



Inulin rich carbohydrates extraction from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers and application of different drying methods



Irene A. Rubel^{a,*}, Carolina Iraporda^a, Rocio Novosad^a, Fernanda A. Cabrera^b, Diego B. Genovese^b, Guillermo D. Manrique^a

^a Departamento de Ingeniería Química, Facultad de Ingeniería, Universidad Nacional del Centro de la Provincia de Buenos Aires, Av. del Valle 5737, B7400JWI Olavarría, Argentina

^b PLAPIQUI (Universidad Nacional del Sur – CONICET). Camino La Carrindanga KM 7, B8000 Bahía Blanca, Argentina

ARTICLE INFO

Keywords:

Jerusalem artichoke tubers

Inulin

Prebiotic food ingredient

Freeze-drying

Spray-drying

ABSTRACT

In this study the operational extraction variables to obtain higher yields of inulin from Jerusalem artichoke tubers (JAT), as well as the optimal conditions to obtain a stable and dispersible powdered product by either spray or freeze drying, were studied. With this purpose, the powder yield, moisture content, water activity and flowability or products obtained by different experimental conditions were analyzed. Inulin rich carbohydrates (IRC) extraction was performed from lyophilized and ground tubers employing distilled hot water as solvent. It was proved that the solid:solvent ratio (S:S) was the critical variable in the extraction process, followed by temperature. Thus, the IRC extraction was optimal without ultrasound assistance, at 76 °C, employing a S:S of 1:16, during 90 min. In addition, the powder obtained by freeze-drying of the IRC extract showed advantages respect to powders obtained by spray-drying regarding the yield and considering that maltodextrin was not necessary as encapsulation agent. In another hand, spray drying process provided IRC powered materials with appropriate flow properties, and taking into account cost and time of production, this method should be considered as an alternative of freeze-drying.

1. Introduction

Jerusalem artichoke (*Helianthus tuberosus* L.) is used for many purposes such as human food, animal feed and the production of bioenergy and biochemical products (Li, Wang, Du, & Qin, 2013), and it is a crop that can be developed under different environment conditions (Rébora, 2008). Jerusalem artichoke tubers (JAT) accumulate high levels of inulin and fructo-oligosaccharides (FOS) (16–20% of fresh weight) (Van Loo et al., 1999). The chain length, composition, and polydispersity of inulin depend on plant species, harvesting conditions, and extraction and post-extraction processes (Ronkart et al., 2007; Rubel, Pérez, Genovese, & Manrique, 2014), and these parameters determine its biological and technological properties (Roberfroid, 2005). Inulin is a water soluble fiber that consists of a mixture of oligo- and/or polysaccharides of $\beta(2 \rightarrow 1)$ linked D-fructose units with a terminal glucose residue (Causey, Feirtag, Gallaher, Tungland, & Slavin, 2000) which are classified as fructans. Inulin-type fructans are considered as prebiotic

compounds, meaning that they can preferentially stimulate the growth and activity of a limited number of desirable bacteria in the colon, which in turn improve host health (Bach, Jensen, Kidmose, Sørensen, & Edelenbos, 2013; Causey et al., 2000; Gibson & Roberfroid, 1995).

JAT represents a valuable alternative source of prebiotic compounds (Marx, Nosberger, & Frehner, 1997; Rubel et al., 2014). Inulin is used as an ingredient in the development of functional foods given its technological properties and its beneficial effects on health (Rastall, 2010; Roberfroid, 1993). Many application of inulin from different sources in food products can be mentioned (Di Criscio et al., 2010; Quattrucci, Bruschi, Manzi, Aromolo, & Panfili, 1997; Sánchez et al., 2009). In particular, Rubel, Pérez, Manrique, and Genovese (2015) analyzed the enrichment of wheat bread with JAT inulin, finding that the dose applied had significant consequences on dough machinability and gassing power, while Khuenpet, Jittanit, Sirisansaneeyakul, and Srichamngong (2017) investigated the effect of fortifying commercial products with

Abbreviations: DOE, Design of experiments; FOS, Fructooligosaccharides; IGR, Inulin Orafiti®GR; IHP, Inulin Orafiti®HP; IRC, Inulin rich carbohydrates; JAT, Jerusalem artichoke tubers; M, Maltodextrin; RSM, Response surface methodology; S:S, Solid:solvent; SS:M, Soluble solids:maltodextrin; TLC, Thin layer chromatography; WAI, Water absorption index; WSI, Water solubility index

* Corresponding author.

E-mail addresses: irubel@fio.unicen.edu.ar (I.A. Rubel), fcabrera@plapiqui.edu.ar (F.A. Cabrera), dgenovese@plapiqui.edu.ar (D.B. Genovese), gmanrique@fio.unicen.edu.ar (G.D. Manrique).

<http://dx.doi.org/10.1016/j.foodres.2017.10.041>

Received 1 June 2017; Received in revised form 30 August 2017; Accepted 22 October 2017

Available online 23 October 2017

0963-9969/ © 2017 Elsevier Ltd. All rights reserved.

inulin powder obtained from JAT, and revealed that the improvement in either product formula or JAT inulin quality especially in matter of color must be carried out before launching these inulin fortified products to the market.

Different methods for obtaining several types of prebiotic oligosaccharides have been proposed, including: isolation and purification from plant sources, microbiological production or enzymatic synthesis, and enzymatic degradation of polysaccharides (Crittenden & Playne, 1996; Gulewicz et al., 2003). Although inulin serves as storage polymers in many members of the Compositae such as *Cichorium intybus* (chicory), *Inula helenium* (elecampane), *Taraxacum officinalis* (dandelion), and *Helianthus tuberosus* L. (Jerusalem artichoke) (Waterhouse & Chatterton, 1993). At present, both inulin and oligofructose are mainly obtained as commercial food ingredients from dahlia and chicory (Franck, 2002), however the endeavor to improve the extraction quality of other crops does not bring to a stop. So, it seems to be promising the study of alternative plant sources for obtaining these prebiotic ingredients that could stimulate the exploitation of crops with high agronomic potential.

