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Д.В.Сокольский атындағы «Жанармай,  
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## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН  
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## NEWS

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**SELECTING OPTIMAL MODES OF KNOTWEED RAW  
MATERIALS PRESSING OUT AND DEVELOPING TECHNOLOGY  
FOR OBTAINING DRY EXTRACT**

**Abstract.** A complex of studies on the choice of optimal conditions for extraction of medicinal plant raw materials of poultry mountain and the development of technology for obtaining dry extract from plant raw materials was concluded. There is a tendency to develop resource-saving technologies in ways of creating new drugs from plant raw materials. This is ensured by the use of various extractants, schemes and extraction modes, using equipment that can significantly increase the release of radioactive and active substances. Several variants of extraction of raw materials by the proposed technology using different temperature regimes and time exposures were studied. We used 40, 50, 60, 70% aqueous solutions of ethyl alcohol as an extractant and determined that 60% aqueous solution of ethyl alcohol has a better ability with respect to the amount of flavonoid grass red tape to select the optimal method of extraction of plant raw materials. Quantitative determination of the amount of flavonoids in the herbs of the red ribbon is made in four Parallels and their metrological characteristics are calculated. According to the experimental data, the optimal number of particles was 2 mm.

**Keywords:** Flavonoid, avicularin, hyperoside, quercitrin, isoquercitrin, carotene, rutin.

**Introduction**

Knotweed, *Polygonum aviculare* L. is a plant that belongs to the families of alpine fleecflower blooming since June throughout the summer. Most of them ripe in the second half of August and the flowers are small and green. Knotweed has a wide range of biological activities. It is an annual herbaceous plant up to 30 cm in height, rough, up to 100 cm long. Pale scaly tracheas are observed in the nodes. It is most common in all the regions of Kazakhstan. They can be found on the roadsides, in plowed places, in places of walks, in river Sands, in courtyards, in gardens, arrays.

Grass is used for medicinal purposes. The upper part of plants up to 40 cm in length is stocked up when flowering with a sharp knife or wrapper. The storage term is 3 years [1].

The plant of the knotweed contains flavonoids (avicularin, hyperoside, quercitrin and isoquercitrin), tannins (up to 0.4%), vitamins (ascorbic acid, up to 0.9% by dry weight, carotene, vitamin K), silicic acid compounds (up to 4.5%), as well as resins, essential oils, drying, oils, sugar.

It is used for treating colds, bronchitis, pneumonia, pleurisy, cough cracks, bronchial asthma, pulmonary tuberculosis, insomnia, inflammation of the oral mucosa and red chin, gastritis, dysentery, liver, gastrointestinal tract, biliary tract, skin diseases, urinary tract burns, rheumatism, wounds.

One of the urgent tasks of modern pharmacotherapy is the problem of choosing medicines of natural origin for treating the hepatobiliary system. Socio-economic living conditions of the population and environmental pollution, the use of food containing preservatives, medication without a doctor's prescription leads to stress on the liver and contribute to the likelihood of the development of certain diseases.

Currently, the pharmaceutical market is increasing the range of medicines used in treating diseases of liver and biliary tract, of which there are more than a thousand items. The pharmaceutical industry in different countries releases hepatoprotective agents of knotweed for treating the liver pathology.

In recent years the approaches to the development of new products of plant materials have tended to develop resource-saving technologies, which is ensured by the use of various extractants, extraction schemes and modes, the use of equipment that allows significant increasing the yield of extractive and active substances.

The development and introduction into practice of a resource-saving method of extraction (due to the maximum depletion of raw materials), as well as preparing stable preparations based on plant extracts, is an urgent task.

The aim of the work is selecting the optimal modes of extraction of the knotweed plant material and developing a technology for producing a dry extract.

### Experimental part

At present there are a large number of various extraction schemes designed to increase the yield of active substances, to ensure maximum depletion of raw materials and enrichment of the extract with target biologically active substances (BAS). Among them there is a scheme of extraction of raw materials with purified water, proposed by Sargin B.V. et al., implemented on examples of raw materials rich in ascorbic acid (fruits of viburnum ordinary, *Viburnum opulus*), water-soluble phenolic compounds (weeping birch leaves, *Betulapendula* Roth).

