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Developing emulsion gels by incorporating Jerusalem artichoke inulin and investigating their lipid oxidative stability

Feijie Li¹, Aynur Gunenc¹ and Farah Hosseinian^{1,2*}

Abstract

This study investigated physical, chemical and lipid oxidative properties of emulsion gels (W/O) incorporating Jerusalem artichoke (JA) inulin. Primary purified inulin extract (PPIE, 1%) improved the homogeneity of emulsion gel (with no syneresis) and developed smaller particle size droplets (average 40 μm) than control (average size 60 μm). HPLC revealed that PPIE had 80.28% inulin content compared with commercial inulin (CI, 100%). Crude inulin extract (CIE, 0.08–0.33 mg/mL) delayed linoleic acid oxidation because of higher total phenolic content (4.96 ± 0.01 , mg GAE/g), compared with PPIE (0.72 ± 0.03). Lipid oxidative stability of emulsion gels with inulin samples was in the order of CI > PPIE > CIE ($P < 0.05$) by Rancimat analysis, which agreed with volumetric gel index results. This study suggests that emulsion gels with JA inulin (PPIE) could act as a potential fat replacement in food systems.

Keywords: Inulin, Emulsion gel, Reduced-fat, Liposome, Lipid oxidation, Rancimat

Highlights

- Emulsion gel with 1% primary purified inulin extract (PPIE) developed smaller droplets size and had no syneresis.
- Emulsion gels with higher inulin content meant longer induction time.
- Crude inulin extract could delay linoleic acid oxidation using liposome model.
- Crude inulin extract contained higher amount of phenolics than PPIE.

Introduction

Traditionally, solid fat is derived from saturated animal or vegetable fat (Hartel et al. 2018), or from partially hydrogenated oil which might be associated with an increased risk of coronary heart disease (Mozaffarian et al. 2006). With increased health concerns, people prefer to choose low-fat or reduced-fat food. However, fat has a significant effect on the physical properties of food

products (Dave 2012). Low-fat or reduced-fat foods with grainy and undesirable texture have less organoleptic properties than full-fat foods (Hamilton et al. 2000). One of the promising strategies is using fat replacers that have similar physical and organoleptic properties as fat. It is reported that succinyl chitosan in cakes formulations (Rios et al. 2018), hydroxypropyl methylcellulose in muffins (Oh & Lee 2018), soy protein hydrolysate/xanthan gum in low-fat ice cream (Liu et al. 2018), chia and oat emulsion gels in low-fat sausages (Pintado et al. 2018) were used as fat replacers.

Emulsion gel comprised of inulin and monoglycerides is a potential fat replacer. Inulin could form a gel in aqueous solution at high concentrations (> 15%) throughout the heating-cooling process (Kim et al. 2001). In addition, monoglycerides and vegetable oil could form a semi-solid aggregated network structure when cooling from heating (Ojijo et al. 2004).

Inulin (Fig. 1) is a dietary fiber (polysaccharide) in which D-fructose units linked by β (2 \rightarrow 1) linkages with the end of glucose residue (Li et al. 2015). The degree of polymerization (DP) of inulin varies from 2 to 60, depending on plant resources, harvest time, storage and process conditions (Saengthongpinit & Sajjaanantakul 2005), which determines the biological and physical

* Correspondence: farah.hosseinian@carleton.ca

¹Food Science and Nutrition, Chemistry Department, Carleton University, Ottawa, ON, Canada

²Institute of Biochemistry, Carleton University, 1125 Colonel By Drive, Ottawa, ON K1S 5B6, Canada



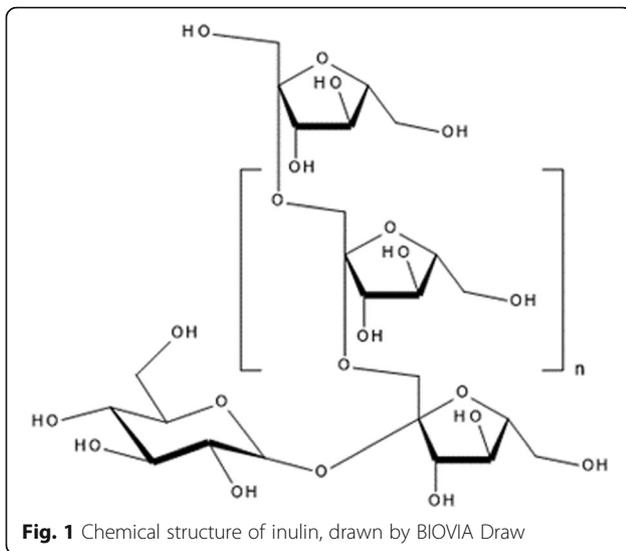


Fig. 1 Chemical structure of inulin, drawn by BIOVIA Draw

properties of inulin (Rubel et al. 2018). High DP inulin has good ability to form gels (Kim et al. 2001). Also, inulin has amazing water-binding activity, and which could reduce syneresis in spread (Fadaei et al. 2012).

On the other hand, sugars and sugar-like compounds play an important role in defending oxidative stress in plant cells (Bolouri-Moghaddam et al. 2010). Stoyanova et al. (2011) revealed that inulin and stevioside have super capacity of scavenging hydroxyl and superoxide radicals, which were more effective than mannitol that is well-known as an antioxidant (Stoyanova et al. 2011).

Lipid oxidation can be measured with different methods including spectrophotometry for conjugated dienes (Liégeois et al. 2000) and 2-thiobarbituric acid (TBA) value (Zeng et al. 2017), Rancimat method for oil stability index (Tavakoli et al. 2019), chromatographic analysis for changes in reactants (Bazina & He 2018), Fourier transform infrared (FTIR) method for peroxide value; differential scanning calorimetry (DSC), iodometric titration, and others (Shahidi & Zhong 2005). Conjugated dienes with strong absorption of UV radiation at 234 nm are produced during oxidation of lipid that contains dienes or polyenes, which is a good index for measuring lipid oxidation (Vieira & Regitano-D'arce 1998). Rancimat method is normally used to test oil stability under accelerated storage conditions to estimate the shelf life of food products (Farhoosh et al. 2008).

The objectives of this work were to: (i) analyze JA inulin extracts by HPLC-IR; (ii) formulate emulsion gels by incorporating JA inulin; (iii) evaluate the effects of inulin extracts against lipid oxidation using liposome model and emulsion gel; (iv) inspect total phenolic content in inulin extracts; (v) investigate oxidative stability of emulsion gels by Rancimat method.

Materials and methods

Chemicals and reagents

Soy lecithin and monoglycerides (MGs) (Alphadim® 90 SBK) were kindly provided by Grain Process Enterprises limited (Scarborough, ON, CA) and Caravan Ingredients, Inc. (Lenexa, Kansas, USA), respectively. Extra virgin olive oil was purchased from Amazon.ca. Tween 80 (polyoxyethylene-20-sorbitan monooleate) and sucrose (ultra-pure) were from Fisher Scientific (CA). Activated charcoal (powder, 100 particle size), Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), linoleic acid ($\geq 99\%$), Tween 20 (polyoxyethylene sorbitan monolaurate), inulin from chicory (commercial inulin) and Folin & Ciocalteu phenol reagent were from Sigma-Aldrich (St Louis, MO, USA). CaCl_2 was from Sigma Chemical Co. (St. Louis, MO, USA). Na_2CO_3 was from VWR International Co. (Mississauga, ON, CA). Glucose (CAS: 50–99-7) was from Sigma-Aldrich (UK). D-fructose was from BioShop Canada Inc. AAPH (2,2'-Azobis (2-amidinopropane) dihydrochloride, 98%) was from ACROS ORGANICS (New Jersey, USA). Gallic acid was from Sigma Chemical (St Louis, MO, USA).

