Supporting Information

Optimization of Ultrasound Assisted Extraction of Functional Food Fiber from Canadian Horseweed (*Erigeron canadensis* L.)

Sebastian Balicki[†,*], *Izabela Pawlaczyk-Graja*[†], *Roman Gancarz*[†], *Peter Capek*[‡], *Kazimiera A. Wilk*[†,*]

[†]*Department of Engineering and Technology of Chemical Processes, Faculty of Chemistry, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland*

[‡]*Institute of Chemistry, Center for Glycomics, Slovak Academy of Sciences, Dubravská cesta 9, 845 38 Bratislava, Slovakia*

* Corresponding authors. Department of Engineering and Technology of Chemical Processes, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland

E-mail addresses:

kazimiera.wilk@pwr.edu.pl (K.A. Wilk), Tel.: +48 71 320 28 28;

sebastian.balicki@pwr.edu.pl (S. Balicki), Tel.: +48 71 320 33-94

Content of the Electronic Supporting Information

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Text S1. Conventional isolation of the Canadian horseweed crude extracts.[1]

Preparation of the sample was done in the same way as described in the section *Ultrasound assisted extraction procedure*. The plant material was delivered by the manufacturer already dried and minced into appropriate particle size. It was stored in original package in dark, dry space, i.e. 15 – 20 ºC, air humidity 30-40%". Afterwards, the mixture was heated at 97 °C for 6 h, solid plant residues were filtered off and the resulting supernatant was centrifuged for 20 min, at 1850×*g*. The solution obtained was then neutralized with 1.0 M HCl and concentrated to a smaller volume under reduced pressure to produce a raw aqueous plant extract.

Table S1. *I*-optimal design with corresponding variables, process parameters levels of the conducted experiment with determined energy density and coding of the raw horsewood food fibre.

process parameters		**ultrasound assisted extraction**			
independent variable	**number of levels**	**value levels**			
		1	**2**	**3**	**4**
A: time [min]	4	10	20	40	60
B: power [W]	4	30	60	120	240
constant parameters					
temperature [°C]		25			
frequency [kHz]		20			
comminution of the raw material		single-fractioned			
solvent:dry mass ratio		12:1			
Energy density (J/cm^3) values for each combination of the independent variables					
(A_1B_1) = 15	(A_2B_1) = 30	(A_3B_1) = 60	(A_4B_1) = 90		
(A_1B_2) = 30	(A_2B_2) = 60	(A_3B_2) = 120	(A_4B_2) = 180		
(A_1B_3) = 60	(A_2B_3) = 120	(A_3B_3) = 240	(A_4B_3) = 360		
(A_1B_4) = 120	(A_2B_4) = 240	(A_3B_4) = 480	(A_4B_4) = 720		
Coding of the raw horseweed food fiber: 1’ (A_1B_1); ‘2 (A_1B_2); 3’ (A_1B_3); 4’ (A_1B_4); 5’ (A_2B_1); 6’ (A_2B_2); 7’ (A_2B_3); 8’ (A_2B_4); 9’ (A_3B_1); 10’ (A_3B_2); 11’ (A_3B_3); 12’ (A_3B_4); 13’ (A_4B_1); 14’ (A_4B_2); 15’ (A_4B_3); 16’ (A_4B_4);					

Table S2A. Conventional process of horseweed treatment. Consumption of time, water, and input electric energy for 100 g feedstock.

unit process		electric device	time requirement (h)	energy use (kWh)	cumulative energy demand (MJ)	water requirement (dm^3)
solid-liquid extraction	maceration		24.0	–	–	3.0
	hot alkaline extraction	heating mantle	6.0	1.37	4.93	–
	cooling		5.0	–	–	20.0
	filtration	filter press	2.0	–	–	–
	centrifugation	centrifuge	0.5	0.08	0.29	–
	neutralization	pH-meter	0.5	0.01	0.04	–
	condensation	rotary evaporator	4.0	0.13	0.46	–
		heating bath	4.0	2.85	10.26	–
		membrane pump	4.0	0.24	0.86	–
TOTAL			**50**	**4.7**	**16.8**	**23.0**

Table S2B. UAE optimized horseweed treatment. Consumption of time, water, and input electric energy for 100 g feedstock.

unit process		electric device	time requirement (h)	energy use (kWh)	cumulative energy demand (MJ)	water requirement (dm^3)
solid-liquid extraction	maceration		24.0	–	–	3.0
	ultrasound assisted extraction	ultrasound generator	1.0	0.04	0.86	–
	cooling	circulation pump	1.0	0.04	0.14	0.5
	filtration	filter press	2.0	–	–	–
	centrifugation	centrifuge	0.5	0.08	0.29	–
	neutralization	pH-meter	0.5	0.01	0.04	–
	condensation	rotary evaporator	4.0	0.13	0.46	–
		heating bath	4.0	2.85	10.26	–
		membrane pump	4.0	0.24	0.86	–
TOTAL			**41**	**3.4**	**12.2**	**3.5**

