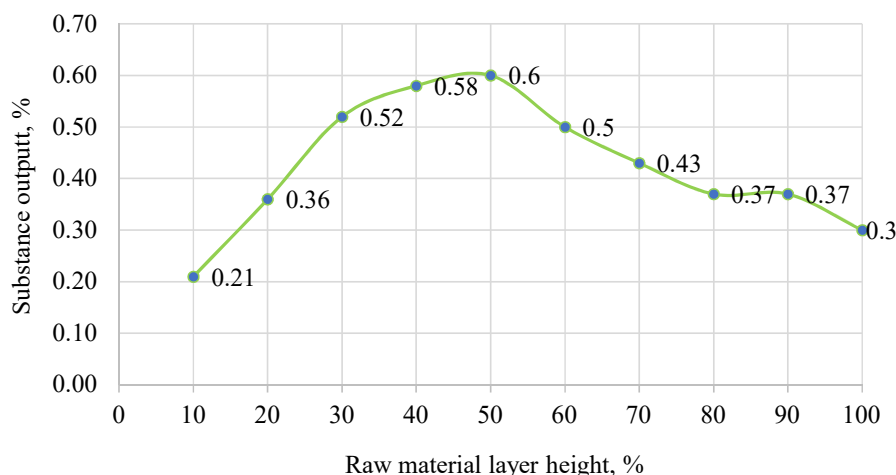


Fig. 7. Graph of the dependence of the yield of essential oil on the height of the layer of raw materials

*The duration of the extraction time.*

When extracting the essential oil, observations were made on the dependence of the product yield on the extraction time. With an increase in the processing time

of the kidneys, the yield of essential oil increases. The most effective time interval is 1.5–2.5 hours. Then the product yield starts to decrease sharply, which indicates the inexpediency of using a longer extraction time (Table 5). The experiment was carried out 5 times. In each case, practically equal results were obtained.

The optimal extraction time of raw materials is 2 hours.

Graphically, the extraction time's dependence on essential oil yield is shown in Fig. 8.

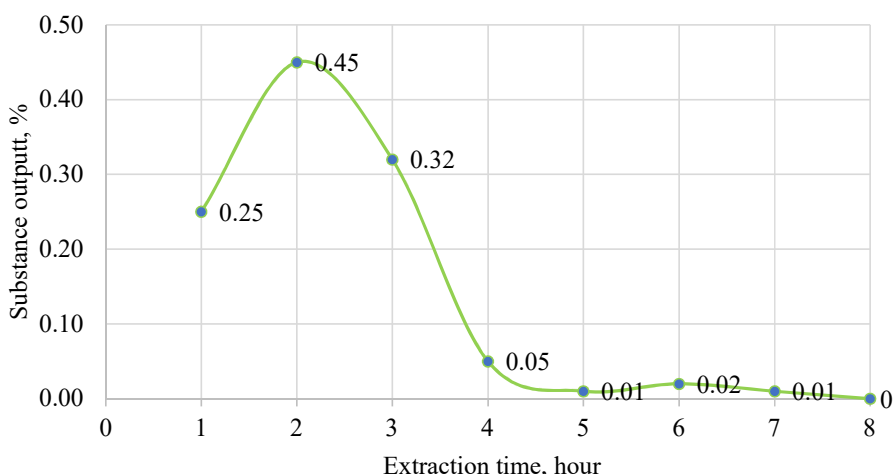


Fig. 8. The plot of essential oil yield versus extraction time

Based on the foregoing, an example of one installation cycle can be given.