Inulin extraction

Sample preparation

Fresh Jerusalem artichoke (JA) tubers were sliced, freeze-dried and milled to obtain powdered tubers. It was stored in a desiccator at room temperature until further analysis (Srinameb et al. 2015).

Extractions

Crude inulin extract (CIE) JA tuber powder was extracted with distilled water (1:20 w/v) at 80 °C for 2 h. After cooling to room temperature, the mixture was filtered and centrifuged (Thermo Fisher Scientific, Langensfeld, Germany) at a speed of 4500 g at 23 °C for 15 min. Supernatant was freeze-dried (Labconco, Kansas, USA) and stored in the fridge until further analysis (Srinameb et al. 2015).

Primary purified inulin extract (PPIE) CIE was further purified by deproteinization (Huang et al. 2011) and decolouration (Hongxin 2008). Briefly, CaCl_2 (0.1%, w/v) was added into CIE solution and mixed until flocculi were no longer produced. Na_2CO_3 was added to precipitate redundant CaCl_2 . Color was removed by adding 2.0% (w/v) activated charcoal at 80 °C for 15 min, then filtered and freeze-dried to obtain PPIE.

Secondary purified inulin extract (SPIE) For further purification, 4 parts of ethanol were added to 1 part of PPIE water solution (5%) to get precipitated inulin (SPIE) by filtration (Ku et al. 2003).

HPLC-MS

Chromatographic analysis was carried out using high-performance liquid chromatography (HPLC, Waters e2695, Waters Corporation, Milford, USA) with a refractive index (RI) detector (Waters 2414, Waters Corporation, Milford, USA). Commercial inulin (CI, 0.25 mg/mL), PPIE (1.0 mg/mL) and SPIE (1.0 mg/mL) were separated by Sugar-Pak™ I column (6.5 × 300 mm, 10 μm, Waters, USA) at 60 °C with 10 μL injection volume. Milli-Q water (Millipore) was used as mobile phase with a flow rate of 0.6 mL/min. Glucose, fructose and sucrose were used as standards.

The mass spectrometry (MS) analysis of inulin extract (PPIE) was conducted in Carleton Mass Spectrometry Center. Four microliters inulin sample was placed into the Proxeon nanoelectrospray emitter and detected by a hybrid quadrupole TOF MS. The spectra of samples were recorded with the voltage of ESI (electrospray ionization), and the data was analyzed by ChemStation software.

Emulsion gel preparations

Inulin gel

CI, PPIE and CIE were dissolved in distilled water (20%, w/v) separately at 80 °C for 5 min, and then cooled to room temperature (23 °C) for 24 h to set gel structure. In order to compare gel formation degree, volumetric gel index (VGI) was calculated for each sample. VGI is defined as the ratio of gel volume to total volume (Eq. 1) (Kim et al. 2001).

$$\begin{aligned} \text{Volumetric gel index (VGI)} \\ = (\text{gel volume})/(\text{total volume}) \times 100\% \end{aligned} \quad (1)$$

Oleogel

Monoglycerides were dissolved in olive oil (5%, w/v) at 80 °C until totally melted, and then cooled at room temperature to form oleogel (23 °C).

Inulin emulsion gel

The ingredients of emulsion gels included 57.0% olive oil, 3.0% monoglycerides, 0.8% Tween 80, 39% distilled water and 1% inulin samples (CI, PPIE, CIE). An equal amount of distilled water instead of inulin samples acted as a control. These formulations A (CI), B (PPIE), C (CIE) and D (control) of emulsion gels were designed and modified according to preliminary experimental results. Specifically, monoglycerides were dissolved in oil completely at 80 °C, and then the water phase including inulin was added to the oil phase at the same temperature, followed by strong vortex (Mini-vortexer VM-3000, VWR, Radnor, USA) until a desired consistency was achieved. Emulsions were cooled to

form emulsion gel at room temperature (23 °C) until further analysis.

Microscopic observations

Microstructures of inulin gel and oleogel were observed under PLM (polarized light microscope, Axioplan 2 imaging and Axiophot 2 universal microscope). Emulsion gels (with 1% PPIE and control) were visualized both by PLM and cryo-SEM (cryo-scanning electron microscope, Nano Imaging Facility Laboratory of Carleton University, Ottawa, ON) after prepared immediately to see their morphological differences, so that effect of 1% PPIE was achieved.

Antioxidant activity

Conjugated dienes

Aqueous dispersion of liposome model Liposome dispersion was prepared by the film hydration method (Hosseinian et al. 2006). Specifically, 0.5% (v/v) lipid dispersion was prepared by dissolving 20 μL linoleic acid and 30 μL soy lecithin into a vial containing 2 mL chloroform. A thin lipid film was formed at the bottom of the vial after chloroform evaporated in a fume hood overnight. Phosphate buffer solution (10 mL of 0.05 mol/L, pH 7.4) containing 0.5% (v/v) Tween 20 was added into the vial (Liégeois et al. 2000) and agitated in ultrasonic bath (PS-20, Qingdao, Shandong, China) for 15 min, mixed by a mini-vortexer until it became cloudy. Unilamellar liposome vesicles (ULVs) were produced by the extrusion method using Liposofast mini-extruder (Avestin, Inc., Ottawa, ON, CA) as explained by Hosseinian et al. (2006) and established in this lab.

Emulsion gel Four emulsion gels (A, B, C, D) consisted of CI, PPIE, CIE and control were prepared as in [Inulin emulsion gel](#) section. They were diluted 4 times with phosphate buffer solution (10 mL of 0.05 mol/L, pH 7.4) containing 0.8% (v/v) Tween 20 until further use.

Antioxidant test Antioxidant activities were determined by the method of Liégeois et al. (2000) with modification. As for the liposome model, 50 μL of 0.5% ULVs dispersion and 50 μL of 5 mg/mL sample (either PPIE, CIE, CI, trolox or control) were added into a quartz cuvette which contained 2.75 mL phosphate buffer solution (pH 7.4, 50 mmol/L). For the emulsion gel, 100 μL 4-fold diluted emulsion gel (containing either PPIE, CIE, CI or control) was added to the quartz cuvette and other conditions were the same. The oxidation reaction was initiated at ambient temperature by adding 150 μL of 40 mmol/L AAPH which acted as a free radical generator. Antioxidant activities were tested kinetically using a Cary 50 Bio UV-visible spectrophotometer (Varian Inc.,

Australia). In the liposome model without any antioxidant, lipid oxidation was carried out in the presence of the same amount of phosphate buffer solution (PBS, blank control). Absorbances of samples themselves and AAPH in buffer at 234 nm were all subtracted (Liégeois et al. 2000). Each test was performed in triplicate and the averages were used as final results.

TPC assay

Crude inulin extract (CIE), primary purified inulin extract (PPIE) and commercial inulin (CI) were dissolved in distilled water separately and formed 5 mg/ml sample solutions. Fifty microliters of standard (gallic acid), sample solution or blank (distilled water) was added into a 1.5 mL Eppendorf tube, followed by 475 μL of 10-time diluted Folin & Ciocalteu phenol solution, and mixed thoroughly. Then 475 μL of 6% sodium carbonate solution was added and mixed again. All these mixtures were incubated in darkness at ambient temperature for 2 h and measured at 725 nm using a microplate reader with

Gen 5 software (BioTek, USA). Each sample was performed and analyzed in separate triplicates, and the averages were used as final results expressed as mg gallic acid equivalency per gram sample (mg GAE/g sample) (Gunenc et al. 2015).

