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Ecology

Functional Dietary Fibers from Subtropical Fruit Byproducts

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(Presented by Academy Member Vano Papunidze)

Abstract. The byproducts generated from the industrial processing of fruits present a significant ecological challenge and pose a considerable problem for the food industry. Byproducts derived from mandarin fruit (*Citrus unshiu* Marc.), mandarin flowers (*Citrus unshiu*), and persimmon fruit (*Diospyros kaki* L.) are rich in sugars, vitamins, minerals, carotenes, phenolic compounds, and water-insoluble fibrous polysaccharides. Prebiotic dietary fibers extracted from these byproducts are notable for their phytochemical indicators and functional properties, including water retention capacity (WRC), fat absorption capacity (FAC), sorption properties, total flavonoid content (TFC), total phenolic content (TPC), and mineral element composition. Among the dietary fiber supplements analyzed, mandarin fruit fiber exhibited the highest water and fat binding capacities, as well as the greatest lead ion adsorption, with values of 14.5 ± 0.02 g/g, 2.7 ± 0.02 g/g, and 20.6 ± 0.3 mg/g, respectively. The highest cholic acid adsorption capacity was observed in mandarin flower fiber (9.2 ± 0.2 mg/g), followed by mandarin fruit fiber (8.9 ± 0.3 mg/g). Mandarin fiber was also distinguished by its total flavonoid content, with P vitamin levels of 9.1% in mandarin fiber and 6.5% in flower fiber. Phenolic compounds were most abundant in persimmon peel fiber, with a concentration of 953.4 mg/%. Both mandarin flower and persimmon fibers were notable for their mineral content. These findings provide valuable and reliable data supporting the use of high-purity dietary fibers from fruit byproducts as functional and prebiotic ingredients in food products. © *2025 Bull. Natl. Acad. Sci. Georg.*

Keywords: subtropical fruit crops, food fibers, food additive, circular economy

Introduction

By adopting a circular economy model, low-cost and, high-biological-value waste is increasingly being reintegrated into production processes, thereby fostering new business opportunities and enabling the creation of innovative products (Buljeta, 2021; Russo, 2021). This model not only reduces the environmental burden of agro-industrial waste, but also aligns with global efforts to promote sustainable development and resource efficiency in the food industry.

The global dietary fiber market is currently valued at USD 2.5 billion and is projected to grow at an annual rate of 9.5%, reaching USD 17.3 billion by 2032. The demand for dietary fibers derived from fruits and vegetables is steadily increa-

sing. These fibers are widely used as raw materials in the production of functional foods, bakery products, beverages, and pharmaceutical preparations. Their incorporation enhances not only nutritional value but also product texture, stability, and shelf life – making them highly attractive for both health-conscious consumers and manufacturers.

Recent research focuses on the application of modern green technologies for byproduct processing. Studies have shown that conventional chemical methods can disrupt glycosidic bonds in dietary fibers, leading to the complete degradation of soluble dietary fiber, a 30-40% reduction in hemicellulose content, and a 10-20% decrease in cellulose content. While ultrasound-assisted extraction improves the efficiency of bioactive compound recovery, it also presents limitations, including high operational costs and limited research on its long-term effects on fiber structure and human health (Bansal, 2025). Therefore, identifying cost-effective, eco-friendly processing techniques remains a critical area of interest.

Mandarin fruit (*Citrus unshiu* Marc.), mandarin flowers (*Citrus unshiu*), and persimmon fruit (*Diospyros kaki* L.) are rich in biologically active compounds known for their health benefits (Hosseininejad, 2022), (Sarrou, 2013), (Martínez-Las Heras, 2017). These include dietary fibers, phenolic compounds, flavonoids, and carotenoids, which demonstrate antioxidant, anti-inflammatory, and cholesterol-lowering properties in previous studies.

The aim of this research is to obtain the high-purity dietary fiber from the byproducts of industrial processing of mandarin fruit, mandarin flowers, and persimmon fruit and to conduct a comparative characterization of the resulting dietary fibers as physiologically active food additives. This study contributes to both waste valorization and the development of natural, functional ingredients with potential applications in the health food and nutraceutical sectors.

