

Comparison of the Chemical Composition and Physicochemical Properties of Different Fibers Prepared from the Peel of *Citrus sinensis* L. Cv. Liucheng

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Fiber-rich fractions (FRFs) including soluble and insoluble dietary fibers (SDF and IDF), alcohol-insoluble solid (AIS), and water-insoluble solid (WIS) were isolated from the peel of *Citrus sinensis* L. cv. Liucheng for analysis and tests. The peel was rich in insoluble FRFs (IDF, AIS, and WIS; 476–515 g kg⁻¹ of peel), which were mainly composed of pectic substances and cellulose, and also contained pectic polysaccharide-rich SDF (94.1 g kg⁻¹ of peel). These insoluble FRFs had water-holding capacities (15.5–16.7 mL g⁻¹), oil-holding capacities (2.35–5.09 g g⁻¹), cation-exchange capacities (454–997 mequiv kg⁻¹), and swelling properties (14.6–21.1 mL g⁻¹) much higher than those of cellulose. These results recommended the consumption of these peel insoluble FRFs of desired physicochemical properties as sources of food fibers or low-calorie bulk ingredients in food applications requiring oil and moisture retention. Further investigations on the physiological functions of these peel FRFs using animal-feeding experiments are underway.

KEYWORDS: Dietary fiber; alcohol-insoluble solid; water-insoluble solid; fiber-rich fractions; composition; physicochemical properties; *Citrus sinensis* L. cv. Liucheng; peel

INTRODUCTION

On the basis of the research effort in the past two decades, dietary fibers as plant-cell-wall polysaccharides were acknowledged to be beneficial to normal gastrointestinal and physiological functions (1, 2). The importance of food fibers has led to the development of a large and potential market for fiber-rich products and ingredients. In recent years, there is a trend to find new sources of dietary fiber as food ingredients for the food industry.

Dietary fibers obtained from different methods and sources (i.e., cultivar, genus, or species) might vary in their chemical composition and physicochemical properties, which subsequently affects their uses as ingredients in food applications (3–5). Both the chemical and physical characteristics of dietary fibers would provide clues to their physiological responses (6).

Citrus sinensis L. cv. Liucheng (Liucheng sweet orange) is an important fruit indigenous to Taiwan and usually used for orange juice production. After the juice extraction process, thousands of tons of pomace in the form of peel are produced from the Liucheng sweet orange. This agricultural byproduct is generally discarded as feed. Although cereal is the common source of dietary fiber in many fiber-enriched foods, previous studies have indicated that fruits and vegetables could also be good sources of food fibers (4, 5, 7–10). The composition, physicochemical properties, and physiological effects of some

pomace fibers from fruits and vegetables including apple, celery, guava, Japanese quince, mango, pineapple, and rutabaga have been evaluated to explore their potential applications and physiological activities (3–5, 11–15).

Because the method of processing of agricultural byproducts might determine the functionality of dietary fibers, the aim of the present work was to evaluate and compare the chemical composition and physicochemical properties of various fiber-rich fractions (FRFs) prepared from the Liucheng sweet orange peel via different methods. Differences among the dietary fibers, alcohol-insoluble solid, and water-insoluble solid as well as the potential applications of these peel FRFs as sources of food fibers will be discussed in this study.

MATERIALS AND METHODS

Sample Preparation. The peel sample of *C. sinensis* L. cv. Liucheng (Liucheng sweet orange, LSO) was obtained from CHIA-MEEI Food Industrial Corp., Taiwan. After the juice extraction process, the LSO peel sample was collected and dried in an air-oven at 40 °C for 48 h for composition analysis. The moisture content of the dried peel sample was 7.57 ± 0.42 g kg⁻¹. The dried sample was then finely ground to 0.5 mm in size and kept in a desiccator until used.

Proximate Analysis. The ash content was estimated according to AOAC method 4.1.10 (16). Moisture was determined by drying to a constant weight at 105 °C. The crude lipid content was quantified by extracting the sample with petroleum ether in a Soxhlet apparatus. The crude protein content was calculated by multiplying the nitrogen content obtained from a CHN-OS rapid element analyzer (Heraeus F002, Hanau, Germany) with a factor of 6.25.