Rancimat

Accelerated oxidative stability of emulsion gels (CIE, PPIE, CI) was tested by Rancimat (Metrohm). Stability parameters were set at 121 °C and 20 mL/h airflow rate. This test was performed in duplicate.

Statistics analysis

One-way ANCOVA (analysis of covariance) followed by post-hoc Tukey’s test was used to find significant differences (α = 0.05). Results were significantly different when *P* < 0.05.

Results and discussion

HPLC-MS analysis

HPLC-IR chromatograms of the samples (PPIE: primary purified inulin extract, SPIE: secondary purified inulin

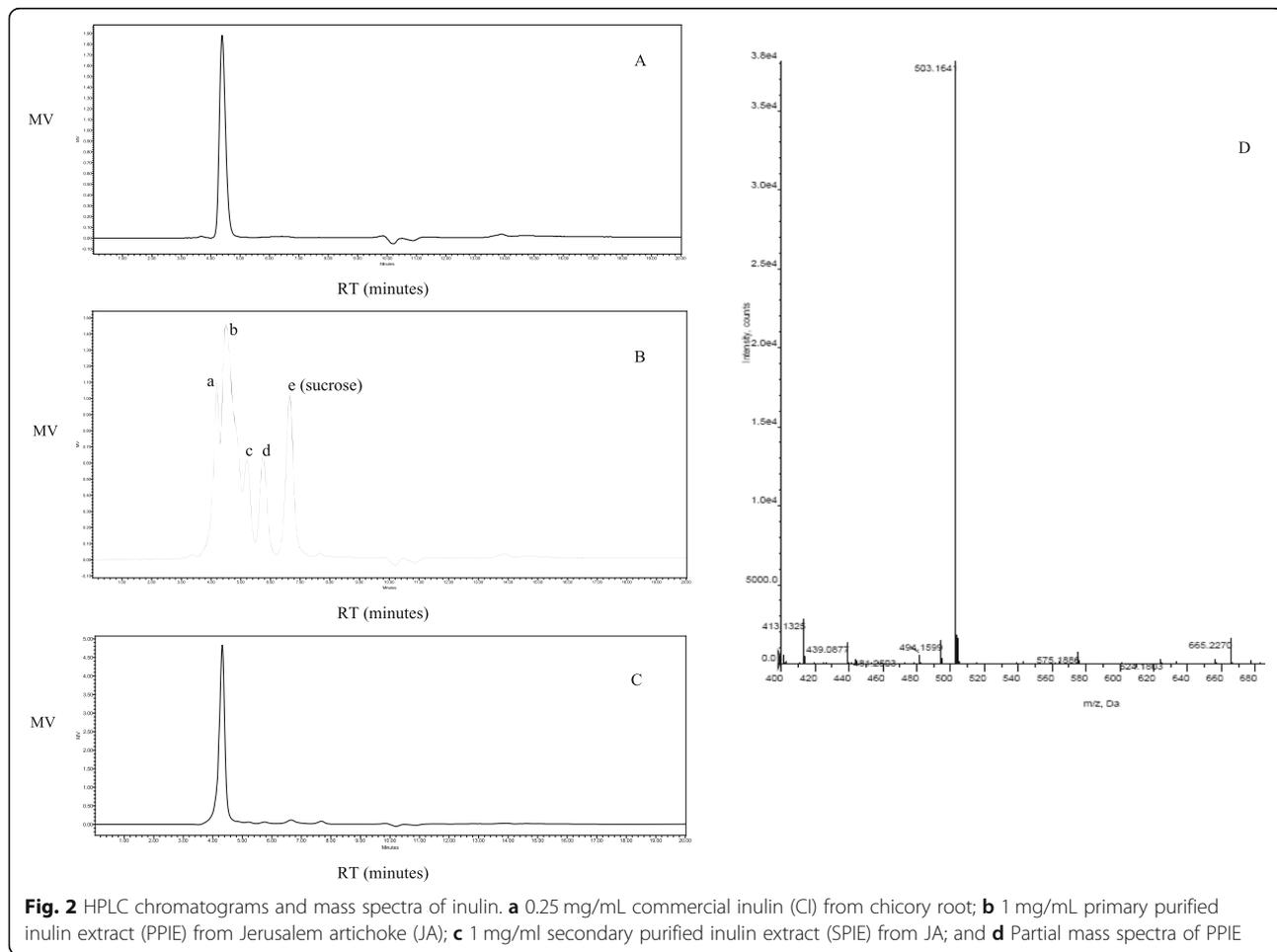


Fig. 2 HPLC chromatograms and mass spectra of inulin. **a** 0.25 mg/mL commercial inulin (CI) from chicory root; **b** 1 mg/mL primary purified inulin extract (PPIE) from Jerusalem artichoke (JA); **c** 1 mg/ml secondary purified inulin extract (SPIE) from JA; and **d** Partial mass spectra of PPIE

extract) and control (CI: commercial inulin) were shown in Fig. 2. The chromatogram of crude inulin extract (CIE) was not shown because CIE contains many impurities. Figure 2a showed only one peak at 4.4 min, which could be inulin with several DPs (degree of polymerizations) that had close retention time. DP of inulin varies from 2 to 60, depending on plant resources, harvest time, storage and process conditions (Saengthongpinit & Sajjanantakul 2005). It was reported that commercial chicory inulin (from Sigma-Aldrich the same as CI used in this study) had an average DP between 13 and 30 analyzed by HPAEC-PAD (Böhm et al. 2005).

The chromatogram of PPIE sample (Fig. 2b) mainly showed five peaks (a, b, c, d and e); peak a to d (higher DP inulin, DP >2) constituted 80.28%, and peak e (sucrose) took up 17.92% according to their peak areas. From all those peaks, the peak “b” that accounted for 45.48% had the same retention time as the one in CI (Fig. 2a). Similarly, (Beirão-da-costa et al. 2005) analyzed inulin with the same detector and column (HPLC-RI with Sugar pak I), and found that higher DP came out from the column earlier than lower DP, and fructose

and glucose came out at last (Beirão-da-costa et al. 2005). Other peaks in Fig. 2b were possibly inulin with higher DPs. In Fig. 2d, the MS analysis confirmed PPIE contained DP 3 (503 Da) and DP 4 (665 Da). In general, PPIE had wider distributed peaks than CI.

Interestingly, the chromatogram of SPIE (Fig. 2c) had almost the same peak with CI (Fig. 2a). This confirmed that addition of ethanol to PPIE solution precipitated inulin with higher DP, which agreed with the literature (Temkov et al. 2015). Although SPIE had higher purity than PPIE, its yield (7.85%) was negligible. Therefore, PPIE was chosen to prepare emulsion gel instead of SPIE.

Inulin emulsion gel

Figure 3 shows powders, gels of CI, PPIE, CIE, MGs, and emulsion gels (A, B, C, D) after heating-cooling process.