Materials and Methods

This research focuses on dietary fibers derived from the byproducts of mandarin fruit (*Citrus unshiu* Marc.), mandarin flowers (*Citrus unshiu*), and persimmon fruit (*Diospyros kaki* L.). These byproducts, traditionally considered waste, are rich in bioactive compounds and were selected due to their abundance and potential value in circular bio-economy models.

Technological Schemes for obtaining dietary fiber. Dietary fibers preparations are extracted using the following schemes:

Technological Scheme I: Mandarin fruit byproducts → Drying at 60-65°C for 40-60 minutes → Grinding.

Technological Scheme II: Mandarin fruit byproducts → Crushing → Multiple washes with water at 50-60°C to remove simple sugars → Drying at 60-65°C for 40–60 minutes → Grinding.

Technological Scheme III: Persimmon fruit peel byproducts → Crushing → Drying at 50-60°C → Multiple washes with water at 40-50°C to remove simple sugars → Drying at 50-55°C for 60 minutes → Grinding.

Technological Scheme IV: Mandarin flower byproducts → Drying at 40-50°C for 60 minutes → Grinding.

The waste from mandarin flowers after the production of semi-finished flower extract contains 1-1.5% simple sugars and does not require washing with water. All extracted fibers were packaged in oxygen-free containers and stored at +4°C to preserve their chemical integrity for further analysis.

Evaluation of functional properties. Water Retention Capacity (WRC) and Fat Absorption Capacity (FAC) were determined using the gravity method described by Núñez-Gómez et al. (Núñez-Gómez, 2024). WRC – 1 g of sample was mixed with 20 mL of distilled water and maintained at 25°C for 24 hours, then centrifuged at 3000 × g for 15 minutes. The supernatants were removed, the

mass of the residue after hydration was measured and WRC was determined.

$$WRC\left(\frac{g}{g}\right) = \frac{m_2 - m_1}{m_1},$$

where m_1 – the mass of the dry sample (g) before hydration, m_2 – the mass of the sample (g) after hydration.

FAC – 0.5 g of sample was mixed with 20 mL of sunflower oil in the centrifuge tube and incubated at 37°C for 1 hour. After incubation, the sample was centrifuged at 3000 rpm for 15 minutes. The supernatant was removed, and the residue was weighed.

Fat Absorption Capacity (FAC) is measured by the amount of absorbed oil (in grams) using the following formula:

$$FAC\left(\frac{g}{g}\right) = \frac{m_2 - m_1}{m_1},$$

where m_1 – is the weight of the sample (g) before incubation with oil, m_2 – is the weight of the sample (g) after incubation with oil.

Sodium cholate binding capacity (NaChBC) – 0.2 g of dietary fiber and 0.2 g of sodium cholate were mixed with 100 mL of NaCl solution (0.15 mol/L, pH 7.0) in a 250 mL flask. A control sample (without dietary fiber) was also prepared. All samples were incubated at 37°C for 2 hours with intermittent shaking at 120 rpm in a water bath. After incubation, the samples were centrifuged at 4000 × g for 20 minutes. Then, 0.5 mL of the supernatant was mixed with 4.5 mL of 42% sulfuric acid and maintained at 70°C for 20 minutes. The concentration of sodium cholate was determined according to Zhanmei et al. (Zhanmei, 2022) by measuring the absorbance at 387 nm using a calibration curve.

The NaChBC of dietary fiber was calculated using the following formula:

$$NaChBC\left(\frac{mg}{g}\right) = \frac{C_1 - C_2}{m} * V,$$

where C_1 is the sodium cholate concentration in the control sample (mg/mL), C_2 is the sodium cholate concentration in the fiber-containing sample (mg/mL), m is the mass of dietary fiber (g), V is the total volume of the sample (mL).

