LWT - Food Science and Technology 154 (2022) 112624

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt

Utilization of stalk waste separated during processing of sun-dried figs (*Ficus carica*) as a source of pectin: Extraction and determination of molecular and functional properties

Elif Çavdaroğlu, Ahmet Yemenicioğlu *

Department of Food Engineering, Faculty of Engineering, Izmir Institute of Technology, 35430, Gülbahçe Köyü, Urla, Izmir, Turkey

ARTICLE INFO

Keywords:
Fig stalk
Fig pectin
Citrus pectin
Acidic extraction
Functional properties

ABSTRACT

This study aimed the utilization of fig stalk waste as an alternative pectin source. For this purpose, the characteristics of extracted stalk waste pectin (SP) were compared with those of citrus pectin (CP) and pectin extracted from defected substandard whole sun-dried figs (FP). The SP had a higher extraction yield (11.7%) than FP (9.4%). The galacturonic acid content and degree of esterification of SP (32.3 and 50.1%) were higher than those of FP (19.9 and 38.8%), but lower than those of CP (79.3 and 56.2%), respectively. The SP and CP had different sugar compositions (D-glucose, L-rhamnose, D-galactose and L-arabinose) and weight average molecular weights, but similar FTIR profiles. The SP showed almost 1.9 and 1.6-fold higher Trolox equivalent antioxidant capacity (TEAC), and 2.7 and 2.0-fold higher water absorption capacity than CP and FP, respectively. SP at 3% (w/w) showed the second highest viscosity after CP and the highest emulsion stability. Gels of SP and CP at 1.75–3% range had similar firmness, but SP formed more fracturable gels than CP. Sun-dried fig stalk waste is a better source of pectin than defected substandard whole sun-dried figs. The SP could be utilized to develop functional food with alternative textural and rheological properties.

1. Introduction

Pectin is an indispensable ingredient for the food, biomedical, drug, cosmetics and nutraceuticals industry not only due to its techno-functional properties but also health-promoting effects as soluble fiber (Gilani et al., 2008; Muñoz-Almagro et al., 2020; Noreen et al., 2017; Rezvanain et al., 2017; Yang et al., 2015). Thus, to meet the growing demands of the global pectin market, extensive studies have been directed towards alternative sources of pectin (Ciriminna et al., 2016; Cui et al., 2021; Khedmat et al., 2020; Liu et al., 2020). For example, sunflower heads and sugar beet pulp are emerging sources of pectin while pectins from tomato, carrot and pumpkin waste, passion and banana fruit peels, and watermelon rinds (Dranca & Oroian, 2018) are other potential pectin sources under investigation. The molecular architecture and functional properties of pectin from different sources are unique. Thus, studies for characterization of functional properties of alternative novel pectins (e.g., from pomelo, berries, hawthorn, etc.) and discovering novel uses of pectin are continuing (Gamonpilas et al., 2021; Li et al., 2021; Muñoz-Almagro et al., 2021; Reichembach & Lúcia de Oliveira Petkowicz, 2021).

Turkey, with its 89,000 metric tons of production in the 2019/20 season, is the largest producer and exporter of sun-dried figs in the world (Anonymous, 2020). Sun-drying is an ancient process to dry fruits that highly affected by the climate and field practices, thus, causes a great variation in the quality of fruits classified as extra quality, class I and class II (UNECE., 2016). A considerable part of the fruits is also separated as substandard and those seriously damaged by insects, rotting, sun-scalding, split and torn, or excessive drying are used for molasses production or utilized as sugar source in alcohol fermentation. The standard sun-dried fruits with 18–22% moisture content are brought to factory and fumigated, washed, examined (for fungal decay and metabolites) and kept in cold storage until packaging. The sun-dried figs have long been known for their rich soluble dietary fiber content that is formed mainly by pectin (Trad et al., 2014). Due to their high dietary fiber content and bioactive phenolic constituents, fig fruits are historically used as a natural laxative and have been considered as a functional food having positive health benefits on gastrointestinal disorders (Rtibi et al., 2018; Simmons & Preedy, 2016). Therefore, the production of fig pectin and characterization of its technological properties and health benefits have attracted interest of different researchers. For example,

* Corresponding author.
E-mail address: ahmetyemenicioglu@iyte.edu.tr (A. Yemenicioğlu).

https://doi.org/10.1016/j.lwt.2021.112624
Received 15 June 2021; Received in revised form 17 September 2021; Accepted 12 October 2021
Available online 14 October 2021

Gharibzahedi et al. (2019a, 2019b) extracted and characterized pectin from peels of fresh figs. Moreover, Çavdaroğlu et al. (2020) extracted pectin from whole sun-dried figs using classical hot acidic extraction with a yield between 8 and 9% and characterized its edible film properties and applicability as a fruit coating.

Recently, a new and rapidly developing trend in the processing of sun-dried figs is that the extra quality sun-dried fruits rehydrated to intermediate moisture levels (~35%) are portion packed, and then they are pasteurized to obtain ready-to-eat, soft, and juicy shelf-stable fruits. The stalks of these premium fruits processed by this emerging method are cut and removed manually before processing. These fig stalks contain part of the flesh tissue that changes between 1 and 1.5% of total fruit weight depending on the experience of the workers employed in stalk-cutting. Thus, there is an increased interest by the industry to valorize stalk wastes for the production of value-added products. In the current study, pectin from waste sun-dried fig stalk has been extracted and characterized for its molecular and functional properties. The functional properties of fig stalk pectin were also compared with those of commercial citrus pectin to understand its industrial relevance, and those of seriously defected substandard sun-dried figs that are currently processed into molasses or utilized in alcohol fermentation (Çavdaroğlu et al., 2020). This work is original in that it is the first study related to the functional properties of pectin from sun-dried fig stalk waste and whole sun-dried figs.

2. Materials and methods

2.1. Materials

Citrus pectin (CP, P9135, galacturonic acid ≥79%, methoxy content ≥8%) was purchased from Sigma-Aldrich (St. Louis, MO, USA). All other chemicals were reagent grade. The stalk wastes (contain the stalk and a piece of attached fruit flesh that corresponds to 1–1.5% of fruit flesh) remained from the processing of high-quality sun-dried figs destined for portion pack pasteurization, and seriously damaged substandard whole sun-dried figs (both Sarilop cultivar) were kindly supplied by KFC Gıda Tekstil Sanayi İthalat İhracat Yatırım A.Ş (Menemen, Turkey). All samples were passed from UV inspection. The proximate composition of stalk waste was found as follows: moisture (by vacuum oven method, AOAC, 934.06), protein (by micro Kjeldahl method), oil (by Soxhlet method, AOAC, 963.15), ash (by AOAC, 940.26) and carbohydrate (by calculation) contents were 20.9, 4.6, 0.7, 2.5 and 71.3%, respectively. All analyses for proximate composition were performed in triplicate.

2.2. Methods

2.2.1. Extraction of pectin

The acid extraction method used for sun-dried fig fruits by Çavdaroğlu et al. (2020) was applied to extract fig stalk pectin. The flow diagram was provided in Supplementary Figs. S–1. Briefly, pectin was extracted via hot acidic extraction with 1:3 (w/v) stalk waste or fig fruit (both at almost 20% moisture content) to liquid ratio using 1, 3 or 6% (w/v) citric acid (CA) solutions (pH of 2.23, 1.98 and 1.83) at 95 °C for 1 or 3 h, respectively. The optimal conditions (CA concentration of 6% and 1 h) selected for extraction of pectin from fig stalks (SP) were also applied for extraction of pectin from whole figs (FP).

2.2.2. Characterization of molecular properties and composition

The galacturonic acid (GA) content and degree of esterification (DE) of pectins were determined by spectrophotometric and titrimetric methods given by Çavdaroğlu et al. (2020), respectively. The GA, DE as well as degree of acetylation were also determined by High Performance Liquid Chromatographic methods (HPLC). The GA content of pectins was analyzed according to Rumpunen et al. (2002) by using the Perkin Elmer Series 200 HPLC system with auto-injector (20 μL), column oven, refractive index detector (RID) and Aminex HPX-87H (1,300 × 7.8 mm, 9 μm) column. To prepare HPLC samples, the enzymatic degradation was applied by mixing 10 mg of pectin with 1 mL of 2% (v/v) commercial enzyme (Pectinex Ultra SP-L, Novozyme, Denmark). The test tubes were then carefully shaken at 240 rpm for 3 h using an orbital shaker (IKA, OS 5 basic, Germany) placed in an incubator working at 48 °C. The serial concentrations of pectin (0.5–20 g/L) were degraded by Pectinex Ultra SP-L and used for standard curve of galacturonic acid area vs. pectin concentration. Duplicate measurements were used to calculate the results as % (g GA/100 g of pectin). The flow rate during HPLC was isocratic at 0.6 ml/min with 5 mM H_2SO_4 as mobile phase at 65 °C column temperature. The DE and acetylation of the fig pectins were determined according to the method described by Voragen et al. (1986) with slight modifications. 30 mg of pectin was suspended in a 1 mL 0.25 M NaOH solution and held at ambient temperature for 2 h. The suspensions were then centrifuged at 10,000×*g* at 4 °C for 10 min, and 20 μL of the clear supernatant was injected on the HPLC column (Aminex HPX-87H, Biorad, 300 × 7.8 mm). The column was operated at 35 °C with a flow rate of 0.6 mL/min for 5 mM H_2SO_4 used as mobile phase. Components eluted from the column were detected with a refractive index detector which was performed in duplicate. The DE and acetylation (DA) were expressed as the percent molar ratio of methanol or acetic acid to GA quantified by the HPLC method, respectively. The moisture and ash contents were determined according to AOAC (AOAC., 1990). The soluble protein contents of pectins were determined according to the Bradford method (Bradford, 1976). Total carbohydrates were determined by the phenol-sulfuric acid method using D-glucose as a standard (Dubois et al., 1956).

