Development of an express method for obtaining inulin

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SUMMARY

Inulin is a natural plant polyfructosan that is in demand in modern medicine, pharmacy and food industry. Available patented technologies for inulin production are characterized by low product yield and significant duration. The aim of the study was to develop an express method for the isolation and quantitative determination of inulin from dandelion roots. To accelerate the process of extracting biologically active substances from the roots of medicinal dandelion, as well as to increase the yield of inulin, it was decided to use an ultrasonic bath. By varying the parameters of the process, it was possible to select the optimal conditions for the extraction of inulin from the roots of dandelion medicinal under the conditions of sonication: the grinding of the raw material is 0.2-0.5 mm, the temperature is 80 ° C, the extraction ratio is 3, the duration of the extractions is 40 minutes. ultrasound frequency - 35 kHz, the ratio of raw materials and extractant 1 g per 10 ml. We also selected the optimal conditions for the purification of the polysaccharide complex of dandelion roots to obtain pure inulin. The proposed method allows to intensify the process of obtaining inulin from the roots of dandelion medicinal and to reduce the time spent on it to 6-7 hours, as well as to increase the product yield to 22.36% in terms of absolutely dry raw materials. The technique can be used for express analysis of the quality of dandelion roots, as well as for the industrial production of inulin from this type of raw material. The proposed method allows to intensify the process of obtaining inulin from the roots of dandelion medicinal and to reduce the time spent on it to 6-7 hours, as well as to increase the product yield to 22.36% in terms of absolutely dry raw materials. The technique can be used for express analysis of the quality of dandelion roots, as well as for the industrial production of inulin from this type of raw material. The proposed method allows to intensify the process of obtaining inulin from the roots of dandelion medicinal and to reduce the time spent on it to 6-7 hours, as well as to increase the product yield to 22.36% in terms of absolutely dry raw materials. The technique can be used for express analysis of the quality of dandelion roots, as well as for the industrial production of inulin from this type of raw material.

Key words: inulin, water-soluble polysaccharides, ultrasound, medicinal dandelion.

RESUME

Inulin is a natural plant polyphructosan, the importance of which in modern medicine and pharmacy, as well as the food industry, is difficult to overestimate. Having patented technologies for producing inulin, they are characterized by low product yield and significant duration. The aim of the study was to develop an express method for isolating and quantifying inulin from the roots of dandelion medicine. To accelerate the process of extracting biologically active substances from the roots of dandelion medicine, as well as increasing the yield of inulin, it was decided to use an ultrasonic bath. Varying the process parameters, it was possible to select the optimal conditions for extracting inulin from the roots of the dandelion drug in ultrasound treatment conditions: grinding of the raw materials 0.2–0.5 mm, temperature - 80 ° C, extraction rate - 3, extraction duration - 40 minutes, ultrasound frequency - 35 kHz, ratio of raw materials to extractant 1 g per 10 ml. The optimal conditions for purification of the polysaccharide complex of dandelion roots of the drug to obtain pure inulin were also selected. The proposed method allows intensifying the process of inulin production from dandelion roots and reducing the time spent on it to 6–7 hours, as well as increasing the product yield to 22.36% in terms of absolutely dry raw materials. The procedure can be used for express analysis of the quality of dandelion roots of medicinal, as well as in industrial production of inulin from this kind of raw material. The optimal conditions for purification of the polysaccharide complex of dandelion roots of the drug to obtain pure inulin were also selected. The proposed method allows intensifying the process of inulin production from dandelion roots and reducing the time spent on it to 6–7 hours, as well as increasing the product yield to 22.36% in terms of absolutely dry raw materials. The procedure can be used for express analysis of the quality of dandelion roots of medicinal, as well as in industrial production of inulin from this kind of raw material. The optimal conditions for purification of the polysaccharide complex of dandelion roots of the drug to obtain pure inulin were also selected. The proposed method allows intensifying the process of inulin production from dandelion roots and reducing the time spent on it to 6–7 hours, as well as increasing the product yield to 22.36% in terms of absolutely dry raw materials. The procedure can be used for express analysis of the quality of dandelion roots of medicinal, as well as in industrial production of inulin from this kind of raw material. 36% in terms of absolutely dry raw materials. The procedure can be used for express analysis of the quality of dandelion roots of medicinal, as well as in industrial production of inulin from this kind of raw material. 36% in terms of absolutely dry raw materials. The procedure can be used for express analysis of the quality of dandelion roots of medicinal, as well as in industrial production of inulin from this kind of raw material.

Keywords: inulin, water-soluble polysaccharides, ultrasound, dandelion medicinal.