Inulin gel and oleogel

Gel can be defined as an intermediate (semi-solid) material between liquid and solid (Nishinari 2009). Inulin solution formed a gel slowly as temperature decreased,

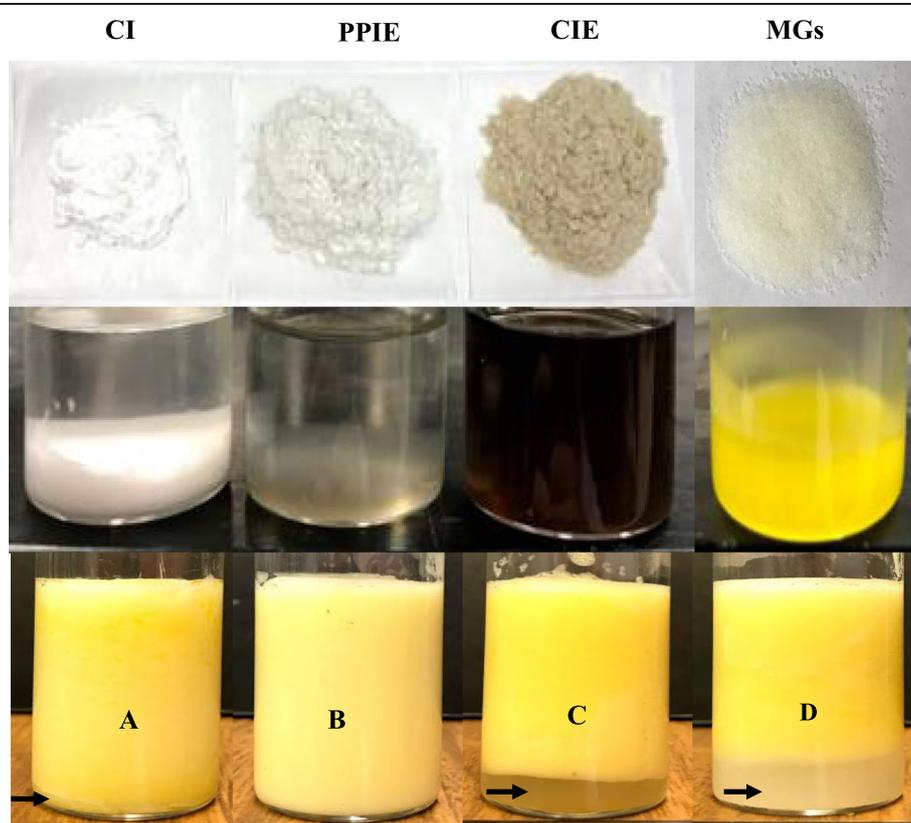


Fig. 3 The powders, gels and emulsion gels formed by inulin and/or MGs. The first row from the top shows the pictures of CI (commercial inulin from chicory root), PPIE (primary purified inulin extract from JA), CIE (crude inulin extract from JA) and MGs (monoglycerides) powders; The middle row shows the pictures of gels formed by corresponding powders solution through the heating-cooling process. The bottom row shows the pictures of emulsion gels; **a** contains CI; **b** contains PPIE; **c** contains CIE; **d** is control (no inulin). Black arrow shows separations in emulsion gel **a, c and d**

displaying a typical sol-gel transition (Kim et al. 2001). In the middle row of Fig. 3, PPIE and CIE gels were darker than CI, possibly due to remaining inherent color or products of enzymatic browning reactions in JA tuber (Tchoné et al. 2005). As mentioned earlier, the degree of gel formation was expressed as volumetric gel index (VGI). The VGI of CI gel (26%) was higher than PPIE gel (22%) and CIE gel (18%), which was likely due to different DPs and inulin content. Monoglycerides structured oil and formed gel-like material (oleogel). In this process, oil transferred into a thermo-reversible three-dimensional gel network in the presence of monoglycerides (Mert & Demirkesen 2016).

Inulin emulsion gel

Emulsion gel can be regarded as a gelled emulsion. Emulsion gel with PPIE (B) had better appearance than the other three. Emulsion gel with CI (A) had slight separation, whereas those with CIE (C) and control (D) presented apparent separations. PPIE improved the homogeneity of emulsion gel with no syneresis, which might be due to its wider DP distribution (inulin peaks a-d in HPLC).

Microstructure

PLM

Inulin gel and oleogel Microscopic images of inulin (CI, PPIE and CIE) gels and oleogel were shown in Fig. 4. CI

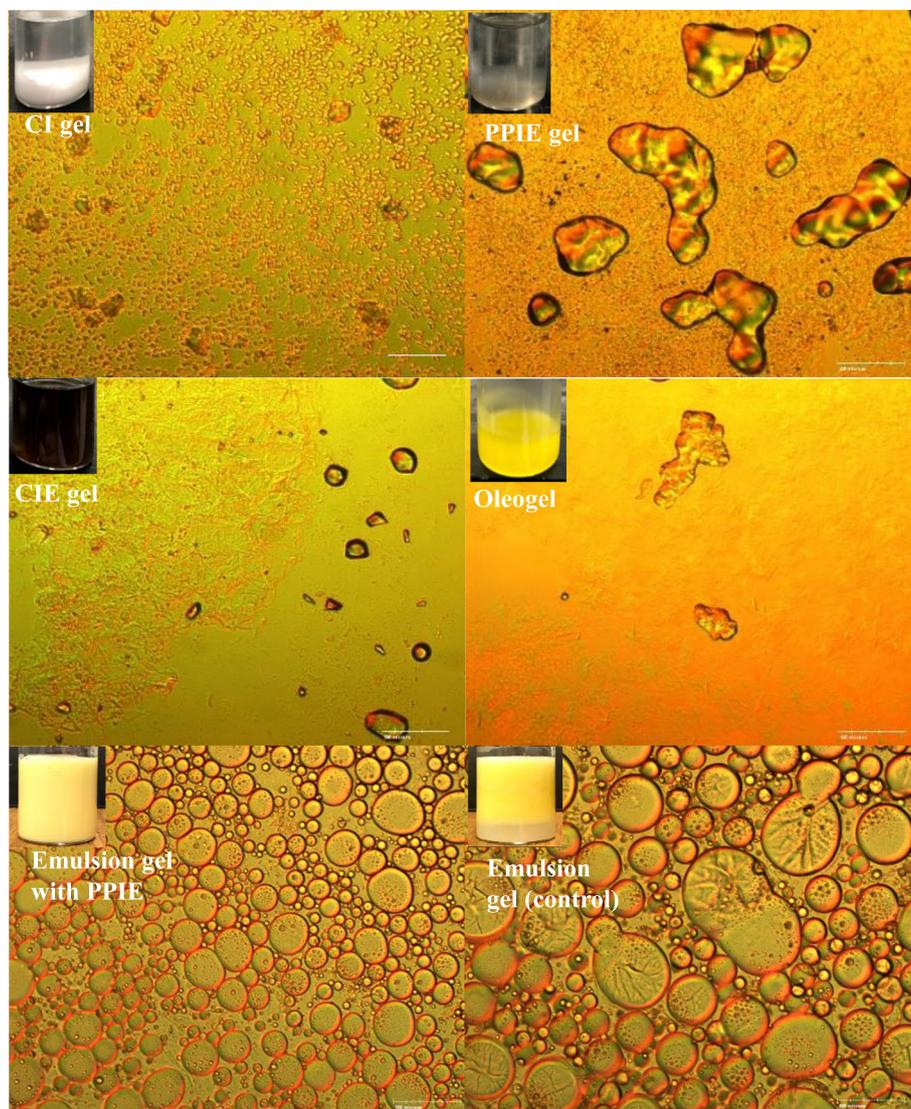


Fig. 4 Polarized light microscope images of gels and emulsion gels. The first two rows from the top indicate inulin gels and/or oleogel; the third row shows emulsion gel with 1% PPIE and without PPIE (control). CI: commercial inulin from chicory root; PPIE: primary purified inulin extract from JA; CIE: crude inulin extract from JA; MGs: monoglycerides; oleogel is formed by MGs; magnification: 200x; scale bar =100 μ m

gel had homogeneous crystals with approximate 10 μm particle size which was two times larger than the small crystals (5 μm) in PPIE gel. Besides small crystals, PPIE gel also consisted of large bulky crystals with 20–200 μm particle size. It was possibly due to inulin (PPIE) with different chain lengths that dispersed and packed efficiently in spatial arrangement and connected to become large crystals. Interestingly, CIE gel had large pieces of crystals similar to that in oleogel. It was likely attributed to the connection of inulin with some other compounds, such as peptides remaining in CIE (Rubel et al. 2018).

