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DEVELOPMENT OF INDUSTRIAL TECHNOLOGY OF INULIN PRODUCTION FROM DANDELION ROOTS (*Taraxacum officinale* WIGG.)

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A method of extraction with forced circulation of extractant was used for exhaustive extraction of inulin from dandelion roots (*Taraxacum officinale* Wigg.). Inulin samples were obtained by three methods that were evaluated by inulin yield and purity. The results were used to develop an industrial technology for inulin production from dandelion roots that consisted of extraction of roots with water at 80°C, circulation of the extractant at a rate of 90 L/h, concentration of the extract to 70% dry residue, precipitation of inulin by adding 95% EtOH in a concentrate(EtOH volume ratio of 1:4, and sedimentation for 8 h. Inulin was produced in 30.0% yield of the raw material mass with a purity of at least 80%. The following characteristics were determined: mass loss on drying ≤5.0%; ash content ≤2%; heavy metals ≤0.001%; inulin content ≥80.0%. The resulting product complied with standards for the contents of toxic elements, pesticides, and radionuclides and was safe for use in the pharmaceutical and food industries.

Keywords: dandelion (*Taraxacum officinale* Wigg.), inulin, extraction, precipitation, technology.

Inulin is an effective prebiotic. Daily use of it significantly increases the number of bifidobacteria in the intestines. It also improves glucose utilization; stimulates the synthesis of protein, cholesterol, and bile acids; and helps to detoxify toxic substances in the intestines and blood [1 – 3].

Inulin has potential applications in pharmaceutics as an additive to tablet dosage forms to increase the dissolution rate of the active ingredient and to target delivery to the colon and as a vaccine stabilizer and adjuvant. Inulin also increases the stability of proteins, which was demonstrated using hemagglutinin from flu vaccine as an example. The hemagglutinin was well preserved in the presence of inulin after freeze drying [4, 5].

Inulin is a biopolymer, does not have irritating and allergenic action, and is readily absorbed and utilized *in vivo* in humans. Inulin forms a cream-like gel upon dissolution in H_2O. This is responsible for the ability to use it as a thickener in the manufacturing of soft dosage forms, blood replacements, and emulsion stabilizers [6].

Inulin was included in the United States Pharmacopeia (USP, Inulin, CAS RN® 9005-80-5; UNII: JOS53KRJ01) [7]. Combined drugs containing inulin are registered on the Uzbekistan pharmaceutical market and include the eubiotic preparation Rotabiotic [caps. N10 (1×10), N20 (2×10); blisters; Reg. No. DV/X 09945/06/22; caps. N6, N12 (blisters), N25 (vials); Reg. No. DV/X 04481/06/18; Rotapharm Ltd. (Great Britain)]; the antidiarrhea agent Rotabiotic Baby [powder for preparing solution for internal use, 3 g N10 (packet); Reg. No. DV/X 04481/06/18]; and the probiotic Opefera [caps. N10, N20 (blisters); Reg. No. DV/X 09433/11/21; World Medicine Ltd. (Great Britain)] [8].

Inulin is produced on industrial scales mainly from chicory roots, Jerusalem artichoke tubers, and agave. However, the increasing demand for inulin has prompted a search for promising sources of this polysaccharide. One such source is dandelion (*Taraxacum officinale* Wigg., Asteraceae), a perennial herbaceous plant, the roots of which are used as the raw material. The chemical composition of dandelion is varied, i.e., inulin (up to 40%), sugar (up to 20%), malic acid

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(0.25%), rubber (2 – 3%), ascorbic acid, resins, fatty oil, poorly studied triterpenes, alcohols, and sterols [9 – 11].

Many methods for producing inulin that differ in the type of pretreatment of the plant raw material, nature of extractant, and methods of extraction, purification, and drying have now been developed. The main technology includes extraction using diffusion of hot water and purification and drying of the extract to produce pure inulin powder [12 – 15]. Methods for extracting inulin at high temperatures not only by water but also salt solutions and at low temperatures by organic solvents are known [15 – 21].

Purification of the inulin-containing plant extract uses activated carbon, H_3PO_4, $CaCO_3$, ion-exchange resins, and chromatographic and filtration methods [15, 19, 20, 22, 23]. Russian scientists proposed a method for precipitating inulin with EtOH of various concentrations [18]. According to published data, sublimation and spray drying and drying at room temperature are widely used to manufacture inulin powder [24 – 26].

Considering the above, the aim of the present research was to develop the industrial technology for producing inulin from dandelion roots.

