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Technology of Production and Study of Lilacs Flowers Thick Extract

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Abstract : The physicochemical and technological properties of lilac flowers have been studied to develop the optimal technology for extracts obtaining, as well as the efficiency of the extraction process, the forecasting and rationing of the finished product quality. It was established: humidity – 5.92%; bulk density – 0,23 g/cm^3; grinding ratio – 3-5 mm; specific gravity 1.40 g/cm^3; bulk mass – 0.50 g/cm^3; porosity – 0.59 g/cm^3; fenestration – 0.54 g/cm^3; the free volume of the layer – 0.81 g/cm^3; absorption coefficient of extragent: water – 4.4 ml / g; 40% ethanol – 3.9 ml/g; 70% ethanol is 3.1 ml/g.
The process of lilac flowers filtration extraction to obtain dense extract has been studied. The best yield of extractive substances from lilac flowers was observed under the following conditions of filtration extraction at laboratory conditions: the mass of the loaded raw material – 150.0 g; extragent – 50% ethanol; extraction temperature – 20±2 ºC; extraction rate – 3-4 ml/min; the ratio «raw material:extractant» (DER) – 1:5.
The obtained data can be used in the development of the technological specification, the quality specification of the extract, and also when calculating the material balance of the technological riles.
Keyword : lilac flowers, extraction, the ratio of «raw materials:extractant», extractives.

Introdution

Common lilac (*Syringa vulgaris* L.) belongs to the genus Lilacs (*Syringa*) Olive family (*Oleaceae*). Of all the species, the common lilac is the most widespread, presented currently in the gardens include 500 varieties of the most diverse color. The plant, especially the flowers, contains many useful substances due to which it is used in folk medicine as a wound healing, sweating, antipyretic, analgesic and antimalarial agent.

In scientific medicine, common lilac is practically not used. But in folk medicine, leaves and lilac flowers are recommended as a diaphoretic, anti-inflammatory, antipyretic and analgesic for febrile illnesses (flu, ARVI and malaria). At prolonged administration, the lilac is considered an effective remedy for epilepsy. In addition, lilac buds are recommended to use for the treatment of diabetes and urolithiasis. Infusion of fresh lilac leaves can be used for compresses in the treatment of wounds, panaricians and boils as an anti-inflammatory and wound-healing agent. Lilac is also recommended for the treatment of fever and diabetes[1,2].

Flowers contain farnesol, essential oil, traces of alkaloids; In flowers, leaves, buds – derivatives of coumarin, phenol glycoside syringin, flavonoids, tannins, resins, ascorbic acid, phytoncides[3,4,5].

For a more efficient process of extraction, predicting and normalizing of the extracts quality it is

necessary to know the technological properties of medicinal plant raw material[6].

The aim of the study was to develop a technology of lilac flowers extract obtaining for further use as an active pharmaceutical ingredient in a semisolid dosage form.

Material and Method

In studying the technological parameters of lilac flowers, pharmacopoeial and non-pharmacopoeial technological methods were used, which are described in the experimental part of given article.

When developing the technology of various medicinal forms from medicinal plant raw materials (MPRM) obtaining it is necessary to take into account its properties, which make it possible to extract the biologically active substances as much as possible in order to provide the necessary pharmacological effect.

In addition, the experimental data obtained are used to calculate the material balance in order to ensure an appropriate level of profitability of phytopreparation production.

Therefore, the determination of the technological parameters of the used plant material is topical.

Material

The object of the study were the flowers of lilac harvested during the flowering period in 2017. The flowers were released from the base of the inflorescence, dried first under the sun light, and then under a canopy in the air. The finished raw materials were stored in a dry place.

Physico-chemical and technological research

Determination of moisture content and content of extractives in medicinal plant raw materials was carried out according to the methodology of the State Pharmacopoeia of Ukraine (SPU)[7].

To study the optimal conditions for extracts from lilac flowers obtaining was determined the technological parameters of MPRM: humidity, bulk density; grinding ratio; specific gravity; bulk mass; porosity; fenestration; the free volume of the layer; absorption coefficient of extragent.

Determination of the specific gravity.