Emulsion gel Microstructures of emulsion gel with 1% PPIE and control (without 1% PPIE) were shown in the third row of Fig. 4. Full and round droplets in emulsion gel with PPIE were smaller (average size 40 μm) and more homogeneous than the droplets that partially crumpled in control (average size 60 μm). This phenomenon indicated that addition of 1% PPIE could prevent emulsion from syneresis and made it more homogenous, which also corresponded with the observation on a macro level: emulsion gel with PPIE had better appearance. It could be due to the excellent water-binding ability of inulin (Fadaei et al. 2012).

Cryo-SEM

The morphological characteristics of emulsion gels with 1% PPIE and control (without 1% PPIE) were also observed by cryo-SEM and shown in Fig. 5. Emulsion gel incorporating PPIE displayed a porous network with smaller pore sizes, which probably contributed to its creamy and smooth texture, whereas the control exhibited an uneven surface with large pore sizes. When emulsion gels were observed by cryo-SEM, temperature and pressure were –65 $^{\circ}\text{C}$ and 18–30 Pa, respectively. Water underwent partially frozen and sublimated according to the typical

pressure-temperature phase diagram of water. In this case, it was assumed that the remaining matrix was mainly formed by inulin, monoglyceride, Tween 80, as well as partial fat and ice. Incorporation of PPIE inhibited ice from forming large crystals, which could have potential applications on ice cream (Aleong et al. 2008).

Antioxidant activity

Conjugated dienes

Liposome model *Different inulin samples with the same concentration*

Antioxidant properties of CIE, PPIE and CI were evaluated by measuring their capacity to inhibit or delay linoleic acid oxidation, which was detected kinetically by increased absorbance of products (conjugated dienes hydroperoxides) at 234 nm (Vieira & Regitano-D'arce 1998), and results were presented in Fig. 6a.

CIE had the highest antioxidant activity amongst all inulin samples and was similar to trolox as there was no significant difference ($P < 0.05$). CIE started to differentiate from the control at ~50 min, and its absorbance was lower than control throughout the tested time. Whereas PPIE had a similar but weaker antioxidant activity compared with CIE as statistical analysis showed no significant differences, but PPIE had significantly lower activity than trolox (Fig. 6a). This result was parallel to total phenolic content (TPC) results: CIE had approximately 7 times as many TPC values as PPIE. Antioxidant activity of CIE and PPIE might be from remaining phenolic compounds (Tchoné et al. 2005) or peptides (Bhagia et al. 2018). Besides phenolics that remained in CIE and PPIE, another possible mechanism could be due to the interactions of CIE with other compounds, acting as physical barriers that encapsulated linoleic acid (de Barros Fernandes et al. 2016). Hinchá et al. (2000) revealed inulin had a capacity of stabilizing liposome in combination with glucose (Hinchá et al. 2000).

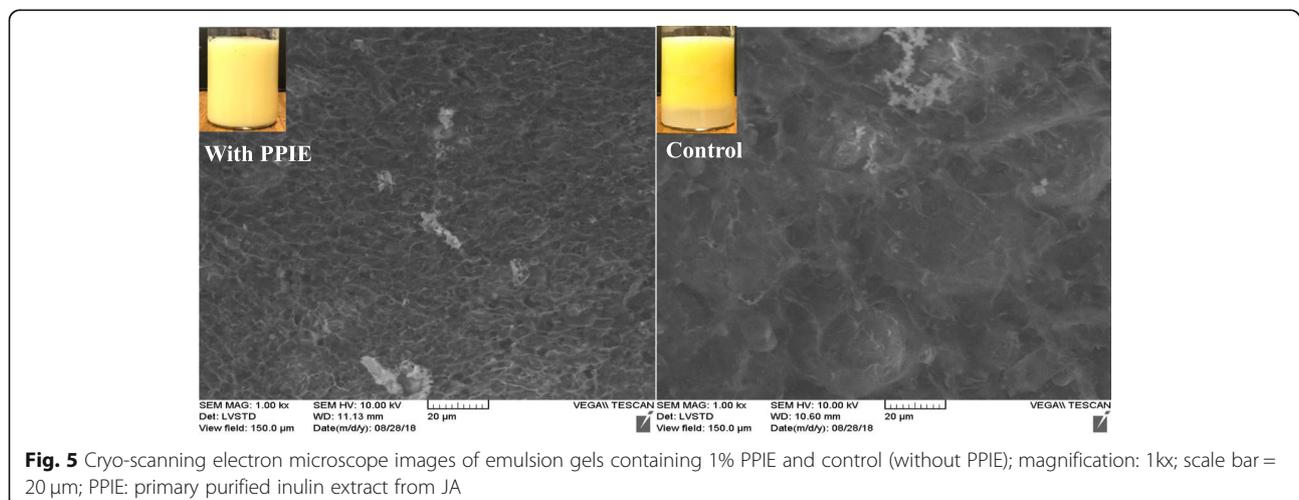


Fig. 5 Cryo-scanning electron microscope images of emulsion gels containing 1% PPIE and control (without PPIE); magnification: 1kx; scale bar = 20 μm ; PPIE: primary purified inulin extract from JA