EXPERIMENTAL PART

The experiments used raw material with an inulin content of 30.6%. The raw material was harvested on Sept. 10 – 20, 2022, in Bostanliq District, Tashkent Region.

The content of dry residue in solutions was determined on a Pocket refractometer (Japan).

The inulin content was determined using a spectrophotometric method [27].

Three samples of inulin from dandelion roots were obtained by us using various methods. Extraction with forced circulation of the extractant (purified H_2O) was used in all tests for efficient extraction of inulin.

Sample I. Raw material (10.0 kg) and H_2O (60.0 L) were loaded into an extractor with a sleeve for supplying steam and an installed pump for circulation of the extractant. Steam was fed into the extractor sleeve. The extraction was performed at 80°C with extractant circulation rate 90 L/h. The extractant was circulated by withdrawing it from the bottom of the extractor and feeding it from above as a shower. The first extraction lasted 4 h. Then, the extract (40.0 L) was drained. A new portion of purified H_2O (40.0 L) was added to the extractor. The extraction lasted for 3 h at 80°C with the extractant circulating at 90 L/h. The second extract was drained. A third extraction was performed analogously to the second. The obtained extracts were cooled, combined, filtered in a Nutsche filter filled with belting. The filtered extract was concentrated (vacuum (0.8 to (0.6 kgs/cm^2, 60°C) to a content of 10% dry mass. Then, the concentrate was dried in a nozzle-type spray dryer (Anhydro No. 2) with a drying-agent temperature at the inlet of 170 – 180°C; at the outlet, 85 – 90°C; solution feed rate, 5 L/h; solution feed pressure, 0.2 MPa.

Sample II. Raw material (10.0 kg) was loaded into an extractor and extracted (3×) with H_2O, standing for 5 h at room temperature. The temperature was 18 – 22°C during the extraction. Then, the rinsed raw material was extracted (3×) with H_2O at 80°C for 3 h with extractant circulation rate 90 L/h. The extracts were combined, filtered, concentrated, and dried under the drying conditions for sample I.

Sample III. The extract obtained analogously to sample I was concentrated to a content of 70% dry residue. The concentrate was vigorously stirred and treated with 95% EtOH in a 1:5 concentrate(EtOH volume ratio. The mixture was left for 6 h. The precipitate was filtered off and dried in a drying cabinet at 60°C.

Experiments to determine the ratio of EtOH and H_2O to precipitate inulin were conducted as follows. Raw material (12 kg) was extracted under conditions analogous to those for producing sample I. The obtained extracts were combined, filtered, and divided into 10 portions. Each portion was concentrated to a different content of dry residue. Inulin was precipitated from each concentrate by adding various amounts of 95% EtOH. The aqueous EtOH solution in each experiment was left for 12 h. The precipitates were filtered off and dried in a drying cabinet at 60°C.

The time for precipitation was determined by concentrating the next five portions of extract to a content of 70% dry residue, adding 95% EtOH with stirring in a 1:4 volume ratio, and leaving the mixtures for various times. When the time expired, the precipitates were filtered off and dried as in the preceding experiment.

Physicochemical parameters such as mass loss on drying, ash content, and heavy metal content were determined using methods given in the *State Pharmacopoeia of the Republic of Uzbekistan* [28].

Determination of safety parameters. Samples were prepared for mineralization according to GOST 26929(94 “Raw material and food-stuffs. Preparation of samples. Decomposition of organic matter for analysis of toxic elements” [29]. Mercury content was determined according to GOST 26927(86 “Raw material and food-stuffs. Methods for determination of mercury” [30]. Arsenic content was determined according to GOST 26930(86 “Raw material and food-stuffs. Method for determination of arsenic content” [31]. The contents of Cd and Pb were determined using an inversion voltammeter.

RESULTS AND DISCUSSION

Based on the literature, inulin is extracted from samples by hot water (80°C). Inulin samples were obtained using three methods. The first method was based on direct drying of the aqueous extract. The second method used two extractions of the plant raw material with low-molecular-mass polysaccharides removed in the first step to enrich the raw