The extraction is the first key step for the inulin production from JAT (Franck, 2002). Many different sources and conditions for the extraction process of inulin have been reported in the literature. For instance, Lorenzo, Navia, and Neiditch (1999) applied boiling water extraction 10–15 min for inulin extraction from JAT, while Lingyun et al. (2007) found that a temperature of 76.65 °C during 20 min were the optimum process parameters predicted to obtain the highest yield for the same source material. Saengthongpinit and Sajjaanantakul (2005) applied distilled water at 80 °C for 1 h for inulin extraction from JAT and then they precipitated it by alcohol addition prior drying. Taneli, Park, Ramalho, Murr, and Fabbro (2008) extracted inulin from dry chicory roots applying a hot water diffusion procedure at an average temperature of 80 °C for 1 h with continuous stirring. The second step for inulin production consists in the obtaining of inulin powder suitable to be employed as an ingredient in the food industry. For the production of inulin powder various drying methods, including spray- and freeze-drying, could be used. Spray drying allows the transformation of a solution into a dried powder in a single operation, where usually maltodextrin as drying agent is used. Beyond being a rapid drying process, this technique is also inexpensive, its operation is simple and continuous and reduced time-to-market due to scale up benefits and better product quality, with no batch to batch variations. Nevertheless, this technology requires adequate adjustment of operating conditions as well as of the composition of feed solution that contains the active principles. Also, the drying temperature must be controlled considering that high temperatures in this process may lead to the obtaining of burned powders, as well as the low temperatures might produce unstable and sticky powders (Barbosa et al., 2015; Quek, Chok, & Swedlund, 2007). On the other hand, freeze-drying is a process in which the water is removed from a frozen solution by sublimation under reduced pressure giving rise to high quality dried products (Ratti, 2001). In this process, flavours, smells and nutritional content generally remain unchanged, however, it is expensive and time-consuming (Castro, Teixeira, & Kirby, 1997).

The aim of this study was to determine the operational variables to obtain higher yields of extraction of inulin rich carbohydrates from Jerusalem artichoke tubers, and to analyze the properties of the powdered products obtained either by spray- or freeze-drying, in order to assess its suitability to be used as ingredient in functional foods. With this purpose, the powder yield, moisture content, water activity and flowability of powdered samples were analyzed.

2. Materials and methods

2.1. Tuber handling

Jerusalem artichoke tubers (JAT), Bianca variety, were grown in

Córdoba, Argentina, were kindly provided by Nacional Institute of Agricultural Technology (INTA, Argentina) and harvested ten months after planting. Ten days after harvest, the tubers were washed and brushed manually to eliminate soil residues. In order to reduce superficial microorganisms, the tubers were soaked in a 0.038 M sodium hypochlorite solution for 30 min, and then they were dried on a surface exposed to a fan stream with an air velocity of 48 m/min at room temperature. Clean and dry tubers were packed in plastic woven bags and stored in a chamber at 4–5 °C and > 98% relative humidity until use. The tubers were cut into slices of 3 mm thickness with a domestic food processor (Moulinex, Fresh Express). Slices were frozen at – 20 °C and freeze-dried (LA-B3-C, Rificor, Argentina). The lyophilized material was stored in Ziploc® bags under nitrogen atmosphere, kept in a cool and dry place (15–25 °C, 40% RH) and utilized within a month. The chemical composition of JAT employed (carbohydrates, protein, lipid and ash content), was previously assessed and published (Rubel et al., 2014).

2.2. Inulin rich carbohydrates extraction

For the experimental design the function of MINITAB statistical software (Version 17, Minitab Inc., Minitab Software, USA) was employed. Response surface methodology (RSM) consists of a group of mathematical and statistical procedures that can be used to study relationships between one or more responses and a number of independent variables. RSM defines the effect of the independent variables, alone or in combination, on the process. Furthermore, the Advanced Design of Experiments (DOE) tool, which allowed determining the relevant factors of the process, was applied. A fractional factorial design (FFD) 2^k was employed, where k = 3 (temperature, solid:solvent ratio, ultrasound) with 2 levels for each factor: – 1 (low level) and 1 (high level), defining the yield of total solids as the response parameter. The FFD was employed to identify the most important extraction parameters and finally the central composite design to optimize the extraction process. The experimental design included three steps of study. The first step consisted in the analysis of 75 different conditions of extraction combining 3 selected factors. Factorial ANOVA analysis was applied to define the effect of each factor on the response, with a significance level $\alpha = 0.05$. The inulin rich carbohydrates (IRC) extraction from lyophilized and ground JAT using hot distilled water in a batch stirred system (Magnetic stirrer FBR, Decalab SRL, Argentina) at 200 rpm was optimized. The levels employed on the FFD (defined as low and high) were 70 and 85 °C for temperature, 1:8 and 1:20 for the S:S, and 0 and 30 min for indirect ultrasound assistance (42 Khz and 100 W, 8891-26 Cole-Parmer Ultrasonic bath, Cole Parmer Instruments, USA). For the indirect ultrasound pre-treatment the sample in a plastic tube with a diameter of 30 mm, was immersed in an ultrasound bath, the tube was shaken periodically with an orbital shaker and the liquid level inside the tube was about 1 cm below the liquid surface in the bath.

The extraction time was initially set as a constant (120 min) and then adjusted employing the optimal values of the other parameters analyzed. The working ranges of the extraction variables were loaded into the DOE and they were processed using the Box-Wilson composite central design, yielding 75 specific experiments with different combinations of working conditions, which were carried out. The corresponding yields were calculated as g of total solids/100 g tubers (dry weight) and introduced as a response to the system, allowing to predict the conditions to reach the highest yield. The RSM was employed to optimize multiple variables to predict the condition that produced the highest yield with a minimum number of experiments (Kong, He, Chen, & Chen, 2004; Shi, Xu, & Cen, 2006). The pH of the IRC extracts during the extraction process was kept natural, at 6.59 ± 0.15 . Once selected the temperature, the ultrasound assistance, the S:S, the kinetic of the process (different extraction times) as well as three independent extraction steps were also analyzed. The three independent extraction

steps were carried out consecutively under the same experimental conditions, using the solid material from the previous step and adding a new batch of fresh solvent.

2.3. Proximate analysis of IRC extracts

Moisture content of the IRC extracts was determined using an electronic moisture analyzer (Kern, DBS 60-3 N, Germany), protein content by the micro-Kjeldahl method, using an automatic digester and distillation units (B-316, Büchi, Suiza) (AOAC, 1990) with a factor of 6.25 for converting nitrogen to protein. Fats were extracted with *n*-hexane (b.p. 68–72 °C) in a Soxhlet apparatus following IUPAC Standard Method 1.122 (1992), ash content was evaluated by the technique AOCs Ba 5a-49 (1993), and total carbohydrates content was calculated by difference method. Proximate analysis was conducted in duplicate. Finally, soluble solids content was determined employing a refractometer (ABBE 1 T, ATAGO, Japan) and expressed as °Brix.

