Journal of Food and Drug Analysis

Volume 19 | Issue 3 Article 4

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Journal of Food and Drug Analysis, Vol. 19, No. 3, 2011, Pages 318-323 藥物食品分析 第十九卷 第三期

Preparation and Physicochemical Properties of Fiber-Rich Fraction from Pineapple Peels as a Potential Ingredient

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(Received: November 29, 2010; Accepted: March 29, 2011)

ABSTRACT

Pineapple (*Ananas comosus* L. Merr.) peels as agricultural wastes represent around 35% of the whole fruit mass. By different preparation methods, the pineapple peel fiber was analyzed and evaluated for its composition and functional properties, which would provide a clue to its physiological function. The pineapple peel contained an appreciable amount of insoluble fiber-rich fraction (FRF) (41.8 - 48.0 g/100 g) including insoluble dietary fiber (IDF), alcohol-insoluble solids (AIS), and water-insoluble solids (WIS), which primarily consisted of cellulose, pectic substances and hemicellulose. These fractions also contained notable proportions of lignin (60.7 - 65.8 g/100 g). Compared with cellulose, these FRFs exhibited the greater water- and oil-holding capacities (7.94 - 12.3 mL/g and 5.84 - 8.64 g/g, respectively), swelling properties (10.6 - 18.4 mL/g), and cation-exchange capacities (102 - 120 mequiv/kg). The results indicate that the physicochemical properties of various fibers are dependent on the preparation method and composition. Every insoluble FRF studied possesses interesting characteristics, suggesting possible uses in the development of functional food ingredients for reduction of calories or dietary fiber enrichment.

Key words: pineapple peel, dietary fiber, fiber-rich fractions, composition, physicochemical properties

INTRODUCTION

Dietary patterns are obviously associated with the prevention of chronic disease. More and more studies show that potential chronic diseases such as cardiovascular disease, diabetes, colon cancer, and constipation syndrome might be prevented by consumption of the dietary fiber found in fruits and vegetables[1,2]. Currently, considerable attention is being paid to dietary fiber, especially for its practical nutritional effects. Dietary fiber is composed of remnants of plant cell walls that are not hydrolyzed by intestinal enzymes in human. Note that the composition and physicochemical properties of dietary fiber depend on the extraction methods and sources[3]. In addition to the physicochemical properties given by high dietary fiber-rich food, studies in experimental animals and humans have suggested that fiber component can provide physiological effect such as reducing blood sugar content and decreasing serum lipid concentration[4]. In view of the potential benefits of dietary fiber, a wide range of fiber-rich products could be developed as functional food ingredients.

The production of pineapple (*Ananas comosus* L. Merr.) is increasing in tropical regions, and the annual yield in Taiwan is more than 400,000 metric tons. Currently, pineapples are commonly processed into canned fruit and juice, in addition to being eaten fresh. Since the peel of a pineapple accounts for the 34.7% of the whole fruit mass, there is a great interest in utilizing this biomasses as a source of dietary fiber, instead of feeding it to livestock. In previous studies[5,6], dietary fiber prepared from seeds, peels, and pomace from fruits has been found to possess excellent physicochemical properties. Given that dietary fiber with desirable nutritional and physicochemical properties is necessary for the food industry, it is worthwhile to process agricultural by-products in order to exploit fiber-rich fraction (FRF) as a potential functional food ingredient.

The objective of this study was to compare the composition and functional properties of FRFs derived from pineapple peel by various preparation methods. In addition, the reasonable use of FRFs in food applications will be discussed in this study. Results of this study provide insight into the differences in the composition and physicochemical properties of the FRFs that prepared from a potential source of biomass in order to develop valuable fiber ingredients suitable for usage in functional food.