INTRODUCTION

Medicinal dandelion (Taraxacum officinale FHWigg.) Is a perennial, ubiquitous in the European part of Russia, in particular in the Voronezh region, a herb about 30-40 cm high with a slightly branched taproot about 50-60 cm long. Dandelion roots have been used since ancient times in medicine in as a choleretic, antispasmodic, laxative, antitumor, antimicrobial, antifungal, antiviral, antiparasitic, antidiabetic agent that improves appetite and digestion. Widespread use is due to the rich chemical composition of this type of medicinal plant material, which is based on polysaccharides (up to 44% inulin), monosaccharides (fructose, glucose), triterpenoids, sesquiterpenoids, rubber, flavonoids, phenolcarboxylic acids, lactones (taraxoside), tannins,

Inulin - in demand in modern medicine, pharmacy and food industry natural plant polyfructosan. It is an active sorbent capable of removing various toxins from the human body - from heavy metals, radionuclides to excess low density lipoproteins. Inulin has prebiotic properties, contributing to the rapid restoration of intestinal normal flora, and also exhibits prokinetic, immunomodulatory, hepatoprotective activity. A large number of drugs and biologically active additives, including domestic ones, are produced on the basis of inulin [6, 7].

Inulin is obtained exclusively from plant objects by extraction with water followed by purification. The available patented technologies for the production of inulin are distinguished by a low yield of the product and a significant duration; the extraction of raw materials takes up to 3-5 days [8, 9]. The main industrial sources of inulin today are specially grown raw materials - chicory roots (up to 40% inulin), Jerusalem artichoke tubers (up to 18% inulin). At the same time, inulin obtained from chicory has contraindications. Other sources of inulin are also widely known, in particular, such accessible plant objects with significant raw materials in the territory of the Russian Federation, such as medicinal dandelion [1, 10, 11].

A highly effective method of accelerating the extraction process is a method based on the use of mechanical vibrations in the ultrasonic range. It was found that ultrasound with a frequency of 20–40 kHz can be used to extract coumarins, anthocyanins, phenolic glycosides from plants with a reduction in the extraction process by 1–2 orders of magnitude [12]. At the same time, not only a significant acceleration of the process of isolating biologically active substances from plant raw materials is noted, but also an increase in the yield of the main product [11, 13].

Objective of the study: development of an express method for obtaining inulin from dandelion roots medicinal using an ultrasonic bath.

MATERIALS AND METHODS

To intensify the process of extracting water-soluble polysaccharides (WSPP), an ultrasonic bath "Grad 40–35" was used. Purified water was used as an extractant; the rest of the process parameters were selected experimentally.

When developing the methodology, the roots of medicinal dandelion were used, harvested in the Anninsky district of the Voronezh region at the beginning of May 2016 in the natural biocenosis, far from highways, industrial enterprises and other economic activities, observing all pharmacopoeial rules for the preparation and drying of this type of medicinal plant material [14].

RESULTS

Initially, the optimal conditions for extracting the medicinal sum of the VSPR from the dandelion roots were selected using an ultrasonic bath. The temperature regime of extraction, the grinding of the raw material, the duration and frequency of extraction, the ratio of the raw material and the extractant, and the frequency of ultrasonic waves were experimentally selected. Each experiment was repeated three times. The results of the determinations are shown in table. 1, 2, 3.

The optimal conditions for extracting VSPR from the roots of medicinal dandelion in an ultrasonic bath are as follows: the size of the raw material is 0.2-0.5 mm, the temperature is 80 ° C, the extraction ratio is 3, the duration of the extractions is 40 minutes, the ultrasound frequency is 35 kHz, the ratio of the raw material is and extractant 1 g per 10 ml [11].

The precipitate obtained after the precipitation of water-soluble polysaccharides with ethanol contains impurities of pectin, some pigments, and some organic acids. To remove pectins, it was decided to interact with a calcium salt after dissolving the obtained precipitate of WSP in water, and to remove pigments with finely dispersed aluminum oxide [15]. After filtering the resulting precipitate of impurities under vacuum, it was decided to remove the remaining impurities by passing the solution through columns of cation and anion exchangers, for which we chose ion exchange columns with anion exchanger in the hydroxyl form AB178 and cation exchanger in the hydrogen form KU28.

Table 1

Content of VRPS (in terms of absolutely dry raw materials,%)
in the roots of medicinal dandelion by varying the fineness of the raw material and the temperature of the ultrasonic bath (with three-fold extraction for 40 minutes with a ratio of raw materials and extractant 1 g per 10 ml and an ultrasound frequency of 35 kHz)

Temperature, ° C	Grinding of raw materials, mm		
	0.2-0.5	0.5-1.0	1.0-2.0
60	18.39 ± 0.41	14.37 ± 0.41	12.03 ± 0.42
70	23.29 ± 0.38	20.81 ± 0.59	17.19 ± 0.39
80	32.89 ± 0.32	27.74 ± 0.38	21.37 ± 0.31

table 2

Content of VRPS (in terms of absolutely dry raw materials,%) in the roots of dandelion medicinal with varying the frequency and duration of extraction (with a crushing of raw materials 0.2-0.5 mm, the temperature of an ultrasonic bath 80 ° C with a ratio of raw materials and extractant 1 g per 10 ml and an ultrasound frequency of 35 kHz)