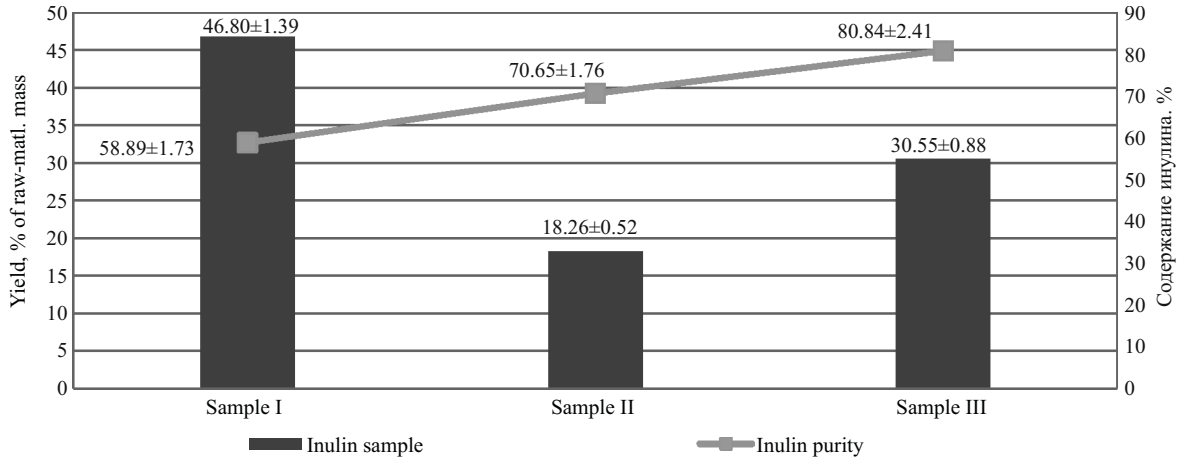

Fig. 1. Yield and purity of inulin samples from dandelion roots as functions of production method.

TABLE 1. Effect of Condensation of Extract and Amount of Added 95% EtOH on Inulin Yield and Purity

Expt. No.	Content of dry residue in concentrated extract, %	Amount of added 95% EtOH in concentrate(EtOH volume ratios	Inulin yield, % of raw-matl. mass	Inulin purity, %
1	60	1:4	22.75 ± 0.46	72.94 ± 2.19
2	60	1:5	26.12 ± 0.53	76.55 ± 2.26
3	60	1:6	29.85 ± 0.74	80.07 ± 2.38
4	70	1:3	27.68 ± 0.55	79.56 ± 2.32
5	70	1:4	30.14 ± 0.85	81.38 ± 2.40
6	70	1:5	30.48 ± 0.87	80.79 ± 2.44
7	70	1:6	31.65 ± 0.92	75.36 ± 2.25

material before inulin was subsequently extracted by hot water. The third method purified inulin via precipitation by EtOH. The inulin yield and purity were analyzed for each method (Fig. 1).

The experimental results in Fig. 1 show that the inulin yield for the first method was 90.07% of the raw-material content; for the second method, 42.16%; and for the third method, 81.47%. The results indicated that part of the inulin was lost if the raw material was rinsed with cold water. Sample III had a higher inulin content than the other samples. The results also showed that ~9% of the inulin was lost via precipitation by EtOH. Thus, the method for producing sample I was selected for producing inulin from dandelion roots. It consisted of extraction of the raw material at 80°C with extractant circulating at a rate of 90 L/h, filtration, combination of the extracts, concentration, and precipitation of inulin by adding EtOH.

TABLE 2. Effect of Standing Time of Aqueous EtOH Solution on Inulin Yield and Purity

Expt. No.	Standing time, h	Inulin yield, % of raw-matl. mass	Inulin purity, %
1	6	28.44 ± 0.71	81.48 ± 2.43
2	8	30.25 ± 0.74	81.07 ± 2.42
3	10	30.48 ± 0.76	80.82 ± 2.40
4	12	30.94 ± 0.79	80.30 ± 2.38
5	14	31.57 ± 0.81	78.42 ± 2.34

The efficiency of the inulin precipitation depended on factors such as the thickness of the extract, the amount of added EtOH, and the standing time. The effect of the ratio of aqueous solution and EtOH on inulin precipitation in the first step was studied (Table 1).

TABLE 1 shows that the amount of precipitated inulin was directly proportional to the amount of added EtOH. Simultaneously, increasing the amount of EtOH also led to a reduced inulin content in the final product. The desired results were obtained in experiments Nos. 3, 5, and 6. Considering the usage of EtOH in the precipitation, the conditions used in test No. 5 were selected. Thus, concentration of the obtained aqueous extract to a content of 70% dry residue

