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PHYSICOCHEMICAL CHARACTERISTICS OF INULINS FROM NATURAL SOURCES

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Abstract
Inulin-type fructans are used as prebiotic dietary fibres and as texturising, fat-replacing and encapsulating agents, but their technological behaviour depends strongly on botanical origin and isolation conditions. Onopordum acanthium L. (Scotch thistle, Asteraceae) is a widespread wild plant in Bulgaria, but data on its fructans are scarce. The aim of this study was to isolate inulin from O. acanthium flower heads by microwave-assisted (MW) and classical hot-water (CL) extraction and to compare its physicochemical and functional properties with those of commercial chicory inulin. The isolates were characterised for yield, purity (HPLC-RID), degree of polymerisation (DP, HPLC-SEC), colour (CIELAB), particle morphology (SEM), wettability, swelling capacity, angle of repose, water- and oil-holding capacity and hygroscopicity. The classical extraction gave a higher yield (6.56 vs. 4.67 %), whereas the 5-min microwave treatment resulted in higher purity (32 vs. 20 %) and longer chains (DP 30 vs. 20). Both isolates are long-chain inulins with DP exceeding that of the commercial standard (DP 12). Compared with chicory inulin, O. acanthium inulins showed 12–13 times higher water-holding (4.94–5.32 g/g) and 18–20 times higher oil-holding capacity (7.36–7.85 g/g), high swelling capacity (8.79–10.96 cm^3/g), slower wetting, moderate hygroscopicity (5.1–5.2 %) and loose, spongy particle morphology. O. acanthium is a promising alternative source of long-chain inulin for texturising and fat-replacing applications.

Keywords: *Onopordum acanthium*, inulin, degree of polymerization, microwave-assisted extraction, functional properties, SEM.

INTRODUCTION

Inulin is a polydisperse fructan composed mainly of β(2→1)-linked fructofuranosyl units, usually terminated by an α(1→2)-linked glucose residue. It is not hydrolysed in the upper gastrointestinal tract and in the colon selectively stimulates the growth of bifidobacteria and lactobacilli, which is the basis of its prebiotic effect [1]. In foods, inulin acts as soluble dietary fibre, sugar and fat replacer, texturiser, stabiliser and carrier for encapsulation of bioactive compounds [2, 3]. Its functionality is governed largely by chain length: short-chain fractions (DP ≤ 10, oligofructose) are more soluble and slightly sweet, whereas long-chain inulins (DP ≥ 23) are less soluble and form particle gels suitable for fat replacement [2, 3].

Industrial inulin is obtained mainly from chicory roots and Jerusalem artichoke tubers, and its DP depends on the source – 6–10 for Jerusalem artichoke, 10–14 for chicory and 13–20 for dahlia [3]. The growing demand for inulin has stimulated the search for alternative plant sources [4]. Even high-purity inulins from agave, chicory and Jerusalem artichoke differ markedly in solubility and water-holding

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capacity (1.68–5.40 g/g) [5], so the physicochemical and technological properties have to be determined for every new source. Wild plants of the Asteraceae family are of particular interest – burdock roots contain fructans with DP 20–24 [6], and inulin from dandelion roots isolated by "green" techniques showed DP 18–25 and a specific functional profile [7].

Onopordum acanthium L. (Scotch thistle, Asteraceae) is a biennial plant widespread in Bulgaria. It is used in folk medicine as a diuretic and cardiotonic agent, and the roots, shoots and young inflorescences are consumed as an artichoke substitute [8]; species of the genus are also used as milk coagulants in cheese making [9]. The plant contains sesquiterpene lactones, flavonoids (apigenin, luteolin, quercetin) and triterpenes [8], and flower extracts are rich in chlorogenic acid and inhibit α-glucosidase [10]. Data on its carbohydrates are scarce: flower heads of the related *O. tauricum* contain 7.90 g/100 g dw total fructans and 4.50 g/100 g dw inulin [11], and 1.35–2.9 g/100 g dw inulin has been found in *O. acanthium* flower heads [12]. However, inulin has not been isolated from this species and its DP and functional properties have not been reported.

The aim of the present study was to isolate inulin from *O. acanthium* flower heads by microwave-assisted and classical extraction and to compare its basic physicochemical characteristics, particle morphology and functional properties with those of commercial chicory inulin.