The scheme proposed by Sargin B.V. et al., was taken as the basis of our studies with some modifications. In the variant proposed by Sargin B.V., the general conditions of the primary and subsequent heating of the extracted plant material are indicated in a fairly wide range of temperatures and heating time. The cooling conditions are not specified, only the time interval is given, however, the final temperature of the extracted mixture is not indicated.

### Results and discussion

In this regard, several variants of extracting raw materials according to the proposed technology with the use of various temperature regimes and temporary exposures are given in Table 1.

At stage 3 of the extraction process (initial heating of the mixture) there were selected the conditions for gradual increasing the temperature from 20 °C to 40 °C by 1 °C and 5 °C in various time intervals. Further, at stages 4, 6 (infusion of the raw material at a constant temperature), the possibility of extracting the raw material by holding the extractable mixture at a constant temperature, as well as the possibility of reducing the extraction time by eliminating this stage, was evaluated.

Table 1 – Selecting an optimal scheme of the raw material extracting

Stage No.	Mode	Scheme 1 (n=5)	Scheme 2 (n=5)	Scheme 3 (n=5)
1	The plant material was placed in a ceramic vessel.	The vessel is pre-aged in an oven-thermostat at 20 °C for 1 hour		
2.	Filled with the calculated volume of the extractant, taking into account the coefficient of absorption of raw materials			
3.	The plant material heating	Increasing the temperature of the extracted mixture from 20 °C to 40 °C within 2 hours	Increasing the temperature of the extracted mixture from 20 °C to 40 °C within 2 hours	Increasing the temperature of the extracted mixture from 20 °C to 40 °C for 2 hours
4.	Infusion of raw materials at a constant temperature	Holding at the temperature 40°C within an hour		None
5.	Subsequent heating	With a gradual increase in temperature to 90 °C within 2-6 hours	With a gradual increase in temperature to 90 °C within 8 hours (by 1°C every 10 min)	With a gradual increase in temperature to 90 °C within 8 hours (by 3°C every 30 min)
6.	Infusion of raw materials at a constant temperature	Holding at the temperature 90°C within an hour		Without holding
7.	Cooling	Within 6 hours	Gradual cooling from 90 °C to 20 °C within 10.5 hours (by 1 °C every 10 min)	At the end of the extraction time, the ceramic vessel was removed from the thermostat and cooled at room temperature.

At stage 5 of the extraction process, the possibility of gradually increasing the temperature of the extracted mixture from 40 °C to 90 °C for 8 hours, by 1 °C every 10 minutes and by 3 °C every 30 minutes, was evaluated. Later, at stage No. 7, the conditions for cooling the mixture were specified.

Thus, three variants of obtaining water extracts are considered: the first one according to the scheme described in the patent, the 2<sup>nd</sup> and the 3<sup>rd</sup> by the modified method. In addition, when performing the first option, cooling was performed without removing the extraction tank from the thermostat, but only turning it off.

In all three cases, the raw material of knotweed was used in the dry-air state, previously crushed, mixed and sifted from dust; the first stage for all three extraction schemes was left unchanged.

Table 2 – Comparative assessment of the extractable matters content and the leading BAS groups in the ready extract and the infusion of knotweed, (M ± m)

The studied indicator	Water extract Scheme 1 (n=5)	Water extract Scheme 2 (n=5)	Water extract Scheme 3 (n=5)	Infusion (n=5)
The content of extractives, %	26.4±0.3	28.6±0.4*.**,Δ	14.9±0.03	10.2±0.05
The content of polyphenolic compounds in terms of rut in, %	1.81±0.02	2.03±0.02*.Δ	1.4±0.01	0.9±0.02
The content of tannins in terms of gallic acid,%	13.07±0.02	14.01±0.02*.**,Δ	9.86±0.03	6.15±0.03

Note: \* - significant difference with infusion (p <0.05); \*\* - significant difference with the aqueous extract obtained according to scheme 3 (p <0.05); Δ - significant difference with the aqueous extract obtained according to scheme 1 (p <0.05)

Then, to select the optimal method of pressing out the plant raw material of knotweed, we used 40, 50, 60, 70% aqueous solutions of ethyl alcohol as an extractant and found that a 60% aqueous solution of ethyl alcohol has the best extracting ability with respect to the sum of bird's mountaineer (Table 3) [5].