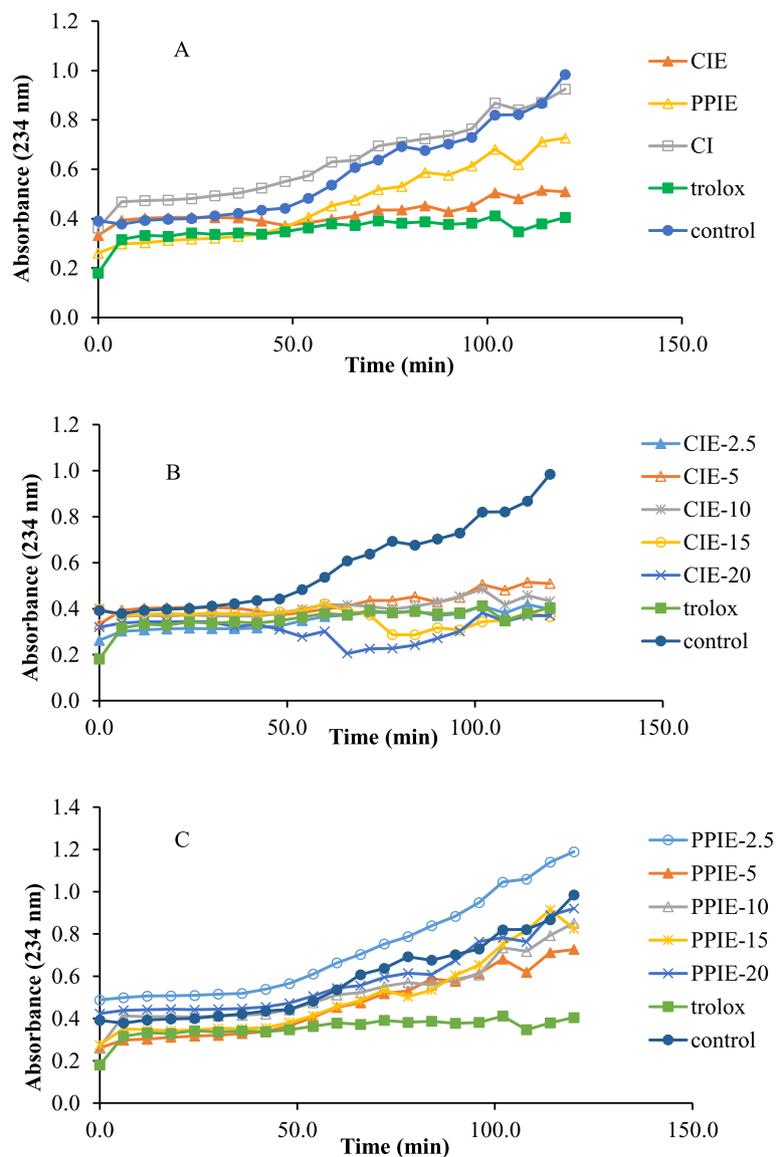


Fig. 6 Conjugated dienes formation from lipid oxidation in aqueous system influenced by adding inulin. **a** CIE (crude inulin extract from JA), PPIE (primary purified inulin extract from JA) and CI (commercial inulin from chicory root) solutions with the same concentration; **b** Different concentrations of CIE; **c** Different concentrations of PPIE. Each data point represents mean ($n = 3$). One-way ANCOVA followed by post-hoc Tukey's test is used to find significant differences ($\alpha = 0.05$). Results are different when $P < 0.05$

Trolox curve was almost stable and increased slightly during 120 min run. For blank control (PBS), it increased dramatically after 50 min, indicating that lots of conjugated dienes were produced. Whereas this curve kept stable during the first 50 min, which was likely because linoleic acid was encapsulated with lecithin (liposome model) and protected from oxidation at the beginning of reaction.

In summary, CIE had stronger antioxidant activity than other tested inulin samples, and this result was in agreement with the TPC results.

CIE and PPIE-different concentrations

It is well-known that activity is generally dose-dependent (Karadag et al. 2009). To investigate the effect of concentration, CIE samples with five different concentrations (in between 2.5–20 mg/mL) were tested in the same assay, and results were shown in Fig. 6b. All tested CIE were significantly different from blank control, but similar with trolox (Fig. 6b). It indicated that CIE had antioxidant activity in the tested concentration range.

Similarly, different concentrations of PPIE were also investigated. Five different concentrations (in between 2.5–

20 mg/mL) were run in the same assay, and results were shown in Fig. 6c. PPIE with 5 mg/mL showed antioxidant activity, and it was the only treatment concentration that was significantly ($P < 0.05$) different than control.

Emulsion gel model The above measurements were based on the liposome model in which linoleic acid was dispersed in aqueous buffer solution stabilized by soy lecithin, and inulin samples were dissolved in buffer solution and added to the reaction system separately. In the following experiment, emulsion gel was used in which oil and inulin samples were incorporated, and the whole emulsion gel was dispersed in buffer solution.

Antioxidant assessment of emulsion gels (PPIE, CIE, CI and control) were performed by the conjugated dienes method (the same method as liposome model), and the result was presented in Fig. 7. In order to eliminate spectral interference by emulsion gel itself, absorbances at 22 h were subtracted since emulsion gels had the lowest absorbances at that point. For all samples, absorbances began to increase at 22 h, indicating that oxidative reactions started at this time and more conjugated dienes hydroperoxides accumulated. Compared with Fig. 6a in which oxidation started at ~ 50 min, emulsion gels delay lipid oxidation much longer (oxidation started at ~ 22 h) than the liposome model. In addition, emulsion gel with PPIE had lower absorbance than control after 94 h, meaning that it slowed down the oxidation at that time. It was possibly related to the increased stability and homogeneity of emulsion gel with PPIE (Fig. 3).

TPC

TPC values of PPIE and CIE were expressed as milligram gallic acid equivalency per gram of sample (mg

GAE/g sample). The CIE sample had a higher TPC value (4.96 ± 0.01) than PPIE (0.72 ± 0.03), and commercial inulin had no TPC data to present, which was parallel to the results of antioxidant activity. Our result was close to the study of Petkova et al. (2014) in which TPC value was between 4 and 10 mg GAE/g dry weight in JA tuber water extraction (Petkova et al. 2014) but lower than the work of Yuan et al. (2012) in which TPC value was 22.40 ± 0.63 mg GAE/g dry weight (Yuan et al. 2012).

Rancimat

Accelerated oxidative stability of emulsion gels (containing CI, PPIE, or CIE) were tested by Rancimat, and results were displayed in Fig. 8. Emulsion gel containing CI had longer induction time (14.83 ± 0.69 h) than that containing PPIE (5.19 ± 0.64 h) or CIE (2.92 ± 0.21 h) ($P < 0.05$). It is probably because of complex components (Bhagia et al. 2018) in PPIE and CIE. Some impurities inside were possibly oxidized very fast under the accelerated oxidation condition, which produced more volatile compounds and increased conductivity. Therefore, emulsion gel containing inulin with higher purity had longer induction time. Rancimat result was in agreement with the volumetric gel index result.

Conclusion

This work examined the formulation and lipid oxidative stability of emulsion filled gels developed by incorporating Jerusalem artichoke inulin. HPLC results showed that the chromatogram of primary purified inulin extract (PPIE) had wider distributed peaks than commercial inulin (CI) and secondary purified inulin extract (SPIE). Physical investigation showed that addition of 1% PPIE

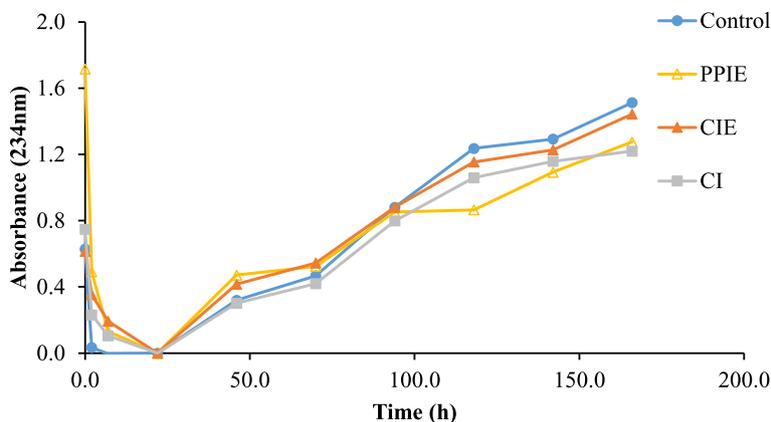


Fig. 7 Conjugated dienes formation from lipid oxidation in emulsion gels influenced by adding inulin. Emulsion gel containing PPIE (primary purified inulin extract from JA); CI (commercial inulin from chicory root); CIE (crude inulin extract from JA) and control (without inulin). Data points represent means ($n = 3$). One-way ANCOVA followed by post-hoc Tukey's test is used to find significant differences ($\alpha = 0.05$). Results are different when $P < 0.05$