About 5.0 grams (accurately weighed) of grinded raw material was placed in 100 ml pycnometer poured purified water up to 2/3 of the volume and incubated on a boiling water bath for 1,5-2 hours, stirring occasionally to remove any air from the raw material. After this, the pycnometer cooled to 20 °C and diluted to volume with purified water. Thus determine the mass of the pycnometer with the raw materials and purified water. Previously determine the mass of the pycnometer with water.

The specific gravity is calculated by the formula:

$$d_Y = \frac{P \times d_w}{P + G - F}, \text{ g/cm}^3,$$

P – the mass of absolutely dry grinded raw material, g;
G – mass of pycnometer with water, g;
F – mass of the pycnometer with water and raw material, g;
d_W – the specific gravity of water, g/cm^3 (d_w = 0.9982 g/cm^3).

Determination of volume mass.

About 10.0 grams (accurately weighed) of grinded raw material quickly immerse in a graduated cylinder with water purified and determined the amount. By the difference in the volumes in the graduated cylinder determine the volume occupied by the raw material.

Volume mass is calculated by the formula:

$$d_0 = \frac{P_0}{V_0}, \text{ g/cm}^3,$$

P_0 – mass of unmilled raw material at a certain humidity, g;
V_0 – the volume occupied by raw material, cm^3.

Determination of bulk density.

In a measuring cylinder loaded grinded raw, shaking slightly to align the raw material and determine the total volume that it occupies. After this, raw material is weighed.

Bulk density is calculated as follows:

$$d = \frac{P_н}{V_н}, \text{ g/cm}^3,$$

P_H – mass of unmilled raw material at a certain humidity, g;
V_H – volume occupied by the raw material, cm^3.

The porosity of the raw material is calculated as follows:

$$P_C = \frac{d_V - d_0}{d_V},$$

d_V – the specific gravity of raw materials g/cm^3;
d_0 -– the volume mass of raw material, g/cm^3.

The fractional void volume of the layer is calculated using the formula:

$$P_V = \frac{d_0 - d_1}{d_0},$$

d_0– the volume weight of raw material g/cm^3;
d_1 – bulk weight of raw material g/cm^3.

The free volume of the layer is calculated by formula :

$$V = \frac{d_Y - d_H}{d_Y},$$

d_Y – the specific gravity of raw material g/cm^3;
d_H – bulk mass of raw material g/cm^3.

The volume and bulk density should be considered to determine the volume occupied by the dry and swollen raw material, external juice, which allow to set the ratio of raw material and the extragent, change in the volume of external and internal juices at raw swerlling, the concentration of substances in the internal and external juices at changing of their volumes. Bulk weight is a measure of volume occupied by a unit weight of the grindes raw material.

The filling ratio of the raw material is the amount of liquid required to fill the interstices between the particles of unit mass of dry, compacted material. Displacement ratio of raw material is the volume of liquid displaced by immersion therein of a unit mass of dry material[8].These indicators of the technological properties of raw materials are determined simultaneously.

Approximately 50,0 g of raw material placed in a stoppered cylinder of capacity 500 cm^3 and compacted until the termination of volume change, fixed volume, and poured into cylinder 400 cm^3 of extractant.The contents of the cylinder stirred for 2 minutes to remove air bubbles from the surface of the

particles of raw material, by the level of liquid in the cylinder fixed total volume of the extractant and raw material, then stoppered and allowed to stand for 24 hours to swell.The raw was then pressed by lattice, taking to the initial volume, poured extraction fixing the volume[8].

The absorption coefficient of the raw material is the volume of extractant absorbed by a unit mass of the raw material at its swelling. Internal juice formation factor- the volume of the internal juice, formed in a unit mass of raw materials, when dissolved in the extractant absorbed capillary moisture and extractives. Magnification volume by dissolving extractives – increase in the extractant by dissolving it in a unit mass of extractives.

100.0 g of lilac flowers were placed in pre-weighted cone. The raw material is compacted and weighed. After removing the plug when the valve is closed, the raw material is poured by extragent to form a liquid layer on the surface of the raw material 5 cm. Is pressed against the surface of the grate raw close diffuser cap and weighed. Maceration is carried out for 24 hours, stirring occasionally. Then poured into a pre-weighed extraction cylinder fixed volume cylinder with extraction weighed. 25 ml of the filtered extract was placed in a preweighed weighing bottle and weighed. Extraction was evaporated to dryness, adjusted to a constant weight at 100 °C for 3 hours[8].

