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Soluble dietary fiber from *Canna edulis* Ker by-product and its physicochemical properties

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ABSTRACT

Using *Canna edulis* Ker by-product as raw materials, soluble dietary fiber (SDF) was prepared using six different methods, including chemical, physical–chemical, enzymatic, physical–enzymatic, chemical–enzymatic and physical–chemical–enzymatic methods. As main component in the *C. edulis* by-product composed of cellulose, glucose converts to other single sugars, which form a series of compounds in the SDF. The treated methods have impact effects on single sugar composition, metal ion content, molecular size distribution, chemical bonds and groups in the structure, thermal property and color of the final product. In view of security, high yield and homogeneity as well as good thermal stability of final product, physical–enzymatic method will be a best choice for the production of SDF from *C. edulis* by-product. The SDF obtained can be used as dietary supplement and additive in the food industry.

1. Introduction

It has been proved that intake of diet containing high dietary fiber (DF) is negatively related to the incidence rate of chronic diseases, such as obesity, diabetes mellitus, large bowel cancer, cardiovascular disease, colonic diverticulosis and constipation, so on (Eshak et al., 2010; Isken, Klaus, Osterhoff, Pfeiffer, & Weickert, 2010; Kim, 2000). However, the average worldwide ingestion of this component is still considerably lower than the recommended daily intake levels. So, it is necessary to add DF to food products. As a kind of newly bioactive constituent, DF has become a hot topic in the research field. Moreover, it has been reported that soluble dietary fiber (SDF) is more beneficial to human than insoluble dietary fiber (IDF) (Chawla & Patil, 2010). Additionally, it is also recommendable to explore new sources of fiber in the sight of precise technological and physiological functionality of the fiber depending on its sources and chemical composition.

Canna edulis Ker, belonging to the genus *Canna* (Cannceae), is largely cultivated in South America, Vietnam, Thailand and China (Chansri, Puttanlek, Rungsadthong, & Uttapap, 2005). The dry rhizome of *C. edulis* contains 70–80% starches which are reported more digestible than other kinds of starches (Perez, Lares, & Gonzalez, 1997). After starch extraction, abundant residues discarded as waste are potentially an environmental problem because they are highly susceptible to putrefaction. Additionally, residue also has a high moisture content (80%), making it difficult to handle and too expensive to dry by conventional means. According to our knowledge, this by-product might be mainly consisted of fiber, which is a good source for the preparation of SDF. Consequently, it is greatly significant to exploit SDF from *C. edulis* by-product.

Therefore, the objective of this work was to recycle and utilize *C. edulis* by-product after the extraction of starch. SDF was prepared by six different kinds of methods from the by-product, and its physicochemical properties including color, ionic content, single sugar composition, molecular weight and thermal characteristics were determined.

2. Materials and methods

2.1. Samples and reagents

Fresh rhizomes of *C. edulis* were obtained from Guizhou Ziyun Jiahe Chemical Co. Ltd. in the Guizhou Province of China. *C. edulis* by-product was obtained from residues of rhizomes after the extraction of starch. The by-product was washed with water several times and dried at ambient temperature for 24 h. The products were grounded with a mortar and pestle to a fine powder passing through a 60 mesh sieve. The powder was transferred to airtight plastic bags and stored in a desiccator at room temperature for further analysis. Cellulase was purchased from Yakult Co. Ltd. (Japan). The other enzymes and standards were obtained from

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Sigma–Aldrich Chemical Co. Ltd. (USA). All other reagents were of chromatographic or analytical quality.

2.2. Sample preparation

C. edulis SDF was prepared through six kinds of methods, including physical, enzymatic, chemical methods and their different combinations.

2.2.1. Chemical method

C. edulis by-product (1 g) was solubilized in 2% HAc (20 mL), followed by the addition of 5% H_2O_2 (10 mL), and the mixed solution was heated at 60 °C for 2 h. After cooled, the mixture was adjusted to pH 10 with sodium hydroxide and filtrated after 2 h. Then ethanol (200 mL) was added into the filtrate and stay overnight. The precipitation obtained was washed with ethanol until liquid became neutral, and freeze-dried to give SDF, named CSDF.

2.2.2. Enzymatic method

C. edulis by-product (1 g) was solubilized in PBS buffer (20 mL 0.2 M pH 6.9). The mixture was incubated at 37 °C for 2 h with the addition of 5 mL of porcine amylase solution, which was prepared through solubilization of porcine pancreatic α-amylase powder (Type VI-B from porcine pancreas (≥10 units solid)) (10 mg) in 10 mL of PBS buffer (0.2 M pH 6.9), and adjusted pH value to 5.0. Then 65 mg of cellulase was added and the mixture was hydrolyzed at 40 °C. After 4 h, the pH value was adjusted to 5.2. The mixture was incubated at 37 °C for 2 h, after the addition of mixed liquid of pancreatin (A0585, from porcine pancreas, Sigma) and amyloglucosidase (A3306, from *Aspergillus niger*, Sigma), which was prepared freshly through mixture of amyloglucosidase (100 μL) and supernatant liquid of pancreatin obtained through centrifugation of mixed solution of pancreatin powder (200 mg) and PBS buffer (10 mL 0.2 M). After cooled, the mixture was adjusted to pH 10 with sodium hydroxide and filtrated after 2 h. Then ethanol (200 mL) was added into the filtrate and stay overnight. The precipitation obtained was washed with ethanol until liquid became neutral, and freeze-dried to give SDF, named ESDF.