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Separation and Analysis of Dietary Fiber (DF). According to the Mes-Tris AOAC method 991.43 (16), the DF content was determined with the fiber assay kit (Megazyme K-TDFR, Wicklow, Ireland). In brief, peel sample suspended in Mes-Tris buffer was sequentially digested by heat-stable α -amylase, protease, and amyloglucosidase to remove starch and protein. Insoluble dietary fiber (IDF) was recovered from the enzyme digestate after filtration. Soluble dietary fiber (SDF) in the filtrate was precipitated with ethanol and filtered. All DF fractions collected were dried by solvent exchange and air at 80 °C. Total dietary fiber (TDF) was calculated as the sum of IDF and SDF. All DF contents were corrected for residual protein, ash, and blank.

Measurement of Starch. Following the method of dietary fiber separation, the content of digestible starch was obtained from the difference between the total glucose content in the filtrate after the enzymatic digestion process and the amount of glucose present in the filtrate without enzymatic digestion (control).

Separation of Alcohol-Insoluble Solid (AIS). According to the method of Thomas et al. (5) with slight modifications, peel sample was homogenized in boiling alcohol (85%, v/v) using the Osterizer (Sunbeam-Oster, Chicago, IL) at the "Hi" speed for 1 min. The peel-to-alcohol ratio was 1:30 (w/v). The suspension was then boiled for another 40 min, and the entire extraction process was done in twice. AIS was filtered, washed with 70% ethanol, and finally dried with solvent exchange and air at 30 °C.

Separation of Water-Insoluble Solid (WIS). According to the method of Massiot and Renard (17) with slight modifications, WIS was prepared by homogenizing the peel sample in cold distilled water using the Osterizer (Sunbeam-Oster) at the "Hi" speed for 1 min. The peel-to-water ratio was 1:10 (w/v). After it had been filtered and washed with cold distilled water, WIS was dried by solvent exchange and air at 30 °C.

Chemical Analysis of Fiber Components. According to the methods described by Englyst et al. (18) and Southgate (19), the neutral sugar profiles of the peel FRFs were determined using allose as an internal standard. The FRFs were first hydrolyzed with 12 M H₂SO₄ at 35 °C for 60 min and further boiled in 2 M H₂SO₄ for another 60 min. The released monosaccharides were quantified as alditol acetates using a gas chromatograph (Hitachi G-5000, Tokyo, Japan) fitted with a flame ionization detector. The conditions were as follows: capillary column, Quadrex 007-225 (15 m \times 0.53 mm i.d.); oven temperature, initially held at 100 °C for 3 min, then raised to 220 °C at a rate of 4 °C min⁻¹; injector and detector temperatures, 270 °C; gas flow rates, 2.1 mL min⁻¹ (carrier gas, nitrogen) and 500 mL min⁻¹ (air).

According to AOAC method 45.4.11 (16), uronic acids in the acid hydrolysate were determined by colorimetry using D-galacturonic acid monohydrate as reference. The pectin content in the sample was estimated by the amount of uronic acids, which were measured as polysaccharide residues.

For the determination of noncellulosic glucose, FRFs were hydrolyzed with 2 M H₂SO₄ at 100 °C for 60 min. Cellulose content was calculated from the difference between the amount of glucose released from 12 M (followed by 2 M) H₂SO₄ and 2 M H₂SO₄ hydrolysis.

Physicochemical Properties. Both the bulk density (grams per milliliter) and cation-exchange capacity (milliequivalents per kilogram) of the insoluble FRFs were determined as described by Chau (20). The swelling property of the insoluble FRFs was determined using the bed volume technique (21). According to the method of Chau et al. (22) with slight modifications, the water-holding capacity (WHC) and oil-holding capacity (OHC) were determined by mixing the insoluble FRFs with distilled water (1:10, w/v) for 24 h and with vegetable oil (1:5, w/v) for 30 min, respectively. After centrifugation at 1006g, the WHC and OHC were expressed as milliliters of water held by 1 g of FRF and grams of oil held by 1 g of FRF, respectively. The density of vegetable oil is 0.88 g mL⁻¹.

Statistical Analysis. Data collected from this study were analyzed by the Duncan test using the Statistical Analysis System (SAS). An α level of 0.05 was set to determine statistical significance.