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Journal of Food and Drug Analysis, Vol. 19, No. 3, 2011

MATERIALS AND METHODS

I. *Peel Samples*

Pineapples (hybrid, Tai-Nong-17, average weight of 1.45 ± 0.17 kg) were purchased from the supermarket with an. The peel of about 1 cm thick was manually cut using sharp knife and the collected peel sample was dried in an air oven (40°C) for 48 h, ground to pass through a screen of 0.5 mm and kept in a desiccator until used.

II. *Proximate Analysis*

The moisture was determined as the weight loss after an air-oven drying at 105°C until a constant weight was obtained[7]. The crude protein was estimated by multiplying the nitrogen content, which is determined by a CHNS-OS rapid element analyzer (Heraeus F002, Hanau, Germany) with a factor of 6.25. The ash content was determined as the weight loss after incarnation overnight at 550°C[7]. The crude lipid was determined using a Soxhlet apparatus with petroleum ether.

III. *Preparation and Analysis of Dietary Fiber (DF)*

According to AOAC method 991.43[7], DF was analyzed using the fiber assay kit (Megazyme K-TEFR, Wicklow, Ireland). Peel sample suspended in Mes-Tris buffer was sequentially digested by heat-stable α-amylase, protease, and amyloglucosidase to remove starch and protein. After centrifuged at 6510 × g for 10 min, the residue was the insoluble dietary fiber (IDF). The soluble dietary fiber in the supernant was further precipitated with four volumes of 95% ethanol and recovered by centrifugation at 6510 × g for 10 min. All DF fractions were dried by solvent-exchange and under air-oven overnight at 40°C. The DF contents were corrected for residual protein, ash, and blank.

IV. *Separation of Water-Insoluble Solids (WIS)*

Following the method of Massiot and Renard[8] with slight modifications, peel sample was homogenized in cold distilled water using Waring blender for 1 min (peel-to-water ratio of 1 : 30, w/v) at high speed. After filtration, the residue was washed with 70% ethanol, dried by solvent-exchange and air-oven at 40°C for 48 h. The content of WIS was corrected for residual protein and ash contents.

V. *Separation of Alcohol-Insoluble Solids (AIS)*

According to the method of Thomas *et al.*[9] with slight modifications, AIS was prepared by homogenizing the peel sample in boiling alcohol (85%, v/w) using Waring blender for 1 min at high speed. The peel-to-alcohol ratio is 1 : 30 (w/v). The suspension was boiled for another 40 min and then filtrated. The residue was washed with 70% ethanol, and then air-dried at 40°C for 48 h. The content of AIS was corrected for residual protein and ash contents.

VI. *Analysis of Fiber Component*

The sugar composition of all FRFs was based on the method described by Englyst *et al.*[10] and Southgate[11]. All FRFs (20 mg) were pre-hydrolyzed with 12 M H_2SO_4 at 35°C for 60 min and further boiled in 2 M H_2SO_4 for another 60 min. The individual neutral sugars were reduced, acetylated, and analyzed by gas chromatography (Thermo FOCUS GC series, Milan, Italy) equipped with a flame ionization detector and a capillary column (Quardex 007-225; 15 m × 0.53 mm i.d.), using allose as an internal standard. Nitrogen was used as carrier gas with a flow rate of 2.0 mL/min. The temperature program was set as follows: the initial column temperature of 100°C was held for 3 min and then the temperature was increased at 4°C/min to 160°C, and held for 5 min. Thereafter, the temperature was increased again at 3°C/min to 220°C, and held for 1 min. Detector and injector temperatures were held at 280°C and 270°C, respectively.

For the determination of noncellulosic glucose, FRFs were hydrolyzed with 2 M H_2SO_4 at 100°C for 60 min. The amount of cellulose glucose was calculated by subtraction of noncellulosic glucose from the total glucose present in the 12 M (followed by 2 M) H_2SO_4.

According to AOAC method 45.4.11[7], the uronic acid content in the acid hydrolysate was determined colorimetrically with 3,5-dimethylphenol. D-galacturonic acid was used as a standard. The pectin content in the sample was estimated by the amount of uronic acids, which were measured as polysaccharide residues.