Installation cycle:

- 1) filling the container with raw materials to a height of 40–50 %;
- 2) power on;
- 3) filling the steam generator with water;
- 4) heating water in the steam generator to the boiling point;
- 5) forcing pressure in the steam generator, due to the formation of steam in the steam generator up to 1.5–1.8 atm.;
- 6) turning on the refrigerator;
- 7) warming up the reactor with the resulting steam (when valve 4 is opened, steam is supplied from the steam generator to the reactor, and then the steam supply is stopped by it);
- 8) draining the condensate formed as a result of heating through valve 5 from the reactor;
- 9) injection of the reaction atmosphere in the reactor by opening valve 4;
- 10) upon reaching a pressure value of 1.25 atm. the collection of an aqueous emulsion of an essential oil be-

gins through valve 10 (we slightly open the valve and monitor the temperature of the water at the refrigerator outlet, in case of heating, we increase the flow of water). When valve 10 is opened, the rate of pressure increase in the reactor decreases. It is necessary to support its growth, not allowing a decrease in pressure;

11) upon reaching a pressure value of 1.6–1.7 atm. collection of thick substances begins through valve 5. The collection takes place together with condensate, which is separated after cooling;

12) upon reaching a pressure value of 1.8 atm. and above, gradually opening valve 10 to the end, we collect an aqueous emulsion of essential oil (continuing to monitor the temperature of the water in the refrigerator);

13) in addition, throughout the entire time of operation at the installation (within 2 hours), it is necessary to monitor the water level in the steam generator; when the water level decreases by less than half, turn off the power;

14) with a gradual decrease in pressure, we continue to collect both an aqueous emulsion and a thick substance without stopping monitoring the temperature of the water in the refrigerator;

15) upon reaching the pressure value of 0 atm. in the steam generator, we remove the container with raw materials from the reactor;

16) we extract the waste material from the container.

Table 5

The effect of extraction time on the yield of essential oil

Extraction time, hour	1	2	3	4	5	6	7	8
Substance output, %	0.25± ±0.01	0.45± ±0.03	0.32± ±0.02	0.05± ±0.05	0.01± ±0.01	0.02± ±0.03	0.01± ±0.02	0

## 5. Discussion

As a result of the study of buds and shoots of birch (*Betula pendula* Roth.), it was found that the time of collection significantly affects the content of essential oil. In autumn birch buds, essential oil content is 38 % less than in spring samples; in winter samples, the content is 10 % less than in spring ones. The content of essential oil in birch shoots collected in spring is 50 % higher than in shoots collected in winter and autumn. Therefore, it is advisable to use samples of spring shoots and buds in this study. The content of extractive substances in different fractions of birch buds was determined by extraction with various solvents of polar and non-polar nature. The highest yield of extractives was obtained using a non-polar solvent – petroleum ether (33.6 %). Among the polar solvents, ethyl alcohol (5.51 %) isolated the largest number of extractives. These results indicate that in the composition of birch buds, the largest amount of biologically active substances are lipid compounds (fatty acids, sterols, terpene compounds), and phenolic compounds are contained in raw materials in a smaller amount. The content of extractive substances in birch shoots was also determined by extraction with various solvents of polar and non-polar nature. Compared with the kidneys, the total yield of extractives decreased from 46.32 % to 13.09 %. Moreover, the highest yield of extractives was obtained using a polar solvent – ethyl alcohol

(8.34 %). This result suggests that in shoots, compared with buds, the largest amount of biologically active substances are phenolic compounds. Studies by Korean scientists on the extraction of biologically active substances from birch shoots by extraction with ethyl alcohol showed similar results – 5.9 % [26]. In studies by Czech scientists, it is also reported that the highest yield of extractable substances from birch shoots occurs during extraction with 80 % ethyl alcohol [6]. The essential oil of balsamic poplar buds was obtained by the barothermic method at various pressure indicators. The highest yield (0.50–0.57 %) was recorded at a pressure of 1.5 to 1.8 atm. Also, in the framework of this experiment, we checked the dependence of the height of the layer of raw materials and the effect of extraction time on the yield of essential oil. The highest yield of raw materials (0.58–0.60 %) was obtained with a layer height of raw materials from 6.0 to 7.5 cm. The optimal extraction time is 1.5–2.5 hours, since, during this time, the highest yield of the extracted essential oil is observed. The barothermal method is used to extract biologically active flavonoids. At the same time, the completeness of extraction of balsam poplar bud flavonoids is achieved by the barothermal method. This is confirmed by the studies of Karaganda scientists [27].