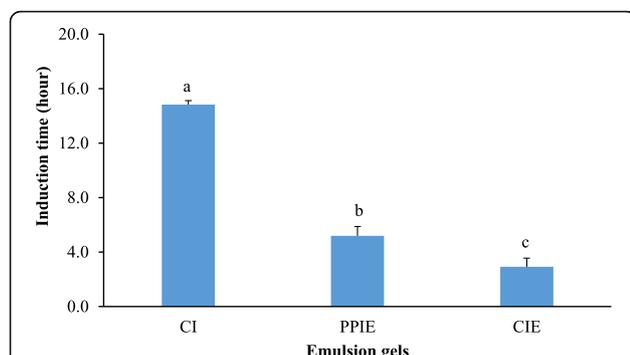


Fig. 8 Induction times of emulsion gels containing CI, PPIE, or CIE tested by Rancimat. PPIE: primary purified inulin extract from JA; CIE: crude inulin extract from JA; CI: commercial inulin from chicory root. Results represent mean ($n = 2$), and standard deviation value is used as error bar. Values with different superscript letters are significantly different ($p < 0.05$)

improved the appearance and stability of emulsion gel, which characterized by smaller droplets size (average $40 \mu\text{m}$) than control (average size $60 \mu\text{m}$). Rancimat test indicated that emulsion gel with high purity inulin (CI) had longer induction time than those with PPIE and CIE, which was in agreement with volumetric gel index results. Most importantly, CIE had antioxidant ability against linoleic acid oxidation using liposome model by delaying or inhibiting the production of conjugated dienes with a tested range of $2.5\text{--}20 \text{ mg/mL}$ (final concentration $0.04\text{--}0.33 \text{ mg/mL}$). It might be attributed to remaining phenolics as the TPC assay showed that CIE had high total phenolic content. In the future study, applications and characterization of emulsion gel with JA inulin (PPIE) used in food system as a potential fat replacement need to be investigated.

Abbreviations

CI: Commercial inulin from chicory root; CIE: Crude inulin extract; DP: Degree of polymerization; GAE: Gallic acid equivalency; HPLC: High performance liquid chromatography; JA: Jerusalem artichoke; MGs: Monoglycerides; MS: Mass spectrometry; PBS: Phosphate buffer solution; PLM: Polarized light microscope; PPIE: Primary purified inulin extract; RI: Refractive index; SEM: Scanning electron microscope; SPIE: Secondary purified inulin extract; TPC: Total phenolic content; VGI: Volumetric gel index; W/O: Water in oil

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Authors' contributions

FL carried out this study and drafted the manuscript. AG participated in modification and discussion of the manuscript. FH designed the study and modified the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

All data supporting this study are included in this article. Further details are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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References

- Aleong, J. M., Frochot, S., & Goff, H. D. (2008). Ice recrystallization inhibition in ice cream by propylene glycol monostearate. *Journal of Food Science*, 73(9), E463–E468. <https://doi.org/10.1111/j.1750-3841.2008.00954.x>.
- Bazina, N., & He, J. (2018). Analysis of fatty acid profiles of free fatty acids generated in deep-frying process. *Journal of Food Science and Technology*, 55(8), 3085–3092. <https://doi.org/10.1007/s13197-018-3232-9>.
- Beirão-da-costa, M. L., Januario, M. I. N., Simão, F. M. S., & Leitão, A. E. B. (2005). Characterisation of inulin from chicory and salsify cultivated in Portugal. *Alimentos E Nutrição Araraquara*, 16(3), 221–225.
- Bhagia, S., Ferreira, J. F. S., Kothari, N., Nunez, A., Liu, X., da Silva Dias, N., et al. (2018). Sugar yield and composition of tubers from Jerusalem Artichoke (*Helianthus tuberosus*) irrigated with saline waters. *Biotechnology and Bioengineering*, 115(6), 1475–1484. <https://doi.org/10.1002/bit.26582>.
- Böhm, A., Kaiser, I., Trebstein, A., & Henle, T. (2005). Heat-induced degradation of inulin. *European Food Research and Technology*. <https://doi.org/10.1007/s00217-004-1098-8>.
- Bolouri-Moghaddam, M. R., Le Roy, K., Xiang, L., Rolland, F., & Van Den Ende, W. (2010). Sugar signalling and antioxidant network connections in plant cells. *FEBS Journal*, 277(9), 2022–2037. <https://doi.org/10.1111/j.1742-4658.2010.07633.x>.
- Dave, P. (2012). *Rheological properties of low-fat processed cheese spread made with inulin as a fat replacer*. (Doctoral dissertation, University of Wisconsin–Stout), Menomonie, WI 54751, USA.
- de Barros Fernandes, R. V., Botrel, D. A., Silva, E. K., Borges, S. V., de Oliveira, C. R., Yoshida, M. I., et al. (2016). Cashew gum and inulin: new alternative for ginger essential oil microencapsulation. *Carbohydrate Polymers*, 153, 133–142. <https://doi.org/10.1016/j.carbpol.2016.07.096>.
- Fadaei, V., Poursharif, K., Daneshi, M., & Honarvar, M. (2012). Chemical characteristics of low-fat wheyless cream cheese containing inulin as fat replacer. *European Journal of Experimental Biology*, 2(3), 690–694.
- Farhoosh, R., Niazmand, R., Rezaei, M., & Sarabi, M. (2008). Kinetic parameter determination of vegetable oil oxidation under Rancimat test conditions. *European Journal of Lipid Science and Technology*, 110, 587–592.
- Gunenc, A., HadiNezhad, M., Farah, I., Hashem, A., & Hosseini, F. (2015). Impact of supercritical CO₂ and traditional solvent extraction systems on the extractability of alkylresorcinols, phenolic profile and their antioxidant activity in wheat bran. *Journal of Functional Foods*, 12, 109–119. <https://doi.org/10.1016/j.jff.2014.10.024>.
- Hamilton, J., Knox, B., Hill, D., & Parr, H. (2000). Reduced fat products – Consumer perceptions and preferences. *British Food Journal*, 102(7), 494–506. <https://doi.org/10.1108/00070700010336454>.
- Hartel, R. d. W., von Elbe, J. H., & Hofberger, R. (2018). Fats, oils and emulsifiers. In *Confectionery science and technology* (pp. 85–124). Cham: Springer. https://doi.org/10.1007/978-3-319-61742-8_4.
- Hincha, D. K., Hellwege, E. M., Heyer, A. G., & Crowe, J. H. (2000). Plant fructans stabilize phosphatidylcholine liposomes during freeze-drying. *European Journal of Biochemistry*, 267(2), 535–540. <https://doi.org/10.1046/j.1432-1327.2000.01028.x>.
- Hongxin, W. (2008). Study on the decoloration of chicory inulin by active carbon method. *Journal of Anhui Agricultural Sciences*, 36(7), 2626–2628.
- Hosseini, F. S., Muir, A. D., Westcott, N. D., & Krol, E. S. (2006). Antioxidant capacity of flaxseed lignans in two model systems. *Journal of the American Oil Chemists' Society*, 83(10), 835–840.
- Huang, G., Chen, Y., & Wang, X. (2011). Extraction and deproteinization of pumpkin polysaccharide. *International Journal of Food Sciences and Nutrition*, 62(6), 568–571. <https://doi.org/10.3109/09637486.2011.560566>.
- Karadag, A., Ozcelik, B., & Saner, S. (2009). Review of methods to determine antioxidant capacities. *Food Analytical Methods*, 2(1), 41–60. <https://doi.org/10.1007/s12161-008-9067-7>.