Lignin was determined gravimetrically as Klason lignin. After acid hydrolysis, the residual material was dried and weighed.

VII. *Physicochemical Properties*

The bulk density was determined according to the methods described by Chau[12]. A known amount of FRFs was packed in 10 mL graduated cylinder by gently tapping cylinder on the bench top 20 times. While the volume of contents did not reduced further, the volumetric measurement was precisely recorded and the result was expressed as grams per milliliter.

The swelling property of FRFs was measured by bed volume technique[13]. Insoluble FRFs (1 g) was hydrated in a known volume of distilled water (10 mL), in a calibrated cylinder (1.5 cm diameter) at room temperature. After equilibration (24 h), the bed volume was recorded and the swelling property was expressed as milliliter of swollen sample per gram of dry initial sample. Oil-holding capacity (OHC) of FRFs was determined by the method of Chau *et al.*[14] with slight modifications. One gram of insoluble FRFs was mixed with 10 mL of vegetable oil in a 50-mL centrifuge tube. After centrifuged at 3720 × g for 10 min, the free oil was decanted and weighed. OHC was expressed as grams of oil held by 1 g of FRF. The density of vegetable oil is 0.88 g/mL.

Water-holding capacity (WHC) of FRFs was measured as described by Chau *et al.*[14] with slight modifications. One gram of FRFs was soaked in 10 mL of distilled water for 24 h and then centrifuged at 3720 × g for 10 min. The supernatant was carefully decanted into the graduated cylinder and the volume of excess water was read. Hence, WHC was expressed as milliliters of water held by 1 g of FRF.

For the determination of cation-exchange capacity, FRFs was converted to the protonated form by treatment with 2 M HCl under magnetic stirring overnight at 4°C according to the method of Chau[12]. After extensive washing, the solution was titrated with 0.1 N NaOH and cation-exchange capacity was calculated as milliequivalents per kilogram of FRFs.

In the measurement of physiochemical properties, the cellulose (MP Biomedicals, Solon, OH, USA) of food grade was used as reference.

VIII. *Statistical Analysis*

Data obtained from this study were analyzed by one-way analysis of variance using the Statistical Analysis System (SAS). Values of $p < 0.05$ were considered to be significant.

RESULTS AND DISCUSSION

The content of peel and pulp in the fresh pineapple were about 34.7 ± 1.29 and 65.3 ± 1.29 g/100 g, respectively. The proximate composition of pineapple peel is shown in Table 1. The moisture content in the pineapple peel was 82.7 g/100 g for fresh fruit. TDF was present in large quantities (42.2 g/100 g), of which IDF was the major constituent (36.3 g /100 g), with only minor amounts of SDF (5.90 g/100 g). In terms of dry weight, pineapple peel was low in crude protein (9.13 g/100 g), ash (4.81 g/100 g), and lipid (1.57 g/100 g). As compared to the agricultural by-products from pears, peaches, and oat bran, with TDF of 23.8 - 36.1 g/100 g and IDF of 20.2 - 26.1 g/100 g[15], pineapple peels was found to have higher TDF content, and to be particularly rich in IDF. This suggests that DF obtained from pineapple peel may be used as an abundant source of IDF. Some studies have reported that IDF is characterized by its ability to decrease intestinal transit and increase fecal weight [16,17].

Table 2 presents the yield of the FRFs obtained from pineapple peel by various preparation methods. The AIS content (48.0 g/100 g) was significantly ($p < 0.05$) higher than the WIS content (41.8 g/100 g), which is in agreement with a previous investigation on fruit such as apple tissue[8]. Accordingly, preparation of AIS is suitable for cell-wall polysaccharides from fruits and vegetables, in which low amounts of starch and intra-cellular proteins are present[18]. On the other hand, the higher protein content in the AIS (3.69 g/100 g) relative to other insoluble FRFs (2.63 g/100 g of WIS and 1.25 g/100 g of IDF, respectively) could be ascribed to cell-wall proteins or co-precipitated intracellular proteins (Table 3). It is clear that relatively small amounts of impurities (such as protein and ash) remained in all three FRFs after the process of homogenization or enzymatic digestion.