2.2.3. Determination of sugars, molecular composition and sugar molar ratios

The glucose, arabinose, galactose, and rhamnose contents used to evaluate the molecular composition of pectins (HG and RG-I ratios and contents) and their sugar molar ratios were determined spectrophotometrically using enzymatic kits (for glucose using GAGO20, Sigma, and for arabinose (Ara) and galactose (Gal), and rhamnose (Rha) using K-ARGA and K-RHAMNOSE, Megazyme, Ireland, respectively) according to the manufacturer instructions in duplicate samples (Gawkowska et al., 2019). Briefly, the pectin samples (100 mg ± 0.1 mg) were first hydrolyzed chemically with 1.3 M HCL (5 mL) in sealed glass tubes at 100 °C for 1 h. After the tubes were cooled to room temperature, they were neutralized by adding 1.3 M NaOH and adjusted to 100 mL with distilled water. After centrifugation at 10,000 rpm for 10 min, the samples were analyzed according to the instruction manuals of each kit. Sugar molar ratios (R-1, R-2, R-3, R-4), and homogalacturonan (HG) and rhamnogalacturonan-I (RG-I) contents of the pectins were calculated according to M'Sakni et al. (2006). The equations used in calculations are as follows: HG (mol%) = GalA (mol%) – Rha (mol%); RG-I (mol%) = GalA (mol%) – HG (mol%) + Rha (mol%) + Ara (mol%) + Gal (mol%); R-1 (mol%, linearity of pectin) = GalA (mol%)/(Rha (mol%) +Ara (mol %) +Gal (mol%)); R-2 (mol%, RG-I fraction content of pectin) = Rha (mol%)/GalA (mol%); R-3 (mol%, degree of branching of RG-I) = (Ara (mol%) + Gal (mol%))/Rha (mol%); R-4 (mol%, length of Gal branching in RG- I) = Gal (mol%)/Rha (mol%).

2.2.4. Determination of molecular weight

The weight average molecular weight (Mw), number average molecular weight (Mn), and polydispersity index (Mw/Mn) of SP and CP were determined by gel permeation chromatography (GPC) using a Viscotek GPCmax VE 2001 pump system (Malvern Panalytical, Malvern, UK) connected to the Viscotek TDA 302 detector system with refractive index (660 nm) and right-angle light scattering (670 nm) detectors used for on-line SEC signal detection. A sample solution of 100 μL was injected into a TSK-Gel G3000PWxl column (Tosoh, Tokyo, Japan). The column temperature was 22 °C, and the flow rate was 0.8 mL/min. The mobile phases were 50 mM phosphate buffer saline (PBS) containing 0.15 M NaCl (pH 6–7) and acetate buffer with 0.01 M CH_3COOH and

0.15 M NaCl (pH 5.0) containing 0.05% NaN_3 to prevent biological degradation of the columns. Detectors were calibrated using with a series of polyethylene oxide (PEO) polymer standards with Mp values of 235; 2,100; 18,600 and 222,000 Da (Malvern Panalytical, Malvern, UK) in a mobile phase of PBS at 0.8 ml/min flow rate. OmniSEC software was used for the acquisition and analysis of SEC data. Experiments were repeated 2 times for each sample.

2.2.5. FTIR analysis

Fourier transform infrared (FTIR) spectra of pectin samples were analyzed with a Perkin Elmer FTIR spectrometer (Perkin Elmer – Spectrum 100). The pectin:KBr pellets were prepared with a ratio of 1:100. Spectra were collected as an average of 32 scans in the range 4000–650 cm^{-1}, with a resolution of 4 cm^{-1} (Jafari et al., 2017). The background was taken under the same conditions with air. Ten replications of the FTIR analysis were performed for each pectin sample.

2.2.6. Characterization of functional properties

The TEAC method was used for the determination of the free radical scavenging-based antioxidant activity of pectins using ABTS as a free radical according to Re et al. (1999). Briefly, 10, 20 and 30 μL of 5 g/L pectin samples were mixed with 2 mL of ABTS free radical cation solution prepared in PBS at pH 7.4. The reduction in absorbance values was recorded at 734 nm with a spectrophotometer (Shimadzu Model 2450, Japan) for test periods of 1, 3, 6, 9, 12, and 15 min. The Trolox was used as a standard. The area under the curve (AUC) values of triplicated samples were calculated to find TEAC values, and antioxidant activity was expressed as mmol Trolox equivalents per 100 g of pectin.

The water (WAC) and oil absorption capacity (OAC), and foaming capacity (FC) and stability (FS) of pectin samples were determined according to Aydemir and Yemenicioğlu (2013).

The viscosity of 3% (w/v) pectin solutions was determined with a Haake VT 550 Rheometer (Haake MessTechnik Gmbh Co., Karlsruhe, Germany) with a SV-DIN sensor at 23 °C (Monsoor, 2005).

The emulsifying capacity (EC) and stability of pectins were assessed according to Raji et al. (2017) with slight modifications. Emulsions were prepared by mixing 5 mL of sunflower oil with 5 mL of pectin solution (1–3%, w/w) in the presence of 0.01%, w/v Na-azide. The mixtures were homogenized with ultraturrax at 15,000 rpm for 3 min at room temperature and stored at 25 °C for 1 week. The EC was determined by measuring the volume of the emulsified layer as mL (Wv) after 30 min (time necessary to observe the initial sharp phase separation). Emulsion stability (ES) was determined from the ratio of the emulsified layer volume (ELV) at day 1 and day 7 to the total volume (Wv).

$$ES\ \% = \frac{ELV}{Wv} x100$$

The stability of emulsions at 3% pectin was also determined by monitoring the particle size distributions at the beginning and after 7 days at 25 °C using dynamic light scattering (DLS) system (NanoPlus DLS, Micromeritics Instrument Corporation, GA, USA). The surface mean diameter (D[3,2]) values were recorded. The data were reported as an average of 3 repeated measurements of two replicates.

The gelation properties of pectin solutions at different concentrations (0.4%–3% w/w) were studied by preparing standard gels for high methyl ester and low methyl ester pectin gelation methods in the presence of 64 and 30% (w/w) sucrose according to Food Chemicals Codex (Food Chemicals Codex, 1972), respectively. Before analysis, the least gelling concentrations of each pectin were determined using a classical flow test in reversed test tubes with 5 g gel. Then, the hardness, fracturability, gumminess and chewiness of gels were determined by texture profile analysis (TPA) using glass back extrusion cells (100 mm high, 52 mm internal diameter) filled with 50.8 g of gel and incubated at 4 °C for 1 day before TPA was conducted using a texture analyzer (TA-XT2, Stable Microsystems, Godalming, UK). The gel temperature was brought to 25 °C (checked with a thermocouple) by 30 min incubation in an incubator before measurements. The compression test was applied at a constant speed of 0.50 mm/s to a distance of 15 mm from the gel surface using a P/1R cylinder (Delrin radiused) plunger (diameter 25.4 mm). Duplicate samples were tested for each gel.

2.2.7. Statistical analysis

Statistical analysis was conducted by using variance analysis (one way-ANOVA) and Fisher's post-hoc test ($p \leq 0.05$) with Minitab (ver.18.1., Minitab Inc., United Kingdom). All experiments were triplicated for each sample unless otherwise was stated.

3. Results and discussions

3.1. Extraction yield of fig stalk pectin

The SP extraction yields at different conditions are presented in Fig. 1. The extraction yields at 95 °C for different CA concentrations and periods changed between 6.30 and 12.40%. The pectin extraction yield increased in a concentration-dependent manner as CA concentration increased from 1% to 6%. The increase of extraction period from 1 to 3 h caused significant increases in yields in presence of 3% CA, but the extraction period had no significant effect on yield at 1 and 6% CA. Thus, the CA concentration of 6% and 1 h extraction was used to extract SP in the current work. The analysis of extraction data with the response surface methodology also proved these findings (see supplementary files for ANOVA table and plot given in Tables S–1 and Fig.S-2, respectively). The extraction yield of 11.68% for SP obtained with 6% CA by 1 h in the current study is higher than that of 9.38% obtained at the same conditions for FP that is frequently processed into molasses or utilized as a source of sugar for alcohol fermentation. Besides, the yield of SP is also higher than that reported by (Gharibzahedi et al. 2019b, 2019a) for fresh fig peel pectin obtained with hot acidic extraction (Yield: 6%) or microwave-assisted extraction (Yield: 9.26%), but it is lower than that of combined ultrasound-microwave assisted extraction of peels optimized with response surface methodology (Yield: ~14%). However, it should be reminded that the yields reported by Gharibzahedi et al. 2019b, 2019a were for a different fig cultivar and section, and it was in fresh form. These results suggested that the stalk wastes obtained from sun-dried fig processing are good sources of pectin.

3.2. Molecular properties and composition of fig stalk pectin

The GA and DE of SP and CP determined by the spectrophotometric and titrimetric methods were 32.0 and 50.1%, and 79.3 and 56.2%, respectively (Table 1). The GA and DE values determined for pectins by the HPLC methods were found similar to GA and DE determined by the spectrophotometric and titrimetric methods, respectively. Thus, the SP stood just at the border of high and low methoxyl pectins with significantly lower GA than CP. The GA and DE of SP were also significantly higher than those of FP extracted at the same conditions. However, the GA of SP was lower than that of fresh fig peel pectin (GA: 52.5%), but DE of SP was higher than that of fresh fig peel pectin (DE: 39%) reported by Gharibzahedi et al. (2019a) who also employed hot acidic extraction. The degree of acetylation (DA) of SP (11.56%) and FP (7.54%) were considerably higher than that of CP (3.23%). The percentage of acetyl groups in pectins showed a great variation depending on the source of extraction. For example, the DAs for sugar-beet, citrus and pear pectins were reported as 3, 2 and 14%, respectively (Voragen et al., 1986). It was reported that the high degree of acetylation interfered with the gelation of pectins since the presence of acetyl groups caused steric hindrance for chain association (Vriesmann & Petkowicz, 2013). The highest total carbohydrate content was determined for CP (82.4%) followed by SP (75.7%) and FP (54.0%). The FP contained the highest amount of protein (~11%) since it contains protein-rich seeds of fig fruit while SP (6.3%) and CP (6.2%) contained lower, but similar amounts of protein. Finally, the SP and FP contained significantly higher