2.4. Thin layer chromatography

The IRC extracts were analyzed by thin layer chromatography (TLC). This assay was performed on Silica gel 60 plates (Merck, Germany) using butanol:isopropanol:water:acetic acid (7:5:4:2) as mobile phase (Lingyun et al., 2007). Carbohydrates were visualized as blue spots by spraying a solution of *p*-amino benzoic acid (7 g/L) and *o*-phosphoric acid (30 g/L) in methanol (Zweig & Sherma, 1978) and heating at 85 °C for about 10 min. Standards of fructose, sucrose, FOS; 1-kestose, nystose and inulin from dhalia (Sigma-Aldrich, USA) 0.1% w/v were employed. Inulin Orafit®GR (IGR) and Orafit®HP (IHP) (Beneo-Orafit, Belgium) kindly donated by Saporiti SA (Argentina), were also analyzed.

2.5. IRC extracts drying

The IRC extracts were dried either by spray- or freeze-drying using laboratory-scale equipment. For freeze-drying, each sample of 100 mL was initially frozen at –20 °C and then desiccated under vacuum for 2 days in a freeze dryer (FD-1A-50, Beijing Boyikang Instruments Co. Ltd., China).

The IRC extracts were atomized in a lab-scale spray dryer (Mini Spray Dryer B-290, BÜCHI, Flawil, Switzerland) equipped with a standard cyclone. A two-fluid nozzle with a cap-orifice diameter of 0.5 mm was used. The extracts were constantly stirred to keep the samples homogeneous and fed into the drying chamber through the nozzle by means of a peristaltic pump. The following conditions were used during the procedure: air inlet temperature (co-current flow) 140, 165, 170, 175 and 180 °C; drying air flow rate: 35 m³/h; liquid feed flow rate: 3–4 mL/min and atomization air flow rate: 670 L/h. These conditions were selected based on preliminary assays using other matrices (data not shown), considering that the outlet temperature must not exceed the degradation temperatures of the product. The process yield was estimated as the collected powder weight respect to the total solid content fed to the spray drier.

The powder recovery was calculated applying the following equation:

% powder recovery = (Solid weight of powder after drying/Total solid weight in feed material before drying) × 100. Each experiment was conducted in duplicate.

The IRC extracts, with a given content of soluble solids (SS), were mixed with maltodextrin (M) as drying adjuvant. For both drying process, different SS:M ratios (1:1.25, 1:1, 1:0.5, 1:0.25, 1:0) were analyzed.

2.6. Analysis of powders

2.6.1. Water activity

The water activity (a_w) of powders obtained was measured in a water activity meter (AquaLab, Dew Point Water Activity Meter 4TE, USA) at a constant temperature of 25 ± 0.1 °C.

2.6.2. Moisture content

The moisture content was determined using an electronic moisture analyzer (Kern, DBS 60-3N, Germany). The moisture content was measured immediately after the drying process. Approximately 1 g of powder was heated until the weight change was < 1 mg in 90 s.

2.6.3. Bulk density

Bulk density was determined by adding 2–4 g of powder to a 15 mL graduated tube and holding on a vibrator for 1 min. The bulk density was calculated by dividing the mass of powders by the volume occupied in the tube as described by Jirayucharoenak et al. (2015).

2.6.4. Water solubility index and water absorption index

Water solubility index (WSI) and water absorption index (WAI) were determined adding 2 g of powder sample in 25 mL of distilled water at 25 °C, left for 30 min in an orbital shaker at 240 rpm (M-23, Vicking, Argentina), and then centrifuged at 5000 g for 10 min (Sorvall Legend X1, Thermo Scientific, Germany). The supernatant was separated out whereas the remaining sediment was weighed. The WAI was calculated by the following formula:

$$WAI = \frac{S}{D_s} \times 100$$

where, *S* correspond to the sediment weight, and *D_s* correspond to the dry weight of the initial powder sample.

The WSI was calculated by the following formula:

$$WSI = \frac{D_{ss}}{D_s} \times 100$$

where, *D_{ss}* correspond to the amount of dry solids in the separated supernatant, and *D_s* correspond to the dry weight of the initial powder sample.

2.7. Statistical analysis

For the statistics analysis of data, InfoStat Software (Version 2011p, Córdoba, Argentina) was employed. Results were expressed as the mean value ± standard deviation of two or more independent experiments. Analysis of the variance (ANOVA) followed by the LSD Fisher's test was applied and significant differences were considered when *p*-value < 0.05.

3. Results

3.1. IRC extraction optimization

It has been described that the main factors that influence the yield of inulin extraction from JAT employing hot water as solvent include temperature, extraction time and solid to solvent ratio, (Apolinario et al., 2014; Khuenpet, Jittanit, et al., 2017; Khuenpet, Fukuoka, Jittanit, & Sirisansaneeyakul, 2017; Paseephol, Small, & Sherkat, 2007; Saengkanuk, Nuchadomrong, Jogloy, Patanothai, & Srijaranai, 2011). In this work, RSM was employed to optimize the IRC extraction conditions from JAT. Lyophilized JAT were employed in order to avoid changes in the carbohydrates composition and distribution during storage (Rubel et al., 2014; Saengthongpinit & Sajjaanantakul, 2005). The percentage of total solids obtained in the extracts was employed to calculate the yield for each set of experimental conditions and it was considered the dependent variable or response. This parameter varied

markedly for the different assays with values from 22 to 72 g total solids/100 g dry weight. The minimum total solids extraction yield was obtained with the highest solid:solvent ratio (S:S) 1:8 at 85 °C, even when the maximum time of ultrasound pretreatment (30 min) was applied. The data clearly showed that the decrease of the S:S ratio as well as the increase of the temperature resulted in significantly higher yields of total solids. In all the experiments pH value was measured at the beginning and at final with control purpose (6.59 ± 0.15). Moreover, according to the results of the FFD regression analysis, the ultrasound assistance did not significantly affect the total solid extraction ($p < 0.05$), which may be explained taking into account the pretreatments of drying and milling of tubers employed. In the same way, Lingyun et al. (2007) observed that indirect ultrasound-assistance did not affect the maximum extraction yield of inulin from fresh JAT, however they informed that the ultrasound-assistance would be useful for short times of extraction process. Moreover, Pourfarzad, Najafi, Khodaparast, and Khayyat (2015) showed that the indirect sonication extraction is a suitable method for fructan extraction from *Eremurus spectabilis* tubers. It was reported that direct sonication caused a decrease in the degree of polymerization of fructans extracted from different sources (Abbasi & Farzanmehr, 2009; Milani, Koocheki, & Golimovahed, 2011; Pourfarzad et al., 2015).

