

Document downloaded from:

<http://hdl.handle.net/10251/183677>

This paper must be cited as:

Silva Espinoza, MA.; Ayed, C.; Camacho Vidal, MM.; Foster, T.; Martínez-Navarrete, N. (2021). Impact of Maltodextrin, Gum Arabic, Different Fibres and Starches on the Properties of Freeze-Dried Orange Puree Powder. *Food Biophysics*. 16(2):270-279.
<https://doi.org/10.1007/s11483-021-09667-x>



The final publication is available at

<https://doi.org/10.1007/s11483-021-09667-x>

Copyright Springer-Verlag

Additional Information

Impact of maltodextrin, gum Arabic, different fibres, and starches on the properties of freeze-dried orange puree powder

Marilú Andrea Silva-Espinoza^a, Charfedinne Ayed^b, María del Mar Camacho^a, Timothy Foster^b, Nuria Martínez-Navarrete^a

^aFood Technology Department, Food Investigation and Innovation Group, Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain.

^bDepartment of Food, Nutrition and Dietetics, School of Biosciences, University of Nottingham, Sutton Bonington Campus, Loughborough LE12 5RD, UK

Abstract

Fruits are essential components of a healthy diet contributing to the prevention of different diseases. Nevertheless, their consumption is limited among the population for reasons of convenience, among others. High-quality products are obtained by freeze-drying; however, dehydrated fruit presents a physical stability problem associated with the glass transition of its amorphous matrix. A common technique to prevent the rubbery state is the incorporation of biopolymers that contribute to increasing the glass transition temperature or that may exert a steric role. Nevertheless, the chemical composition and physical properties of these biopolymers may affect the quality of the dehydrated product which could compromise its use for specific applications. This work studies the impact of gum Arabic, bamboo fibre, maltodextrin, pea fibre, starch substituted with octenyl succinic groups and native corn starch added to an orange puree, on powder flowability and rehydration behaviour of the freeze-dried fruit powder. As regards the flowability, according to the angle of repose values (37-42°), all powder formulations were considered in the range of 'acceptable' powders. However, both samples containing maltodextrin showed significantly the lowest angle of repose value (37-38°), and therefore a better flowability. Samples containing gum Arabic, bamboo fibre and maltodextrin showed the lower wetting time (175-570 s), which is desired for rehydration, compared to those formulated with OSA (914-1887 s). Moreover, sample with gum Arabic showed the lowest viscosity after rehydration (0.199 Pas), desired to be consumed as a juice. According to the obtained results, if to get an orange puree powder with a good flowability is desired, the use of maltodextrin may be recommended. However, if rehydration is preferred, the use of gum Arabic is more recommended.

Keywords: powder flowability, rehydration properties, gum Arabic, maltodextrin, fibre.

1. Introduction

Fruits and vegetables are a rich source of vitamins, minerals, dietary fibre, and other beneficial non-nutrient bioactive compounds which seem to be able to prevent a wide range of pathologies, such as cancer, cardiovascular disease and degenerative diseases connected to aging processes (Tavarini, Degl'Innocenti, Remorini, Massai, & Guidi, 2008). According to FAO, a minimum of 400 g of fruits and vegetables per day should be taken to prevent chronic diseases and mitigate several micronutrient deficiencies. However, according to the last Food Consumption Report in Spain, the average intake of fruits and vegetables was 248 g and 233 g, respectively, in 2018 (MAPA, 2017). It is thought that the population does not consume fruit and vegetables for various reasons: cost, convenience, and taste, among others (FAO, 2003).

As a result of the composition, seasonality, and the geographic distribution of the fruits, transformation is required to extend its accessibility while preserving its nutritional benefits. Freeze-drying is a dehydration technique that operates at low temperatures and under vacuum, so oxidation reactions and the degradation of thermolabile compounds are minimized, many of those compounds responsible for the aromas and nutritional value of the fruits (Ratti, 2013). Since the freeze-drying is known for providing high added value products (Hammami et al., 1999), in this study this technology is proposed to obtain powdered fruit as a different way to offer fruit to the consumer. The powdered fruit may have a target audience among those people who are demanding in convenient foods with high nutritional value, spanning athletes, children, or the elderly.