2.2.3. Physical–chemical method

C. edulis by-product (1 g) was solubilized in 2% HAc (20 mL), followed by the addition of 5% H_2O_2 (10 mL), and ultrasonicated for 15 min at room temperature. Then the reactive mixture was further treated using the method described in Section 2.2.1, and the final product obtained was named PCSDF.

2.2.4. Physical–enzymatic method

C. edulis by-product (1 g) was solubilized in PBS buffer (20 mL 0.2 M pH 6.9) and ultrasonicated for 15 min at room temperature. Then the mixture was further treated using the method described in Section 2.2.2, and the final product obtained was named PESDF.

2.2.5. Chemical–enzymatic method

C. edulis by-product (1 g) was solubilized in 2% HAc (20 mL), followed by the addition of 5% H_2O_2 (10 mL). The mixed solution was heated at 60 °C for 2 h. After cooled, the mixture was adjusted to pH 6.9 with sodium hydroxide and further treated using the method described in Section 2.2.2. The final product was named CESDF.

2.2.6. Physical–chemical–enzymatic method

C. edulis by-product (1 g) was solubilized in 2% HAc (20 mL), followed by the addition of 5% H_2O_2 (10 mL), and ultrasonicated for 15 min at room temperature. The mixed solution was further treated using the method described in Section 2.2.5. The final product was named PCESDF.

2.3. Ionic content

The ionic contents including kalium, sodium and calcium were determined using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICPAES) (IRIS Advantage 1000, Thermo Electron, USA).

2.4. Single sugar composition

SDF obtained was hydrolyzed, and the released sugars were transformed into alditol acetates with acetic anhydride in the presence of 1-methylimidazol according to the method of Blakeney, Harris, Henry, and Stone (1983). Quantification was performed in a Shimadzu GC-2010 gas chromatograph (Shimadzu Co., Kyoto, Japan) equipped with a hydrogen flame ionization detector. The column used was Rtx-5 (30 m × 0.25 mm × 0.25 μm) and nitrogen served as carrier gas. Temperatures of injector and detector were 290 and 280 °C, respectively. An oven temperature program of initial temperature 100 °C with a hold of 5 min, followed by a temperature rise of 20 °C/min to 190 °C, 3 °C/min to 260 °C, and 10 °C/min to 280 °C with a final hold of 5.17 min was conducted. Data were collected and processed with an Agilent Chem Station software system (Agilent Technologies, Waldrom, Germany) and β-D-allose was used as internal standard.

2.5. Molecular weight

The samples were dissolved in HPLC grade water and centrifuged at 10,000 rpm for 10 min. The supernatant obtained was analyzed for molecular weight distribution using a Waters™ 650E Advanced Protein Purification System (Waters Corporation, Milford, MA, USA) assembly running 10 μL sample at 0.5 mL/min. TSK gel 2000 SWXL (7.8 mm i.d. × 300 mm) column was regulated at 30 °C. A calibration curve was obtained with bovine carbonic anhydrase (29,000 Da), horse heart cytochrome C (12,500 Da), aprotinin (6500 Da), bacitracin (1450 Da), gly-gly-tyr-arg (451 Da) and gly-gly-gly (189 Da). With the help of elution time of calibration materials, the linear regression equation was obtained for the calculation of molecular weight. The results were processed with Millennium32 Version 3.05 Copyright © 1998 (Waters Corporation, Milford, MA, USA) (Wu, Wang, & Xu, 2007).

2.6. Fourier transforms infrared spectroscopy

FT-IR spectra were obtained on an Equinox 55 Fourier Transform Infrared-Raman spectrometer (Bruker Co., Germany). The spectra were recorded in transmission mode from 1200 to 800 cm^{-1} at a resolution of 0.44 cm^{-1} at room temperature. Samples were diluted with KBr (1:100, v/v) before acquisition, and the background value from pure KBr was acquired before the sample was scanned.

2.7. Thermal analysis

The simultaneous thermal analysis (differential scanning calorimetry DSC and thermogravimetry TG) was carried out using a STA 409 C-device (Netzsch, Selb, Germany) according to Einhorn-Stoll, Kunzek, and Dongowski (2007), using the following conditions: linear heating rate 10 K/min from 20 to 550 °C, dynamic inert nitrogen atmosphere (75 mL/min), empty crucible as reference, sample weight approximately 10–20 mg. All runs were performed at least in duplicate. The extrapolated onset, peak and offset temperatures as well as the maximum heat flow E_{max} and the maximum degradation velocity v_{max} were calculated with the Netzsch software.

Table 1
Single sugar composition and metal ion content of soluble *Canna edulis* Ker dietary fibers.