Table 3 –Selecting the extractant concentration

Ethyl alcohol concentration, %	Optical density of the complex with AlCl <sub>3</sub> /HCl at 411 nm	Content of the total flavonoids in the extract, %
40	0.446	0.024 ± 0.001
50	0.460	0.025± 0.002
60	0.577	0.031± 0.001
70	0.419	0.022± 0.003
80	0.384	0.020± 0.004

The optimal size of the particles according to experimental data made 2 mm (see Table 4).

Table 4 –Raw material grinding degree

Grass particles size, mm	Optical density of the complex with AlCl <sub>3</sub> /HCl at 411 nm	Content of the total flavonoids in the extract, %
1	0.381	0.020± 0.003
2	0.598	0.032± 0.001
3	0.420	0.022± 0.002
4	0.345	0.018± 0.004
5	0.488	0.026± 0.001

From the experimental data it can be seen that as the extraction temperature increases, a proportional increase in product recovery is observed (Table 5) [5,6]. This indicates that the phenolic compounds in the grass of a bird highlander easier go into solution at higher extraction temperatures of 40-80 °C and at the ratio of raw material-extractant 1:15 (Table 6), the duration of extraction is 90 minutes (Table 7).

Table5 -Determining extraction temperature

Extraction temperature, °C	Optical density of the complex with AlCl <sub>3</sub> /HCl at 411 nm	Content of the total flavonoids in the extract, %
40	0.339	0.018± 0.0002
50	0.355	0.019± 0.0003
60	0.385	0.020± 0.0004
70	0.391	0.021± 0.0001
80	0.405	0.022± 0.0005

Table 6 –Selecting the extraction multiplicity and the raw material to extractant ratio

Raw material and extractant ratio	Optical density of the complex with AlCl <sub>3</sub> /HCl at 411 nm	Content of the total flavonoids in the extract, g
1:6	0.389	0.0615 ± 0.00004
1:10	0.236	0.0625 ± 0.00005
1:15	0.161	0.0637 ± 0.00003

Таблица 7 – Selecting the extraction duration

Heating time at the boiling temperature, min	Optical density of the complex with AlCl <sub>3</sub> /HCl at 411 nm
30	0.233 ± 0.0003
60	0.234 ± 0.0001
90	0.274 ± 0.0002

The quantitative determining of the total flavonoids in the grass of knotweed was carried out in four parallels and their metrological characteristics were calculated. The relative error of determining ( $\epsilon$ ) is 2-10% at the confidence level of 95[7,8]. The high relative error of a single determination ( $\epsilon_1$ ) is due to the fact that in differential spectrophotometry, aliquots of solutions containing substances absorbing in UV light are taken not only for the tested extract and standard substance, but also for comparison solutions.

### Conclusion

Thus, we carried out a set of studies for selecting the optimal conditions for extracting the knotweed medicinal plant raw materials and developing a technology for producing dry extract from plant raw materials. The optimal particle size, according to experimental data, is 2 mm, the ratio of raw materials-extractant is 1:15, the duration of extraction is 90 minutes with 60% ethyl alcohol.

The data presented in Table 2 indicate that the content of extractive substances in the aqueous extract obtained according to scheme 2 is 1.1 times and 1.9 times respectively higher than that in extracts obtained according to scheme 1 and 3 and the content of extractive substances is 2.8 times higher in the infusion. At the same time, the content of polyphenolic compounds in terms of rutin (%) in the extract obtained according to scheme 2 exceeded the content of those in the extracts obtained according to schemes 1 and 3 and in water extraction of the knotweed raw material is 1.1 times, 1.5 times and 2.3 times higher, respectively.