Table 3 shows that all insoluble FRFs contained varying amounts of total sugars, from 29.2 to 32.7 g/100 g, but differed slightly in their respective contents of monomeric sugars. In terms of sugar composition, these insoluble FRFs contained a considerable amount of cellulosic glucose (8.33 - 10.7 g/100 g), followed by xylose (7.83 - 9.23 g/100 g) and uronic acid (6.83 - 8.26 g/100 g). It can thus be deduced that these fractions contained certain polysaccharides, such as cellulose, hemicellulose (xylans or xyloglucan), and pectic substances, as major constituents. Significant differences in several monomeric sugars, namely xylose, mannose, noncellulosic glucose, and uronic acid, were observed in SDF and insoluble FRFs in the pineapple peel. Considering the distribution of monomeric sugars released from polysaccharides[19], the sugar components of pectic polysaccharides are galacturonic acid, rhamnose, arabinose, and galactose, whereas those of hemicelluloses are glucose, xylose, and mannose. As the arabinose, rhamnose, galactose, and uronic acids in the SDF accounted for approximately 76.1% of the total sugar content, it is assumed that SDF fraction in the pineapple peel was mainly composed of arabinose-rich pectic polysaccharides. Among the three insoluble FRFs, the significantly ($p < 0.05$) higher level of cellulosic glucose in IDF can probably be attributed to ameliorated depolymerization of cellulose, which swells readily and disperses in acid after thermal process by the AOAC method[20,21]. Furthermore, WIS and IDF (65.8 g/100 g and 64.1 g/100 g, respectively) had a higher quantity of lignin than AIS (60.7 g/100 g).

Table 1. Proximate composition of the pineapple peels[a]

Composition	g/100 g peel, dry wt
Crude protein[b]	9.13 ± 0.25
Crude lipid[b]	1.57 ± 0.13
Total dietary fiber (TDF)[b,c]	42.2 ± 0.89
Insoluble dietary fiber (IDF)[b,c]	36.3 ± 0.79
Soluble dietary fiber (SDF)[b,c]	5.90 ± 0.19
Ash[b]	4.81 ± 0.03
Carbohydrate[d]	42.3

[a] The moisture content of pineapple peel is 82.7 ± 1.91.
[b] Results are mean ± SD of triplicate analyses.
[c] The fiber contents have been corrected for protein and ash.
[d] Carbohydrate was calculated as 100 - (ash + protein + lipid).

Table 2. Distribution of fiber-rich fractions prepared from pineapple peels

Fiber-rich fractions	g/100 g peel, dry wt
Water-insoluble solid (WIS)[a,b]	41.8 ± 0.14
Alcohol-insoluble solid (AIS)[a,b]	48.0 ± 0.12

[a] Results are mean ± SD of triplicate analyses.
[b] FRFs contents have been corrected for protein and ash.

Journal of Food and Drug Analysis, Vol. 19, No. 3, 2011

Table 3. Monosaccharide composition[a] and constitutes[a] of fiber-rich fractions[b] prepared from the pineapple peels