Then an experimental series of face creams with extracts of poplar and birch buds with a concentration of 1 % was developed. This cream is based on lanolin with the addition of poplar and birch bud resin. Lanolin is a waxy natural substance that is used in pharmaceuticals as the base of many ointments. This wax has one feature – it dissolves water well, thereby providing good skin hydration. In addition, it contains unsaturated fatty acids with high biological activity, regulating the metabolism in the skin of the face. To obtain the base of the cream, it is necessary to use a number of components that emulsify, stabilize, and disinfect the base of the cream. The composition was created using one technology, which is shown in Fig. 9.

**Research limitations.** The proposed barothermal

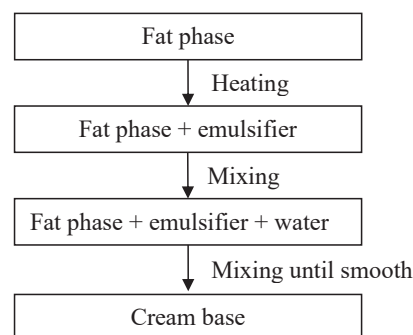


Fig. 9. Technological scheme for creating cosmetic bases

method for extracting biologically active substances from plant buds cannot be used to extract biologically active substances from other parts of plants due to the specificity of this method.

**Prospects for further research.**

The created semi-industrial plant will provide the basis for the creation of new original domestic products in industrial volumes. The obtained biologically active



complexes from various vegetative organs of poplar and birch, on the basis of which cosmetic compositions of the form will be created in the future, can be used as domestic cosmeceutical products.

## 6. Conclusions

1. The yield of essential oil depends on the time of collection of raw materials, and the highest content of essential oil was found in spring buds, which amounted to 0.21 % of dry raw materials. In autumn buds, the yield was 0.13 %, and in winter buds, 0.19 %. In the shoots, the essential oil is found in much smaller quantities. Its highest content also fell in the spring period (0.08 %). The yield of essential oil of autumn and winter shoots did not change from the time of collection and amounted to 0.04 % of the mass of dry raw materials

2. The optimal solvents for the extraction of biologically active complexes from the vegetative organs of birch and poplar have been determined. Petroleum ether extracts the greatest amount of substances from birch buds (33.60 %). In birch shoots with ethanol (8.34 %).

3. For the development of biologically active complexes, a semi-industrial plant has been created that al-

lows you to effectively obtain essential oil and a thick substance from wood processing waste. The optimal cycle of work and the parameters of the plant operation are determined.

## Conflict of interests

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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## Data availability

Data will be made available on reasonable request.