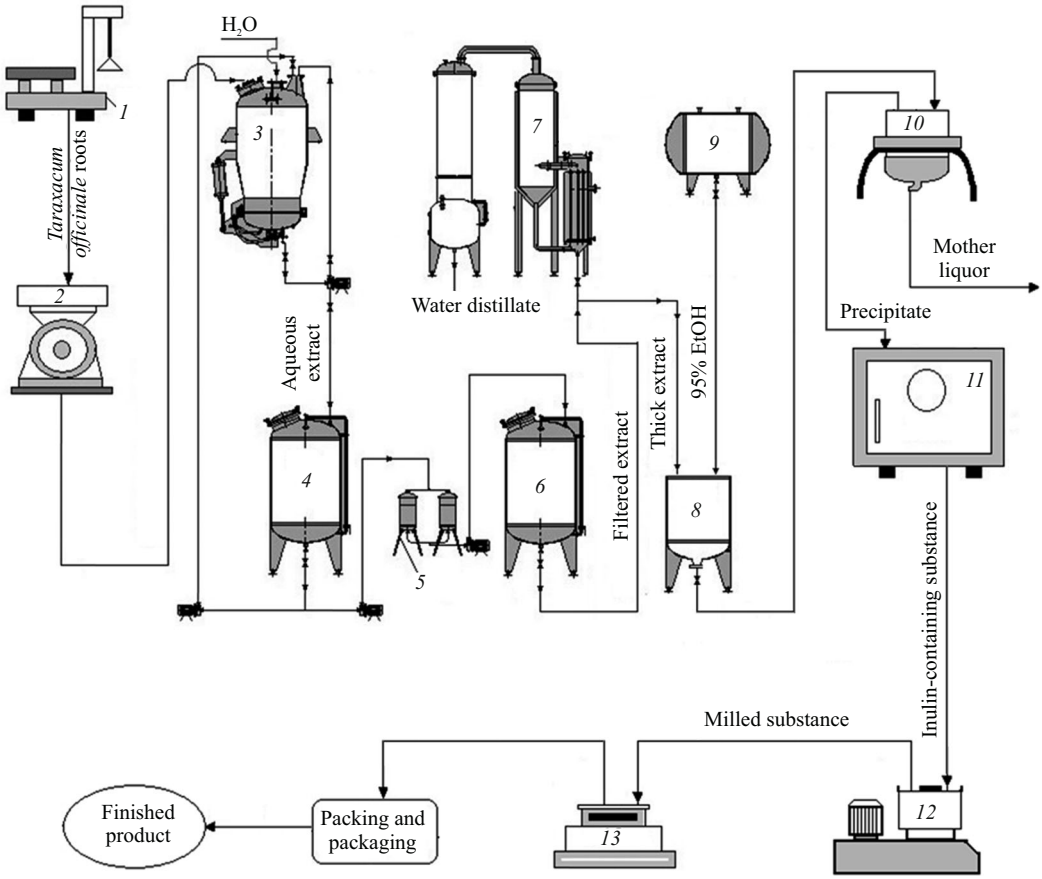

Fig. 2. Diagram of apparatus for inulin production from dandelion roots: balance (*1*), hammer mill (*2*), extractor (*3*), collectors (*4*, *6*), press filter (*5*), vacuum evaporator (*7*), reactor (*8*), feed tank (*9*), Nutsche filter (*10*), drying cabinet (*11*), knife mill (*12*), sieve (*13*).

and addition of EtOH in a 1:4 volume ratio was recommended.

The next stage of the research on selection of the optimal conditions for inulin precipitation studied the effect of the standing time of the aqueous EtOH solution of inulin on the yield and quality of the target product (Table 2).

The results in Table 2 indicated that inulin was precipitated most 8 h after the addition of EtOH. Storing the aqueous EtOH solution for >12 h led to precipitation of ballast substances that lowered the quality of the target product.

The experimental results allowed the development of the industrial technology for producing inulin from dandelion roots (Fig. 2).

Technological description. Air-dried dandelion roots (105.0 kg) with 6% moisture content were weighed on a balance (1) and milled in a pneumatic discharge hammer mill (2). The degree of milling of the raw material was 80% passage through a 6-mm sieve to produce ground dandelion roots (100.0 kg) with 30.6% inulin content. The milled raw material was manually packed into bags and transferred to the stage for inulin extraction from it.

A 1000-L extractor (3) with a sleeve for supplying steam and an installed pump for circulation of extractant was loaded with milled raw material (100.0 kg). Purified H_2O (600.0 L) was poured into the extractor. Steam was fed into the extractor sleeve. The pump was turned on to circulate the extractant at a rate of 90 L/h. The extraction was performed at 80°C for 6 h, after which the aqueous extract (400 L) was decanted into a collector (6). The extractor was filled with a new portion of purified H_2O (400 L). The second extraction was analogous to the first. A third extraction was performed in the same manner. The combined extracts from the collector (6) were filtered through a press-filter (5) filled with belting using inert-gas pressure into a collector (7).