MATERIALS AND METHODS

Plant material and standards

Dried flower heads of *Onopordum acanthium* L. were supplied by "Bilki" Ltd. (Sinitovo, Bulgaria), ground to particle size below 1 mm and stored in closed containers in the dark. Commercial chicory inulins Frutalit HD and Raftiline HD were used as reference samples for the physicochemical and the functional analyses, respectively.

Isolation of inulin

Classical extraction (CL). The plant material was extracted with boiling distilled water (1:10, w/v) for 30 min under stirring; the extraction was repeated twice. The combined filtrates were cooled and inulin was precipitated with acetone (acetone : extract = 4:1, v/v) at 4 °C for 4 h. The precipitate was separated on a Büchner funnel and dried at 40 °C.

Microwave-assisted extraction (MW). Fifty grams of plant material and 500 mL of hot distilled water were treated in a microwave oven for 5 min; the extraction was repeated with 250 mL water. The combined extracts were precipitated with acetone (2:1) at 4 °C for 72 h, and the crude inulin was dried at 40 °C and recrystallised from hot water/acetone [7, 13]. The yield was calculated as:

$$\text{Yield, \%} = 100 \cdot m_i / m_s \qquad (1)$$

where m_i is the mass of dry inulin and m_s the mass of dry plant material, g. The samples are designated OA, MW and OA CL.

Physicochemical characterisation

The carbohydrate composition of the raw material and the purity of the isolates were determined by HPLC-RID on an ELITE LaChrom system (Hitachi, Japan) with a Chromaster 5450 refractive index detector, Shodex SUGAR SP0810 column (85 °C) and ultrapure water as mobile phase (1.0 mL/min). The molecular mass distribution was determined by HPLC-SEC on a Shodex OHpak SB-806M column (30 °C) with 0.1 M $NaNO_3$ (0.8 mL/min), calibrated with pullulan standards, and DP was calculated as $(M_w - 18)/162$. Colour was measured with a WR-10QC portable colorimeter in the CIELAB system (four replicates).

Particle morphology was studied by scanning electron microscopy (ZEISS SEM-LYRA I XMU (Tescan), Brno, Czech Republic) after sputter-coating with a 5 nm gold layer, at an accelerating voltage of 10 kV and magnification 100×, using secondary electron (SE1) and high-resolution backscattered electron (HDBSD) detectors.

Functional properties

Wettability was measured as the time (s) needed for 50 mg of powder placed on 20 mL distilled water (20 °C) to sink completely [14]. *Swelling capacity* (cm^3/g) was determined from the volume occupied by 100 mg of sample after 24 h in 10 mL distilled water [15]. The *angle of repose* was calculated from the height (h) and radius (r) of the cone formed by 10 g of powder flowing through a funnel fixed 10 cm above a flat surface [16]:

$$\alpha = \arctan(h / r) \quad (2)$$

Water-holding (WHC) and *oil-holding capacity* (OHC) were determined by hydration of 200 mg of sample with 10 mL distilled water or sunflower oil for 24 h at 20 °C, centrifugation at 3500 min^{-1} for 15 min and weighing of the sediment, and were expressed as g liquid per g dry sample [17]. *Hygroscopicity* was determined as the moisture uptake (%) of 1 g of sample stored for 7 days at 25 °C in a desiccator over saturated NaCl solution (75 % relative humidity) [18]. All analyses were performed at least in duplicate and the results are presented as mean values ± SD where available.

RESULTS AND DISCUSSION

Yield, purity and degree of polymerisation

HPLC-RID analysis of the raw material showed that inulin is the dominant carbohydrate in *O. acanthium* flower heads (2.57 g/100 g dw), accompanied by fructose (1.14 g/100 g dw), sucrose and glucose (0.33 g/100 g dw each), while 1-kestose and nystose were not detected. This value is in the range reported earlier for this species [12] and lower than that for *O. tauricum* flower heads (4.50 g/100 g dw) [11].