Sorption capacity of dietary fibers toward lead ions. Lead ions were used in the form of analytical grade $Pb(NO_3)_2$ solution (0.025 mol/L). One gram of dietary fiber was mixed with 50 mL of the metal salt solution. Six flasks were incubated for 5, 10, 15, 20, 25, and 30 minutes, respectively, to obtain the sorption kinetics. The quantitative analysis of lead cations in the solution was carried out by titrimetric method (Ryabinina, 2015). From each flask, 2 mL of the test solution was transferred into a titration vessel using a volumetric pipette. Then, 0.1-0.2 g of dry hexamethylenetetramine (urotropine) was added to adjust the pH to 5.0, followed by three drops of Xylenol Orange indicator. The solution was then titrated with a standard EDTA solution (0.025 M) until the color changed from violet to lemon yellow.

To obtain sorption kinetic curves, 1 g of sorbent (m) was placed in a series of test tubes and treated with 50 mL (V) of aqueous metal salt solution. Contact time varied from 5 minutes to 1 hour. At specified time intervals, the solution was separated from the sorbent, and the concentration of metal ions (C_t) in the filtrate was determined using titration. The sorption capacity (A) of the sorbents at each time point was calculated using the following formula:

$$A = \frac{(C_0 - C_t) * V}{m},$$

where C_0 is the initial metal ion concentration (mol/L), C_t is the metal ion concentration at time t (mol/L), V – the volume of the solution (L), m – the mass of the sorbent (g).

The removal efficiency (α) of lead ions was calculated using the equation:

$$a = \frac{(C_0 - C_t)}{C_0} * 100\%,$$

Phytochemical analyses. The quantitative content of vitamin P in dietary fiber samples was determined using the cyanidin reaction method (Papunidze, 2012). To determine the hesperidin content, an

accurately weighed analytical sample was transferred into a porcelain mortar, moistened with hot 50% ethanol, and dissolved. The solution was quantitatively transferred using an additional 60 mL of hot 50% ethanol into a 100 mL volumetric flask. Subsequently, 0.5 mL of hydrochloric acid was added. The flask was placed in a water bath, connected to a reflux condenser, and boiled for 20 minutes. After cooling to room temperature, the solution was brought to volume with 50% ethanol and filtered through a suitable membrane filter. A standard hesperidin solution was prepared by weighing 0.01 g of hesperidin (melting point 259-260°C), previously dried to constant weight at 100-105°C. Six test tubes (120 mm in height, 25 mm in diameter) were each charged with 200 mg of magnesium powder. Two tubes were filled with 5 mL of the test solution, two with 5 mL of the standard solution, and the remaining two with 5 mL of 50% ethanol as blank controls. The test tubes were placed into a rotating rack and immersed in an ice bath. After 10 minutes, hydrochloric acid was added dropwise to each test tube from a burette attached to a vertical stand, starting with test tube No. 1. The addition continued until each test tube received a total of 2 mL of HCl (added dropwise). The rotating rack with the test tubes was then transferred to a water bath maintained at 30-40°C for 30 minutes. After incubation, the test tubes were cooled to room temperature. The optical density (absorbance) of each solution was measured in the range of 530-550 nm using the blank solution as reference (Romanenko, 1966).

The hesperidin content (X, in %) in the test sample was calculated using the following formula:

$$X = \frac{D_x}{D_{st} * a},$$

where D_x is the average optical density of the two test sample measurements, D_{st} is the average optical density of the two standard solution measurements, a is the weight of the test sample (g).

The total phenolic content of the derived dietary fiber from persimmon was quantified using the Folin-Ciocalteu method (Núñez-Gómez, 2024). 0.1 mL of the ethanolic extract of the sample was mixed with 7.5 mL of distilled water, followed by the addition of 0.5 mL of Folin-Ciocalteu reagent and 1 mL of 35% sodium carbonate solution (Na_2CO_3). The total volume of the reaction mixture was adjusted to 10 mL with distilled water and thoroughly mixed.

The mixture was incubated at room temperature for 30 minutes. The absorbance was measured at 725 nm using a UV spectrophotometer. Quantification was carried out using a standard calibration curve of gallic acid. The results were expressed as milligrams of gallic acid equivalents (GAE) per gram of dry weight (DW) of the sample.