- Kim, Y., Faqih, M. N., & Wang, S. S. (2001). Factors affecting gel formation of inulin. *Carbohydrate Polymers*, 46(2), 135–145. [https://doi.org/10.1016/S0144-8617\(00\)00296-4](https://doi.org/10.1016/S0144-8617(00)00296-4).
- Ku, Y., Jansen, O., Oles, C. J., Lazar, E. Z., & Rader, J. I. (2003). Precipitation of inulins and oligofructose by ethanol and other solvents. *Food Chemistry*, 81, 125–132.
- Li, W., Zhang, J., Yu, C., Li, Q., Dong, F., Wang, G., et al. (2015). Extraction, degree of polymerization determination and prebiotic effect evaluation of inulin from Jerusalem artichoke. *Carbohydrate Polymers*, 121, 315–319. <https://doi.org/10.1016/j.carbpol.2014.12.055>.
- Liégeois, C., Lermusieau, G., & Collin, S. (2000). Measuring antioxidant efficiency of wort, malt, and hops against the 2,2'-azobis (2-amidinopropane) dihydrochloride-induced oxidation of an aqueous dispersion of linoleic acid. *Journal of Agricultural and Food Chemistry*, 48(4), 1129–1134. <https://doi.org/10.1021/jf9911242>.
- Liu, R., Wang, L., Liu, Y., Wu, T., & Zhang, M. (2018). Fabricating soy protein hydrolysate/xanthan gum as fat replacer in ice cream by combined enzymatic and heat-shearing treatment. *Food Hydrocolloids*, 81, 39–47. <https://doi.org/10.1016/j.foodhyd.2018.01.031>.
- Mert, B., & Demirkesen, I. (2016). Reducing saturated fat with oleogel / shortening blends in a baked product. *Food Chemistry*, 199, 809–816. <https://doi.org/10.1016/j.foodchem.2015.12.087>.
- Mozaffarian, D., Katan, M. B., Ascherio, A., Stampfer, M. J., & Willett, W. C. (2006). Trans fatty acids and cardiovascular disease. *The New England Journal of Medicine*, 354(15), 1601–1613.
- Nishinari, K. (2009). Some thoughts on the definition of a gel. In *Gels: Structures, properties, and functions* (Vol. 136, pp. 87–94). Berlin: Springer.
- Oh, I. K., & Lee, S. (2018). Utilization of foam structured hydroxypropyl methylcellulose for oleogels and their application as a solid fat replacer in muffins. *Food Hydrocolloids*, 77, 796–802. <https://doi.org/10.1016/j.foodhyd.2017.11.022>.
- Ojijo, N. K. O., Neeman, I., Eger, S., & Shimoni, E. (2004). Effects of monoglyceride content, cooling rate and shear on the rheological properties of olive oil/ monoglyceride gel networks. *Journal of the Science of Food and Agriculture*, 84(12), 1585–1593. <https://doi.org/10.1002/jsfa.1831>.
- Petkova, N., Ivanov, I., Denev, P., & Pavlov, A. (2014). Bioactive substance and free radical scavenging activities of flour from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers—a comparative study. *Turkish Journal of Agricultural and Natural Sciences*, 1(2), 1773–1778.
- Pintado, T., Herrero, A. M., Jiménez-Colmenero, F., Cavalheiro, C. P., & Ruiz-Capillas, C. (2018). Chia and oat emulsion gels as new animal fat replacers and healthy bioactive sources in fresh sausage formulation. *Meat Science*, 135, 6–13. <https://doi.org/10.1016/j.meatsci.2017.08.004>.
- Rios, R. V., Garzón, R., Lannes, S. C. S., & Rosell, C. M. (2018). Use of succinyl chitosan as fat replacer on cake formulations. *LWT-Food Science and Technology*, 96, 260–265. <https://doi.org/10.1016/j.lwt.2018.05.041>.
- Rubel, I. A., Iraporda, C., Novosad, R., Cabrera, F. A., Genovese, D. B., & Manrique, G. D. (2018). Inulin rich carbohydrates extraction from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers and application of different drying methods. *Food Research International*, 103, 226–233. <https://doi.org/10.1016/j.foodres.2017.10.041>.
- Saengthongpinit, W., & Sajjaanantakul, T. (2005). Influence of harvest time and storage temperature on characteristics of inulin from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers. *Postharvest Biology and Technology*, 37(1), 93–100. <https://doi.org/10.1016/j.postharvbio.2005.03.004>.
- Shahidi, F., & Zhong, Y. (2005). Lipid oxidation: Measurement methods. In *Bailey's industrial oil and fat products*.
- Srinameb, B. O., Nuchadomrong, S., Jogloy, S., Patanothai, A., & Srijaranai, S. (2015). Preparation of inulin powder from Jerusalem artichoke (*Helianthus tuberosus* L.) tuber. *Plant Foods for Human Nutrition*, 70(2), 221–226. <https://doi.org/10.1007/s11130-015-0480-y>.
- Stoyanova, S., Geuns, J., Hideg, É., & Van Den Ende, W. (2011). The food additives inulin and stevioside counteract oxidative stress. *International Journal of Food Sciences and Nutrition*, 62(3), 207–214. <https://doi.org/10.3109/09637486.2010.523416>.
- Tavakoli, H. R., Naderi, M., Jafari, S. M., & Naeli, M. H. (2019). Postmarketing surveillance of the oxidative stability for cooking oils, frying oils, and vanaspati supplied in the retail market. *Food Science and Nutrition*, 7(4), 1455–1465. <https://doi.org/10.1002/fsn3.982>.
- Tchoné, M., Bärwald, G., & Meier, C. (2005). Polyphenoloxidases in Jerusalem artichoke (*Helianthus tuberosus* L.). *British Food Journal*, 107(9), 693–701. <https://doi.org/10.1108/00070700510615071>.
- Temkov, M., Petkova, N., Denev, P., & Krastanov, A. (2015). Characterization of inulin from *Helianthus tuberosus* L. obtained by different extraction methods—comparative study. *Scientific Works of University of Food Technologies*, 461–464. <https://doi.org/10.13140/RG.2.1.4093.7040>.
- Vieira, T. M., & Regitano-D'arce, M. A. (1998). Stability of oils heated by microwave: UV-spectrophotometric evaluation. *Ciência e Tecnologia de Alimentos*, 18(4), 433–437.
- Yuan, X., Gao, M., Xiao, H., Tan, C., & Du, Y. (2012). Free radical scavenging activities and bioactive substances of Jerusalem artichoke (*Helianthus tuberosus* L.) leaves. *Food Chemistry*, 133(1), 10–14. <https://doi.org/10.1016/j.foodchem.2011.09.071>.
- Zeng, T., Wu, Z. L., Zhu, J. Y., Yin, S. W., Tang, C. H., Wu, L. Y., & Yang, X. Q. (2017). Development of antioxidant pickering high internal phase emulsions (HIPes) stabilized by protein/polysaccharide hybrid particles as potential alternative for PHOs. *Food Chemistry*, 231, 122–130. <https://doi.org/10.1016/j.foodchem.2017.03.116>.

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