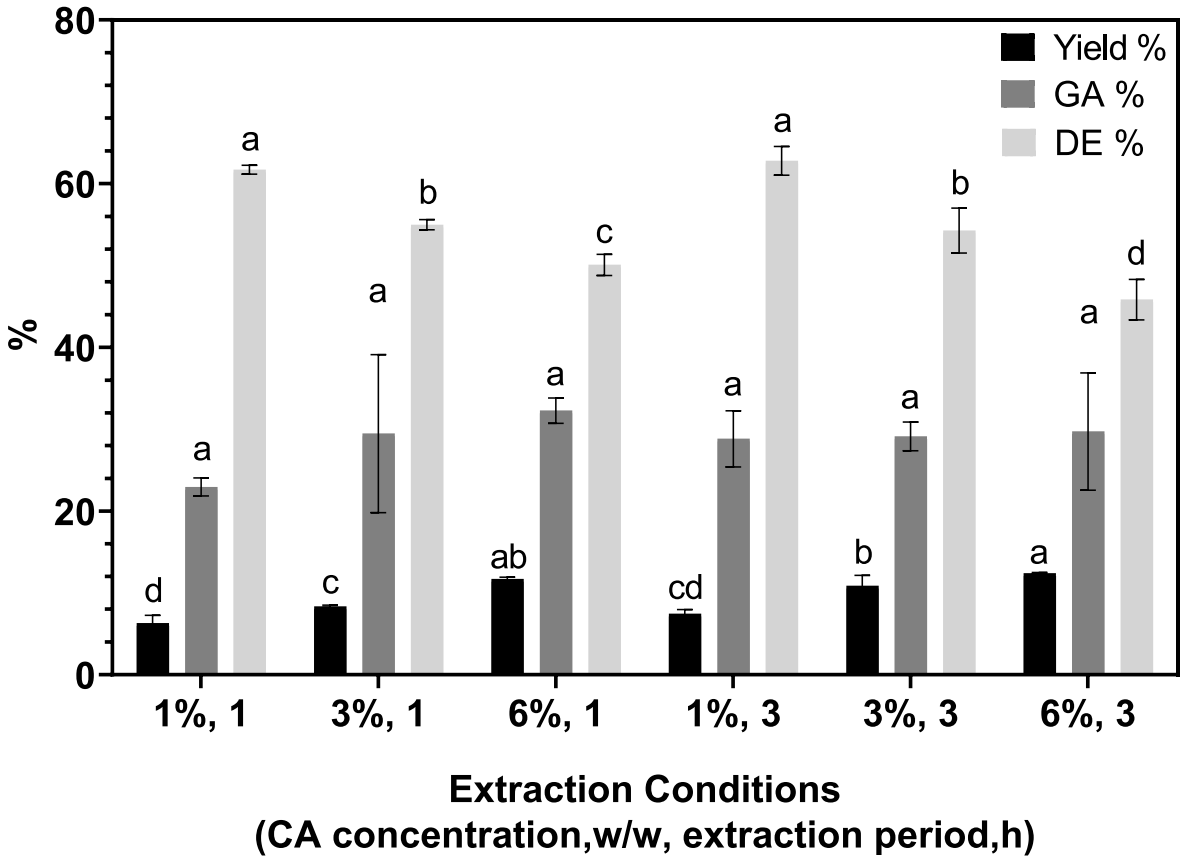

Fig. 1. Yield, galacturonic acid content and degree of esterification of pectins extracted from sun-dried fig stalk waste at different conditions. Values followed by different letters are significantly different at $p < 0.05$.

Table 1
Different characteristics of sun-dried fig pectins and commercial citrus pectin.

Characteristics	CP[a]	SP[a]	FP[a]
Yield (%)[b]	–	11.68 ± 0.23^{A}	9.38 ± 1.01^{B}
GA (%)- spectrophotometric	79.30 ± 1.49^{A}	32.27 ± 1.55^{B}	19.85 ± 0.04^{C}
GA%-HPLC	77.10 ± 0.21^{A}	30.52 ± 0.08^{C}	21.34 ± 0.13^{D}
DE%-titrimetric	56.20 ± 1.63^{A}	50.07 ± 1.29^{B}	38.75 ± 0.59^{C}
DM%- HPLC	58.03 ± 1.85^{A}	53.37 ± 5.67^{A}	29.83 ± 4.15^{B}
DA%-HPLC	3.23 ± 0.68^{C}	11.56 ± 0.04^{A}	7.54 ± 1.64^{B}
Total carbohydrate (%)	82.44 ± 5.32^{B}	75.68 ± 8.29^{A}	53.97 ± 8.84^{B}
Moisture content (%)	11.44 ± 0.18^{A}	8.43 ± 0.04^{B}	7.73 ± 0.03^{C}
Ash (%)	3.61 ± 0.30^{B}	6.39 ± 0.01^{A}	6.80 ± 0.06^{A}
Protein (%)	6.23 ± 0.27^{B}	6.29 ± 0.53^{B}	10.62 ± 0.36^{A}
Color–L*	72.30 ± 0.44^{A}	59.25 ± 1.18^{B}	49.39 ± 0.17^{C}
Color–a*	6.20 ± 0.13^{C}	6.86 ± 0.43^{B}	7.58 ± 0.15^{A}
Color–b*	22.55 ± 0.14^{A}	16.83 ± 0.95^{B}	14.88 ± 0.17^{C}
Mn-(10^4Dalton)	3.27 ± 1.56^{A}	3.94 ± 0.87^{A}	-[c]
Mw-(10^4Dalton)	13.44 ± 1.77^{B}	19.20 ± 0.87^{A}	–
Mw/Mn	4.50 ± 1.61^{A}	4.98 ± 0.88^{A}	–

[a] Values are shown as mean ± standard deviation. Values at each row indicated by different letters are significantly different ($p \leq 0.05$).
[b] All values are expressed as % on dry basis of pectin powder except moisture content.
[c] The measurements cannot be conducted due to turbidity formed by high protein content.

Table 2
The composition of critical neutral sugars in different pectins.

Sugar (g/100g dry sample) /Parameter	CP[a]	SP[a]	FP[a]
D-Glc	0.32 ± 0.073^{C}	3.70 ± 0.004^{A}	2.46 ± 0.009^{B}
L-Rha	0.26 ± 0.015^{B}	0.43 ± 0.054^{A}	0.28 ± 0.044^{B}
D-Gal	3.31 ± 0.485^{A}	2.23 ± 0.081^{B}	4.00 ± 0.00^{A}
L-Ara	2.19 ± 0.251^{A}	1.51 ± 0.042^{B}	2.49 ± 0.084^{A}
R-1	11.8	6.63	2.52
R-2	0.004	0.016	0.017
R-3	20.8	8.49	22.6
R-4	11.6	4.68	13.00
HG (mol %)	91.5	77.2	64.3
RG-I (mol %)	8.13	13.1	27.0
HG:RG-I	11.3	5.90	2.38

[a] Values are shown as mean ± standard deviation. Values at each row indicated by different letters are significantly different ($p \leq 0.05$).

(1.8–1.9-fold) ash content than CP, possibly due to the high mineral content of fig fruit (Doymaz, 2005).

The amount of glucose (Glu), and rhamnose (Rha), galactose (Gal), and arabinose (Ara) used to calculate sugar molar ratios (R1, R2, R3 and R4), and estimate amounts of HG and RG-I fractions of pectins are also shown in Table 2. As expected, FP and SP contained significantly higher glucose contents since almost half of the total soluble sugar (about 43%) in sun-dried figs is glucose (Çalişkan & Aytekin Polat, 2011). Most of the glucose in fig pectins originated from free glucose that remained in the pectin matrix during alcohol precipitation and washing steps. The CP and FP contained similar amounts of Rha, Gal and Ara while SP contained significantly higher Rha (1.5–1.7-fold), but lower Gal and Ara than these two pectins ($p<0.05$). In general, the majority of plant pectins are formed by HG (~65%) and rhamnogalacturonan-I (RG-I, ~20–35%) fractions while rhamnogalacturonan-II (RG-II, ~10%) is a very complex minor fraction (Alba & Kontogiorgos, 2017; Chandrayan, 2018; Yapo, 2011). Therefore, the theoretical calculations mostly ignore the RG-II (M'sakni et al., 2006). The R-1 and R-2 represent the linearity of pectin and RG-I fraction content of each pectin, respectively while R-3 and R-4 are used to estimate the degree of branching of RG-I, and length of Gal branching in RG- I, respectively (Denman & Morris, 2015; Houben

et al., 2011; Kpodo et al., 2017). Thus, the calculated sugar molar ratios suggested that CP was the most, and FP is the least linear pectin molecules while SP had intermediate linearity between these two pectins. As a result, FP showed the highest RG-I content followed by SP and CP. Moreover, it seemed that the RG-I in CP and FP had similar degrees of branching and linearity of Gal branches in RG-I while RG-I in SP had less branching and Gal branch length compared to the other two pectins. The estimated HG and RG-I contents also showed parallelism with sugar molar ratios. The highest amount of HG was calculated for CP (91.5%) followed by HGs of SP (77.2%) and FP (64.3%) while amounts of calculated branchy RG-I regions of pectins were almost 8.1, 13.1 and 27.0%, respectively. These findings suggested that the SP have intermediate HG and RG-I levels between those of CP and FP. Also, the FP is a pectin with limited amount of HG and much higher RG-I content and branching than SP. Further studies are needed to characterize the RG-I fraction of fig pectins. Moreover, the RG-II content of fig pectins should also be investigated to unveil complete molecular structure of novel fig pectins.

Molecular weight characteristics of SP and CP were determined by GPC, but the chromatography of FP cannot be performed due to intense and stable turbidity of samples caused probably by high protein content (Qiu et al., 2015). The examples of chromatographic profiles of CP and SP were given as Supplementary in Fig.S-3.4. Although the Mw of SP (19.20×10^4 Da) was significantly higher than CP (13.40×10^4 Da) ($p < 0.05$), the Mn values of these two pectins were not found statistically different ($p > 0.05$). In the literature, data about Mw and Mn of sun-dried fig pectin are scarce, but Gharibzahedi et al. (2019a) reported that the molecular weight of fresh fig peel pectin extracted by different methods was in the 5.3–6.9 $\times 10^3$ kDa range. The different molecular weight as well as DE and GA of fresh fig peel pectin than fig stalk waste pectin could be due to the differences in extracted fruit sections and/or cultivars (Wongkaew et al., 2021; Zhong, Jin, Lai, Lin, & Jiang, 2010). Moreover, the peel pectin studied by Gharibzahedi et al. (2019a) was from a fresh peel tissue that might contain highly active pectinases that could affect molecular properties of pectin at pre- and post-extraction stages. The Mw of SP is also slightly higher than that of 8.8×10^4 Da reported for dragon fruit (Muhammad et al., 2014), but lower than that of $2.4–16.5 \times 10^5$ Da determined for kiwi fruit (Yuliarti et al., 2011). It is also important to note that the Mw of 13.8×10^4 Da reported by Muhammad et al. (2014) was quite similar with Mw for CP determined in the current work. The Mw/Mn ratios of SP and CP determined in the current work also suggested that both SP and CP were heterogenous pectins consisted of different fractions.