The qualitative and quantitative analyses of mineral elements in mandarin and persimmon fruits and their byproducts, as well as in mandarin flower and its byproduct, were performed using a plasma atomic emission spectrometer (ICPE-9820) (Papunidze, 2023).

Statistical analysis. All measurements were performed in triplicate. Data were processed using R Studio (version 4.0.5) supported by the R Foundation for Statistical Computing. Mean values and standard deviations were calculated. Differences among means were evaluated using Stewart's t-test at a significance level of $p < 0.05$. Pearson correlation coefficients were used to assess relationships between functional and phytochemical properties.

Results and Discussion

A gentle extraction method using distilled hot water was employed to obtain dietary fibers from mandarin fruit (*Citrus unshiu* Marc.), mandarin flowers (*Citrus unshiu*), and persimmon fruit (*Diospyros kaki* L.). A critical stage in the extraction process involved the removal of low-molecular-weight compounds without using chemical reagents-essential for maintaining the natural structure and purity of fibrous polysaccharides.

The dietary fibers obtained from the various technological schemes showed significant differences in purity.

Table 1. The content of fibrous polysaccharides (raw mass)

A way to get dietary fiber	Dry matter %	Simple sugars %	fibrous Polysaccharides %
Technological Scheme I (Mandarin fruit)	85.2±0.1	20.6±0.02	54.6±0.3
Technological Scheme II (Mandarin fruit)	90.7±0.15	1.5±0.01	89.2±0.1
Technological Scheme III (Persimmon fruit peel)	88.5±0.2	3.5±0.02	87.5±0.2
Technological Scheme IV (Mandarin flower)	92.4±0.1	1.2±0.02	89.3±0.1

The dietary fibers obtained from the various technological schemes showed significant differences in purity. As seen in Table 1, Scheme II (mandarin fruit), Scheme IV (mandarin flowers) and Scheme III (persimmon fruit) yielded dietary fibers with the highest content of fibrous polysaccharides (89.2%, 89.3% and 87.5%, respectively) and the lowest content of simple sugars (1.5%, 1.2% and 3.5%, respectively), indicating high-purity dietary fiber. In contrast, Scheme I (mandarin fruit) without washing retained 20.6% simple sugars, resulting in a relatively low purity of 54.6% fibrous polysaccharides. The higher the content of fibrous polysaccharides, the higher their sorption properties and physiological activity. These findings underscore the importance of washing step in removing low molecular weight components, as supported by similar observations in research by Akter et al. (Akter, 2010), who also emphasized hot water extraction as a favorable approach for high-fiber yield and purity.

The functional attributes of the extracted dietary fibers – such as Water Retention Capacity (WRC), Fat Absorption Capacity (FAC), and sorption capacities for bile acids and lead ions are presented in Table 2.

Table 2. Functional indicators of food fibers

Functional indicators	Mandarin byproduct fiber	Mandarin flower byproduct fiber	Persimmon byproduct fiber
WRC (g/g)	14.5±0.02	13.3±0.02	11.0±0.01
FAC (g)	2.7±0.02	2.1±0.05	1.5±0.05
Cholic Acid Sorption Capacity (mg/g)	8.9±0.3	9.2±0.2	8.8±0.2
Lead Sorption Capacity (mg/g)	20.6±0.3	20.1±0.4	18.7±0.2

(Different letters among columns indicate significant differences ($p < 0.05$).

Notably, dietary fiber from mandarin fruit demonstrated the highest values across several indicators: WRC: 14.5 ± 0.02 g/g; FAC: 2.7 ± 0.02 g; Lead adsorption: 20.6 ± 0.3 mg/g; Cholic acid binding: 8.9 ± 0.3 mg/g. Mandarin flower fiber also showed robust functionality, particularly in bile acid binding (9.2 ± 0.2 mg/g), which was slightly higher than mandarin fruit fiber. Persimmon fiber showed slightly lower but still valuable capacities. These results suggest strong potential for these fibers as functional ingredients in foods-especially in formulations aimed at fat reduction, cholesterol management, and detoxification. The performance of mandarin fiber exceeds values reported for lemon peel by Jiaqi Sang et al. (Jiaqi, 2021), supporting its potential application in dairy and bakery industries.