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## References

1. Distribution of forested land by dominant forest species and groups (2020). State accounting of the forest fund of the North Kazakhstan region.
2. Lezhneva, M. Yu. (2006). Terpene compounds of birch, their chemical modifications and biological activity. Astana: Institute of Phytochemistry, 24.
3. Chen, S., Zhao, X., Chen, S. (2022). Identification of the cell wall synthesis genes in *Betula pendula*. *Wood Research*, 67 (4), 519–532. doi: <https://doi.org/10.37763/wr.1336-4561/67.4.519532>
4. Hynynen, J., Niemisto, P., Vihera-Aarnio, A., Brunner, A., Hein, S., Velling, P. (2009). Silviculture of birch (*Betula pendula* Roth and *Betula pubescens* Ehrh.) in northern Europe. *Forestry*, 83 (1), 103–119. doi: <https://doi.org/10.1093/forestry/cpp035>
5. Harr, L., Esper, J., Kirchhefer, J. A., Zhou, W., Hartl, C. (2020). Growth response of *Betula pubescens* Ehrh. to varying disturbance factors in northern Norway. *Trees*, 35 (2), 421–431. doi: <https://doi.org/10.1007/s00468-020-02043-1>
6. Havlik, J., de la Huebra, R. G., Hejtmankova, K., Fernandez, J., Simonova, J., Melich, M., Rada, V. (2010). Xanthine oxidase inhibitory properties of Czech medicinal plants. *Journal of Ethnopharmacology*, 132 (2), 461–465. doi: <https://doi.org/10.1016/j.jep.2010.08.044>
7. Gründemann, C., Gruber, C. W., Hertrampf, A., Zehl, M., Kopp, B., Huber, R. (2011). An aqueous birch leaf extract of *Betula pendula* inhibits the growth and cell division of inflammatory lymphocytes. *Journal of Ethnopharmacology*, 136 (3), 444–451. doi: <https://doi.org/10.1016/j.jep.2011.05.018>
8. Bljajčić, K., Šoštarić, N., Petlevski, R., Vujić, L., Brajković, A., Fumić, B. et al. (2016). Effect of *Betula pendula* Leaf Extract on  $\alpha$ -Glucosidase and Glutathione Level in Glucose-Induced Oxidative Stress. *Evidence-Based Complementary and Alternative Medicine*, 2016, 1–8. doi: <https://doi.org/10.1155/2016/8429398>
9. Rastogi, S., Pandey, M. M., Kumar Singh Rawat, A. (2015). Medicinal plants of the genus *Betula* – Traditional uses and a phytochemical–pharmacological review. *Journal of Ethnopharmacology*, 159, 62–83. doi: <https://doi.org/10.1016/j.jep.2014.11.010>
10. Svanberg, I., Söukand, R., Łuczaj, Ł., Kalle, R., Zyryanova, O., Dénes, A. et al. (2012). Uses of tree saps in northern and eastern parts of Europe. *Acta Societatis Botanicorum Poloniae*, 81 (4), 343–357. doi: <https://doi.org/10.5586/asbp.2012.036>
11. Eddouks, M., Maghrani, M., Lemhadri, A., Ouahidi, M.-L., Jouad, H. (2002). Ethnopharmacological survey of medicinal plants used for the treatment of diabetes mellitus, hypertension and cardiac diseases in the south-east region of Morocco (Tafilalet). *Journal of Ethnopharmacology*, 82 (2-3), 97–103. doi: [https://doi.org/10.1016/s0378-8741\(02\)00164-2](https://doi.org/10.1016/s0378-8741(02)00164-2)
12. Blanco, E., Macía, M. J., Morales, R. (1999). Medicinal and veterinary plants of El Caurel (Galicia, northwest Spain). *Journal of Ethnopharmacology*, 65 (2), 113–124. doi: [https://doi.org/10.1016/s0378-8741\(98\)00178-0](https://doi.org/10.1016/s0378-8741(98)00178-0)
13. Neves, J. M., Matos, C., Moutinho, C., Queiroz, G., Gomes, L. R. (2009). Ethnopharmacological notes about ancient uses of medicinal plants in Trás-os-Montes (northern of Portugal). *Journal of Ethnopharmacology*, 124 (2), 270–283. doi: <https://doi.org/10.1016/j.jep.2009.04.041>
14. Miraldi, E., Ferri, S., Mostaghimi, V. (2001). Botanical drugs and preparations in the traditional medicine of West Azerbaijan (Iran). *Journal of Ethnopharmacology*, 75 (2-3), 77–87. doi: [https://doi.org/10.1016/s0378-8741\(00\)00381-0](https://doi.org/10.1016/s0378-8741(00)00381-0)
15. Bélanger, A., Grenier, A., Simard, F., Gendreau, I., Pichette, A., Legault, J., Pouliot, R. (2019). Dihydrochalcone Derivatives from *Populus balsamifera* L. Buds for the Treatment of Psoriasis. *International Journal of Molecular Sciences*, 21 (1), 256. doi: <https://doi.org/10.3390/ijms21010256>