3.3. FTIR spectra of fig stalk pectin

The FTIR spectra of novel pectins are frequently compared with those of well-known commercial citrus pectin to confirm their molecular compatibility (Jafari et al., 2017; Lira-Ortiz et al., 2014). In general, SP and FP gave identical peaks with CP (Fig. 2). The v-shaped band around the region of 3429 cm^{-1} referred to intermolecular and intramolecular OH bonds in the structure of pectins (Gnanasambandam & Proctor, 2000). These peaks originate probably from –OH groups of galacturonic acid and other sugar residues in pectin structure and water molecules absorbed by the hygroscopic samples. The peak between 2920 and 2850 cm^{-1} reflected the stretching vibrations of the CH bond belonging to CH, CH_2, and CH_3 groups in these pectins (Sinitsya et al., 2002). The two bands around 1630 cm^{-1} and 1747 cm^{-1} showed the free carboxyl (COO-) and ester-carbonyl (C═O) groups are important since they are used to estimate the degree of esterification and galacturonic acid

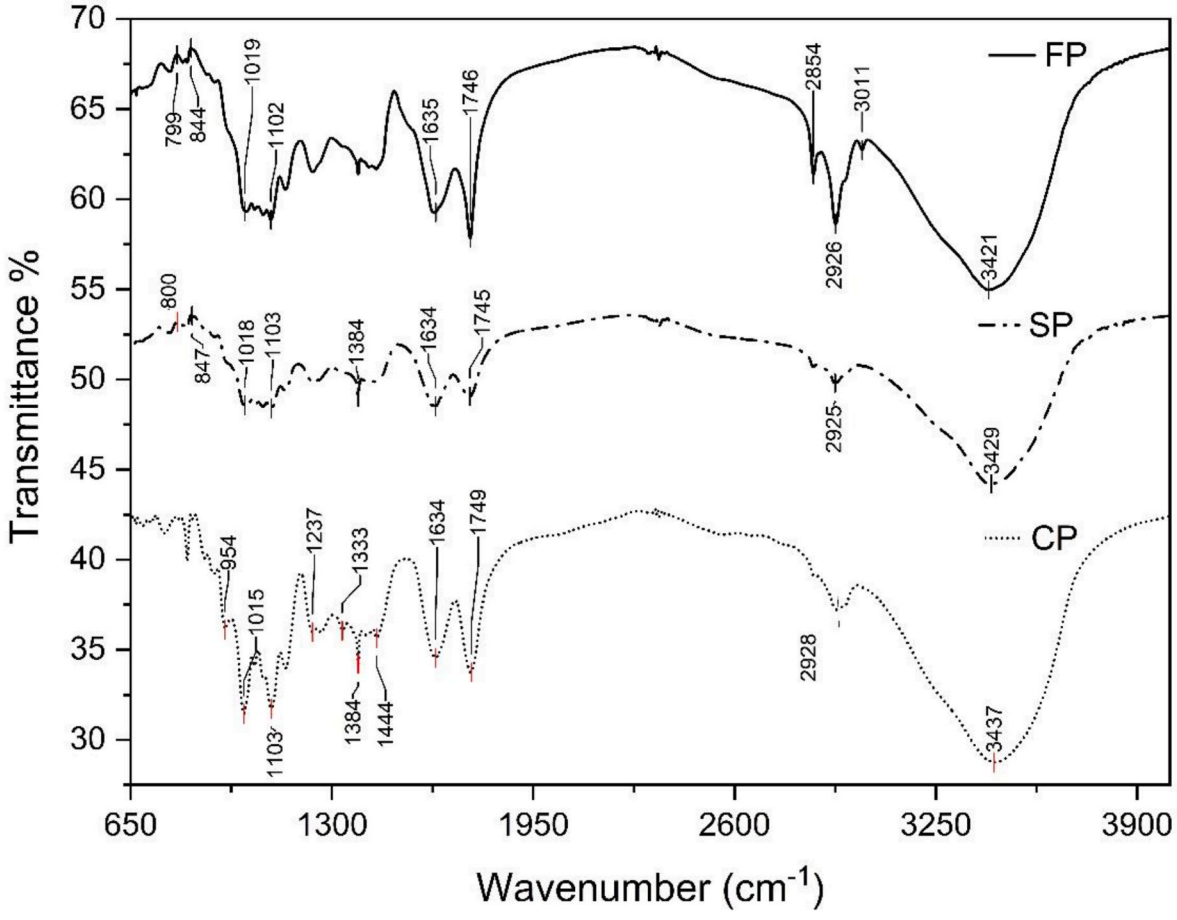

Fig. 2. FTIR spectra of pectin samples.

content of pectins (Fellah et al., 2009; Gnanasambandam & Proctor, 2000; Kyomugasho et al., 2015). The FP that had the lowest DE value (38.8%) gave the lowest transmittance (T) value at 1747 cm^{-1} originating from ester-carbonyl groups (C═O) (T = 29% ± 6.8) while SP and CP that showed comparable DE values (50.1 and 56%) had similar T values at 1747 cm^{-1} (32% ± 5.9 and 34% ± 11.1). Moreover, the GA of pectins (79.3, 32.3, and 19.9% for CP, SP and FP, respectively) showed some correlation with the total transmittance values of 1630 cm^{-1} and 1747 cm^{-1} bands as the highest T was observed for CP (68% ± 22.1) followed descending order by SP (62% ± 12.1) and FP (59% ± 13.6). The CH, C–OH, and α-1,4 glycosidic (COC) bonds in the galacturonic acid chain were also detected at 1384, 1296 and 1197 cm^{-1}, respectively. All pectins studied gave peaks at or closely around these bands with different intensities. It was reported that the band region between 1300 and 800 cm^{-1} of the spectrum is called the fingerprint region that is specific to the material structure and difficult to interpret (Jafari et al., 2017). Therefore, it was proved that the FTIR profiles of SP and FP were highly comparable with those of CP.

3.4. Functional properties of fig stalk pectin

3.4.1. Antioxidant activity

The TEAC based free radical scavenging activity of fig and citrus pectins originated from their polysaccharide structure (e.g, –OH and –COOH groups) (Gharibzahedi et al., 2019b; Wang et al., 2016) and bound antioxidant components (e.g., Maillard reaction products, proteins and polyphenols) (Avila et al., 2018) were given in Table 3. The SP showed almost 1.9 and 1.6-fold higher antioxidant activity than CP and FP, respectively. However, no significant differences existed between the antioxidant activities of CP and FP ($p > 0.05$). Due to differences in antioxidant activity determination methods and expression of results, it is difficult to compare the antioxidant activity of fig pectins with other pectins. However, pectin from fresh fig peel was also reported to show free radical scavenging based on antioxidant capacity (Gharibzahedi et al., 2019b). Some other pectins with reported free radical scavenging activity include okra pectin (Xu et al., 2020), mangosteen rind pectin (Wathoni et al., 2019), grapefruit pectin (Wang et al., 2016) and sweet potato pectin (Ogutu & Mu, 2017).

3.4.2. Water and oil absorption capacity

The WAC and OAC values of fig pectins and commercial citrus pectin are shown in Table 3. The SP showed 2.0 and 2.7-fold higher WAC than FP and CP, respectively. However, the WAC of FP and CP were not found significantly different ($p > 0.05$). The SP also showed higher WAC than those of eggplant peel (6 g/g) and calyx (4.6 g/g) pectins (Kazemi et al., 2019), fiber pectin from tomato pomace (3.57 g/g) (Namir et al., 2015) and olive mill wastewater pectins (3.00 and 2.18 g/g) (Rubio-Senent et al., 2015). Thus, the outstanding WAC of SP could be employed for moisture binding in food formulations. In contrast, all pectins used in this study showed low OACs and were not found significantly different ($p > 0.05$). However, the OAC's of fig pectins were comparable to those of pectins extracted from *Opuntia ficus indica* (1.23 g/g) (Bayar et al., 2018), eggplant calyx (1.46 g/g) (Kazemi et al., 2019) and walnut green husk (1.21 g/g) (Asgari et al., 2020) while pectin from eggplant peel (2.6 g/g) (Kazemi et al., 2019) and sunflower by-product (2.51 g/g) (Ezzati et al., 2020) showed higher OAC than pectins in the current study.

Table 3
Functional properties of pectins.

Properties	CP [a]	SP[a]	FP[a]
TEAC (mmol Trolox/100g)	6.49 ± 0.53^{B}	12.42 ± 1.14^{A}	7.93 ± 1.72^{B}
WAC (g/g)	3.12 ± 0.12^{B}	8.42 ± 0.76^{A}	4.15 ± 0.24^{B}
OAC (g/g)	1.27 ± 0.10^{A}	1.32 ± 0.33^{A}	1.59 ± 0.19^{A}
FC (mL)	3.33 ± 0.58^{A}	3.33 ± 0.76^{A}	1.83 ± 0.29^{B}
FS_{30min}(mL)	80.56 ± 4.81^{B}	79.05 ± 7.19^{B}	100.0 ± 0^{A}
FS_{180min}(mL)	44.44 ± 9.62^{B}	27.86 ± 2.58^{C}	70.00 ± 13.23^{A}
Viscosity (mPa.s)	23.17 ± 0.57^{A}	17.63 ± 0.23^{B}	12.03 ± 1.78^{C}

[a] Data are shown as mean ± standard deviation. Data at each column indicated by different letters are significantly different ($p \leq 0.05$).

3.4.3. Foaming capacity and foam stability

The pectins are frequently tested for their foaming capacity and stability since they mostly contain complexed surface-active protein fractions which show thickening effects that improve the foam stability (Dickinson, 2003). The FC and FS of different pectins are given in Table 3. The CP and SP showed the same FC that was significantly higher (~1.8 mL) than that of FP ($p < 0.05$). In contrast, the FP showed the highest FS by maintaining 100 and 70% of its initial foam volume at the end of 30 min and 180 min, respectively. Although there was no significant difference between FS_{30min} values of CP and SP ($p > 0.05$), CP maintained its foam stability better than SP after 180 min (FS_{180min}). Since carbohydrates are not surface-active molecules, their properties that require surface activity such as foaming and emulsion capacity/stability are often attributed to proteins that are ionically or covalently bound at their surface (Wicker et al., 2014). Therefore, the high FS of FP was possibly associated with its higher protein content than other pectins (see section 3.2). Moreover, it is a well-known fact that polysaccharides can contribute to the stability of the foams by increasing the viscosity in the foam-forming environment (Petkowicz et al., 2017).

3.4.4. Viscosity

The changes in shear stress versus the shear rate of different pectin solutions are shown in Fig. 3, and the viscosities of 3% pectin solutions are given in Table 3. In general, all pectin solutions showed Newtonian behavior and R^2 values of the curves were calculated as 0.9808, 0.9757 and 0.9938 for SP, FP and CP, respectively. During mixing, the pectin chains were attached to each other in the dispersion and entangled, resulting in an increase in viscosity as expected (Lira-Ortiz et al., 2014). The viscosities of the pectin solutions calculated using the slopes of the obtained curves ranged between 12 and 23.2 mPa s. The CP showed the highest viscosity followed descending order by SP and FP. It is noteworthy that SP had a significantly higher viscosity than FP ($p < 0.05$). This result was expected since the GA of SP is significantly higher than that of FP, thus, it might form more entanglement among pectic chains under shear stress. The pectinases as pectin methylesterase are very active in figs and they even survive from the classical sun-drying and processing of dried fruits (Demirbüker et al., 2006). Thus, it appears that GA chains of pectic compounds in the stalk and adjacent fruit tissues are less exposed to the action of pectinases (pectin methylesterase and polygalacturonase) while tissues around the thicker globular part of fruits undergo more effective enzymatic processes due to extensive physical damages (occurred during sun-drying, handling and processing) that spoil enzyme-substrate compartmentation. In the literature, Yuliarti et al. (2015) reported that the viscosity of pectins extracted from gold kiwifruit and its pomace ranged around 30–34 mPa s at the concentration of 1% (w/w). This finding showed significantly higher viscosity of kiwifruit pectins than fig pectins, but it should be noted that the kiwifruit pectins showed a very high galacturonic acid content (>81%, w/w) (Yuliarti et al., 2015). Besides, the green tea leaf pectin that had comparable GA content (26.6–32.4% for different extracts) with SP showed shear-thinning properties with viscosities ranging between 15 and 25 mPa s at the concentration of 5% (w/w) (Zhang et al., 2020).