Vitamin P content was significant in both mandarin-based fibers. Mandarin fruit fiber contained 9.1%, while mandarin flower fiber had 6.5%, confirming the efficacy of the developed method for vitamin P extraction. Compared to the alkaline extraction method used by Padilla et al. (Padilla de la Rosa, 2018), which yielded 70% pure hesperidin with a melting point of 242-244°C, the method

developed by Romanenko E.I. yielded 93.1% pure hesperidin, with a higher melting point of 259-260°C. This demonstrates that the cold method better preserves compound integrity and purity.

The persimmon fiber was particularly rich in phenolic compounds, with a TPC of 953.4 mg/%, including 1.1 mg/% tannins and 40-45 mg/% catechins. This aligns with findings by Gorinstein et al. (Gorinstein, 2001) and supports the fiber's antioxidant and hypolipidemic potential. Carotenoids in persimmon fiber from the total content of carotenoids (raw mass) is: β-carotene accounts for 11.1%, cryptoxanthin – 29.4%, zeaxanthin – 29.2%, and α-carotene – 21.1%. This carotenoid profile further enhances persimmon peel's appeal as a radioprotective and health-promoting additive.

A total of 27 mineral elements were qualitatively and quantitatively identified. As shown in Table 3, all fiber preparations exhibited higher concentrations of both macro- and microelements compared to their corresponding fresh fruits, consistent with findings reported by other researchers (Czech, 2020).

Mandarin flower fiber contained: Ca – 3289 mg/L, K – 8812 mg/L, Mg – 1095 mg/L, P – 1843 mg/L, Fe – 69.2 mg/L. Similarly, persimmon peel fiber was rich in Mn (70.3 mg/L) and K (6980 mg/L). Manganese is especially important for enzyme function and antioxidant defenses. These concentrations exceed those found in persimmons from Israel, as described in comparative studies (Gorinstein, 2001), confirming the superior mineral profile of Georgian fruit waste. Interestingly, mandarin flowers and their byproducts emerged as a particularly mineral-dense source. This supports Chinese agricultural practices where Citrus unshiu flowers are analyzed to diagnose soil nutrient levels and predict fruit yields (Hui-Ping, 2014). All tested samples showed toxic element levels (Pb, Ti, As) below quantifiable limits (ULOQ), confirming the safety of the extracted dietary fibers for food and nutraceutical use.

Conclusion

Dietary fibers obtained from byproducts of mandarin (*Citrus Unshiu* Marc.), mandarin flowers

Table 3. Mineral element content of raw materials and food fiber preparations (mg/L)

Elements	Mandarin fruit	Mandarin fruit, fiber	Mandarin flower	Mandarin flower fiber	Persimmon fruit	Persimmon peel fiber
Macroelements						
Ca	429	762	3120	3289	547	903
K	637	659	7640	8812	5784	6980
P	150	348	1590	1843	569	654
Mg	94.1	109	985	1095	346	414
Na	16.7	50.0	89	107.2	179.2	237
Si	4.46	2.67	105	344	25.8	34.4
Fe	1.69	6.54	50.2	69.2	9.9	14.8
Microelements						
Al	2.46	3.0	32.7	35.2	5.3	15.4
Zn	2.96	3.6	11.2	14.09	3.7	5.91
Cu	0.15	4.4	8.7	10.4	1.44	2.59
Mn	0.41	0.77	12.3	16.8	46.7	70.3
Toxic elements						
Pb	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ
Ti	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ
As	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ	ULOQ