The response surface of Fig. 1, shows that IRC extraction yield reached a maximum value at 76 °C with a S:S 1:20 (w/v), and this model predicted a maximum yield of 73% at this point. The methodology allowed to determine the relative amounts by which the factors have to vary to attain a maximum response, considering also the environmental factor and an improvement observed in the drying process (data not shown), a S:S ratio of 1:16 was chosen as optimal, without a significant variation of extraction yields. The results obtained in the present work are lower than those reported by Lingyun et al. (2007) who informed significant influence of both temperature and S:S on the inulin extraction yield from JAT, and obtained a maximum value inulin extraction yield of 83.6%, at 76.65 °C for 20 min and a solid to solvent ratio of 1:10.56 (w/v). The kinetics of IRC extraction under optimized conditions (76 °C and S:S 1:16 w/v) was analyzed (Table 1). It was observed that with this experimental setting, there were not significant differences in the yield of total solids obtained among the times analyzed; however, at 90 min a significantly higher percentage of sugars in total solids was obtained. It was observed that the reducing sugars were constant during the different extraction times (data not shown), indicating that the IRC were not degraded during the process.

Then, the IRC extraction process in three independent steps (90 min each) was also analyzed. It was observed that no significant differences in the total solids yield were obtained when more than one independent step of extraction was applied, reaching values between 69 and 72% (w/w). This may be explained considering the presence of a weakened physical barrier in the material as a consequence of drying and milling processes of JAT employed, that allowed reaching the maximal yield of IRC extraction in the first step.

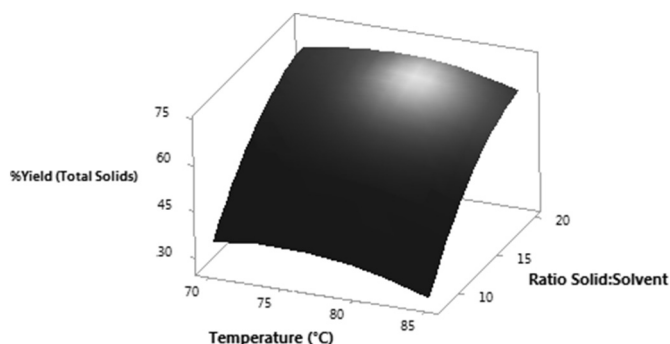


Fig. 1. Inulin rich carbohydrate extraction. Response surface plot of extraction yield (as percentage of total solids), as a function of temperature (°C) and solid:solvent ratio.

Table 1

Analysis of the one step (A) and three steps (B) extraction of IRC from JAT in selected conditions (Solvent: water, Temperature: 76 °C, ratio solid:solvent 1:16).

A		
Kinetic extraction		
Extraction time (min)	Total solids yield (%)	Sugar in total solids (%)
15	71.13 ± 0.88 ^a	66.59 ± 3.72 ^a
30	70.80 ± 0.68 ^a	56.54 ± 1.06 ^{ab}
45	72.98 ± 1.05 ^a	56.45 ± 8.79 ^{ab}
60	68.53 ± 0.68 ^a	53.09 ± 2.56 ^b
90	67.75 ± 1.29 ^a	81.26 ± 1.80 ^c
B		
Step extraction		
Steps (90 min)	Total solids yield (%)	
1	69.37 ± 4.07 ^a	
2	72.54 ± 0.14 ^a	
3	72.54 ± 0.14 ^a	

Different letters in the same column indicates that values are significantly different ($p < 0.05$).

Table 2

Proximal composition of the IRC extracts obtained at 76 °C, S:S ratio 1:16, and 90 min, expressed as % dry weight.

Component	Content
Total carbohydrates ^a (g/100 g _{d.w.})	85.6
Proteins (Nx6,25) (g/100 g _{d.w.})	9.99 ± 0.40
Lipids (g/100 g _{d.w.})	nd
Ash (g/100 g _{d.w.})	4.41 ± 0.66
Soluble solids (°Brix)	6–8

nd: not detectable; d.w.: dry weight.

^a Calculated by difference to 100%.

3.2. IRC extracts composition

Proximal composition of IRC extracts is described in Table 2. The data confirmed that the main components of the extracts were carbohydrates, as expected. The protein content in IRC extracts was 9.99 ± 0.40 g/100 g dry weight, followed by ash (4.41 ± 0.66 g/100 g dry weight), while fats were not detected. Similar results were obtained by Khuenpet, Jittanit, et al. (2017) who described in JAT inulin powder the presence of carbohydrates (84 g/100 g dry weight), proteins (7 g/100 g dry weight), and ash (5 g/100 g dry weight). TLC analysis of IRC extracts (Fig. 2), showed a polydisperse carbohydrate with different DP, and also sucrose was detected. In a previous work, simple sugars in the range 0.09–4.70 g/100 g dry weight were detected in IRC extracts from JAT (Rubel et al., 2014). Also in line with these observations, Khuenpet, Jittanit, et al. (2017) described that glucose, fructose and sucrose were present in JAT inulin powder in concentrations of 0–0.14; 0.15–2.38 and 0.01–0.91 g/100 g dry weight, respectively.

3.3. IRC extracts powder

The IRC extracts were dried either by spray- or freeze-drying. The spray-drying process was analyzed considering some process variables that affect the yield and properties of the powders obtained such as the inlet temperature, the pump feed rate as well as maltodextrin addition

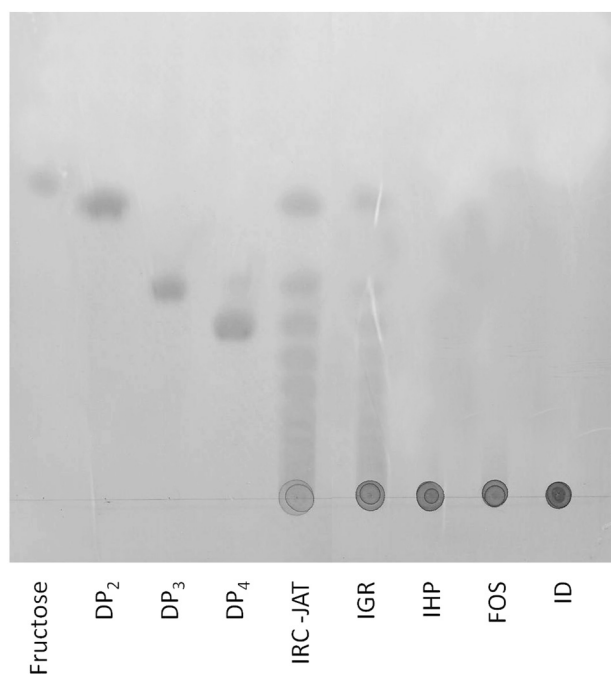


Fig. 2. Thin layer chromatography. DP_n: Degree of polymerization n. IRC-JAT: Inulin rich carbohydrates extracted from Jerusalem artichoke tubers. IGR: Commercial inulin (GR). IHP: Commercial inulin (HP). FOS: Fructooligosaccharides. ID: Inulin from Dhalia.