Duration extractions, min.	Extraction ratio		
	1	2	3
thirty	12.34 ± 0.36	19.39 ± 0.52	25.45 ± 0.43
40	16.95 ± 0.37	23.63 ± 0.41	32.89 ± 0.32
50	17.17 ± 0.46	24.54 ± 0.47	29.42 ± 0.42

Table 3

The content of VRPS (in terms of absolutely dry raw materials,%) in the roots of medicinal dandelion with varying the ratio of raw materials and extractants and the frequency of ultrasound (with three-fold extraction for 40 minutes, an ultrasonic bath temperature of 80 ° C, grinding of raw materials 0.2-0.5 mm)

Raw material ratio and extractant (g: ml)	Ultrasound frequency, kHz		
	15	25	35
1: 5	10.65 ± 0.34	14.63 ± 0.41	16.74 ± 0.58
1:10	23.74 ± 0.27	29.52 ± 0.21	32.89 ± 0.32
1:15	25.60 ± 0.40	24.38 ± 0.37	27.09 ± 0.52

Determination of the degree of purification of the finished product was carried out by thin layer chromatography, comparing with a standard sample of inulin (plates - Silufol, system - 55% ethanol, developer - solutions of resorcinol and sulfuric acid diluted with subsequent heating, Rf ~ 0.81) [16].

The complex of the carried out experimental works makes it possible to propose the following method of isolation and subsequent quantitative gravimetric determination of inulin in the roots of medicinal dandelion. An analytical sample of raw materials is ground to particles with a size of 0.5–1.0 mm. About 1 g (accurately weighed) of the crushed raw material is placed in a flask with a capacity of 50 ml, 15 ml of purified water heated to the boiling point are added, placed in an ultrasonic bath with a frequency of 35 kHz at a temperature of 80 ° C, extracted for 15 minutes. The extraction is repeated 2 more times, adding 15 ml of water. Aqueous extracts are combined and filtered through 3 layers of gauze with a cotton swab placed in a glass funnel 5 cm in diameter.Precipitation is carried out with a threefold amount of 95% ethanol, mix, cooled in a freezer at a temperature of 18 ° C for 1 hour. Then the contents of the flask are filtered through a pre-dried and weighed ashless paper filter inserted into a POR 16 glass filter with a diameter of 40 mm under vacuum at a residual pressure of 0.4–0.8 atm. The resulting precipitate is dissolved in 10 ml of purified water heated to 80 ° C, 5 drops of a 50% solution of calcium chloride and 0.5 g of fine powder of aluminum oxide are added, kept for 20 minutes, then filtered under vacuum at a residual pressure of 0.4-0, 8 atm. The resulting filtrate is sequentially passed through ion-exchange columns with anion exchanger in hydroxyl form AB178 and cation exchanger in hydrogen form KU28, taking into account the capacity of ion-exchange resins to pH 6.5–7.5 of the eluate and inulin purity of 97%.

dried ashless paper filter under vacuum at a residual pressure of 0.4–0.8 atm. The filter cake is sequentially washed with 15 ml of a solution of 95% ethyl alcohol in purified water (3: 1), 10 ml of a mixture of ethyl acetate and 95% ethyl alcohol (1: 1). The filter with the precipitate is dried first in air, then at a temperature of 100–105 ° C to constant weight.

The inulin content in terms of absolutely dry raw materials is calculated using the standard formula: X = (m_1 m_2) - 100 - 100 / m - (100 W),

where m_1 - weight of the dried filter, g; m_2 - weight of the dried filter with sediment, g; m - weight of raw materials, g; W is the loss in the mass of the raw material upon drying,%.

This method makes it possible to intensify the process of obtaining inulin from the roots of medicinal dandelion and to reduce the time spent on it to 6–7 hours, as well as to increase the product yield to 22.36% [17].

Metrological characteristics are given in table. 4. The relative error of the proposed methodology at a confidence level of 95% is 1.64%.

Table 4

Metrological characteristics of the method for the quantitative determination of inulin in the roots of medicinal dandelion

N	f	X	S^2	S	S_x	P,%	$t_{(P, f)}$	Dx	e,%
ten	nine	22.36	0.0055	0.0744	0.0304	95	2.2622	0.19	1.64

CONCLUSION

An express method has been developed for the isolation and quantitative determination of inulin from the roots of dandelion medicinal, which can be used for quality control of this type of raw material and for the industrial production of inulin. The optimal conditions for the extraction of VSPR from the roots of medicinal dandelion were selected: the grinding of the raw material is 0.2-0.5 mm, the temperature is 80 ° C, the extraction rate is 3, the duration of the extractions is 40 minutes, the ultrasound frequency is 35 kHz, the ratio of raw material and extractant is 1 g per 10 ml. Also, the optimal conditions for purification of the polysaccharide complex of dandelion roots were selected, which are reduced to the precipitation of pectins with calcium salts, absorption of pigments by aluminum oxide, followed by passing the extract through ion-exchange columns.

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