Extraction Methods

When studying the process of biologically active substances from medicinal raw material extraction used several methods, one of which is the method of filtration extraction, first proposed by scientists of Borschagovsky CPP, m. Kyiv, Ukraine. Thus, by this method was got a dense extract of the lilac flowers[9].

To determine the optimal extraction conditions was obtained an extract with 50% ethanol. Each of the extracts was withdrawn fractionally with a step DER of 1:1.

The extraction process was carried out in a laboratory filtration extractor. The extractor was loaded with 150.0 g of grinded lilac flowers. The measuring cylinder was filled with ethanol and insisted 24 h. After this, the extraction process was started, setting the rate approximately 3-4 ml/min. Samples of the extract were collected separately with a step DER 1:1. The extraction process was carried out until a total extract of DER 1:10.

For each sample of the extract the main physico-chemical characteristics are determined: the content of dry residue and flavonoids (An, g et En, g) in separate portions of liquid extracts Vn, obtained by the corresponding extragent at the appropriate ratio «raw material:extract» content of dry residue and flavonoids (Bn, g et Fn, g); in the total extracts Vn+1, obtained by the corresponding extragent at the appropriate ratio «raw material:extract»; content of dry residue and flavonoids (Cn,% et Gn,%) in the total extracts Vn+1, obtained by the corresponding extractant at the appropriate ratio «raw material:extract» at the stage; yield of extractives (absolutely dry extract) and flavonoids (Dn, mg% et Ln, mg%) from the extracted raw material at each extraction stage with a suitable extragent at the appropriate ratio «raw material:extract». The methods and formulas for calculating these indicators are given in the article[10].

The quantitative determination of flavonoids in extracts obtained portion wise with a step DER 1:1 was performed by spectrophotometry.

Result and Discussion

Depending on the chemical composition of the medicinal plant raw material and type of used solvent, certain active or ballast substances pass into the extraction.

The solvent, which should be taken when determining extractives, is indicated in the relevant documentation for this type of raw material. Usually it is the same solvent that is used for the preparation of tincture or extract from this raw material. Table 1 shows the results of the raw materials technological parameters determining. Five parallel definitions and statistical data processing were carried out. The obtained results will be used in studies on the development of technology and the production chart for lilac extract.

Table 1. The results of lilac flowers technological parameters determining

№	Technological parameter	Data
1	Humidity, %	5.92±0.02
2	Grinding ratio, mm	3-5
3	Specific gravity, g/sm^3	1.34±0.01
4	Volume density, g/sm^3	0.48±0.02
5	Bulk density, g/sm^3	0.21±0.01
6	Porosity	0.56±0.03
7	Fenestration	0.52±0.04
8	Free layer volume	0.78±0.02
9	Extragent absorption coefficient, ml/g: – water – 40 % ethanol – 70 % ethanol	 4.2±0.01 3.8±0.02 3.1±0.01
10	Coefficient of dry raw materials filling, cm^3/g	3.42±0.02
11	Coefficient of raw materials displacement, cm^3/g	1.04±0.01
12	Coefficient of swollen raw materials filling, cm^3/g	1.92±0.01
13	Coefficient of raw material absorption, cm^3/g	2.76±0.02
14	Coefficient of formation of internal juice, cm^3/g	3.06±0.03
15	Coefficient of volume increase when dissolving extractives, cm^3/g	0.56±0.02

Thus, we studied the technological parameters of lilac flowers, which can be used in the development of the technological specification, the quality specification of the extract, and also in calculating the material balance of the technological rules.

According to the results of the experimental data Figure (1), the largest content of dry residue (extractive substances) is observed when 50% ethanol is used as an extractant. Therefore, further increase in the concentration of ethanol was impractical due to a decrease in the percentage of dry residue. A high extraction ability was shown in ethanol solutions of 40% and 60%.