The yield, purity and DP of the isolated inulins are presented in Table 1. The classical extraction gave a higher yield (6.56 %) than the microwave-assisted one (4.67 %), but a product of lower purity (20 vs. 32 %). Considering the purity, the amount of inulin recovered was 1.49 and 1.31 g/100 g dw for MW and CL, i.e. about 58 and 51 % of the inulin present in the raw material. Thus, the 5-min microwave treatment was at least as efficient as the 2 × 30-min classical extraction in terms of recovered inulin, while the higher mass yield of the CL isolate is due mainly to co-extracted non-fructan compounds. The higher purity of OA MW can be attributed both to the shorter thermal treatment and to the additional recrystallisation step. A similar trend – lower yield but higher purity after microwave-assisted extraction – was reported for dandelion inulin [7]. The purity of both isolates remained far below that of the commercial inulin (82.5 %), which is explained by the high content of phenolic acids, flavonoids and sesquiterpene lactones in the flower heads [8, 10].

Table 1. *Physicochemical characteristics of O. acanthium inulins and commercial chicory inulin*

Parameter	OA MW	OA CL	Frutalit HD
Yield, %	4.67	6.56	–
Purity, %	32	20	82.5
DP (HPLC-SEC)	30	20	12
L^*	42.74 ± 0.66	50.57 ± 0.48	100.00 ± 0.00
a^*	6.42 ± 0.24	8.32 ± 0.10	0.08 ± 0.04
b^*	19.96 ± 0.61	20.55 ± 0.55	6.79 ± 0.10
C^*	20.97	22.18	6.79

OA MW – microwave-assisted extraction; OA CL – classical extraction; Frutalit HD – commercial chicory inulin.

Both *O. acanthium* isolates are long-chain inulins (DP 20–30) with chains considerably longer than those of the commercial chicory inulin (DP 12). The DP values are comparable to those of fructans from other wild Asteraceae – burdock (DP 20–24) [6] and dandelion (DP 18–25) [7] – and higher than the typical DP of chicory (10–14) and dahlia (13–20) inulin [3]. The microwave extraction yielded inulin with higher DP (30 vs. 20), indicating that the prolonged boiling during classical extraction causes partial hydrolysis of the fructan chains. According to the classification of Mensink et al. [2], the obtained products belong to the long-chain inulins, which are less soluble and are preferred as texturisers and fat replacers.

None of the isolates reached the lightness of the commercial inulin ($L^* = 100$). The *O. acanthium* inulins are yellow-brown

powders (a^* = 6.42–8.32; b^* = 19.96–20.55; C^* = 21–22), the MW isolate being darker than the CL one (L^* = 42.74 vs. 50.57) despite its higher purity. This shows that the colour is determined mainly by the type of co-extracted pigments and browning products rather than by the inulin content alone, and that an additional purification step (e.g. activated carbon) is needed for light-coloured food-grade products.

Particle morphology

The SEM micrographs (Fig. 1) show that the commercial inulin consists of relatively uniform, compact particles with smooth surface and narrow size range, typical of spray-dried industrial products. In contrast, the *O. acanthium* inulin forms loose, spongy agglomerates composed of finely dispersed sub-particles with irregular shape and rough surface, as a result of acetone precipitation, oven drying and milling. Similar irregular, flake-like morphology has been described for non-spray-dried inulin powders [19]. The developed surface and the inter-particle capillaries of such agglomerates are expected to retain considerable amounts of liquid.

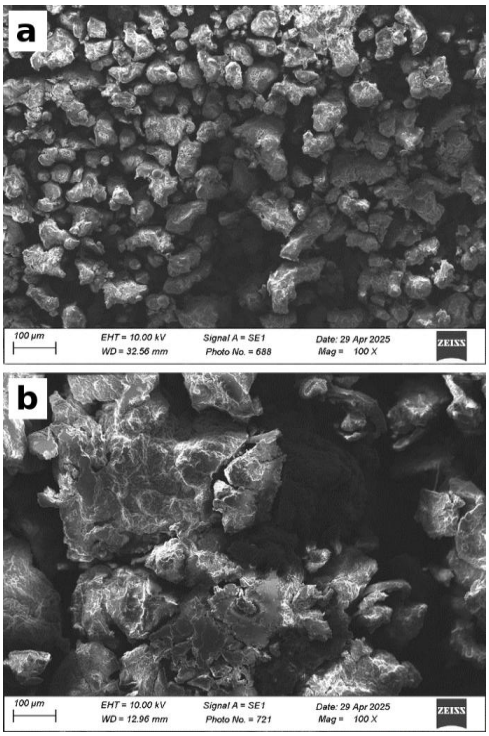


Fig. 1. *SEM micrographs (SE1, 100×) of commercial inulin Frutalit HD (a) and O. acanthium inulin OA MW (b)*