ULOQ – under the limit of quantitation

(*Citrus Unshiu*) and persimmon fruit (*Diospyros Kaki L.*) have different compositions and applications in food industry. Obtaining physiologically active dietary fiber additives, it is crucial to select an extraction technology that preserves the structural integrity of fibrous polysaccharides such as cellulose, hemicellulose, pectin and maximum removal of low molecular weight substances (simple sugars). Highly purified dietary fibers, known for their physiological benefits, can be used as a prebiotic additive in a wide range of low-calorie breads, confectionery, dairy and meat products. In contrast, low-purity dietary fibers, which have a high content of simple sugars, can serve as a physiologically active additive that improves the rheological and organoleptic properties of bread and bakery products. The inclusion of dietary fibers in bakery products not only helps reduce the amount of wheat flour, but also increases the yield of the product, extends the shelf life and improves the functional properties.

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ეკოლოგია

სუბტროპიკული ხილის ნარჩენებიდან მიღებული ფუნქციონალური საკვები ბოჭკოები

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ხილის სამრეწველო გადამუშავების შედეგად წარმოქმნილი ნარჩენები მნიშვნელოვან ეკოლოგიურ გამოწვევას წარმოადგენს კვების მრეწველობისთვის. მანდარინის ნაყოფის (*Citrus unshiu* Marc.), მანდარინის ყვავილის (*Citrus unshiu*), ხურმის ნაყოფის (*Diospyros kaki* L.) სამრეწველო გადამუშავების შემდეგ მიღებული ნარჩენები მდიდარია შაქრებით, ვიტამინებით, კაროტინოიდებით, მინერალური ნივთიერებებით, ფენოლური ნაერთებით, წყალში ხსნადი და უხსნადი ბოჭკოვანი პოლისაქარიდებით. კვლევის შედეგებიდან გამომდინარე, ნარჩენებიდან მიღებული საკვები ბოჭკოები გამოირჩევა მაღალი ფიტოქიმიური მაჩვენებლებით და ფუნქციური თვისებებით: წყლის (WRC), ცხიმის (FAC) შეკავშირების უნარით, სორბციული თვისებებით, საერთო ფლავონოიდების (TFC), საერთო ფენოლების (TPC) და

მინერალური ელემენტების შემცველობით. მიღებულ საკვებ ბოჭკოვან დანამატებს შორის, წყლის და ცხიმის შეკავშირების მაღალი უნარით, ასევე ტყვიის იონების მაღალი ადსორბციით ხასიათდება მანდარინის საკვები ბოჭკო შესაბამისად **14,5 ± 0,02**გ/გ, **2,7 ± 0,02**გ/გ და **20,6 ± 0,3**მგ/გ. მანდარინის ყვავილის ნარჩენიდან მიღებული საკვები ბოჭკო გამოირჩევა ქოლის მჟავას ადსორბციით **9,2 ± 0,2**მგ/გ, უმნიშვნელო სხვაობით მანდარინის ნაყოფის გამონაწურხის საკვები ბოჭკოს მიერ ქოლის მჟავას ადსორბცია შეადგენდა **8,9 ± 0,3**მგ/გ. საერთო ფლავონოიდების (P ვიტამინი) მაღალი შემცველობით გამოირჩეოდა მანდარინის ბოჭკო **9,1%** (მშ. მას.), ხოლო მანდარინის ყვავილის ბოჭკო – **6,5%** (მშ. მას.). ფენოლური ნაერთების მაღალი შემცველობა დაფიქსირდა ხურმის კანიდან მიღებულ საკვებ ბოჭკოში – **953,4**მგ% (მშ. მას.) ადამიანის ორგანიზმისთვის აუცილებელი მინერალური ნივთიერებების შემცველობით გამოირჩეოდა მანდარინის ყვავილის და ხურმის კანის ნარჩენებიდან მიღებული საკვები ბოჭკო. მიღებული შედეგები ადასტურებს ხილის ნარჩენებიდან მიღებული მაღალი სისუფთავის საკვები ბოჭკოების, როგორც დანამატების საკვებ პროდუქტებში ფუნქციურ, პრებიოტიკულ ინგრედიენტებად გამოყენების აუცილებლობას.

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