RESULTS AND DISCUSSION

In this study, the contents of peel, pulp, juice, and seed in the fresh Liucheng sweet orange (LSO) were found to be 262

Table 1. Proximate Composition of the Peel from *C. sinensis* L. Cv. Liucheng

| composition | g kg ⁻¹ of peel, dry wt |
|---|------------------------------------|
| moisture ^a | 753 \pm 10.2 |
| crude protein ^b | 102 \pm 3.70 |
| crude lipid ^a | 22.2 \pm 6.10 |
| total dietary fiber (TDF) ^{bc} | 570 \pm 10.0 |
| insoluble dietary fiber (IDF) ^{bc} | 476 \pm 10.8 |
| soluble dietary fiber (SF) ^{bc} | 94.1 \pm 0.90 |
| ash ^a | 33.0 \pm 0.50 |
| carbohydrate ^d | 273 |
| digestible starch ^b | 50.8 \pm 0.76 |

^a Means \pm SD of triplicates. ^b Means \pm SD of duplicates. ^c Fiber contents were corrected for protein and ash; contents of TDF, IDF, and SDF without correction were 665, 554, and 110 g kg⁻¹ of peel, respectively. ^d Carbohydrate was defined as the residue excluding protein, lipid, TDF, and ash and was calculated by difference (= 100 - protein - lipid - TDF - ash).

Table 2. Contents of AIS and WIS Prepared from the Peel of *C. sinensis* L. Cv. Liucheng

| fiber-rich fraction ^a | g kg ⁻¹ of peel, dry wt |
|--|------------------------------------|
| alcohol-insoluble solid (AIS) ^{b,c} | 515 \pm 11.1 |
| water-insoluble solid (WIS) ^{b,c} | 502 \pm 10.6 |

^a Fiber-rich fractions were determined on weight basis and were not corrected for protein and ash. ^b Means \pm SD of triplicates. ^c Starch contents in AIS and WIS were 1.16 \pm 0.83 and 0.33 \pm 0.16 g kg⁻¹ of FRF, respectively.

\pm 21.6, 275 \pm 34.0, 445 \pm 12.5, and 17.9 \pm 4.00 g kg⁻¹ of fresh fruit, respectively. In **Table 1**, the proximate composition in dry weight revealed that the LSO peel was rich in TDF (570 g kg⁻¹) and possessed low contents of crude protein (102 g kg⁻¹), crude lipid (22.2 g kg⁻¹), and ash (33.0 g kg⁻¹). The TDF content of the LSO peel was found to be higher than those of some other citrus fruit peels (139–140 g kg⁻¹), residues from some orange varieties (354–369 g kg⁻¹), and byproducts from some fruits and greens (358–588 g kg⁻¹) (10, 23, 24). The contents of IDF and SDF in the LSO peel were 476 and 94.1 g kg⁻¹, respectively (**Table 1**). IDF was the dominant fiber fraction (~83.5% of TDF); hence, these peel fibers might possibly give pronounced effects on intestinal regulation and stool volume, which are related to the consumption of insoluble fibers (25). Similarly, IDF was also found to be the larger fiber fraction in the peels or DF concentrates of some other fruits and vegetables (10, 23, 24).

The AIS and WIS were prepared from the LSO peel without enzymatic digestion. **Table 2** shows that the contents of AIS and WIS were 515 and 502 g kg⁻¹, respectively, and comparable to each other. For some other fruit byproducts, the AIS contents of mango and citrus wastes were reported to be 307–497 and 450–750 g kg⁻¹, respectively (12). In **Tables 2** and **3**, AIS and WIS were determined on a weight basis and were not corrected for protein (26.0 and <0.01 g kg⁻¹ of FRF, respectively), ash (45.2 and 30.8 g kg⁻¹ of FRF, respectively), and starch (1.16 and 0.33 g kg⁻¹ of FRF, respectively). The results revealed that both the AIS and WIS were fiber-rich and that relatively small amounts of impurities (31.1–72.4 g kg⁻¹ of FRF) was left in these two FRFs after the homogenization process.