3.4.5. Emulsification capacity, emulsion stability and particle size characteristics

The EC and ES of different pectins between 1 and 3% (w/w) are seen in Fig. 4. The ECs of CP, SP and FP were not significantly different at pectin concentrations of 2 and 3% ($p > 0.05$). However, the reduction of pectin concentration to 1% reduced the ECs of CP and FP more significantly than EC of SP that showed similar values at all pectin concentrations ($p > 0.05$). The ESs of pectins at the end of 1- and 7-days also

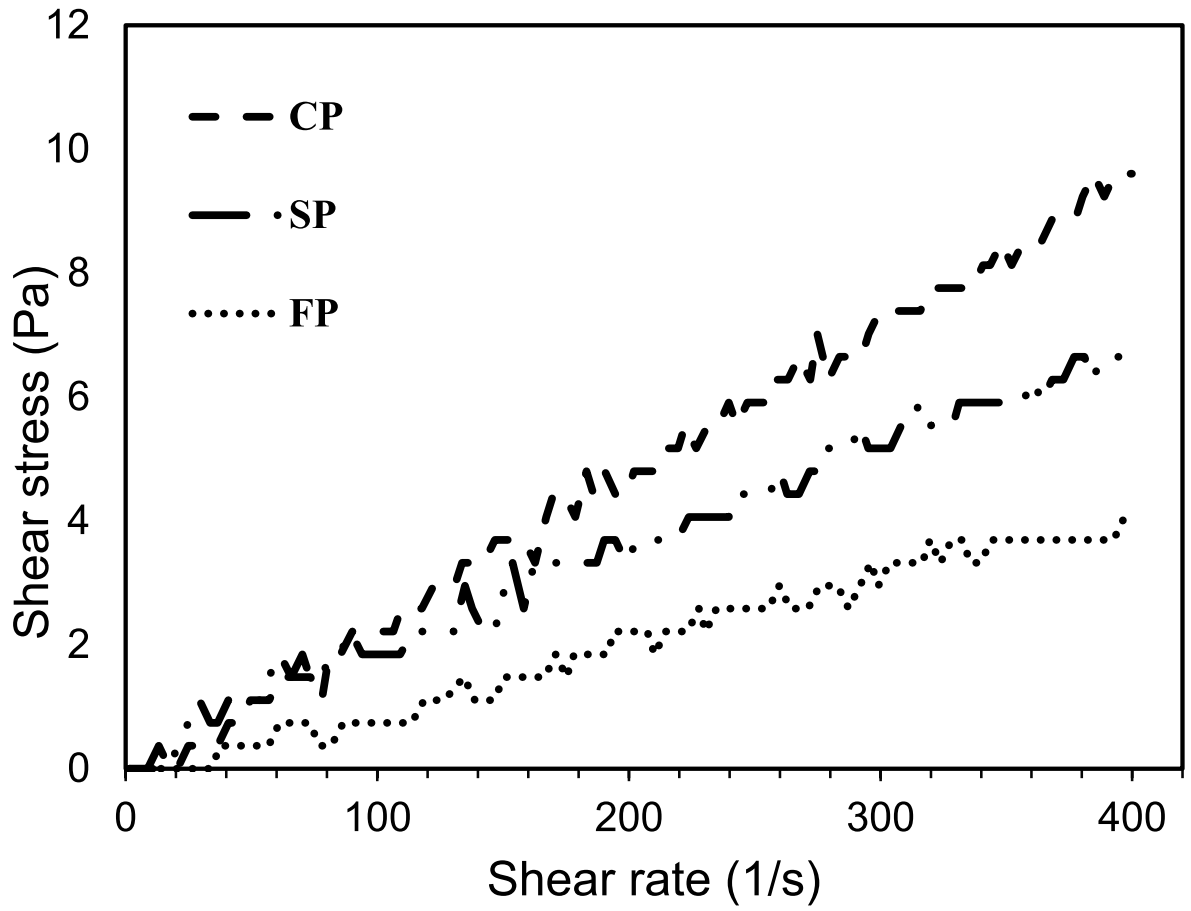

Fig. 3. Flow behavior of 3%(w/v) solutions of different fig pectins and commercial pectins at 23 °C.

showed some differences (Fig.4B and C). The CP and SP at 3% gave significantly higher ES than those at 1 and 2% at the end of 1- and 7-days ($p < 0.05$). The ES of FP at 3% was also higher than those at 1 and 2% at end of 1-day, but the FP concentration did not affect the ESs at the end of 7-days. The results of 7-days storage showed that the SP at 3% showed the highest ES value (~96.16%) while CP at 3% was the second highest value (~82.02%) at the end of 7-days. It is important to note that the SP at 2% and FP at 3% showed similar ESs at the end of 7-days. However, there were no significant differences among ESs of different pectins at 1 and 2% within 7-days. The higher ES of SP than the FP might be in part due to its greater capacity to increase the emulsion viscosity (see section 3.4.4). However, pectins maintain emulsion stability not only by increasing viscosity around emulsified lipid droplets but also by creating steric hindrance and electrostatic interactions (mainly repulsion) among lipid droplets within the emulsion system through their side and main chains (Funami et al., 2011). The higher steric hindrance created by side chains of SP explains its superior ES than CP although the latter had a higher ability to increase viscosity than the former. The amounts of protein of SP and CP determined in the current work were not considerably different. Thus, it appeared that the differences between ESs of SP and CP were also related to their variations in Mw and molecular structure (e.g. HG and RG composition, linearity, RG branching, repulsion created by negatively charged carboxyl groups, etc.) and/or surface activity of protein constituent.

The particle size characteristics of emulsions, D[3,2], formed at 3% pectin concentration are also shown at 0 and 7 days (Fig. 4D). On day 0, D[3,2] of pectin emulsions changed between 17 and 80 μm. The smallest sized emulsion droplet at day 0 was observed for FP (D[3,2] = 17 μm) while emulsions of SP formed the second smallest sized droplets (D[3,2] = 24 μm), and CP emulsions formed the largest sized emulsions (D[3,2] = 80 μm). The differences between the mean diameters of fig and citrus pectin emulsions could be related to different factors. For example, the smaller mean diameter of FP emulsions could be related to its higher protein content (natural surface-active agents) than the others. It is well-known that some polysaccharides such as sugar beet pectin, soluble soybean polysaccharide, and gum Arabic owe their emulsifying properties to the surface activity of complex hydrophobic protein components (Yemenicioğlu et al., 2020). In contrast, the large size of CP emulsion droplets might be related to its structural and conformational differences than the fig pectins (Neckebroeck et al., 2021). It appears that the long smooth chains of CP composed of GA units might initially form a thick layer around the oil droplets, thus, resulted with increased droplet sizes (Funami et al., 2011). No significant changes occurred in the mean diameter of SP emulsion droplets at day 7 while the D[3,2] of FP emulsions reduced slightly from 17 to 12 μm. In contrast, the significant reduction observed in the D[3,2] of CP emulsion from 80 to 44 μm suggested the depletion flocculation of pectin molecules from the interface that reduced the thick pectin layer around emulsified lipid droplets and caused the reduction of average droplet size (Neckebroeck et al., 2021) ($p < 0.05$). However, the stability of CP emulsion has not been considerably affected by this size change indicating that the sufficient emulsion stabilizing capacity of pectin molecules was still deposited around the oil-water interface of lipid droplets. The overall results showed that the SP could be an alternative to commercial citrus pectin to increase the emulsion stability of oil-in-water emulsion foods.

3.4.6. *Least gelling concentration and texture profile analysis of gels*

Before conducting texture profile analysis (TPA) of gels, the least gelling concentration (LGC) of SP and other pectins were determined. The LGC of SP, FP and CP by the standard high methoxyl pectin gelation