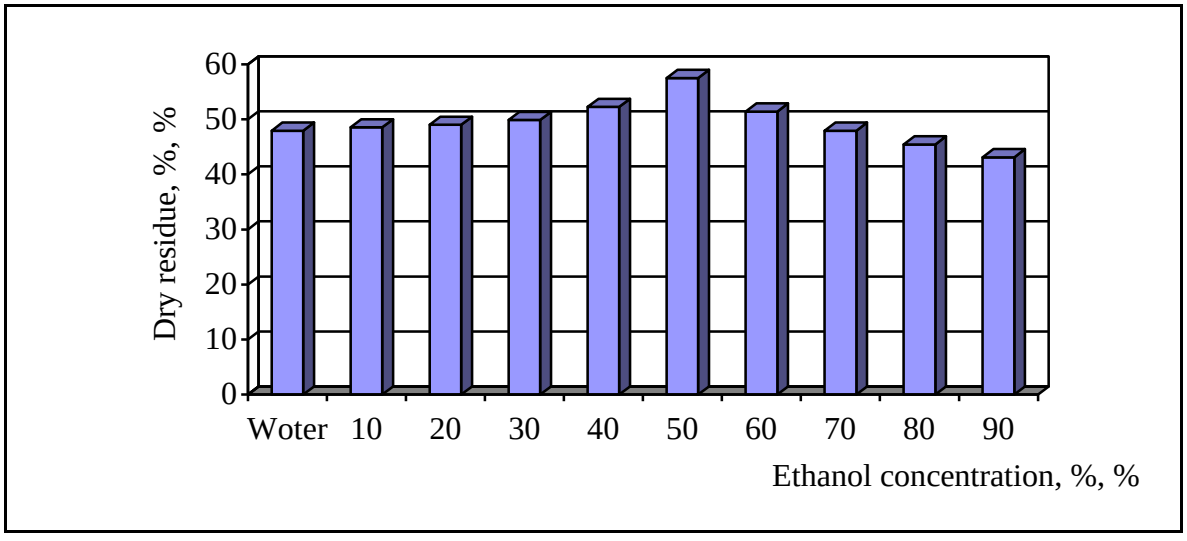

Figure 1. The amount of extractive substance depending on the extractant

The results of lilac flowers conditions extracting by the method of filtration extraction are given in Table (2).

In order to determine the optimum conditions for lilac flowers extraction for each of the experiments were constructed diagrams of the main criteria of extraction process efficiency on the change in the ratio «raw

material: extract» dependence Figure (2-5).

Table 2. The results of lilac flowers extraction

Index	Number of pouring									
	1	2	3	4	5	6	7	8	9	10
The volume of a separate extract portion, Vn	150	150	150	150	150	150	150	150	150	150
The volume of the total extract of Vn+1at the stage, ml	150	300	450	600	750	900	1050	1200	1350	1500
Dry residue content, ωn, g/100 ml	10.53	9.97	8.70	7.24	4.60	2.7	1.32	0.89	0.85	0.84
An, g	15.80	14.9	13.1	10.9	6.90	4.46	1.98	1.34	1.28	1.26
Bn, g	15.80	30.75	43.80	54.66	61.56	66.02	67.99	69.33	70.61	71.86
Cn, %	10.53	10.25	9.73	9.11	8.21	7.33	6.48	5.78	5.23	4.79
Dn, %	10.53	20.50	29.20	36.44	41.04	44.01	45.33	46.22	47.07	47.91
En, mg	19.95	13.85	9.36	3.74	1.08	0.75	0.68	0.54	0.48	0.48
Fn, mg	19.95	33.80	43.16	46.90	47.98	48.73	49.40	49.94	50.42	50.90
Gn, mg%	280.0	275.8	272.2	253.2	236.1	223.3	213.2	203.7	196.0	189.1
Ln, mg%	13.30	22.53	28.77	31.27	31.99	32.49	32.94	33.30	33.62	33.94

The obtained data indicate that the maximum number of extracting steps for obtaining the extract should be considered equal to 5, since a further increase in the portions of the extractant does not lead to a significant increase in the yield of the finished product.