Functional properties

The functional properties of *O. acanthium* inulins compared with Raftiline HD are summarised in Table 2. Both isolates were wetted about 2.5 times more slowly than the commercial inulin (22–24 s vs. 9 s). This is attributed to their longer chains, which hydrate more slowly due to their lower solubility [2], and to the irregular, rough particles (Fig. 1) that hinder water penetration into the powder layer. The swelling capacity of OA MW and OA CL was 10.96 and 8.79 cm^3/g, respectively – about twice the values reported for dandelion inulin (4.40–5.60 mL/g) [7]. Thus, although the *O. acanthium* powders are wetted slowly, once hydrated they form a voluminous, loosely packed sediment, which is consistent with their high water-holding capacity and spongy particle structure (Fig. 1).

Table 2. *Functional properties of O. acanthium inulins and commercial chicory inulin*

Parameter	OA MW	OA CL	Raftiline HD
Wetting time, s	23.72	22.02	9.00
Swelling capacity, cm^3/g	10.96	8.79	–
Angle of repose, °	15.52	12.93	25.64
WHC, g/g	5.32	4.94	0.40
OHC, g/g	7.85	7.36	0.40
Hygroscopicity, %	5.24	5.10	3.70

WHC – water-holding capacity; OHC – oil-holding capacity; Raftiline HD – commercial chicory inulin; – not determined.

The angles of repose of OA MW and OA CL (15.52° and 12.93°) were much lower than that of the commercial inulin (25.64°) and of typical free-flowing powders (25–35°). Such flat cones are characteristic of fine and/or electrostatically charged particles, in which cohesive forces exceed gravity, so the angle of repose of these powders should be interpreted together with bulk and tapped density data [16].

The most pronounced difference from the commercial inulin was found for the liquid-holding capacity. The WHC of *O. acanthium* inulins (4.94–5.32 g/g) was 12–13 times, and the OHC (7.36–7.85 g/g) 18–20 times higher than those of Raftiline HD (0.40 g/g for both). These values also exceed those reported for high-purity inulins from agave,

chicory and Jerusalem artichoke (WHC 1.68–5.40 g/g; OHC 0.87–3.27 g/g) [5]. In both samples OHC exceeded WHC, which is typical of partially purified fructan isolates with developed, rough particle surface (Fig. 1): oil is retained mainly physically in the inter-particle capillaries, whereas water binding depends also on the accessibility of hydroxyl groups, part of which are shielded by co-extracted hydrophobic compounds. The MW isolate, with higher purity and DP, showed slightly higher WHC and OHC than the CL one.

The hygroscopicity of *O. acanthium* inulins (5.10–5.24 %) was higher than that of the commercial inulin (3.70 %), which is explained by the presence of residual low-molecular sugars and phenolic compounds, more hygroscopic than polymeric inulin. The differences between the two extraction methods were negligible. The isolates can be classified as moderately hygroscopic powders that should be stored in packaging with low water-vapour permeability, as moisture uptake lowers the glass transition temperature of amorphous inulin and causes caking [2, 20].

CONCLUSION

Inulin was isolated for the first time from *Onopordum acanthium* L. flower heads by microwave-assisted and classical extraction. The 5-min microwave treatment recovered a similar amount of inulin as the classical extraction, but gave a product with higher purity (32 %) and longer chains (DP 30). Both isolates are long-chain inulins (DP 20–30) with DP higher than that of commercial chicory inulin. Compared with chicory inulin, *O. acanthium* inulins showed loose, spongy particle morphology, slower wetting, high swelling capacity (8.79–10.96 cm^3/g), moderate hygroscopicity and 12–20 times higher water- and oil-holding capacity, which makes them promising texturising and fat-replacing ingredients. Further purification is required to increase purity and to obtain light-coloured products suitable for food applications.

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