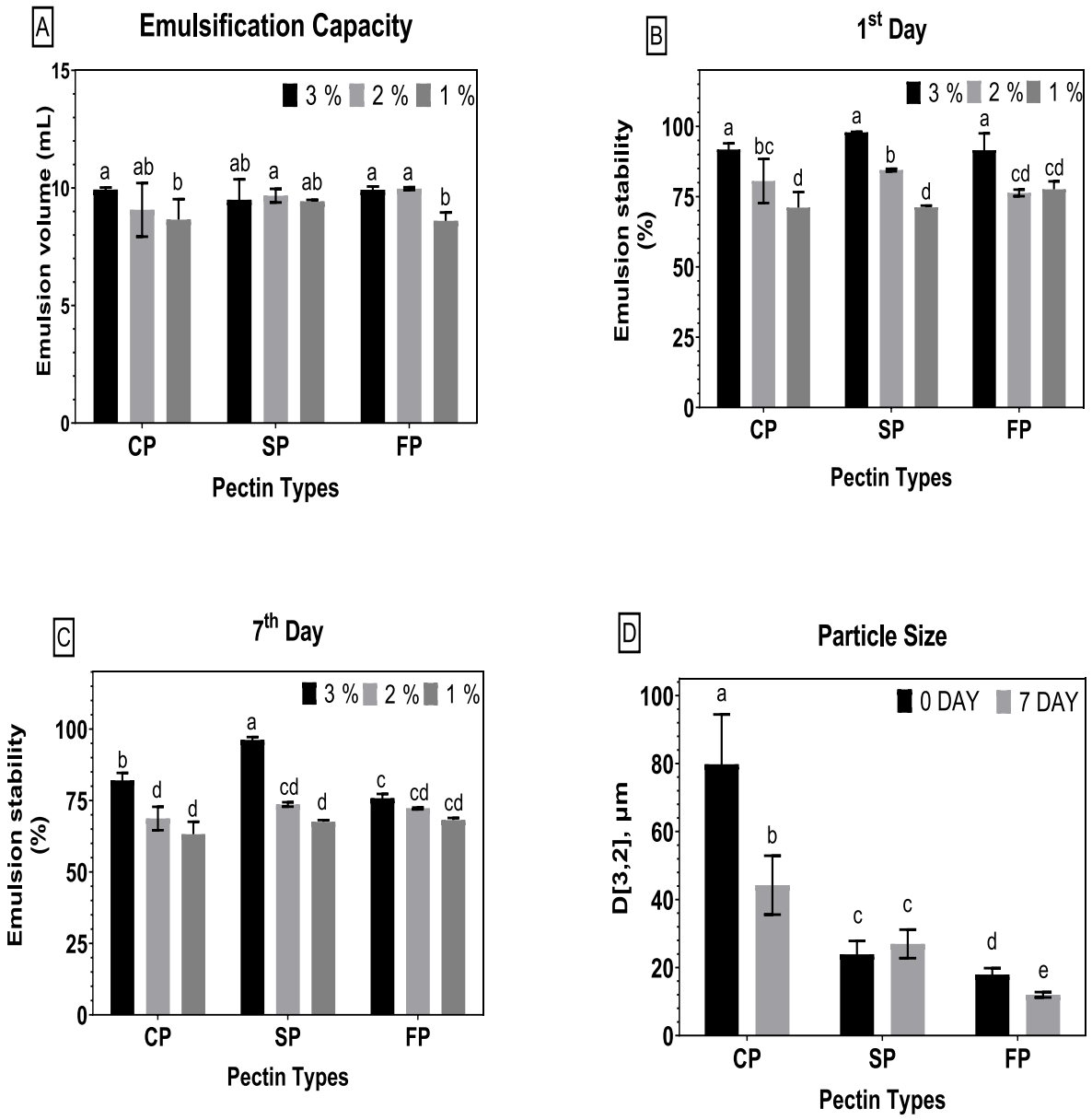

Fig. 4. Emulsification capacity (A); 1st Day of emulsion stability (B); 7 th Day of emulsion stability (C); particle sizes (D) of pectins.

method at 64% sucrose were found as 1.75, 1.2, and 0.4%, respectively. It appeared that the LGC of pectins correlated well with their DA values. Thus, the pectin concentrations between 1.25 and 3%, and 1.75 and 3% for FP and SP were selected to compare TPAs with those of CP gels, respectively. The DE of FP was 38.8%, but it was unable to show gelation in the presence of 30% sucrose by the standard low methoxyl pectin gelation method (Food Chemicals Codex, 1972). The lack of gel formation by FP in the presence of Ca^{++2} atoms should be related to irregular distribution (not blockwise manner) of nonmethoxylated carboxyl groups that are responsible for the formation of necessary junction zones (egg-box-model) during gelation (Fraeye et al., 2010).

The TPA results of pectin gels obtained by the standard high methoxyl pectin gelation method at 64% sucrose are shown in Table 4. The fracturability could not have been determined for the CP since this pectin did not form brittle gels. In contrast, the SP and FP gave brittle gels with fracturability values ranging between 0.30 and 4.22 N. The 2.4–4.6-fold greater fracturability values of the FP clearly showed that the FP gels are considerably more brittle than the SP. Additionally, the highest gel hardness was also obtained for FP followed by CP and SP. The hardness values of FP were 2–4 fold higher than those of CP (between 1.25 and 3%) and 2–6 fold higher than those of SP (between 1.75 and 3%). The hardness values of SP and CP were not significantly different at 2.5 and 3%, but CP at 1.75 and 2% showed slightly higher hardness than SP at the same concentrations. Both the hardness and fracturability of FP increased at a concentration-dependent manner between 1.25 and 3%. In contrast, the hardness of CP gels, and the hardness and fracturability of FP gels did not change considerably up to 2% ($p > 0.05$). However, pectin concentrations at 2.5 and 3% caused significant increases in hardness of CP gels, and in both hardness and fracturability of SP gels ($p < 0.05$).

The gumminess and chewiness of FP gels increased at a concentration-dependent manner between 1.25 and 3% while CP and SP concentrations must have been increased above 2% to have a concentration-dependent increase in these two parameters. The SP between 1.75 and 3% gave the least gummy gels. The FP and CP gels showed 2.6–4.5 and 2.2–2.6-fold higher gumminess than SP. The FP and CP gels had similar gumminess at 1.25, 2.5 and 3%, but FP gave significantly gummier (1.6-2-fold higher) gels than CP gels at 1.5, 1.75

Table 4
Texture profile analysis of pectin gels at different concentrations.

	Conc	Fracturability (N)[a]	Hardness (N)[a]	Gumminess (N)[a]	Chewiness (N.mm)[a]
CP	1.25%	–	0.454 ± 0.023gh	0.370 ± 0.032g	0.364 ± 0.037h
	1.50%	–	0.441 ± 0.043gh	0.338 ± 0.017g	0.328 ± 0.030h
	1.75%	–	0.479 ± 0.025gh	0.383 ± 0.023g	0.364 ± 0.020h
	2%	–	0.551 ± 0.144g	0.436 ± 0.094 fg	0.408 ± 0.088gh
	2.50%	–	1.736 ± 0.680de	1.147 ± 0.324bc	1.054 ± 0.300cd
	3%	–	2.501 ± 0.135c	1.694 ± 0.123a	1.583 ± 0.121 ab
FP	1.25%	0.496 ± 0.012f	0.817 ± 0.023f	0.357 ± 0.004g	0.350 ± 0.005h
	1.50%	0.774 ± 0.015e	1.270 ± 0.096ef	0.533 ± 0.063ef	0.519 ± 0.062 fg
	1.75%	1.117 ± 0.028d	1.865 ± 0.004de	0.761 ± 0.016d	0.737 ± 0.010e
	2%	1.662 ± 0.011c	2.300 ± 0.268cd	0.890 ± 0.120cd	0.858 ± 0.115de
	2.50%	2.631 ± 0.087b	3.483 ± 0.577b	1.266 ± 0.347bc	1.213 ± 0.324bc
	3%	4.224 ± 0.088a	4.994 ± 0.579a	1.817 ± 0.062a	1.764 ± 0.066a
SP	1.75%	0.301 ± 0.027g	0.305 ± 0.062ı	0.174 ± 0.023h	0.174 ± 0.022ı
	2%	0.357 ± 0.073g	0.382 ± 0.095hı	0.195 ± 0.027h	0.195 ± 0.025ı
	2.50%	1.051 ± 0.050d	1.256 ± 0.003ef	0.446 ± 0.026 fg	0.424 ± 0.026gh
	3%	1.790 ± 0.333c	1.993 ± 0.356cd	0.711 ± 0.102de	0.664 ± 0.088ef

[a] Data are shown as mean ± standard deviation. Data at each column indicated by different letters are significantly different ($p \leq 0.05$).

and 2%. The chewiness values of different pectins showed high parallelism with gumminess values. Therefore, FP gave the highest chewiness values followed by CP and SP.

The overall results of TPA clearly showed that SP forms intermediary gels comparable to gels of both FP and CP in several different parameters. The SP gave less gummy and chewable gels than CP, but both gels showed comparable hardness. The SP gels were also less hard and fracturable than FP in nature. The FP gave the highest firmness and fracturability and this may be related to its very low HG, but high RG-I content. It was reported that as the RG-I side chains increased, the formation of the tighter gelling network was promoted due to the increased entanglements among pectin molecules, and the formation of subsequent hydrophobic interactions and hydrogen bonding among HG chains (Sousa et al., 2015).

4. Conclusions

This work revealed that the stalks separated as waste during the processing of sun-dried figs are a better source of pectin than severely defected substandard whole figs since they provided higher extraction yield, degree of esterification, galacturonic acid content, and homogeneity (higher HG, but lower RG and branching). The characterization of stalk waste pectin and comparison of its functional properties with those of commercial citrus pectin clearly showed that this hydrocolloid presents outstanding emulsion stability, water absorption capacity and alternative gelling properties. The fig stalk waste pectin also possesses almost 2-fold higher free radical scavenging-based antioxidant activity than citrus pectin, but further studies are needed to investigate its health benefits such as prebiotic activity and antitumoral activity recently reported by Gharibzahedi et al. (2019b) for pectin extracted from fresh fig peels. This work revealed the potential of producing value-added pectin products from sun-dried fig processing wastes for the first time. The fig stalk waste pectin could be utilized for developing functional foods such as yogurts, yogurt and fruit beverages, dressings and sauces, smoothie balls, and jams and jellies having alternative structural and rheological properties.

CRediT authorship contribution statement

Elif Çavdaroğlu: Conceptualization, Investigation, Methodology, Data curation, Writing – original draft. **Ahmet Yemenicioğlu:** Conceptualization, Methodology, Supervision, Project administration, Writing – review & editing.

Declaration of competing interest

There are no conflicts of interest in this study.

Acknowledgments

This work is part of the Ph.D. thesis of author Elif Çavdaroğlu, and it was funded by The Scientific and Technological Research Council of Turkey (TUBITAK, Project no: 118 O 372). We thank the Yıldız Technical University Department of Bioengineering for their assistance in pectin molecular weight analysis. We thank Integrated Research Center at Izmir Institute of Technology for particle size analysis. Additionally, we thank Assoc. Prof. Dr. Ali Oğuz Büyükkileci for his guidance through the HPLC analysis.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2021.112624.

Ethical guidelines

Ethics approval was not required for this research.

Data availability statement

Research data are not shared.

References

Alba, K., & Kontogiorgos, V. (2017). Pectin at the oil-water interface: Relationship of molecular composition and structure to functionality. *Food Hydrocolloids, 68*, 211–218. https://doi.org/10.1016/j.foodhyd.2016.07.026

Anonymous. (2020). İncir Değerlendirme Raporu (TARIM/Ürün Masaları Ürün Değerlendirme Raporları yayımlandı/İncir Değerlendirme Raporu.pdf) https://www.tarimorman.gov.tr/BUGEM/Belgeler/MİLLİ.

AOAC. (1990). *Official methods of analysis (15th ed.). Washington, DC: Association of official analytical chemists*.

Asgari, K., Labbafi, M., Khodaiyan, F., Kazemi, M., & Hosseini, S. S. (2020). High-methylated pectin from walnut processing wastes as a potential resource: Ultrasound assisted extraction and physicochemical, structural and functional analysis. *International Journal of Biological Macromolecules, 152*, 1274–1282. https://doi.org/10.1016/j.ijbiomac.2019.10.224

Aydemir, L. Y., & Yemenicioğlu, A. (2013). Potential of Turkish Kabuli type chickpea and green and red lentil cultivars as source of soy and animal origin functional protein alternatives. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 50*(2), 686–694. https://doi.org/10.1016/j.lwt.2012.07.023

Bayar, N., Friji, M., & Kammoun, R. (2018). Optimization of enzymatic extraction of pectin from Opuntia ficus indica cladodes after mucilage removal. *Food Chemistry, 241*(April 2017), 127–134. https://doi.org/10.1016/j.foodchem.2017.08.051

Bradford, M. M. (1976). A dye binding assay for protein. *Analytical Biochemistry, 72*, 248–254.