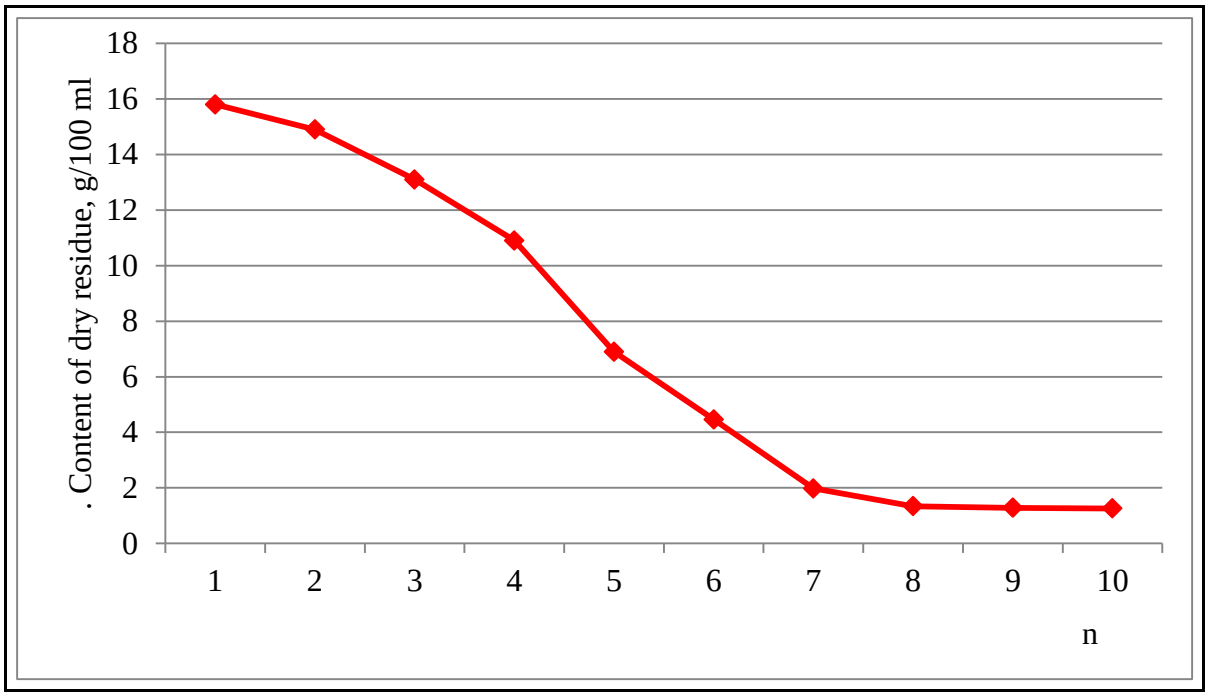

Figure 2. Content of dry residue in n-sample of liquid extract depending on number of pouring