	CSDF	ESDF	PCSDF	CESDF	PESDF	PCESDF
Content[a,b] (%)	9.02 ± 0.60 a	6.34 ± 0.12 b	35.43 ± 0.67 c	28.44 ± 1.07 d	68.71 ± 3.05 e	12.25 ± 1.11 f
Sugar[a,b] (%)						
Rha	0.29 ± 0.03 a	2.51 ± 0.02 b	0.23 ± 0.04 a	4.08 ± 0.09 b	3.83 ± 0.14 c	3.74 ± 0.11 c
Ara	0.21 ± 0.03 a	4.20 ± 0.03 b	0.16 ± 0.03 a	1.02 ± 0.04 c	13.41 ± 0.36 d	0.53 ± 0.03 e
Xyl	4.01 ± 0.02 a	2.83 ± 0.09 b	1.84 ± 0.05 c	6.41 ± 0.09 d	37.00 ± 0.70 e	8.16 ± 0.08 f
Man	1.07 ± 0.01 a	25.14 ± 0.27 b	0.74 ± 0.03 a	21.65 ± 0.90 b	11.31 ± 0.37 b	22.37 ± 0.45 b
Glu	89.38 ± 1.76 a	58.02 ± 0.50 b	93.18 ± 1.50 c	54.48 ± 1.26 d	23.85 ± 0.92 e	53.54 ± 1.24 d
Gal	4.74 ± 0.10 a	6.87 ± 0.24 b	3.08 ± 0.12 c	11.78 ± 0.38 d	10.18 ± 0.25 e	11.75 ± 0.23 d
Metal ions[a,b] (%)						
K	0.067 ± 0.001 a	0.059 ± 0.002 c	0.034 ± 0.001 a	0.085 ± 0.019 b	0.046 ± 0.007 c	0.093 ± 0.001 c
Na	6.62 ± 0.05 a	3.33 ± 0.01 c	2.36 ± 0.02 a	15.44 ± 0.03 b	25.43 ± 0.21 c	3.69 ± 0.01 c
Ca	1.52 ± 0.02 a	2.22 ± 0.01 c	1.90 ± 0.05 a	2.25 ± 0.29 b	0.077 ± 0.000 c	2.20 ± 0.03 c

[a] Values are means ± S.D.
[b] Values not sharing a common letter are significantly different on the same row ($P < 0.05$).

2.8. Color determination

The CIE lab co-ordinates (L^*, a^*, b^*) were directly read in a glass cuvette with spectrophotocolorimeter Mini Scan XETM (HunterLab Inc., Reston, VA, USA). In this coordinate system, the L^* value is a measure of lightness, ranging from 0 (black) to 100 (white); the a^* value ranges from −100 (greenness) to +100 (redness) and the b^* value ranges from −100 (blueness) to +100 (yellowness).

2.9. Statistical analysis

All determinations were triplicates, and mean values and standard deviations were calculated. Analysis of variance (ANOVA) was performed and the mean separation was done by LSD ($P \leq 0.05$). Moreover, correlation matrix and principal component analysis were conducted using SPSS 16.0 program for windows (SPSS Inc., IL, USA).

3. Results and discussion

3.1. Single sugar composition and metal ion content

SDF was prepared through six different methods from *C. edulis* by-product, and its content obtained is shown in Table 1. The prepared methods significantly influence the production of SDF at the statistical level ($P < 0.05$). The content of PESDF (68.71%) is maximized among six SDFs. In view of its higher production than that of ESDF (6.34%), it can be deduced that ultrasonic treatment is beneficial to enzymatic hydrolysis of the by-product. Under ultrasonic treatment, the sample is dispersed and destroyed fully, leaving small and incompact blocks, which are easily attacked. It is the same reason explained for PCSDF (35.43%), in comparison with CSDF (9.02%). The contents of both CSDF (9.02%) and ESDF (6.34%) are lower than that of CESDF (28.44%), suggesting that the addition of H_2O_2 and enzymes improve the formation of SDF. Surprisingly, the content of PCESDF (12.25%) is lower than those of PCSDF, CESDF and PESDF, although the value is higher than those of CSDF and ESDF. It indicates that the combination of three methods has a negative effect on the production of *C. edulis* SDF.

For six different SDFs, their single sugar compositions were analyzed through GC and the corresponding results are exhibited in Table 1. For CSDP and PCSDF, glucose (Glu) is main constituent (about 90%), suggesting the presence of glucan. The sum of mannose (Man) and Glu contents attains to above 75%, which signifies the existence of glucomannan in the ESDF, CESDF and PCESDF. Compared with the three SDFs, CSDP and PCSDF exhibits evidently different compositions of single sugars and it can conclude that enzyme treatment has a significant impact on chemical composition of final product, i.e. SDF. Different from other SDFs, PESDF is mainly composed of xylose (Xyl) (37.00%) and Glu (23.85%), with arabinose (Ara) (13.41%), Man (11.31%) and galactose (Gal) (10.18%). This might indicate the presence of xyloglucan and arabinoxylan. It can draw a conclusion that the course of SDF production from IDF is very complex and correlates with processing method.

Metal ion contents of six SDFs were detected by ICP-MS and the results are shown in Table 1. For all SDFs, Na content maximizes, resulting from Na residue in the process of SDF production. Except for PESDF, the contents of Ca reach to about 2%, which implies that these SDFs could be used as calcium supplement in the functional food. Compared to those of Ca, the contents of K are litter in all SDFs.

As shown in Table 2, the correlation coefficients between each mean variable were computed. The contents of SDF are strongly positively correlated with the values of Ara ($r = 0.766$), Xyl ($r = 0.845$) and K ($r = 0.812$), and negatively with Ca ($r = -0.809$) value. This indicates that the formation of arabinoxylans, also confirmed by correlation between Ara and Xyl ($r = 0.929$), is a key factor to determine SDF yield, and K ion promotes SDF production, inversely, Ca ion prevents. It could be explained by solubility of kalium salt and insolubility of calcium salt. Glu value is clearly negatively correlated with Rha ($r = -0.892$), Ara ($r = -0.792$),

Table 2
Correlation coefficients, *R*, for relationships between values for SDF yield, single sugar composition and metal ion content.