Çalişkan, O., & Aytekin Polat, A. (2011). Phytochemical and antioxidant properties of selected fig (Ficus carica L.) accessions from the eastern Mediterranean region of Turkey. *Scientia Horticulturae, 128*(4), 473–478. https://doi.org/10.1016/j.scienta.2011.02.023

Çavdaroğlu, E., Farris, S., & Yemenicioğlu, A. (2020). Development of pectin–eugenol emulsion coatings for inhibition of Listeria on webbed-rind melons: A comparative study with fig and citrus pectins. *International Journal of Food Science and Technology, 55*(4), 1448–1457. https://doi.org/10.1111/ijfs.14458

Chandrayan, P. (2018). Biological function(s) and application (s) of pectin and pectin degrading enzymes. *Biosciences, Biotechnology Research Asia, 15*(1), 87–100. https://doi.org/10.13005/bbra/2611

Ciriminna, R., Fidalgo, A., Delisi, R., Ilharco, L. M., & Pagliaro, M. (2016). Pectin production and global market. *Agro Food Industry Hi-Tech, 27*(5), 17–20.

Cui, J., Zhao, C., Feng, L., Han, Y., Du, H., & Xiao, H. (2021). Pectins from fruits: Relationships between extraction methods, structural characteristics, and functional properties. *Trends in Food Science & Technology, 110*, 39–54. https://doi.org/10.1016/j.tifs.2021.01.077. October 2020.

Demirbüker, D., Simsek, S., & Yemenicioğlu, A. (2006). Potential application of hot rehydration alone or in combination with hydrogen peroxide to control pectin methylesterase activity and microbial load in cold-stored intermediate-moisture sun-dried figs. *Journal of Food Science, 69*(3), FCT170–FCT178. https://doi.org/10.1111/j.1365-2621.2004.tb13353.x

Denman, L. J., & Morris, G. A. (2015). An experimental design approach to the chemical characterisation of pectin polysaccharides extracted from Cucumis melo Inodorus. *Carbohydrate Polymers, 117*, 364–369. https://doi.org/10.1016/j.carbpol.2014.09.081

Dickinson, E. (2003). Hydrocolloids at interfaces and the influence on the properties of dispersed systems. *Food Hydrocolloids, 17*(1), 25–39. https://doi.org/10.1016/S0268-005X(01)00120-5

Domínguez Avila, J. A., Villegas Ochoa, M. A., Alvarez Parrilla, E., Montalvo González, E., & González Aguilar, G. A. (2018). Interactions between four common plant-derived phenolic acids and pectin, and its effect on antioxidant capacity. *Journal of Food Measurement and Characterization, 12*(2), 992–1004. https://doi.org/10.1007/s11694-017-9714-z

Doymaz, I. (2005). Sun drying of figs: An experimental study. *Journal of Food Engineering, 71*(4), 403–407. https://doi.org/10.1016/j.jfoodeng.2004.11.003

Dranca, F., & Oroian, M. (2018). Extraction, purification and characterization of pectin from alternative sources with potential technological applications. *Food Research International, 113*(June), 327–350. https://doi.org/10.1016/j.foodres.2018.06.065

Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. A., & Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry, 28*(3), 350–356. https://doi.org/10.1021/ac60111a017

Ezzati, S., Ayaseh, A., Ghanbarzadeh, B., & Heshmati, M. K. (2020). Pectin from sunflower by-product: Optimization of ultrasound-assisted extraction, characterization, and functional analysis. *International Journal of Biological Macromolecules, 165*, 776–786. https://doi.org/10.1016/j.ijbiomac.2020.09.205

Fellah, A., Anjukandi, P., Waterland, M. R., & Williams, M. A. K. (2009). Determining the degree of methylesterification of pectin by ATR/FT-IR: Methodology optimisation and comparison with theoretical calculations. *Carbohydrate Polymers, 78*(4), 847–853. https://doi.org/10.1016/j.carbpol.2009.07.003

Food Chemicals Codex. (1972). *Washington, DC: National academy of science*.

Fraeye, I., Colle, I., Vandevenne, E., Duvetter, T., Van Buggenhout, S., Moldenaers, P., Van Loey, A., & Hendrickx, M. (2010). Influence of pectin structure on texture of pectin-calcium gels. *Innovative Food Science & Emerging Technologies, 11*(2), 401–409. https://doi.org/10.1016/j.ifset.2009.08.015

Funami, T., Nakauma, M., Ishihara, S., Tanaka, R., Inoue, T., & Phillips, G. O. (2011). Structural modifications of sugar beet pectin and the relationship of structure to functionality. *Food Hydrocolloids, 25*(2), 221–229. https://doi.org/10.1016/j.foodhyd.2009.11.017

Gamonpilas, C., Buathongjan, C., Kirdsawasd, T., Rattanaprasert, M., Klomtun, M., Phonsatta, N., & Methacanon, P. (2021). Pomelo pectin and fiber: Some perspectives and applications in food industry. *Food Hydrocolloids, 120(April), 106981*. https://doi.org/10.1016/j.foodhyd.2021.106981

Gawkowska, D., Cieśla, J., Zdunek, A., & Cybulska, J. (2019). Cross-linking of diluted alkali-soluble pectin from apple (Malus domestica fruit) in different acid-base conditions. *Food Hydrocolloids, 92*, 285–292. https://doi.org/10.1016/j.foodhyd.2019.02.010. December 2018.

Gharibzahedi, S. M. T., Smith, B., & Guo, Y. (2019a). Pectin extraction from common fig skin by different methods: The physicochemical, rheological, functional, and structural evaluations. *International Journal of Biological Macromolecules, 136*, 275–283. https://doi.org/10.1016/j.ijbiomac.2019.06.040

Gharibzahedi, S. M. T., Smith, B., & Guo, Y. (2019b). Ultrasound-microwave assisted extraction of pectin from fig (Ficus carica L.) skin: Optimization, characterization and bioactivity. *Carbohydrate Polymers, 222*, 114992. https://doi.org/10.1016/j.carbpol.2019.114992. April.

Gilani, A. H., Mehmood, M. H., Janbaz, K. H., Khan, A., & ullah, & Saeed, S. A. (2008). Ethnopharmacological studies on antispasmodic and antiplatelet activities of Ficus carica. *Journal of Ethnopharmacology, 119*(1), 1–5. https://doi.org/10.1016/j.jep.2008.05.040

Gnanasambandam, R., & Proctor, A. (2000). Determination of pectin degree of esterification by diffuse reflectance Fourier transform infrared spectroscopy. *Food Chemistry, 68*(3), 327–332. https://doi.org/10.1016/S0308-8146(99)00191-0

Houben, K., Jolie, R. P., Fraeye, I., Van Loey, A. M., & Hendrickx, M. E. (2011). Comparative study of the cell wall composition of broccoli, carrot, and tomato: Structural characterization of the extractable pectins and hemicelluloses. *Carbohydrate Research, 346*(9), 1105–1111. https://doi.org/10.1016/j.carres.2011.04.014

Jafari, F., Khodaiyan, F., Kiani, H., & Hosseini, S. S. (2017). Pectin from carrot pomace: Optimization of extraction and physicochemical properties. *Carbohydrate Polymers, 157*, 1315–1322. https://doi.org/10.1016/j.carbpol.2016.11.013

Kazemi, M., Khodaiyan, F., & Hosseini, S. S. (2019). Utilization of food processing wastes of eggplant as a high potential pectin source and characterization of extracted pectin. *Food Chemistry*. https://doi.org/10.1016/j.foodchem.2019.05.063

Khedmat, L., Izadi, A., Mofid, V., & Mojtahedi, S. Y. (2020). Recent advances in extracting pectin by single and combined ultrasound techniques: A review of techno-functional and bioactive health-promoting aspects. *Carbohydrate Polymers, 229*. https://doi.org/10.1016/j.carbpol.2019.115474. July 2019.

Kpodo, F. M., Agbenorhevi, J. K., Alba, K., Bingham, R. J., Oduro, I. N., Morris, G. A., & Kontogiorgos, V. (2017). Pectin isolation and characterization from six okra genotypes. *Food Hydrocolloids, 72*, 323–330. https://doi.org/10.1016/j.foodhyd.2017.06.014

Kyomugasho, C., Christiaens, S., Shpigelman, A., Van Loey, A. M., & Hendrickx, M. E. (2015). FT-IR spectroscopy, a reliable method for routine analysis of the degree of methylesterification of pectin in different fruit- and vegetable-based matrices. *Food Chemistry, 176*, 82–90. https://doi.org/10.1016/j.foodchem.2014.12.033

Li, L., Gao, X., Liu, J., Chitrakar, B., Wang, B., & Wang, Y. (2021). Hawthorn pectin: Extraction, function and utilization. *Current Research in Food Science, 4*(June), 429–435. https://doi.org/10.1016/j.crfs.2021.06.002

Lira-Ortiz, A. L., Reséndiz-Vega, F., Ríos-Leal, E., Contreras-Esquivel, J. C., Chavarría-Hernández, N., Vargas-Torres, A., & Rodríguez-Hernández, A. I. (2014). Pectins from waste of prickly pear fruits (Opuntia albicarpa Scheinvar 'Reyna'): Chemical and rheological properties. *Food Hydrocolloids, 37*, 93–99. https://doi.org/10.1016/j.foodhyd.2013.10.018

Liu, J., Bi, J., McClements, D. J., Liu, X., Yi, J., Lyu, J., Zhou, M., Verkerk, R., Dekker, M., Wu, X., & Liu, D. (2020). Impacts of thermal and non-thermal processing on structure and functionality of pectin in fruit- and vegetable- based products: A review. *Carbohydrate Polymers, 250*(2), 116890. https://doi.org/10.1016/j.carbpol.2020.116890

Monsoor, M. A. (2005). Effect of drying methods on the functional properties of soy hull pectin. *Carbohydrate Polymers, 61*(3), 362–367. https://doi.org/10.1016/j.carbpol.2005.06.009

M'sakni, N. H., Majdoub, H., Roudesli, S., Picton, L., Le Cerf, D., Rihouey, C., & Morvan, C. (2006). Composition, structure and solution properties of polysaccharides extracted from leaves of Mesembryanthenum crystallinum. *European Polymer Journal, 42*(4), 786–795. https://doi.org/10.1016/j.eurpolymj.2005.09.014