(Maury, Murphy, Kumar, Shi, & Lee, 2005). This method is the preferred route due its capacity to produce powders with precise specifications (i.e., moisture content, solubility and bulk density) in continuous operations (Souza & Oliveira, 2006). The drying temperature must be controlled considering that high temperatures in this process may lead to actives degradation, while low temperatures might produce unstable and sticky powders (Barbosa et al., 2015; Quek et al., 2007). The moisture content of the final powder influences its cohesiveness (Bhandari, 2013), as well as its storage stability, glass transition temperature and microbiological stability. It was observed that the air inlet temperature was the variable that mostly affected the properties of the powders obtained by spray drying, while variations of liquid feed flow rate (3–4 mL/min) did not improve substantially the powder properties and recovery. Jirayucharoensak et al. (2015) reported that inulin powder recovery from JAT obtained by spray-drying may be increased through an increase in the inlet temperature (190 °C), as well as by increasing the soluble solid content (°Brix) of the fed solution.

In this work it was not possible to completely recover IRC extract dry-material by spray-drying at inlet temperatures of 140 and 180 °C and additionally the powders obtained under these conditions had to be discarded due to its stickiness and/or its dark coloration. By selecting an inlet temperature of 175 °C, a low process yield (35%) was obtained

by spray-drying. Moreover, the resulting powder was unstable, it changed its appearance over time. This behavior was attributed to its high moisture content, high hygroscopicity and low glass transition temperature. It has been reported that the presence of low molecular weight sugars such as fructose and sucrose in the extracts may affect the drying process, since they tend to generate adherence of particles to the internal wall of the drying chamber, resulting in a low process yield and operating difficulties (Bhandari, Datta, & Howes, 1997; Bhandari & Howes, 2005; Hennigs, Kockel, & Langrish, 2001). Also, the heat exposure together with the reducing sugars and amino acids in the IRC extract may result in a browning as a result of the Maillard reactions and caramelization.

Considering the thermal lability of components present in the IRC extracts, the addition of encapsulating agents in the drying process may contribute to obtain powders with higher stability and increase yield. So, the use of maltodextrin as carrier material to IRC extracts before drying was studied. In addition to its suitability as food additive, various properties make maltodextrin as an excellent encapsulating agent in spray-drying processes. Maltodextrin has high aqueous solubility, soft taste, convenient price and availability as well as capability to reduce the stickiness and the tendency to crystallize of the resulting powders (Desobry, Netto, & Labuza, 1997).

It was observed that both, the increase in the inlet temperature and the addition of maltodextrin led to higher powder yields by spray-drying. IRC extract powder yield varied from 23 to 48% when total SS:M ratio was 1:0.25 and 1:1.25 at inlet temperature of 170 °C. The highest yield was $67.15 \pm 1.58\%$ and was reached employing a SS:M ratio 1:1, using a liquid feed flow rate of 3.5 mL/min and an inlet temperature of 175 °C. Conditions that led to an outlet air temperature of 109 °C. As an alternative to obtain maltodextrin-free powders, IRC extracts were dried by freeze-drying. During this drying process there is high preservation of components, nutrients and flavours; also freeze dried products can be easily rehydrated before use (Tsami, Krokida, & Drouzas, 1999). In this drying method the addition of maltodextrin did not significantly affect the powder yield obtained. As expected, in all cases the freeze-dried powder yields were significantly higher than those obtained by spray-drying. Also, as freeze drying is performed at low temperature, the degradation of inulin is negligible. However, as was mentioned previously, freeze-drying is an expensive, discontinuous and time consuming process. Despite the good results obtained for this drying method, it is important to consider the time and cost of production. The spray drying process is commonly employed in industry considering cost, versatility, time consume and the possibility to be a continuous process. Also, powders obtained by spray-drying with maltodextrin addition showed good powders yields, and a pleasant appearance. Other authors have shown that for the spray-drying process, the inlet temperature and the initial concentration are variables that may also influence the powder yield and the physical characteristics of the products obtained (Jirayucharoensak et al., 2015; Khuenpet, Fukuoka, et al., 2017). Then, these parameters may also be

Table 3
Physical characteristics of IRC extract powders obtained by spray- and freeze drying.

	Powder sample (total solids: maltodextrin ratio)	Bulk density (g/L)	WSI (%w/v)	WAI (g/g)	Moisture content (%)
Spray drying	(1:0.25)	321 ± 12 ^a	6.99 ± 0.29 ^a	0.278 ± 0.029 ^c	5.99 ± 0.06 ^{cd}
	(1:1) _{Tinlet = 175 °C}	465 ± 30 ^b	7.50 ± 0.09 ^b	0.095 ± 0.014 ^{ab}	5.64 ± 0.82 ^c
	(1:1) _{Tinlet = 165 °C}	489 ± 6 ^b	8.07 ± 0.20 ^c	0.058 ± 0.040 ^a	5.34 ± 0.06 ^{bc}
	(1:1.25)	577 ± 19 ^c	7.25 ± 0.06 ^{ab}	0.082 ± 0.014 ^{ab}	6.73 ± 0.17 ^d
Freeze drying	(1:0)	875 ± 39 ^f	7.28 ± 0.14 ^{ab}	0.720 ± 0.058 ^d	4.76 ± 0.18 ^{ab}
	(1:0.25)	762 ± 22 ^e	7.50 ± 0.15 ^b	0.100 ± 0.012 ^{ab}	6.21 ± 0.02 ^{cd}
	(1:0.5)	728 ± 1 ^e	7.39 ± 0.42 ^{ab}	0.110 ± 0.022 ^{ab}	5.62 ± 0.34 ^c
	(1:1)	731 ± 5 ^e	7.29 ± 0.15 ^{ab}	0.089 ± 0.002 ^{ab}	5.20 ± 0.36 ^{abc}
IGR		656 ± 7 ^d	7.59 ± 0.33 ^b	0.122 ± 0.037 ^b	4.22 ± 0.05 ^a

Different letters in the same column indicates that values are significantly different with $p < 0.05$. ANOVA followed by LSD Fisher test was applied.