Text S2. Comparison of (**13**) with the conventional FF (K), showed differences in the polyphenolic and saccharide fragments of both the bioproducts studied. In the case of the K product composition, four dominant fractions of molecular weights as 129.0, 26.2, 12.9 and 4.5 × 103 (g×mol^{-1}) were determined (see Fig. S1 (a)). While, the optimal product (shown in Fig. S1 (b)) (**13**) comprised three independent fractions, i.e., F1, F2, and F3

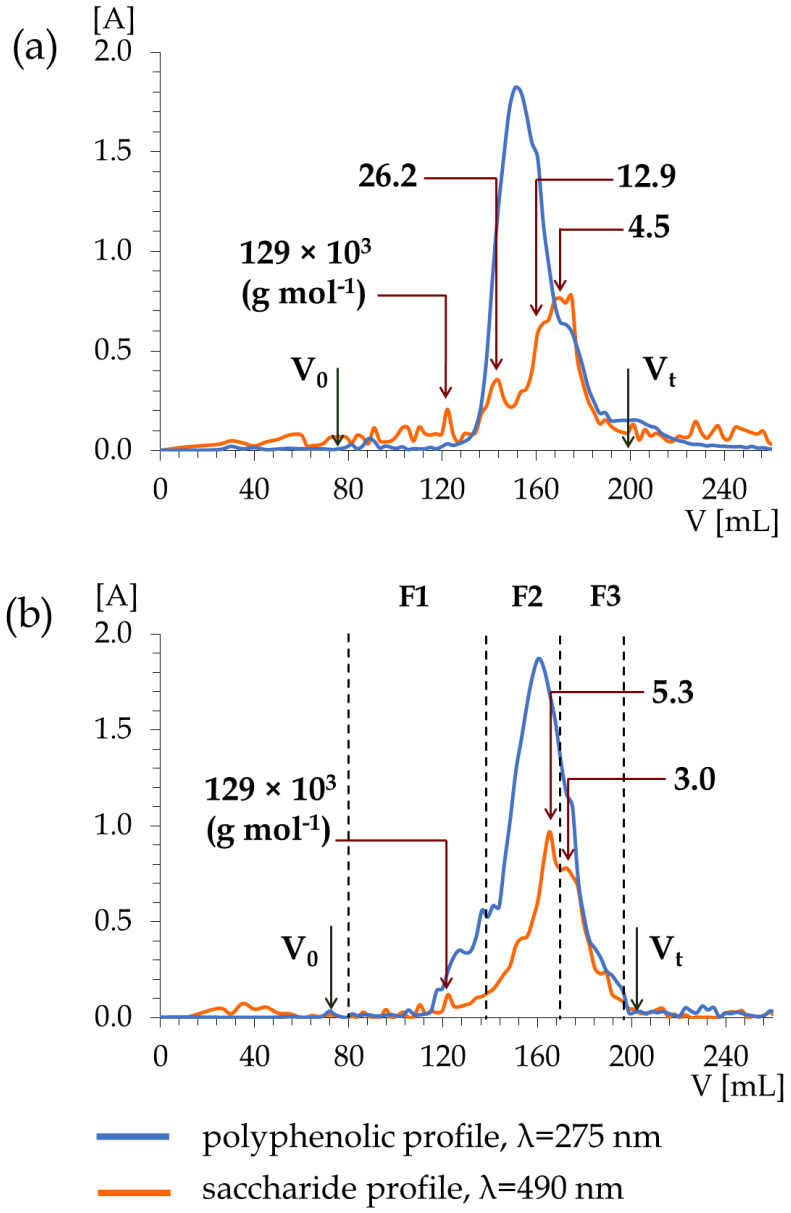

Figure S1. Polyphenolic and saccharide profile of the conventional food fiber (K) with molecular weights estimation (a). Polyphenolic and saccharide profile of the ultrasound assisted optimized food fiber (**13**) with molecular weight estimation and corresponding fractions (F1-F3) (b).

Text S3.

FT-IR spectra were recorded by the ATR technique in the middle region of IR (4000 – 400 cm^{-1}) with a Vertex 70v spectrometer (Bruker Optics Inc., Billerica, USA).

FT-IR spectra (shown in Fig. S2) of the conventional product, optimally obtained product (**13**), and its fraction F2 with the best anticoagulant properties gave bands characteristic for the dietary fiber, i.e., polyphenolic matrices conjugated with polysaccharide chains.[2] In all analyzed spectra a wide band with maximum at 3290 cm^{-1} characteristic for the stretching vibrations of –O-H bonds of saccharide and the phenolic parts of the glycoconjugates, was present.[2] Moreover, a strong and narrow band around 1600 cm^{-1} indicated antisymmetric stretching vibrations typical for carbonyl groups, as well as for skeletal interactions of aromatic rings, possibly derived from uronic acids of carbohydrates and lignin species.[2] In each spectrum at around 1525-1515 cm^{-1} a weak signal accountable for the skeletal vibrations of the ferulic acid derivatives was present. These phenolic structures are usually recognized as those of macromolecular polyphenolic matrix connected to polysaccharide chains through the ester bonds, frequently via arabinose subunits.[3] Finally, bands present at around 890 cm^{-1} responsible for the deformation interactions of β-anomeric carbon atoms of saccharide glycoside bonds[3] were determined at 896 (K), 891 (**13**) and 886 cm^{-1} (F2), respectively.