Table 3 illustrates the contents of various monomeric sugars released from the peel FRFs after acid hydrolysis. The total sugar contents in the insoluble FRFs (IDF, AIS, and WIS) were found to be 67.3, 72.5, and 73.5 wt %, respectively. The quite similar contents of impurities between the AIS and WIS (**Tables**

Table 3. Monosaccharide Composition^a of Different Fiber-Rich Fractions^b Prepared from the Peel of *C. sinensis* L. Cv. Liucheng

| fiber-rich fraction | protein | ash | rhamnose | fucose | arabinose | xylose | mannose | galactose | NC-Glc ^c | C-Glc ^d | uronic acid |
|---------------------|---------|-------|----------|-----------------|-----------|--------|---------|-----------|---------------------|--------------------|-------------|
| IDF | 144x | 57.8x | 25.7x | tr ^e | 46.6xy | 29.3x | 27.2x | 54.6x | 30.1x | 223x | 236x |
| AIS | 26.0y | 45.2y | 19.8y | tr | 46.2y | 27.5x | 23.8x | 38.7x | 36.8x | 199y | 333y |
| WIS | tr | 30.8z | 21.7xy | tr | 46.7xy | 28.6x | 22.2x | 38.2x | 38.7x | 197y | 342y |
| SDF | 208z | | 9.10z | tr | 47.5x | 4.60y | 21.7x | 34.3x | 33.7x | | 246x |

^a Expressed as g kg⁻¹ of fiber-rich fraction. Values in the same column with different letters are significantly different (Duncan, $P < 0.05$). ^b Fiber-rich fractions were determined on weight basis and were not corrected for protein and ash. ^c NC-Glc, noncellulosic glucose. ^d C-Glc, cellulosic glucose. ^e Trace amount (< 0.01).

2 and 3) might partially explain their comparable contents of total sugars. Other components present in the FRFs might be protein, ash, polyphenols, and lignin (5). The percentages of different monomeric sugars including uronic acid, noncellulosic glucose, arabinose, and galactose in these insoluble FRFs were 35.1–48.2, 26.7–33.2, 6.24–6.93, and 5.20–8.12%, respectively. Apparently, arabinose, galactose, and uronic acid constituted ~50.1–60.0% of the total sugars contents. These results revealed that the peel-insoluble FRFs were mainly composed of pectic substances, followed by cellulose. Moreover, the small amounts of noncellulosic glucose and xylose in the insoluble FRFs indicated that hemicelluloses such as xyloglucan and starch were merely minor components. On the other hand, the sum of the three monomeric sugars including arabinose, galactose, and uronic acids accounted for ~82.6% of the total sugar content in the SDF, implying that the SDF was mainly composed of pectic substances (Table 3). The results among the IDF, SDF, AIS, and WIS (Table 3) also revealed that 15.8–17.2% of the LSO peel on a dry weight basis consisted of galacturonic acid, which was determined by uronic acids as polysaccharide residues. Previous findings from Grigelmo-Miguel and Martin-Belloso (23) and Baker (4) indicated that the residues from citrus fruits were strongly associated with pectin, which was mostly derived from the citrus peel. In general, a high similarity in the monosaccharide profiles among the three insoluble FRFs was observed except for a few differences in their uronic acid and cellulosic glucose contents (Table 3). Among the three insoluble FRFs, the significantly ($P < 0.05$) lower level of uronic acids in IDF might be attributed to the AOAC method in which the heating process led to the solubilization of some cell-wall pectic substances by dissolving the middle lamellae and breaking down pectins through β -elimination (26). Moreover, the significantly ($P < 0.05$) higher level of cellulosic glucose in IDF probably resulted from an improved depolymerization of cellulose, which swells more readily and disperses in acid after the thermal treatment in the AOAC method. Such an underestimation of cellulose had also been observed in some other cooked foodstuffs (27, 28).