	WIS	AIS	TDF	
			IDF	SDF
Rhamnose	Tr[f]	0.05 ± 0.01	Tr	0.08 ± 0.01
Fucose	Tr	Tr	Tr	Tr
Arabinose	2.42 ± 0.08w	2.60 ± 0.16w	2.96 ± 0.02x	2.20 ± 0.07y
Xylose	7.83 ± 0.32w	8.70 ± 0.64w	9.23 ± 1.00x	3.92 ± 0.20y
Mannose	0.63 ± 0.05w	0.75 ± 0.03x	0.55 ± 0.02w	3.62 ± 0.07y
Galactose	1.71 ± 0.11w	1.58 ± 0.01w	1.96 ± 0.05x	1.75 ± 0.05w
NC-Glc[c]	0.60 ± 0.06w	0.98 ± 0.02x	0.46 ± 0.06y	1.04 ± 0.04x
C-Glc[d]	8.48 ± 0.10w	8.33 ± 0.05w	10.7 ± 0.03x	–
Uronic acid	7.52 ± 1.42w	8.26 ± 0.73w	6.83 ± 0.48w	23.2 ± 1.26x
Total sugar	29.2	31.3	32.7	35.8
KL[e]	65.8 ± 2.52w	60.7 ± 1.63x	64.1 ± 0.02w	–
Protein	2.63 ± 0.53w	3.69 ± 0.27x	1.25 ± 0.18y	22.8 ± 0.05z
Ash	2.50 ± 0.04w	3.68 ± 0.04x	1.87 ± 0.03y	–

[a] Expressed as g/100 g fiber-rich fraction. Values (Means ± SD) with different letters (w-z) in the same row indicate significantly differences (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] KL, Klason lignin.
[f] Tr, Trace amount (< 0.01).

As reported by Rebole *et al.*[22], many components, such as condensed tannins and protein-tannin complexes associated with the fiber, were found in the lignin fraction of rich-fiber agricultural by-products. Therefore, we can conclude that the pineapple peel fiber would be a good candidate for acquiring functional components.

The physiochemical properties of fibers prepared from some vegetables and fruits are quite well known, but the fibers present in pineapple peel have not been studied. Table 4 shows that the WHC values of various insoluble FRFs resulted from the preparation methods used in the present study. The WHCs of the IDF (11.1 mL/g), WIS (7.94 mL/g), and AIS (12.3 mL/g) were significantly ($p < 0.05$) higher than that of cellulose (2.82 mL/g). In addition, the observed WHCs in the present study were higher than those obtained from fruit by-product fiber from citrus pulp (5.66 g/g) and passion fruit seeds (2.37 - 3.20 g/g)[5,23]. This suggests that the increase in the WHCs of these FRFs was probably due to an increase in the amount of water which could be bound through their structure and to the varied fractional compositions of fibers. In general, lignin fraction possesses hydrophobic properties and binds significantly lower amount of water relative to hydrophilic polysaccharides[24]. Regarding the relationship between lignin content and WHC value in our study, it was found that the higher lignin contents in the WIS (65.8 g/100 g), IDF (64.1 g/100 g) and AIS (60.7 g/100 g) resulted in the lower WHC. As a result, it can be speculated that the amount of lignin in the insoluble FRFs may play a role in their ability to hold water, leading to an apparent difference in WHC among FRFs. Moreover, some studies have demonstrated that WHC of fruit by-product depends on fiber preparation and on its chemical composition and physical structure[15,25,26]. The increase in WHC of the FRFs suggests that they can be used as functional ingredient to reduce drip release and to modify texture in the minced beef[27] and can further be applied to providing the satiety effects of fiber-supplemented products. Generally, WHC has been widely studied in food functionality because water plays a significant role in the food industry, as in baking processes, starch gelatinization, and protein denaturation[28,29]. From the physiological point of view, measuring bound water by centrifugation may be useful for predicting the fecal bulking ability of a fiber source, as bound water will be trapped within the fiber matrix[30].

Table 4 also indicates the swelling capacities of various FRFs from pineapple peel. The swelling capacity ranged from 10.6 to 18.4 mL/g, with IDF giving the highest value ($p < 0.05$). This suggests that the excellent swelling property of IDF is possibly related to the susceptibility of the fiber sample to enzymatic hydrolysis. Among the three FRFs, the trend of reduction in swelling capacities is the opposite of those of bulk density. The greater swelling capacity of FRF obtained from pineapple peel could be ascribed to the destruction of the inner structure of the FRFs matrix during preparation and to discrepancies in bulk density. The swelling capacity of FRFs derived from pineapple peel was higher than that observed in some food by-products such as pea hulls (7.8 - 9.9 mL/g)[30] and cumin spent residue