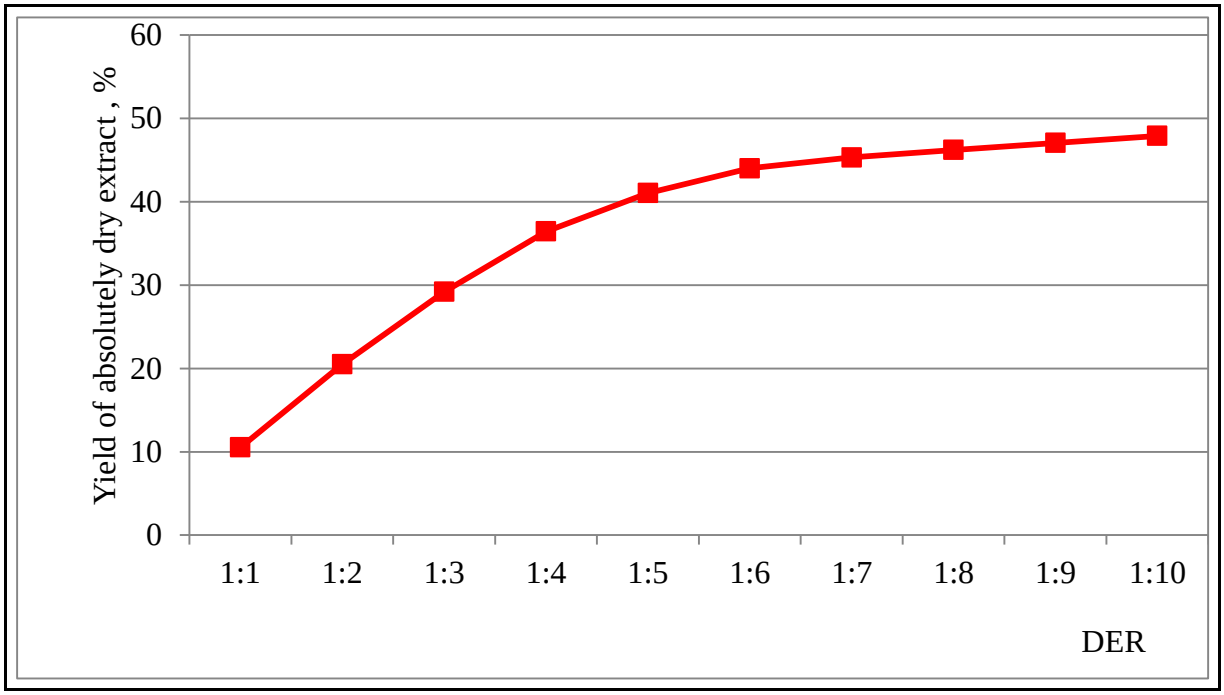

Figure 3. Yield of absolutely dry extract in dependence on DER

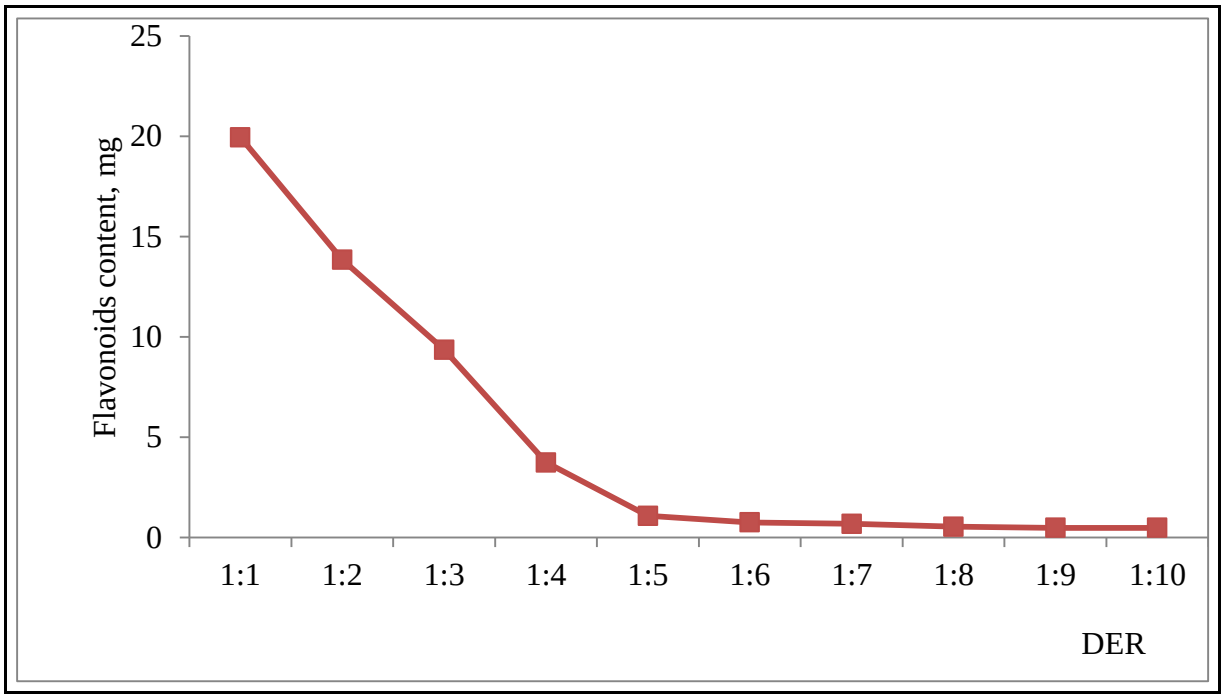

Fig. 4. Content of flavonoids in dependence on DER

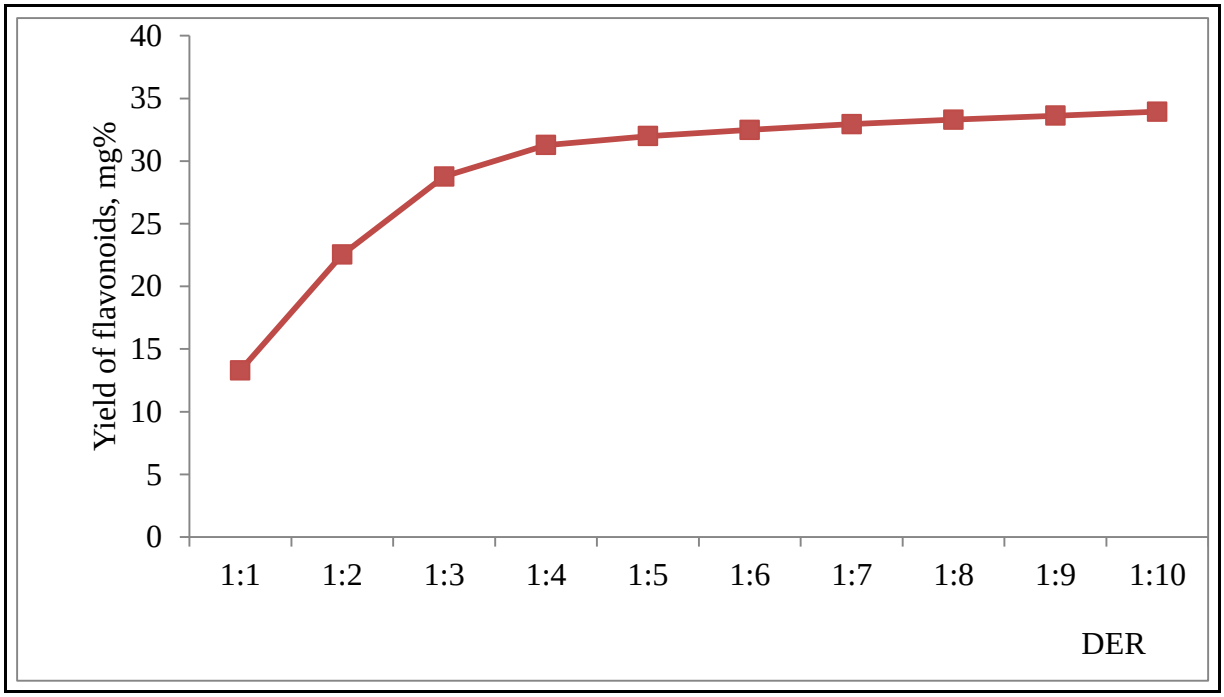

Fig. 5. Yield of flavonoids in dependence on DER

Thus, the best yield of the lilac flowers extract is observed under the following conditions of filtration extraction: the mass of the loaded raw material is 150.0 g; extragent – 50% ethanol; the extraction temperature – 20±2 °C; extraction rate – 3-4 ml/min; the ratio «raw material:extractant» (DER) – 1:5.

Conclusion

The physicochemical and technological properties of lilac flowers have been studied to develop the optimal technology for extracts obtaining, as well as the efficiency of the extraction process, the forecasting and rationing of the finished product quality. It was established: humidity – 5.92%; bulk density – 0,23 g/cm^3; grinding ratio – 3-5 mm; specific gravity – 1.40 g/cm^3; bulk mass – 0.50 g/cm^3; porosity – 0.59 g/cm^3; fenestration – 0.54 g/cm^3; the free volume of the layer – 0.81 g/cm^3; absorption coefficient of extragent: water – 4.4 ml/g; 40% ethanol – 3.9 ml/g; 70% ethanol is 3.1 ml/g.

The process of lilac flowers filtration extraction to obtain dense extract has been studied. The best yield of extractive substances from lilac flowers was observed under the following conditions of filtration extraction at laboratory conditions: the mass of the loaded raw material – 150.0 g; extragent – 50% ethanol; extraction temperature – 20 ± 2 ºC; extraction rate – 3-4 ml/min; the ratio «raw material:extractant» (DER) – 1:5.

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