As shown in Table 4, the bulk densities among the three LSO peel insoluble FRFs were significantly ($P < 0.05$) different from each other (WIS < cellulose < AIS < IDF). All of these insoluble FRFs were found to have comparable WHCs (15.5–16.7 mL g⁻¹), which were significantly ($P < 0.05$) higher than the WHC of cellulose (3.81 mL g⁻¹). The WHCs of these insoluble FRFs were also higher than those (~7–13 mL g⁻¹) obtained for the DF concentrates from some fruit byproducts (10, 23). The comparable WHCs among the insoluble FRFs might be attributed to their similarities in the number and nature of the water-binding sites, structure, and chemical composition (6, 29). In Table 4, the OHCs of the IDF (3.66 g g⁻¹) and AIS (5.09 g g⁻¹) were significantly ($P < 0.05$) higher than that of the reference cellulose (2.76 g g⁻¹), with WIS having its OHC comparable to that of cellulose. The higher OHC of AIS in relation to the IDF and WIS might be due to a larger number

Table 4. Physicochemical Properties of the Insoluble Fiber-Rich Fractions^a Prepared from the Liucheng Sweet Orange Peel Relative to Cellulose

| fiber sample | bulk density (g mL ⁻¹) | water-holding capacity (mL g ⁻¹) | oil-holding capacity (g g ⁻¹) | swelling (mL g ⁻¹) | cation-exchange capacity (mequiv kg ⁻¹) |
|------------------------|------------------------------------|--|---|--------------------------------|---|
| cellulose ^b | 0.38w | 3.81w | 2.76w | 4.97w | 23.0w |
| IDF | 0.53x | 16.2x | 3.66w | 21.1x | 997x |
| AIS | 0.48y | 15.5x | 5.09x | 14.6y | 454y |
| WIS | 0.19z | 16.7x | 2.35w | 16.7z | 523y |

^a Fiber-rich fractions were determined on weight basis and were not corrected for protein and ash. Values in the same column with different letters are significantly different (Duncan, $P < 0.05$). ^b Alphacel-Nonnutritive fiber, ICN Nutritional Biochemicals, Cleveland, OH.

of lipophilic sites released by the hot alcohol (85%, v/v) during the homogenization process. These results were higher than those obtained for some orange DF products (0.9–1.3 g g⁻¹) (23). The differences in the physicochemical properties (e.g., OHC) among the various FRFs and DF products might be attributed to their different chemical and physical structures (30) as well as the different preparation methods. Moreover, both the capillary attraction and hydrophobicity of FRFs would play a role in the physical entrapment of oil as well as the magnitude of OHC (31). According to the above results, the high WHCs and OHCs of the insoluble FRFs prepared from LSO peel suggested their potential uses in food applications as high dietary fiber ingredients to reduce calorie levels.

Table 4 indicates that the cation-exchange capacities of the peel-insoluble FRFs (454–997 mequiv kg⁻¹) were significantly ($P < 0.05$) higher than that of the cellulose (23.0 mequiv kg⁻¹), with IDF having the highest value (997 mequiv kg⁻¹). The stronger ion binding effect of these FRFs compared with that of cellulose might be due to the presence of uronic acids (236–342 g kg⁻¹) (Table 3) because the cation-exchange capacity is related to the uronic acid content of dietary fiber (6, 30). According to Furda (32), fibers with high cation-exchange capacities would entrap, destabilize, and disintegrate the emulsion of lipid, leading to the decrease in diffusion, absorption, and utilization of lipids including cholesterol. In Table 4, the swelling properties of the insoluble FRFs (14.6–21.1 mL g⁻¹) were also significantly ($P < 0.05$) higher than that of the cellulose (4.97 mL g⁻¹). According to the significantly ($P < 0.05$) higher cation-exchange and swelling capacities of the insoluble FRFs relative to cellulose, these insoluble FRFs might be able to create a concerted effect in reducing the number of intact micelles available, the transit time, and consequently the total time available for cholesterol absorption in the small intestine.

This study revealed that the LSO peel was rich in insoluble FRFs (IDF, AIS, and WIS), which were mainly composed of pectic polysaccharides and cellulose. These insoluble FRFs

could be useful in the food industry as high dietary fiber ingredients. Because the physicochemical properties of these peel-insoluble FRFs were generally higher than those of cellulose, it is speculated that these insoluble FRFs might have potential applications as low-calorie bulk ingredients in dietary fiber enrichment, baking, dietetic snacks, noodles, and some other foodstuffs requiring oil and moisture retention. As the fiber-rich LSO peel is available in large quantity as a byproduct in juice production, it could be exploited as a good source of dietary fiber. Further investigations on the physiological functions of these insoluble FRFs using animal-feeding experiments are underway.

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