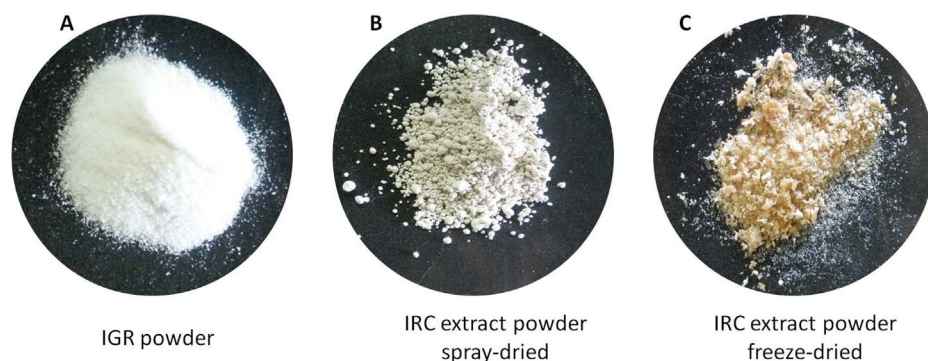


Fig. 3. Digital images of powders obtained by different drying methods. (A) IRC extract freeze-dried without maltodextrin (B) IRC extract spray-dried with maltodextrin 1:1 (inlet = 175 °C) (C) IGR.

considered to complement the analysis.

3.4. Powder physical characteristics

The analysis of physical parameters of the IRC extract powders is relevant to determine its applicability as ingredients in food industry. Physical parameters evaluated are shown in Table 3.

The moisture contents of powders obtained by both drying methods were in the range of 4.76–6.73 g/100 g. IRC extracts powders with 1:1 total solids:maltodextrin relation and without maltodextrin addition, obtained by freeze-drying, did not present significant differences in moisture content with respect to IGR powder (control). Freeze-dried powders obtained with different levels of maltodextrin did not present a clear tendency respect to their moisture contents. All the powders obtained by spray-drying presented moisture contents significantly higher than IGR powder. Jirayucharoensak et al. (2015) described that lower moisture content of powder may result in higher glass transition temperature and subsequently less hygroscopicity and stickiness of product. Given that a mean moisture content for different soluble fiber powders products has been reported as 6% (Yousif & Alghzawi, 2000), the powders obtained from IRC extracts in the present study can be considered stable during storage. Also, powders obtained can be considered safe regarding microbiological requirements ($A_w < 0.6$) (Fontana, 2008), since values were in the range 0.20 to 0.49.

The bulk density could be related to the flowability properties of the powder, being improved with higher bulk density values (Gallo, Llabot, Allemandi, Bucalá, & Piña, 2011). So, the bulk density of the powders obtained from IRC extracts were analyzed here as an indirect indication of the powder particle structure and flowability. The powders obtained by freeze-drying presented a significantly higher bulk density compared to the IGR and spray-dried powders, demonstrating that freeze-drying did not cause shrinkage or toughening of the material being dried. In another hand, the addition of maltodextrin in freeze-drying decreased the bulk density of the powders.

The powder particle structure may also influence its solubility (Caparino et al., 2012). The water solubility index (WSI) at 25 °C of the powders obtained by freeze-drying did not show significant differences with the corresponding to IGR powder, even when this material was obtained by freeze drying (Franck, 2002). The high solubility of freeze-dried powders could be due to the porous structure related to the rehydration capacity and that this process did not cause shrinkage of the products (Barbosa et al., 2015; Ratti, 2001). The WSI of powders obtained by spray drying (at 175 °C inlet temperature) with maltodextrin 1:1 and 1:1.25 did not differ significantly from that of IGR powder, whereas the powder obtained with maltodextrin 1:1 at lower inlet temperature (165 °C), presented significantly higher solubility. In addition, the powder obtained with the lower maltodextrin content (1:0.25) presented significantly lower WSI. Some authors (Cano-Chauca, Stringheta, Ramos, & Cal-Vidal, 2005; Caparino et al., 2012; Goula & Adamopoulos, 2008) found that maltodextrin addition improved powder hygroscopicity and solubility, whereas it deteriorated

slightly its moisture content and density, meanwhile there are also reports where the reduction in maltodextrin concentration improved the solubility (Abadio, Domingues, Borges, & Oliveira, 2004). Thus, results of the effect of maltodextrin addition on solubility are ambiguous and may be related to the properties of the original compound. Moreover, the addition of maltodextrin decreased WAI of powders obtained by both drying-methods.

Powders obtained by spray-drying with a total solids:maltodextrin ratio 1:1 or 1:1.25 with inlet temperature 165 and 175 °C did not present significant differences among them. The lowest maltodextrin addition conducted to significantly higher WAI value. The powder obtained by freeze-drying without addition of maltodextrin had a WAI value significantly higher than all the other powders. The variation in WAI could be due to differences in the degree of engagement of hydroxyl groups to form hydrogen and covalent bonds between carbohydrate chains. In concordance with the results obtained in this work, Ahmed, Akter, Chin, and Eun (2009) informed that WSI and WAI were positively and negatively correlated with maltodextrin addition, respectively in sweet potatoes flour. Jirayucharoensak et al. (2015) informed that drying temperature may also determine WSI as well as WAI, resulting in better WSI and WAI of the powder with the higher spray-drying temperature analyzed (190 °C). Analysis of the microstructure of powders would be necessary to complete and explain the differences in physical characteristics between them.

Images of the powders obtained and the commercial powder are presented in Fig. 3. The commercial powder has as a general characteristic a high degree of uniformity regarding to shape and better particle distribution (smooth and intact surfaces). The powders obtained by spray-drying shown small particle size with strong interaction among particles causing agglomeration, and presented a color similar to the commercial powder. The freeze-dried powder presented a pleasant appearance, adherence among particles, and slightly brown color.

4. Conclusion

In this work it was possible to optimize the extraction of IRC from lyophilized and milled JAT employing the response surface methodology. It was observed that the application of more than one extraction step as well as an ultrasound pretreatment in the process did not effectively enhance the IRC extraction yield. The IRC extract powder obtained by freeze-drying showed advantages regarding the yields obtained and the addition of maltodextrin, in this case, resulted unnecessary. Despite the good results obtained for this drying method, it is important to consider the time and cost of production. The spray drying process is commonly employed in industry considering cost, versatility, time consuming and the possibility to be a continuous process. Understanding how changes in these processing parameters affect flow properties and stability attributes in the powders produced is desirable for further industrial production of solid. So, it would be worthy to complement the study with more spray-drying conditions analyzing others inlet air temperature, airflow, pump speed, liquid flow rate,

atomization and solids concentration, and composition of the solution to be spray-dried, to optimize powders obtaining. This information could be used to promote the use of IRC extracts from JAT, and some other studies about the structure and microstructure would complement the analysis of the powders properties. The present study described a method of extraction and drying for obtaining IRC from JAT possible to be applied at industrial scale allowing the production of an ingredient feasible to be used in the formulation and development of functional foods.