Muhammad, K., Nur, N. I., Gannasin, S. P., Mohd Adzahan, N., & Bakar, J. (2014). High methoxyl pectin from dragon fruit (Hylocereus polyrhizus) peel. *Food Hydrocolloids, 42*(P2), 289–297. https://doi.org/10.1016/j.foodhyd.2014.03.021

Muñoz-Almagro, N., Montilla, A., & Villamiel, M. (2020). Role of pectin in the current trends towards low-glycaemic food consumption. *Food Research International, October, 109851*. https://doi.org/10.1016/j.foodres.2020.109851

Muñoz-Almagro, N., Ruiz-Torralba, A., Méndez-Albiñana, P., Guerra-Hernández, E., García-Villanova, B., Moreno, R., Villamiel, M., & Montilla, A. (2021). Berry fruits as source of pectin: Conventional and non-conventional extraction techniques. *International Journal of Biological Macromolecules, 186*(July), 962–974. https://doi.org/10.1016/j.ijbiomac.2021.07.016

Namir, M., Siliha, H., & Ramadan, M. F. (2015). Fiber pectin from tomato pomace: Characteristics, functional properties and application in low-fat beef burger. *Journal of Food Measurement and Characterization, 9*(3), 305–312. https://doi.org/10.1007/s11694-015-9236-5

Neckebroeck, B., Verkempinck, S. H. E., Van Audenhove, J., Bernaerts, T., de Wilde d'Estmael, H., Hendrickx, M. E., & Van Loey, A. M. (2021). Structural and emulsion stabilizing properties of pectin rich extracts obtained from different botanical sources. *Food Research International, 141*, 110087. https://doi.org/10.1016/j.foodres.2020.110087. July 2020.

Noreen, A., Nazli, Z., i, H., Akram, J., Rasul, I., Mansha, A., Yaqoob, N., Iqbal, R., Tabasum, S., Zuber, M., & Zia, K. M. (2017). Pectins functionalized biomaterials; a new viable approach for biomedical applications: A review. *International Journal of Biological Macromolecules, 101*, 254–272. https://doi.org/10.1016/j.ijbiomac.2017.03.029

Ogutu, F. O., & Mu, T.-H. (2017). Ultrasonic degradation of sweet potato pectin and its antioxidant activity. *Ultrasonics Sonochemistry, 38*, 726–734. https://doi.org/10.1016/j.ultsonch.2016.08.014

Petkowicz, C. L. O., Vriesmann, L. C., & Williams, P. A. (2017). Pectins from food waste: Extraction, characterization and properties of watermelon rind pectin. *Food Hydrocolloids, 65*, 57–67. https://doi.org/10.1016/j.foodhyd.2016.10.040

Qiu, C., Zhao, M., & McClements, D. J. (2015). Improving the stability of wheat protein-stabilized emulsions: Effect of pectin and xanthan gum addition. *Food Hydrocolloids, 43*, 377–387. https://doi.org/10.1016/j.foodhyd.2014.06.013

Raji, Z., Khodaiyan, F., Rezaei, K., Kiani, H., & Hosseini, S. S. (2017). Extraction optimization and physicochemical properties of pectin from melon peel. *International Journal of Biological Macromolecules, 98*, 709–716. https://doi.org/10.1016/j.ijbiomac.2017.01.146

Reichembach, L. H., & Lúcia de Oliveira Petkowicz, C. (2021). Pectins from alternative sources and uses beyond sweets and jellies: An overview. *Food Hydrocolloids, 118*(April). https://doi.org/10.1016/j.foodhyd.2021.106824, 106824.

Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M., & Rice-Evans, C. (1999). Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radical Biology and Medicine, 26*(98), 1231–1237.

Rezvanain, M., Ahmad, N., Mohd Amin, M. C. I., & Ng, S. F. (2017). Optimization, characterization, and in vitro assessment of alginate-pectin ionic cross-linked hydrogel film for wound dressing applications. *International Journal of Biological Macromolecules, 97*, 131–140. https://doi.org/10.1016/j.ijbiomac.2016.12.079

Rtibi, K., Selmi, S., Grami, D., Sebai, H., & Marzouki, L. (2018). Laxative and anti-purgative bioactive compounds in prevention and treatment of functional gastrointestinal disorders, constipation and diarrhea. *Journal of Nutritional Health & Food Engineering, 8*(6), 8–10. https://doi.org/10.15406/jnhfe.2018.08.00312

Rubio-Senent, F., Rodríguez-Gutiérrez, G., Lama-Muñoz, A., García, A., & Fernández-Bolaños, J. (2015). Novel pectin present in new olive mill wastewater with similar emulsifying and better biological properties than citrus pectin. *Food Hydrocolloids, 50*, 237–246. https://doi.org/10.1016/j.foodhyd.2015.03.030

Rumpunen, K., Thomas, M., Badilas, N., & Thibault, J. F. (2002). Validation of a combined enzymatic and HPLC method for screening of pectins in fruits of Japanese quince (Chaenomeles japonica). *Lebensmittel-Wissenschaft Und-Technologie-Food Science and Technology, 35*(6), 490–496. https://doi.org/10.1006/fstl.895

Simmons, M. S. J., & Preedy, V. R. (2016). *Nutritional composition of fruit cultivars*. Academic Press.

Sinitsya, A., Čopıková, J., Prutyanov, V., Skoblya, S., & Machovič, V. (2002). Amidation of highly methoxylated citrus pectin with primary amines. *Carbohydrate Polymers, 42* (4), 359–368. https://doi.org/10.1016/s0144-8617(99)00184-8

Sousa, A. G., Nielsen, H. L., Armagan, I., Larsen, J., & Sørensen, S. O. (2015). The impact of rhamnogalacturonan-I side chain monosaccharides on the rheological properties of citrus pectin. *Food Hydrocolloids, 47*, 130–139. https://doi.org/10.1016/j.foodhyd.2015.01.013

Trad, M., Ginies, C., Gaaliche, B., Renard, C. M. G. C., & Mars, M. (2014). Relationship between pollination and cell wall properties in common fig fruit. *Phytochemistry, 98*, 78–84. https://doi.org/10.1016/j.phytochem.2013.12.011

UNECE. (2016). UNECE Standard DDP-15 concerning the marketing and commercial quality control of Dried Figs. http://www.unece.org/trade/agr/welcome.html.

Voragen, A. G. J., Schols, H. A., & Pilnik, W. (1986). Determination of the degree of methylation and acetylation of pectins by h.p.l.c. *Topics in Catalysis, 1*(1), 65–70. https://doi.org/10.1016/S0268-005X(86)80008-X

Vriesmann, L. C., & Petkowicz, C. L. O. (2013). Highly acetylated pectin from cacao pod husks (Theobroma cacao L.) forms gel. *Food Hydrocolloids, 33*(1), 58–65. https://doi.org/10.1016/j.foodhyd.2013.02.010

Wang, W., Ma, X., Jiang, P., Hu, L., Zhi, Z., Chen, J., Ding, T., Ye, X., & Liu, D. (2016). Characterization of pectin from grapefruit peel: A comparison of ultrasound-assisted and conventional heating extractions. *Food Hydrocolloids, 61*, 730–739. https://doi.org/10.1016/j.foodhyd.2016.06.019

Wathoni, N., Yuan Shan, C., Yi Shan, W., Rostinawati, T., Indradi, R. B., Pratiwi, R., & Muchtaridi, M. (2019). Characterization and antioxidant activity of pectin from Indonesian mangosteen (Garcinia mangostana L.) rind. *Heliyon, 5*(8), Article e02299. https://doi.org/10.1016/j.heliyon.2019.e02299

Wicker, L., Kim, Y., Kim, M. J., Thirkield, B., Lin, Z., & Jung, J. (2014). Pectin as a bioactive polysaccharide - extracting tailored function from less. *Food Hydrocolloids, 42*(P2), 251–259. https://doi.org/10.1016/j.foodhyd.2014.01.002

Wongkaew, M., Kittiwachana, S., Phuangsaijai, N., Tinpovong, B., Tiyayon, C., Pusadee, T., … Cheewangkoon, R. (2021). Fruit Characteristics, Peel Nutritional Compositions, and Their Relationships with Mango Peel Pectin Quality. *Plants, 10* (6), 1148. https://doi.org/10.3390/plants10061148

Xu, K., Martinez, M. M., Yang, B., & Guo, M. (2020). Fine structure, physicochemical and antioxidant properties of LM-pectins from okra pods dried under different techniques. *Carbohydrate Polymers, 241*, 116272. https://doi.org/10.1016/j.carbpol.2020.116272. April.

Yang, X., Guo, J. L., Ye, J. Y., Zhang, Y. X., & Wang, W. (2015). The effects of Ficus carica polysaccharide on immune response and expression of some immune-related genes in grass carp, Ctenopharyngodon idella. *Fish & Shellfish Immunology, 42*(1), 132–137. https://doi.org/10.1016/j.fsi.2014.10.037

Yapo, B. M. (2011). Rhamnogalacturonan-I: A structurally puzzling and functionally versatile polysaccharide from plant cell walls and mucilages. *Polymer Reviews, 51*(4), 391–413. https://doi.org/10.1080/15583724.2011.615962

Yemenicioğlu, A., Farris, S., Turkyilmaz, M., & Gulec, S. (2020). A review of current and future food applications of natural hydrocolloids. *International Journal of Food Science and Technology, 55*(4), 1389–1406. https://doi.org/10.1111/ijfs.14363

Yuliarti, O., Goh, K. K. T., Matia-merino, L., Mawson, J., & Brennan, C. (2015). Extraction and characterisation of pomace pectin from gold kiwifruit (Actinidia chinensis). *Food Chemistry, 187*, 290–296.

Yuliarti, O., Matia-Merino, L., Goh, K. K. T., Mawson, J. A., & Brennan, C. S. (2011). Effect of celluclast 1.5L on the physicochemical characterization of gold Kiwifruit pectin. *International Journal of Molecular Sciences, 12*(10), 6407–6417. https://doi.org/10.3390/ijms12106407

Zhang, C., Zhu, X., Zhang, F., Yang, X., Ni, L., Zhang, W., Liu, Z., & Zhang, Y. (2020). Improving viscosity and gelling properties of leaf pectin by comparing five pectin extraction methods using green tea leaf as a model material. *Food Hydrocolloids, 98*. https://doi.org/10.1016/j.foodhyd.2019.105246. July 2019.

Zhong, X.-K., Jin, X., Lai, F.-Y., Lin, Q.-S., & Jiang, J.-G. (2010). Chemical analysis and antioxidant activities in vitro of polysaccharide extracted from Opuntia ficus indica Mill. cultivated in China. *Carbohydrate Polymers, 82*(3), 722–727. https://doi.org/10.1016/j.carbpol.2010.05.042