	Content	Rha	Ara	Xyl	Man	Glu	Gal	Na	K	Ca
Content	1.000	0.285	0.766*	0.845*	−0.259	−0.535	0.186	−0.509	0.812*	−0.809*
Rha	0.285	1.000	0.440	0.500	0.795*	−0.892*	0.964*	0.532	0.563	−0.097
Ara	0.766*	0.440	1.000	0.929*	0.083	−0.792*	0.285	−0.392	0.794*	−0.854*
Xyl	0.845*	0.500	0.929*	1.000	−0.013	−0.797*	0.426	−0.233	0.877*	−0.905*
Man	−0.259	0.795*	0.083	−0.013	1.000	−0.592	0.732*	0.610	0.034	0.395
Glu	−0.535	−0.892*	−0.792*	−0.797*	−0.592	1.000	−0.796*	−0.189	−0.745*	0.488
Gal	0.186	0.964*	0.285	0.426	0.732*	−0.796*	1.000	0.697	0.510	−0.036
Na	−0.509	0.532	−0.392	−0.233	0.610	−0.189	0.697	1.000	−0.091	0.487
K	0.812*	0.563	0.794*	0.877*	0.034	−0.745*	0.510	−0.091	1.000	−0.769*
Ca	−0.809*	−0.097	−0.854*	−0.905*	0.395	0.488	−0.036	0.487	−0.769*	1.000

* Significant correlation between two variants ($P < 0.05$).

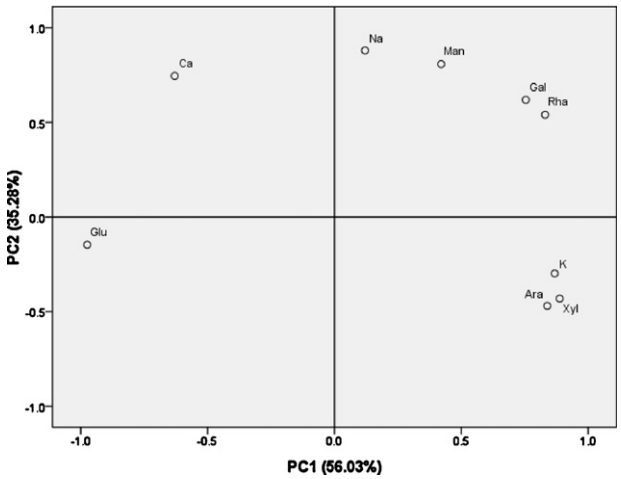

Fig. 1. Principal component analysis biplot of single sugar composition and metal ion content of soluble *Canna edulis* Ker dietary fibers.

Xyl ($r = -0.797$) and Gal ($r = -0.796$) values. This indicates that Glu transforms to other single sugar in the process of SDF. In view of obviously negative correlation between K and Glu values ($r = -0.745$), K ion is beneficial to this chemical reaction, which is also approved by significantly positive correlations between K and Ara ($r = 0.794$), K and Xyl ($r = 0.877$) values. The strongly negative correlations between Ca and Ara ($r = -0.854$), Ca and Xyl ($r = -0.905$), K and Ca ($r = -0.769$) values further confirm that Ca inhibits the transform from IDF to SDF.

Principal component analysis (PCA) was applied for the values of single sugars and metal ions, and the corresponding biplot is shown in Fig. 1. A 91.31% of the total variance could be explained by the greatest eigenvalues of the first two components (PC1 and PC2). Each component is responsible for 56.03% and 35.28%, respectively. Regarding the distribution of the evaluated parameters, the PC1 strongly positively correlates with Rha, Ara, Xyl and K ion values, with negative correlation with Glu value. The PC2 is mainly linked with Man and Na ion values. Strong correlation among Ara, Xyl and K ion values suggests that the existence of K ion benefits arabinoxylans formation in the SDF production.

3.2. Molecular size distribution

Molecular size distributions of SDFs were detected by TSK gel 2000 SWXL – high performance liquid chromatography, and the corresponding results are exhibited in Table 3. The molecular weight of SDF varies largely in the sight of its intricate composition including pentosans, pectin, gums, and mucilage so on (Chawla & Patil, 2010). The process method obviously influences molecular size distributions of SDFs from *C. edulis* by-product. Compared to those of ESDF (weight-average molecular weight (M_w) = 11,460 Da) and PESDF (M_w = 11,431 Da), the main molecular weights of CSDF (M_w = 3888 Da) and PCSDF (M_w = 5278 Da) are much smaller, which implies that, in comparison with enzymatic treatment, chemical treatment (mainly H_2O_2) can severely degrade IDF, such as cellulose, part of hemicellulose and lignin. SDFs obtained by enzymatic treatment are more homogeneous than those prepared through chemical process, considering that ESDF and PESDF present one peak, whereas PCSDF and CSDF give two peaks in the chromatogram. It is surprising that the molecular size of CESDF (M_w = 15,290 Da) maximizes, which could be attributed to enzymatic inactivation effected by residual H_2O_2, and remove of ethanol-soluble substance with low molecular size derived from chemical and enzymatic combination. The incorporation of three treatments seriously leads to heterogeneity of SDF, owing to the presence of three peaks with areas of 15.81%, 27.64% and 56.55%, respectively, and large M_w distribution ranging from 3186 to 164,651 Da for PCESDF. Number-average molecular weight (M_n) and peak molecular weight (M_p) exhibit corresponding changes with M_w.

Table 3
Molecular weight distribution of soluble *Canna edulis* Ker dietary fibers.