Acknowledgements

IAR and CI are research fellow of CONICET (Argentina), RN is a Chemical Engineering advanced student and GM is professor of the UNCPBA and member of research career from CIC (Argentina). Authors are grateful for financial support from Proyecto de Vinculación Tecnológica 115/15 accredited by Ministerio de Educación y Deporte (Argentina).

References

- Abadio, F. D. B., Domingues, A. M., Borges, S. V., & Oliveira, V. M. (2004). Physical properties of powdered pineapple (*Ananas comosus*) juice-effect of malt dextrin concentration and atomization speed. *Journal of Food Engineering*, *64*(3), 285–287.
- Abbasi, S., & Farzaneh, H. (2009). Optimization of extracting conditions of inulin from Iranian artichoke with/without ultrasound using response surface methodology. *Journal of Water and Soil Science*, *13*(47), 423–435.
- Ahmed, M., Akter, M. S., Chin, K. B., & Eun, J. B. (2009). Effect of maltodextrin concentration and drying temperature on quality properties of purple sweet potato flour. *Food Science and Biotechnology*, *18*(6), 1487–1494.
- Apolinario, A. C., de Lima Damasceno, B. P. G., de Macêdo Beltrão, N. E., Pessoa, A., Converti, A., & da Silva, J. A. (2014). Inulin-type fructans: A review on different aspects of biochemical and pharmaceutical technology. *Carbohydrate Polymers*, *101*, 368–378.
- Bach, V., Jensen, S., Kidmose, U., Sørensen, J. N., & Edelenbos, M. (2013). The effect of culinary preparation on carbohydrate composition, texture and sensory quality of Jerusalem artichoke tubers (*Helianthus tuberosus* L.). *LWT - Food Science and Technology*, *54*(1), 165–170.
- Barbosa, J., Borges, S., Amorim, M., Pereira, M. J., Oliveira, A., Pintado, M. E., & Teixeira, P. (2015). Comparison of spray drying, freeze drying and convective hot air drying for the production of a probiotic orange powder. *Journal of Functional Foods*, *17*, 340–351.
- Bhandari, B. (2013). Introduction to food powders. *Handbook of food powders* (pp. 1–25). Oxford: Woodhead Publishing Limited.
- Bhandari, B., & Howes, T. (2005). Relating the stickiness property of foods undergoing drying and dried products to their surface energetics. *Drying Technology*, *23*(4), 781–797.
- Bhandari, B. R., Datta, N., & Howes, T. (1997). Problems associated with spray drying of sugar-rich foods. *Drying Technology*, *15*(2), 671–684.
- Cano-Chauca, M., Stringheta, P. C., Ramos, A. M., & Cal-Vidal, J. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Science & Emerging Technologies*, *6*(4), 420–428.
- Caparino, O. A., Tang, J., Nindo, C. I., Sablani, S. S., Powers, J. R., & Fellman, J. K. (2012). Effect of drying methods on the physical properties and microstructures of mango (Philippine 'Carabao' var.) powder. *Journal of Food Engineering*, *111*(1), 135–148.
- Castro, H. P., Teixeira, P. M., & Kirby, R. (1997). Evidence of membrane damage in lactobacillus bulgaricus following freeze-drying. *Journal of Applied Microbiology*, *82*(1), 87–94.
- Casey, J. L., Feirtag, J. M., Gallaher, D. D., Tunlgand, B. C., & Slavin, J. L. (2000). Effects of dietary inulin on serum lipids, blood glucose and the gastrointestinal environment in hypercholesterolemic men. *Nutrition Research*, *20*(2), 191–201.
- Crittenden, R. G., & Playne, M. (1996). Production, properties and applications of food-grade oligosaccharides. *Trends in Food Science & Technology*, *7*(11), 353–361.
- Desobry, S. A., Netto, F. M., & Labuza, T. P. (1997). Comparison of spray-drying, drum-drying and freeze-drying for β -carotene encapsulation and preservation. *Journal of Food Science*, *62*(6), 1158–1162.
- Di Criscio, T., Fratianni, A., Mignogna, R., Cinquanta, L., Coppola, R., Sorrentino, E., et al. (2010). Production of functional probiotic, prebiotic, and synbiotic ice creams. *Journal of Dairy Science*, *93*(10), 4555–4564.
- Fontana, A. J. (2008). Appendix D: Minimum water activity limits for growth of microorganisms. *Water activity in foods: Fundamentals and applications* (405–405).
- Franca, A. (2002). Technological functionality of inulin and oligofructose. *British Journal of Nutrition*, *87*(S2), S287–S291.
- Gallo, L., Llabot, J. M., Allemandi, D., Bucalá, V., & Piña, J. (2011). Influence of spray-drying operating conditions on *Rhamnus purshiana* (Cáscara sagrada) extract powder physical properties. *Powder Technology*, *208*(1), 205–214.
- Gibson, G. R., & Roberfroid, M. B. (1995). Dietary modulation of the human colonic microbiota: Introducing the concept of prebiotics. *The Journal of Nutrition*, *125*(6), 1401.
- Goula, A. M., & Adamopoulos, K. G. (2008). Effect of maltodextrin addition during spray drying of tomato pulp in dehumidified air: II. Powder properties. *Drying Technology*, *26*(6), 726–737.
- Gulewicz, P., Ciesiołka, D., Frias, J., Vidal-Valverde, C., Frejngel, S., Trojanowska, K., et al. (2003). Simple method of isolation and purification of galactosides from legumes. *Journal of Agricultural and Food Chemistry*, *48*, 3120–3123.
- Hennigs, C., Kockel, T. K., & Langrish, T. A. G. (2001). New measurements of the sticky behavior of skim milk powder. *Drying Technology*, *19*(3–4), 471–484.
- Jirayucharensak, R., Jittanit, W., Sirisansaneeyakul, S., Khuenpet, K., Watchrakorn, T., Pongpinyapibul, T., et al. (2015). Spray-drying for inulin powder production from Jerusalem artichoke tuber extract and product qualities. *Month*, *17*(3).
- Khuenpet, K., Fukuoka, M., Jittanit, W., & Sirisansaneeyakul, S. (2017). Spray drying of inulin component extracted from Jerusalem artichoke tuber powder using conventional and ohmic-ultrasonic heating for extraction process. *Journal of Food Engineering*, *194*, 67–78.
- Khuenpet, K., Jittanit, W., Sirisansaneeyakul, S., & Srichamng, W. (2017). Inulin powder production from Jerusalem artichoke (*Helianthus tuberosus* L.) tuber powder and its application to commercial food products. *Journal of Food Processing and Preservation*, *41*(4), 13097–13110.
- Kong, Q., He, G., Chen, Q., & Chen, F. (2004). Optimization of medium composition for cultivating *Clostridium butyricum* with response surface methodology. *Journal of Food Science*, *69*(7), 163–168.
- Laurenzo, K. S., Navia, J. L., & Neiditch, D. S. (1999). U.S. Patent No. 5,968,365. Washington, DC: U.S. Patent and Trademark Office.
- Li, L., Wang, Y., Du, Y., & Qin, S. (2013). Biorefinery products from the inulin-containing crop Jerusalem artichoke. *Biotechnology Letters*, *35*(4), 471–477.
- Lingyun, W., Jianhua, W., Xiaodong, Z., Da, T., Yalin, Y., Chenggang, C., et al. (2007). Studies on the extracting technical conditions of inulin from Jerusalem artichoke tubers. *Journal of Food Engineering*, *79*(3), 1087–1093.
- Marx, S. P., Nosberger, J., & Frehner, M. (1997). Seasonal variation of fructan- β -fructosidase (FEH) activity and characterization of a β -(2-1)-linkage specific FEH from tubers of Jerusalem artichoke (*Helianthus tuberosus*). *New Phytologist*, *135*(2), 267–277.
- Maury, M., Murphy, K., Kumar, S., Shi, L., & Lee, G. (2005). Effects of process variables on the powder yield of spray-dried trehalose on a laboratory spray-dryer. *European Journal of Pharmaceutics and Biopharmaceutics*, *59*(3), 565–573.
- Milani, E., Koocheki, A., & Golimovahed, Q. A. (2011). Extraction of inulin from Burdock root (*Arctium lappa*) using high intensity ultrasound. *International Journal of Food Science and Technology*, *46*(8), 1699–1704.
- Paseephol, T., Small, D., & Sherkat, F. (2007). Process optimisation for fractionating Jerusalem artichoke fructans with ethanol using response surface methodology. *Food Chemistry*, *104*(1), 73–80.
- Pourfarzad, A., Najafi, M. B. H., Khodaparast, M. H. H., & Khayyat, M. H. (2015). Characterization of fructan extracted from *Eremurus spectabilis* tubers: A comparative study on different technical conditions. *Journal of Food Science and Technology*, *52*(5), 2657–2667.
- Quattrucci, E., Bruschi, L., Manzi, P., Aromolo, R., & Panfilì, G. (1997). Nutritional evaluation of typical and reformulated Italian cheese. *Journal of the Science of Food and Agriculture*, *73*(1), 46–52.
- Quek, S. Y., Chok, N. K., & Swedlund, P. (2007). The physicochemical properties of spray-dried watermelon powders. *Chemical Engineering and Processing: Process Intensification*, *46*(5), 386–392.
- Rastall, R. A. (2010). Functional oligosaccharides: Application and manufacture. *Annual Review of Food Science and Technology*, *1*, 305–339.
- Ratti, C. (2001). Hot air and freeze-drying of high-value foods: A review. *Journal of Food Engineering*, *49*, 311–319.
- Rébora, C. (2008). Topinambur (*Helianthus tuberosus* L.): usos, cultivo y potencialidad en la región de Cuyo. *Avances en Horticultura*, *27*(63), 30–37.
- Roberfroid, M. B. (1993). Dietary fiber, inulin, and oligofructose: A review comparing their physiological effects. *Critical Reviews in Food Science and Nutrition*, *33*(2), 103–148.
- Roberfroid, M. B. (2005). Introducing inulin-type fructans. *British Journal of Nutrition*, *93*(S1), S13–S25.
- Ronkart, S. N., Blecker, C. S., Fourmanoir, H., Fougny, C., Deroanne, C., Van Herck, J. C., et al. (2007). Isolation and identification of inulooligosaccharides resulting from inulin hydrolysis. *Analytica Chimica Acta*, *604*(1), 81–87.
- Rubel, I. A., Pérez, E. E., Genovese, D. B., & Manrique, G. D. (2014). In vitro prebiotic activity of inulin-rich carbohydrates extracted from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers at different storage times by *Lactobacillus paracasei*. *Food Research International*, *62*, 59–65.
- Rubel, I. A., Pérez, E. E., Manrique, G. D., & Genovese, D. B. (2015). Fibre enrichment of wheat bread with Jerusalem artichoke inulin: Effect on dough rheology and bread quality. *Food Structure*, *3*, 21–29.
- Saengkanuk, A., Nuchadomrong, S., Jogy, S., Patanothai, A., & Srijaranai, S. (2011). A simplified spectrophotometric method for the determination of inulin in Jerusalem artichoke (*Helianthus tuberosus* L.) tubers. *European Food Research and Technology*, *233*(4), 609–616.
- 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.
- Sánchez, G. L., Garzón, M. J., Garzón, M. A., Giraldo, F. J., Millán, L., & Villada, M. E. (2009). Aprovechamiento del suero lácteo de una empresa del norte antioqueño mediante microorganismos eficientes. *Producción + Limpia*, *4*(2), 65–74.
- Shi, F., Xu, Z., & Cen, P. (2006). Optimization of γ -polyglutamic acid production by