16. Dudonné, S., Poupard, P., Coutière, P., Woillez, M., Richard, T., Mérillon, J.-M., Vitrac, X. (2011). Phenolic Composition and Antioxidant Properties of Poplar Bud (*Populus nigra*) Extract: Individual Antioxidant Contribution of Phenolics and Transcriptional Effect on Skin Aging. *Journal of Agricultural and Food Chemistry*, 59 (9), 4527–4536. doi: <https://doi.org/10.1021/jf104791t>
17. Pობლოკა-Оlech, L., Inkielwicz-Stepniak, I., Krauze-Baranowska, M. (2019). Anti-inflammatory and antioxidative effects of the buds from different species of *Populus* in human gingival fibroblast cells: Role of bioflavonones. *Phytomedicine*, 56, 1–9. doi: <https://doi.org/10.1016/j.phymed.2018.08.015>
18. Zhang, C., Zheng, H., Liu, G., Hu, F. (2011). Development and validation of HPLC method for determination of salicin in poplar buds: Application for screening of counterfeit propolis. *Food Chemistry*, 127 (1), 345–350. doi: <https://doi.org/10.1016/j.foodchem.2011.01.014>
19. Castaldo, S., Capasso, F. (2002). Propolis, an old remedy used in modern medicine. *Fitoterapia*, 73, S1–S6. doi: [https://doi.org/10.1016/s0367-326x\(02\)00185-5](https://doi.org/10.1016/s0367-326x(02)00185-5)
20. Darra, E., Abdel-Azeim, S., Manara, A., Shoji, K., Maréchal, J.-D., Mariotto, S. et al. (2008). Insight into the apoptosis-inducing action of  $\alpha$ -bisabolol towards malignant tumor cells: Involvement of lipid rafts and Bid. *Archives of Biochemistry and Biophysics*, 476 (2), 113–123. doi: <https://doi.org/10.1016/j.abb.2008.02.004>
21. Piochon-Gauthier, M., Legault, J., Sylvestre, M., Pichette, A. (2014). The Essential Oil of *Populus balsamifera* Buds: Its Chemical Composition and Cytotoxic Activity. *Natural Product Communications*, 9 (2), 257–260. doi: <https://doi.org/10.1177/1934578x1400900231>
22. Kuś, P. M., Okińczyc, P., Jakovljević, M., Jokić, S., Jerković, I. (2018). Development of supercritical CO<sub>2</sub> extraction of bioactive phytochemicals from black poplar (*Populus nigra* L.) buds followed by GC–MS and UHPLC–DAD–QqTOF–MS. *Journal of Pharmaceutical and Biomedical Analysis*, 158, 15–27. doi: <https://doi.org/10.1016/j.jpba.2018.05.041>
23. Simard, F., Legault, J., Lavoie, S., Pichette, A. (2014). Balsacones D-I, dihydrocinnamoyl flavans from *Populus balsamifera* buds. *Phytochemistry*, 100, 141–149. doi: <https://doi.org/10.1016/j.phytochem.2013.12.018>
24. Kabishev, K. E., Sakanyan, E. I. (2002). Determination of extractive substances in medicinal plant raw materials with various technologies for obtaining extracts. *Plant Resources*, 38 (3), 113.
25. Nadirov, R. S., Polyakov, V. V., Adekenov, S. M. (2002). Pre-Pat. No. 11387 KZ. Barotermicheskiy sposob polucheniya masla topolya bal'zamicheskogo. MKP A61K 35/78. published: 15.04.2002, Bul. No. 4
26. Park, K. J., Subedi, L., Kim, S. Y., Choi, S. U., Lee, K. R. (2018). Bioactive triterpenoids from twigs of *Betula schmidtii*. *Bioorganic Chemistry*, 77, 527–533. doi: <https://doi.org/10.1016/j.bioorg.2018.02.006>
27. Adekenov, S. M., Baysarov, G. M., Khabarov, I. A., Polyakov, V. V. (2020). Flavonoids of *populus Balsamifera* L. buds and methods for their isolation. *Chemistry of Plant Raw Material*, 2, 181–188. doi: <https://doi.org/10.14258/jcprm.2020027602>

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