Name	Peak	Area (%)	M_n[a]	M_w[b]	M_p[c]
PCSDF	1	25.59	50,175	87,798	22,302
	2	74.41	2017	5278	2357
PCESDF	1	15.81	105,155	164,651	52,594
	2	27.64	18,089	23,425	8931
	3	56.55	1929	3186	2572
PESDF	1	100.00	3674	11,431	9806
CSDF	1	15.00	39,730	68,488	16,755
	2	85.00	2117	3888	1356
CESDF	1	100.00	3531	15,290	7631
ESDF	1	100.00	2818	11,460	6226

[a] Number-average molecular weight.
[b] Weight-average molecular weight.
[c] Peak molecular weight.

3.3. Structural characteristics

SDFs prepared by six methods were analyzed using Fourier Transform Infrared-Raman spectrometer and the results are exhibited in Fig. 2. The FT-IR spectra can provide the information about chemical bonds and groups in the structure of organic molecule. It is clear that the treated methods obviously influence SDF composition. The spectra of CSDF and PCSDF, ESDF and PESDF are separately similar, which proves the similarity of chemical constituents between them, in accordance with the results from determination for single sugar composition and molecular size distribution. Compared CSDF with ESDF, the two peaks, separately derived from stretching vibration of C=O and C—O—C, have red shifts from 1628 to 1651 cm^{-1} and 1025 to 1043 cm^{-1}, respectively. It could be ascribed to formation of increased hydrogen bonds in the CSDF, composed of smaller molecules than ESDF obtained in the measurement of molecular weight distribution. Furthermore, for ESDF, a new shoulder peak located at 1535 cm^{-1} belongs to bending vibration of N—H, which can be explained by enzyme residue in the treatment. Compared to CSDF and ESDF, CESDF reveals a broad and strong peak at 3700–2200 cm^{-1} and red shifts of two peaks (1654 and 1069 cm^{-1}) belonging to stretching vibration of C=O and C—O—C, respectively. It could be ascribed to the presence of carboxyl group resulting from oxidation of enzymatic hydrolyzed product by H_2O_2. Moreover, due to stronger hydrogen bonds originated from carboxyl in the CESDF, characteristic peak of methyl shows blue shift from 1410 to 1381 cm^{-1}. For PCESDF compared to CESDF, more distinctly broad and strong carboxyl peak and the red shift of another peak at 1705 cm^{-1} for stretching vibration of C=O, suggest more serious oxidation of the final product. In addition, the increased hydrogen bond in the PCESDF results in the blue shift of the stretching vibration peak of C—O—H from 1167 cm^{-1} (CESDF) to 1126 cm^{-1}.

3.4. Thermal property

The thermal characteristic of SDFs was determined and the corresponding results are exhibited in Fig. 3 and Table 4. In the TG spectrum, the first degradation peak between 65 and 140 °C with a maximum at about 90 °C derives from water molecular release, which is a typical endothermic process (DSC). It allows determination of water content in the SDFs through the calculation of the weight loss in the TG signal. It is clear that water content changes from 7% to 12% in the SDFs. Among them, CESDF exhibits