Table 4. Physicochemical properties of the insoluble fiber-rich fractions[a] prepared from the pineapple peels

Fiber samples	Bulk density (g/mL)	Water-holding capacity(mL/g)	Oil-holding capacity(g/g)	Swelling (mL/g)	Cation-exchange capacity (mequiv/kg)
Cellulose[b]	0.28 ± 0.01w	2.82 ± 0.45w	4.30 ± 0.15w	7.70 ± 0.02w	34.5 ± 3.23w
WIS	0.19 ± 0.01x	7.94 ± 1.47x	8.64 ± 0.74x	13.1 ± 0.18x	108 ± 2.97x
AIS	0.37 ± 0.01y	12.3 ± 1.52y	6.03 ± 0.06y	10.6 ± 0.51y	120 ± 1.84y
IDF	0.13 ± 0.01z	11.1 ± 0.12y	5.84 ± 0.82y	18.4 ± 3.01z	102 ± 1.84z

[a] Fiber-rich fractions were determined on weight basis and were not corrected for protein and ash. Values (Mean ± SD) with different letters (w-z) in the same column indicate significantly differences (Duncan, $p < 0.05$).
[b] Alphacel-Nonnutritive fiber, MP Biomedicals, Solon, OH, USA.

(4.5 - 5.0 mL/g)[31]. In addition to the source and preparation method, some physical properties, such as structure, density, and porosity, were considered to be related to swelling property[30].

In Table 4, it can also be seen that FRFs had higher OHC value (5.84 - 8.64 g/g) than cellulose (4.30 g/g) and other agricultural by-products such as flax hulls, wheat bran, and pea hulls (0.8 - 2.0 g/g)[32]. In this case, the increase in the OHC of insoluble FRFs might be due to the high lignin content present in the FRFs. Moreover, the number of hydrophobic groups released in the FRFs is probably affected by preparation methods. Kinsella[33] pointed out that both the capillary attraction and hydrophobic characteristics of FRFs sould have a critical effect in the physical entrapment of components of oily nature. Hence, these insoluble FRFs appeared to stabilize foods with a high percentage of fat and emulsions. The cation-exchange capacity of the various FRF fractions is given in Table 4. The cellulose (34.5 mequiv/kg) had a cation-exchange capacity significantly lower than that of AIS (120 mequiv/kg), WIS (108 mequiv/kg), and IDF (102 mequiv/kg). From the finding on sugar-beet pulp studied by Dronnet *et al.*[34], cation-exchange capacity is related to the uronic acid content of fiber. In light of the above-mentioned observations regarding the sugar composition of insoluble FRFs, a strong linear positive correlation was observed between the uronic acid contents and the cation-exchange capacities ($r = 0.99$; $p < 0.05$) of the fibers. The result obtained from our study confirms that the stronger ion binding effect of these FRFs, as compared with that of cellulose, is the result of the presence of uronic acid. As reported by Furda[35], the high cation-exchange capacities of fibers suggest that these materials could destabilize, entrap, and disintegrate the micelles formed by emulsion of lipid as a result of interference with the diffusion or absorption of micelles caused by fibers.

CONCLUSIONS

This study revealed that pineapple peel is rich in insoluble FRFs which were primarily composed of cellulose, pectic substances and hemicellulose. These FRFs were found to exhibit superior WHCs, OHCs, and cation-exchange capacities over cellulose. The results of our study confirmed that insoluble FRFs from pineapple peel have excellent potential in food applications as a functional ingredient, especially in the development of food reduced in calories and dietary fiber enriched food product. Further studies using animal-feeding experiments are needed to confirm their possible roles in physiological functions.

ACKNOWLEDGMENTS

This study was carried out with financial support from the National Science Council of the Republic of China (NSC 98-2313-B-022-001).

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