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### ҚЫЗЫЛ ТАСПА ӨСІМДІК ШИКІЗАТЫН СЫҒЫНДАУДЫҢ ОҢТАЙЛЫ РЕЖИМДЕРІН ТАҢДАУ ЖӘНЕ ҚҰРҒАҚ СЫҒЫНДЫ АЛУ ТЕХНОЛОГИЯСЫН ӘЗІРЛЕУ

**Аннотация.** Қызыл таспа дәрілік өсімдік шикізатын сығындаудың оңтайлы шарттарын таңдау бойынша және де өсімдік шикізатынан құрғақ сығынды алу технологиясын әзірлеуге зерттеулер кешені жүргізілді. Өсімдік шикізатынан жаңа препараттар жасау тәсілдерінде ресурс үнемдеуші технологияларды әзірлеу үрдісі

байқалады. Бұл әртүрлі экстрагенттерді, экстракцияның схемалары мен режимдерін қолданумен, экстрактивтік және әсер етуші заттардың шығуын едәуір дәрежеде арттыруға мүмкіндік беретін аппаратураны пайдаланумен қамтамасыз етіледі. Әр-түрлі температуралық режимдерді және уақытша экспозицияларды қолдана отырып, ұсынылған технология бойынша шикізатты экстрагирлеудің бірнеше нұсқасы зерделенді. Қызыл таспа өсімдік шикізатын сығындаудың оңтайлы әдісін таңдау үшін бізэкстрагент ретінде 40, 50, 60, 70% этил спиртінің су ерітінділерін пайдаланды және 60% этил спиртінің су ерітіндісі қызыл таспа шөбі флавоноидінің сомасына қатысты ең жақсы алатын қабілеттілікке ие екендігін анықтады. Қызыл таспа шөптеріндегі флавоноидтардың сомасын сандық анықтау төрт параллельде жүргізіледі және олардың метрологиялық сипаттамалары есептелінеді. Тәжірибелік деректер бойынша бөлшектердің оңтайлы мөлшері 2 мм, шикізат – экстрагент арақатынасы 1: 15, экстракция ұзақтығы 90 минут, экстрагент концентрациясы 60% этил спиртін құрады.

**Түйін сөздер:** Флавоноид, авикулярин, гиперозид, кверцитрин, изокверцитрин, каротин, рутин.

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## **ВЫБОР ОПТИМАЛЬНЫХ РЕЖИМОВ ОТЖИМА РАСТИТЕЛЬНОГО СЫРЬЯ ПТИЧЬЕГО ГОРЦА И РАЗРАБОТКА ТЕХНОЛОГИИ ПОЛУЧЕНИЯ СУХОГО ЭКСТРАКТА**

**Аннотация.** Проведен комплекс исследований по выбору оптимальных условий отжима лекарственного растительного сырья птичьего горца и разработке технологии получения сухого экстракта из растительного сырья. Наблюдается тенденция разработки ресурсосберегающих технологий в способах создания новых препаратов из растительного сырья. Это обеспечивается применением различных экстрагентов, схем и режимов экстракции, использованием аппаратуры, позволяющей в значительной степени повысить выброс радиоактивных и действующих веществ. Изучены несколько вариантов экстрагирования сырья по предложенной технологии с использованием различных температурных режимов и временных экспозиций. Птичий горец для выбора оптимального метода отжима растительного сырья использовали в качестве экстрагента 40, 50, 60, 70% водных растворов этилового спирта и определили, что 60% водный раствор этилового спирта обладает лучшей способностью относительно суммы флавоноида травы красной ленты. Количественное определение суммы флавоноидов в травах красной ленты производится в четырех параллелях и рассчитываются их метрологические характеристики. По опытным данным оптимальное количество частиц составило 2 мм, соотношение сырья – экстрагента 1: 15, продолжительность экстракции 90 минут, концентрация экстрагента 60% этилового спирта.

**Ключевые слова:** флавоноид, авикулярин, гиперозид, кверцитрин, изокверцитрин, каротин, рутин.

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