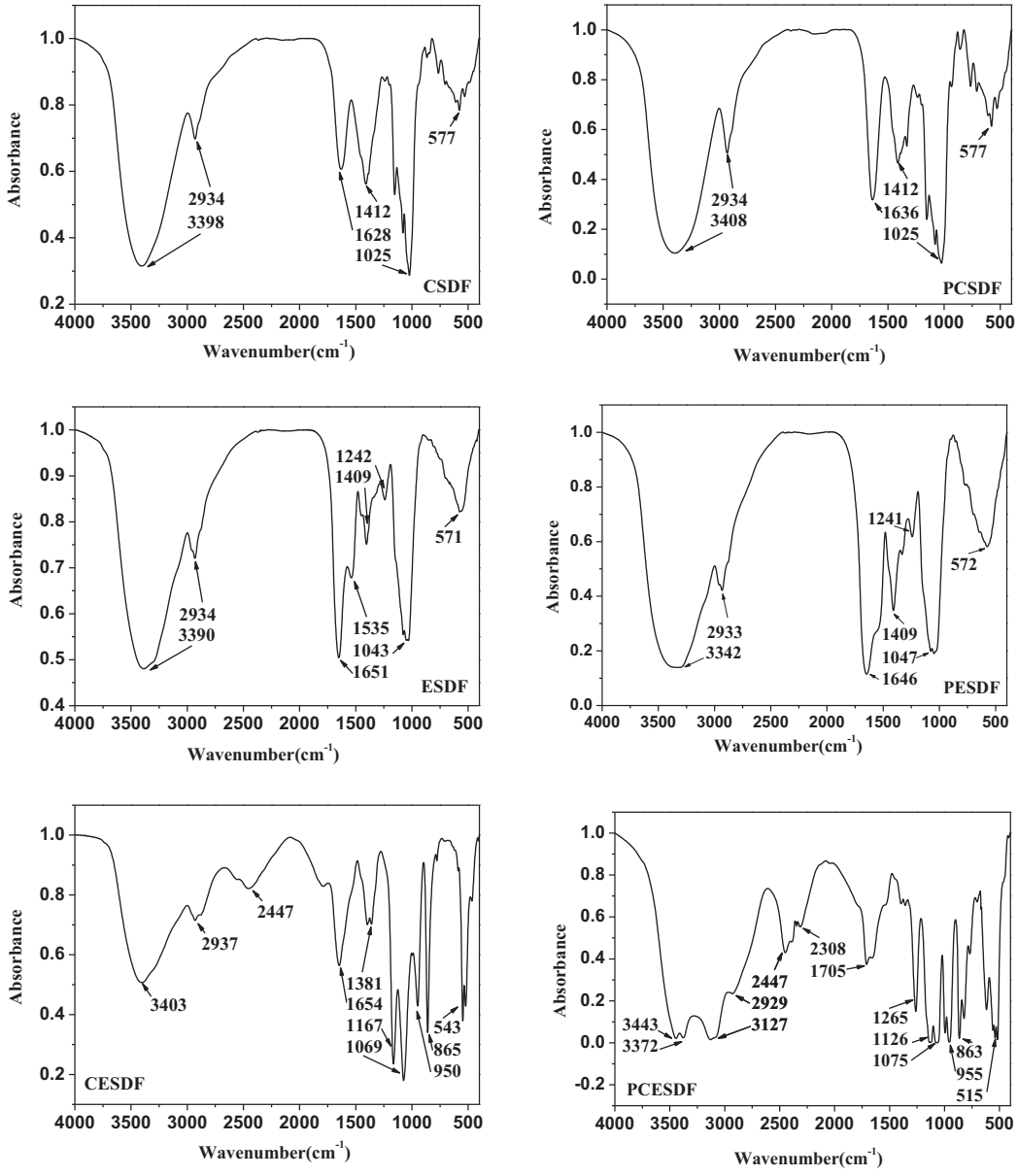

Fig. 2. FT-IR spectra of soluble *Canna edulis* Ker dietary fibers.

maximized changed masses and maximum degradation rate (v_{max}) as well as minimized DTG peak width (ΔT), indicating that the release of water molecule is rapid and drastic. It can be explained by the largest molecular weight, in the sight of the relatively easy release of water molecule in the macromolecule.

In the exception of degradation peak for water molecular release, SDFs also reveal one degradation peak in the TG curves, which can be attributed to the degradation of carbohydrates. With maximized degradation peaks located at 242.83, 280.91 and 243.19 °C, respectively, the pyrolysis of CSDF, PCSDF and PESDF are different from the typical process of cellulose by starting to degrade at 300 °C (Ramanen, Penttila, Svedstrom, Maunu, & Serimaa, 2012). It can be ascribed to the presence of hemicellulose and pectin, and the degradation of the latter is an exothermic reaction, which starts at temperature of about 200 °C and ends at about 240–280 °C, depending on the molecular parameters, degree of modification and physical state (Einhorn-Stoll & Kunzek, 2009; Zhang, Wang, Yu, & Wu, 2011). It can be also explained for the occurrence of shoulder in the second degradation peaks for ESDF, CESDF and PCESDF.

It is well known that the degradation of carbohydrates is consisted of a series of concurrent and consecutive reaction. The intermediate products, such as levoglucosan, go undetected and only final products, like C, CO, CO_2, H_2O and combustive volatiles, can be analyzed. The DSC curves (Fig. 3) display that the combustion of carbon and these combustive volatiles converts an endothermic degradation to an overall exothermic process, which is in accordance with the results reported by Einhorn-Stoll and Kunzek (2009). Furthermore, the endothermic DSC-peak starts earlier than the exothermic DTG-peak for all SDFs, and it indicates that energetic effect could be tested earlier than weight loss, owing to conformation transformation before the degradation weight loss, which requires more energy than that released by the beginning thermal degradation. The conformation change might be the transformation from the stable $^4C_1$ chair conformation of the glucose ring via a $^{1,4}B$ conformation to the inverse $^1C_4$ chair conformation that has higher free energy G. Additionally, though only two weight degradation peaks exhibited in the TG spectra, DSC curves change largely, suggesting that energy varies complicatedly in the process of SDF degradation.