- Bacillus subtilis* ZJU-7 using a surface-response methodology. *Biotechnology and Bioprocess Engineering*, 11(3), 251–257.
- Souza, C. R. F., & Oliveira, W. P. (2006). Powder properties and system behavior during spray drying of *Bauhinia forficata* link extract. *Drying Technology*, 24(6), 735–749.
- Toneli, J. T. C. L., Park, K. J., Ramalho, J. R. P., Murr, F. E. X., & Fabbro, I. M. D. (2008). Rheological characterization of chicory root (*Cichorium intybus* L.) inulin solution. *Brazilian Journal of Chemical Engineering*, 25(3), 461–471.
- Tsami, E., Krokida, M. K., & Drouzas, A. E. (1999). Effect of drying method on the sorption characteristics of model fruit powders. *Journal of Food Engineering*, 38, 381–392.
- Van Loo, J., Cummings, J., Delzenne, N., Englyst, H., Franck, A., Hopkins, M., et al. (1999). Functional food properties of non-digestible oligosaccharides: A consensus report from the ENDO project (DGXII AIRII-CT94-1095). *British Journal of Nutrition*, 81(2), 121–132.
- Waterhouse, A. L., & Chatterton, N. J. (1993). Glossary of fructan terms. *Science and technology of fructans* (pp. 1–7). .
- Yousif, A. K., & Alghzawi, H. M. (2000). Processing and characterization of carob powder. *Food Chemistry*, 69, 283–287.
- Zweig, G., & Sherma, J. (1978). Paper and thin-layer chromatography. *Analytical Chemistry*, 50(5), 50–65.