The pulp was unloaded through the lower door of the extractor (3) into a cart and sent for disposal.

The EtOH extract (1200.0 L) from the collector (6) was fed in portions (100 – 120 L) into a vacuum-circulation evaporation apparatus (7). The evaporation was performed at 60 – 70°C in a vacuum of 0.04 – 0.08 MPa (0.4 – 0.8 kgs/cm^2) to 70% dry residue. The concentrate was drained into a reactor (8). EtOH in a 1:4 concentrate(EtOH volume ratio was fed with stirring from the feed tank (9). The aqueous EtOH inulin solution was left for 8 h. The precipitate was filtered off through a Nutsche filter (10). The precipitate was rinsed with 95% EtOH (5.0 L) at the end of the fil-

TABLE 3. Characteristics of Inulin from Dandelion Roots

No.	Determined parameter	Determined parameter	Results
1	Appearance	Amorphous white powder with cream tint and light-brown inclusions	Complies
2	Solubility	Freely soluble in hot H_2O, poorly soluble in cold H_2O	Complies
3	Identity	1. Thymol solution in H_2SO_4 added to aqueous inulin solution (1 – 2 mL) should give an orangish-red color. 2. α-Naphthol solution (20%) in EtOH and conc. H_2SO_4 added to aqueous inulin solution (1 – 2 mL) should give a purplish-pink color	Complies
4	Mass loss on drying	≤5%	3.56 ± 0.11 %
5	Heavy metals	≤0.001%	Complies
6	Total ash	≤2%	1.47 ± 0.04 %
7	Microbiological purity	Total number of aerobic microorganisms allowed in dry extract (1 g) $\leq 10^4$ CFU; of yeast and mold fungi, $\leq 10^2$ CFU; of enterobacteria resistant to bile, $\leq 10^2$ CFU; without *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Escherichia coli*. *Salmonella* bacteria should be absent in dry extract (25 g)	Complies
8	Inulin purity, %	≥80.0%	81.60 ± 2.42

TABLE 4. Contents of Toxic Substances in Inulin from Dandelion Roots

No.	Parameters	Allowed levels, mg/kg, ≤	Found, mg/kg
1	Toxic elements:		
	Pb	1.0	0.4 ± 0.01
	As	0.2	0.07 ± 0.0017
	Cd	0.1	0.01 ± 0.0002
	Hg	0.03	0.007 ± 0.0001
2	Pesticides:		
	hexachlorocyclohexane (α-, β-, γ-isomers)	0.5	Not detected
	DDT and its metabolites	0.02	Not detected
	heptachlor	Not allowed	Not detected
	aldrin	Not allowed	Not detected
3	Radionuclides, Bq/kg:		
	Cs-137	200	Not detected
	Sr-90	100	Not detected

tration to remove traces of water. The precipitate was loaded into a drying cabinet (11), where it was dried at 60°C for 3 h.

The material being dried was stirred every 15 – 20 min for efficient drying.

The dried material was milled in a mill (12) and sieved through a 0.5-mm sieve (13). The finished inulin was packed and labeled to produce 30.0 kg of packaged inulin.

The organoleptic and physicochemical parameters of the inulin from dandelion roots were established based on experimental data (Table 3).

The safety parameters became important because the inulin-containing preparations were intended for use in the manufacturing of pharmaceuticals. Therefore, the contents of toxic elements, radionuclides, and residual amounts of pesticides in the inulin from dandelion roots obtained via the proposed technology were studied (Table 4).

TABLE 4 shows that the contents of toxic substances in the inulin from dandelion roots met the applicable requirements.

Thus, extraction by H_2O at 80°C with extractant circulating at 90 L/h was proposed for extraction of inulin from dandelion roots. Precipitation of inulin from EtOH was proposed for purification of the extract from ballast substance. Concentration of the extract to a content of 70% dry residue and precipitation of inulin by adding 95% EtOH to a 1:4 concentrate(EtOH volume ratio followed by standing for 8 h was found to produce a product with consistent quality and yield.

An industrial technology for manufacturing inulin from dandelion roots was developed based on the results.

The organoleptic and physicochemical parameters of inulin from dandelion roots that was produced by the developed technology were found. The inulin content was ≥80%. Chemical analysis of inulin produced by the developed technology showed that the content of toxic substances in the product met the requirements of the standards.

Conflict of interest

We declare no conflict of interest.

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Contributions of authors

KNN performed the chemical, analytical, and technological research; ESK performed the statistical processing of the results; RMKh performed the technological research. All authors contributed equally to the writing of the article and interpretation of the research results.

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