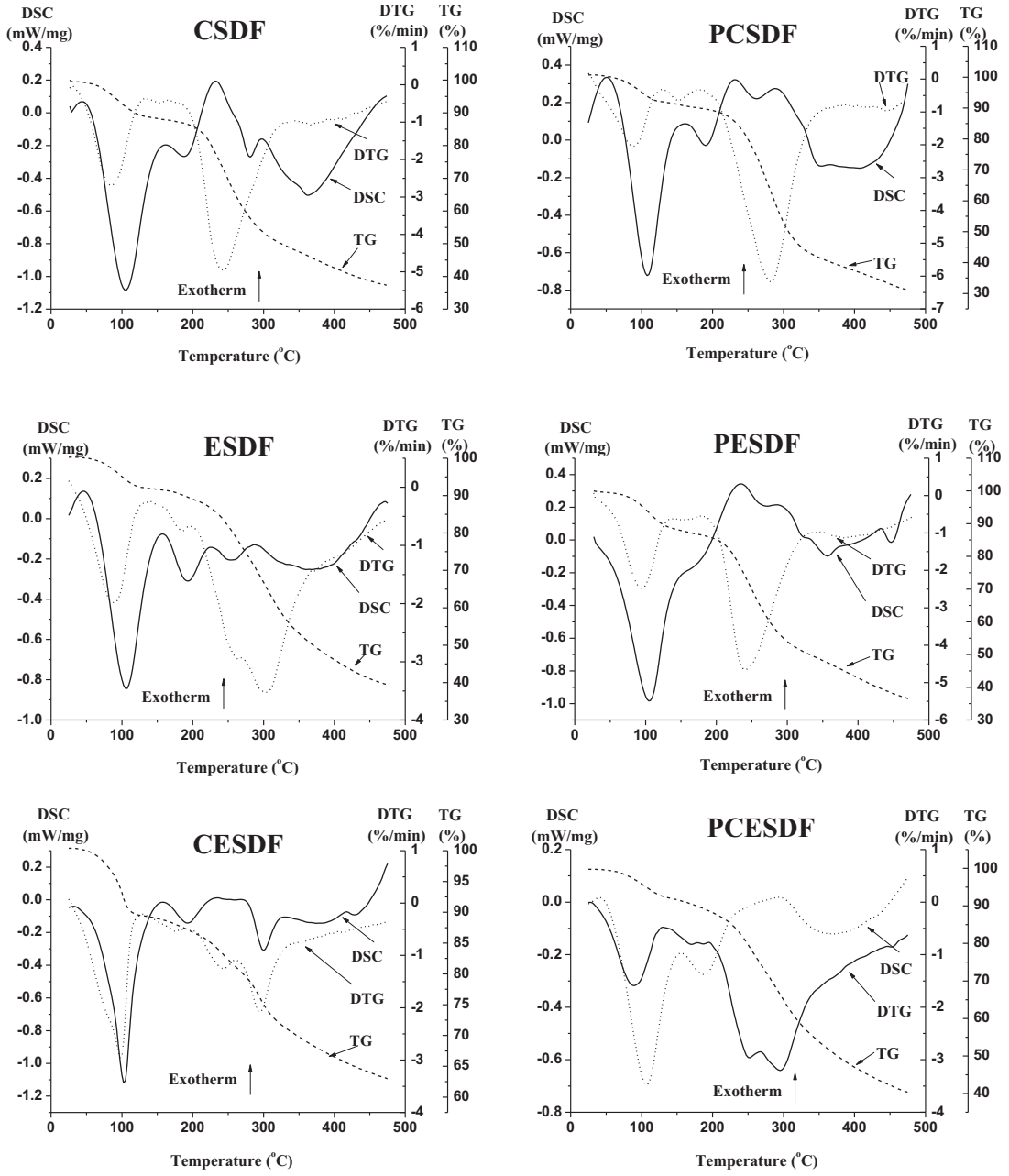

Fig. 3. Thermograms of soluble *Canna edulis* Ker dietary fibers.

In Table 4, PCSDF and PESDF separately show the higher v_{max} values (−6.19 and −4.64%/min, respectively) than those of CSDF and ESDF (−4.92 and −4.64%/min, respectively). Furthermore, lower residual masses (38.34 and 43.31%, respectively) are observed for PCSDF and PESDF, in comparison with those of CSDF and ESDF (45.18% and 45.94%, respectively). These results reveal that the ultrasonic treatment leads to increase of the homogeneity of final product, which also be further confirmed by higher thermal stability of PESDF, in view of its later extrapolate onset temperature (T_{on}) (241.62 °C), an indicator of the thermal stability, than ESDP (216.12 °C). Moreover, ultrasonic treatment results in the disappearance of shoulder in the degradation peak in the ESDF, and relatively high uniformity of PESDF. Other than water release peak, CESDF gives one degradation peak located at 294.72 °C with a shoulder at 243.62 °C, corresponding to combination of CSDF and ESDF, which separately exhibit the degradation peaks at 242.83 and 301.92 °C. Compared to CSDF (−4.92%/min) and ESDF (−3.54%/min for peak and −3.08%/min for shoulder, respectively), CESDF gives lower v_{max} value (−2.30%/min for peak and −1.24%/min for shoulder, respectively). It indicates that the combination of chemical and enzymatic methods has a negative effect, which is also proved by the lowest changed masses (−15.93% for peak) in the CESDF. The negative effect could be explained by the decrease of enzymatic activity influenced by the addition of H_2O_2. Compared to CESDF, PCESDF shows higher v_{max} value (−3.22%/min for peak and −3.01%/min for shoulder, respectively) and changed masses (−35.74% for peak), It approves that ultrasonic treatment plays a key role on the thermal stability and homogeneity of SDF, in agreement with above results for comparison between CSDF and PCSDF, ESDF and PESDF. Due to relatively low residual weight of PCSDF (38.34%) and PESDF (43.31%), chemical–physical and physical–enzymatic methods can be choose for production of SDF from *C. edulis* by-product. However, in view of insecurity of chemical treatment, it can come to a conclusion that

Table 4
Thermal analysis of soluble *Canna edulis* Ker dietary fibers.