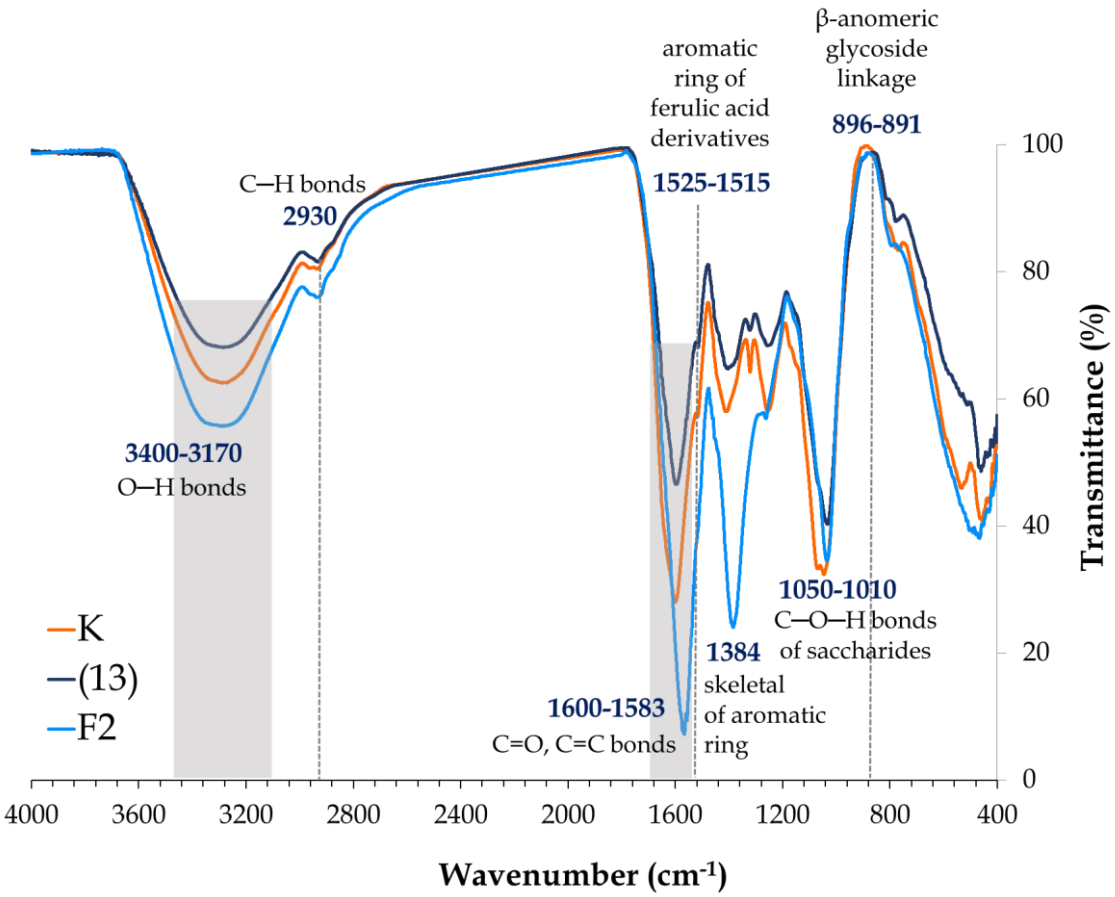

Figure S2. FT-IR spectra of the conventional horseweed functional food fiber, ultrasound assisted optimal food fiber (**13**) and its most anticoagulant active fraction obtained after gel chromatography separation (F2).

Text S4. The analysis of the monosaccharide composition was done according to the GC-MS technique,[1,4] with alditol acetate derivatives of the neutral monosaccharides obtained in the acidic hydrolysis of the received food fiber. This technique is routinely applied in mono-, oligo- and polysaccharides qualitative and quantitative analysis, with good precision and good accuracy. Obtained chromatograms of the mixtures were analyzed by the surface area of detected and determined peaks, based on library of mass spectra and retention times for various alditol acetate derivatives of neutral monosaccharides. Then, the surface areas were expressed as percentage of the total peak area, and recalculated to percentage of each neutral monosaccharide. The contents of uronic acids were determined with *m*-hydroxybiphenyl assay, with galacturonic acid as the standard.[5] A sum of both neutral monosaccharides and uronic acids was treated as carbohydrate part of particular food fiber preparation.

References

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