Sample	CSDF	PCSDF	ESDF	PESDF	CESDF	PCESDF
Release of humidity						
DSC						
T_{on} (°C)	61.63	67.62	63.12	62.93	75.86	61.23
T_p (°C)	104.31	107.22	104.53	104.44	103.62	104.26
ΔT (°C)	81.40	71.71	78.73	70.52	44.04	81.47
E_{max} (mW/mg)	1.07	0.97	0.90	0.89	1.12	0.64
TG						
T_{on} (°C)	67.35	76.03	67.12	75.36	77.73	68.26
ΔT (°C)	53.42	40.52	53.48	53.48	33.63	69.97
Changed masses (%)	−11.06	−9.32	−8.73	−12.21	−11.41	−7.15
v_{max} (%/min)	−2.81	−1.83	−2.02	−2.61	−3.02	−1.54
SDF degradation						
DSC						
Peak 1						
T_{on} (°C°C)	197.52	195.63	202.3	182.86	196.36	200.86
T_p (°C)	233.73	227.92	222.4	225.23	224.56	227.51
ΔT (°C)	82.74	56.73	43.36	62.36	43.87	57.26
E_{max} (mW/mg)	0.50	0.23	0.12	0.17	0.04	0.08
Peak 2						
T_{on} (°C)	286.91	262.43	263.23	275.63	218.36	258.03
T_p (°C)	297.82	296.81	286.96	297.92	277.12	296.36
ΔT (°C)	36.73	77.41	41.36	38.73	77.47	76.06
E_{max} (mW/mg)	0.18	0.19	0.09	0.10	0.18	0.07
TG						
Peak						
T_{on} (°C)	216.12	241.62	288.26	215.42	282.63	281.69
ΔT (°C)	67.53	72.18	47.63	68.47	27.43	38.55
Changed masses (%)	−44.80	−52.71	−43.15	−44.91	−15.98	−35.74
v_{max} (%/min)	−4.92	−6.19	−3.54	−4.64	−2.30	−3.22
Shoulder						
T_{on} (°C)	–	–	236.2	–	223.82	230.45
ΔT (°C)	–	–	32.06	–	31.54	32.65
v_{max} (%/min)	–	–	−3.08	–	−1.24	−3.01
Residual mass (%)	45.18	38.34	45.94	43.31	66.16	47.40

physical–enzymatic method will be a best choice for IDF transform into SDF.

3.5. Color determination

The colors of SDFs were determined through spectrophotocolorimeter and the results are presented in Table 5. The prepared method plays a key role on the color of final products. The colors of all SDFs are much better than DF concentrate from Allig varieties reported (Ismail, Haffar, Baalbaki, & Henry, 2008). CSDF is characterized by higher L^* and a^* as well as lower b^* than PCSDF. These suggest that CSDF is lighter, greener and bluer than PCSDF, which could be due to a more intense browning (Maillard reactions) and oxidation process resulting from the increased contact between SDF and oxygen in air under ultrasonic treatment. This can be also explained for PCESDF, which gives lower L^* as well as higher a^* and b^* in comparison with CESDF. However, ultrasonic treatment has a positive effect on PESDF, which exhibits much higher L^* as well as lower a^* and b^* than ESDF. It well indicates that the light of PESDF increases, which could be attributed to accelerated enzymatic hydrolysis under ultrasonic condition. The ultrasonic treatment promotes the homogeneity of reactive solution and increases the rate of collision between IDF and cellulase, resulting in hydrolysis of colored IDF and production of light colored SDF. CESDF is lighter than ESDF, but darker than CSDF, suggesting the negative correlation between H_2O_2 oxidation and enzymatic hydrolysis, in agreement with the results in the thermal analysis. Among all SDFs investigated, CSDF gives the highest L^* as well as the lowest a^* and b^*, owing to bleaching action of H_2O_2. Concerning the SDFs, the discolors could be due, on the one hand, to the wash operations during the extraction of SDFs and, on the other hand, to the solubility of pigments responsible for the dark units of color.

Table 5
The CIE lab values (L^*, a^*, b^*) of soluble *Canna edulis* Ker dietary fibers.

Name	Colour[a,b]		
	L^*	a^*	b^*
CSDF	74.19 ± 1.01 a	3.78 ± 2.42 ab	4.83 ± 0.67 a
PCSDF	71.93 ± 0.53 b	3.09 ± 0.64 ab	15.44 ± 0.06 b
ESDF	49.22 ± 0.97 c	10.60 ± 1.69 c	18.83 ± 0.16 c
PESDF	69.99 ± 0.09 d	4.70 ± 0.21 ab	7.26 ± 0.08 d
CESDF	70.65 ± 0.12 d	4.28 ± 0.61 ab	14.51 ± 0.11 d
PCESDF	62.62 ± 0.02 e	7.61 ± 0.02 d	21.10 ± 0.15 e

[a] Values are means ± S.D.
[b] Values not sharing a common letter are significantly different in the column ($P < 0.05$).

4. Conclusions

When *C. edulis* by-product was used as raw materials, SDF was successfully prepared using six methods, including chemical, enzymatic and physical methods as well as their different combinations. The maximized yield (68.71%) was obtained using physical–enzymatic method. CSDF and PCSDF are mainly consisted of glucan, and glucomannan is chief composition for ESDF, CESDF and PCESDF. Different from other SDFs, PESDF is largely composed of xyloglucan and arabinoxylan. In the production of SDF, Glu transforms to other single sugars, and the formation of arabinoxylans is a key factor to determine final yield. K ion benefits SDF manufacture, inversely, Ca ion inhibits. The main weight-average molecular weights of CSDF, PCSDF and PCESDF range from 3888 to 5278 Da, and they are much smaller than those of ESDF, PESDF and CESDF (from 11,431 to 15,290 Da). ESDF and PESDF are more homogeneous than CSDF, CESDF, CESDF and PCESDF, among which, PCESDF is most heterogeneous. In the SDFs, the numbers of carboxyl and hydrogen bonds are ranked as follows: ESDF and PESDF < CSDF and PCSDF < CESDF < PCESDF. The maximized degradation peaks range from 242 to 302 °C in the SDFs, the water contents of which change from 7% to 12%. PESDF shows the best thermal stability and homogeneity, in view of the highest T_{on} (241.62 °C) and v_{max} (−6.19%/min) values. CSDF is lightest in the SDFs. In the sight of security, high yield and homogeneity, good thermal stability, physical–enzymatic method will be a best choice for the production of SDF from *C. edulis* by-product. The production of SDF will not only increase economic value of *C. edulis* by-product, but also provide a new additive for research and development of functional food.

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