Due to the increasing amount and variety of powdered products produced in the food industry and their complexity, there is a need for information on their handling and processing characteristics. Powdered fruits could be consumed as an ingredient to complete some other foods or after rehydration as a juice. In this sense, the powdered product must achieve different characteristics according to the format of use. Therefore, its flow behaviour both in air and in water, its compressibility, or its rehydration capability are important properties to be known. Despite the microbiological stability of powdered foods, due to their low water activity, in the case of fruits, they present physical problems associated with the development of stickiness phenomena (Telis & Martínez-Navarrete, 2012). This is due to their high content of sugars and organic acids, which makes their glass transition temperature (T_g) being very low, so that they are often in a rubbery state at usual consumption and storage temperatures. A common technique to increase the T_g is the addition of biopolymers of high molecular weight, such as gums, starches, maltodextrins, among others (Fongin et al., 2019, Pacheco et al., 2020, Silva-Espinoza et al., 2020, Telis & Martínez-Navarrete, 2012). Furthermore, Agudelo et al. (2017) observed that the functional quality of grapefruit powder was improved when a filler like a fibre is also added. Additionally, rehydrated powder quality can be improved also by using fillers as dispersants. If these fillers can serve to provide added fibre, then a nutritionally rounded product could be produced. In this study, gum Arabic (GA), maltodextrin 19DE (MD) and starch modified with octenylsuccinic anhydride (OSA) have been used as T_g modifiers, and bamboo fibre (BF), native corn starch (NCS) and pea fibre (PF) as fillers.

The biopolymers studied and their combination were selected based on previous studies. In a first study, GA and FB were selected and the ratio GA:FB:grapefruit puree was optimized in order to obtain a freeze-dried powder with the highest content of bioactive compounds and antioxidant activity, the lowest water content, hygroscopicity and porosity, together with an appropriate colour (Agudelo et al., 2017). Although the results obtained were highly satisfactory from the point of view of the product quality, the great structural heterogeneity of both GA and FB together with the high cost of the former, led us to look for possible alternatives to these biopolymers suitable to be added to the orange powder. MD is a cheaper biopolymer that has been widely used in spray-drying for its effect on properties such as wetting, tackiness, performance and hygroscopicity that it imparts to powdered products (Fang & Bhandari, 2012). Modified starches with improved properties, high solubility, and relatively low viscosity are becoming increasingly important, not only because of their low cost but also because of their numerous industrial applications (Dokić et al., 2012). In this sense, OSA has already been used as a substitute for some food components such as gum Arabic, fats, and proteins and is characterized as an amphiphilic polymer, obtained by chemical modifications to improve the chemical, physicochemical, and functional properties of native starch. All the native starches are also widely used in the industry since it contributes to stabilize food, helps emulsification, and improves texture (Luallen, 1985). In particular, the insolubility of NCS may suggest its use as filler. As regards pea fibre, it was selected based on the popularity of pea within the organic and health food markets, as it is a good source of dietary fibre and other important nutrients (Powers et al., 2020), and from the sustainable point of view of pulse crops (Stagnari et al., 2017). In any case, despite the benefits of using different biopolymers as to increase the stability of the dried products, all of them will affect the properties of the raw and powdered fruit so that the best combination of them must be selected considering its final use.

As to ensure the effectiveness of the biopolymers selected for the study, the Tg-water content-water activity relationships of the orange product obtained by the freeze-drying of different mixes of 100:5:1 puree:Tg modifier:filler were studied, in relation to the stability of its texture and colour (Silva-Espinoza et al., 2020). As stated by Silva-Espinoza et al. (2020), any of the mixtures of the biopolymers considered in this study permit a reduction in hygroscopicity and an increase in the Tg of the freeze-dried orange snack, without any of them being more or less effective than the others. What remained to be studied was whether any of these mixtures provided a powdered product with better flowability and rehydration behaviour, which was the objective of this study. Besides the particle size distribution, the powder flowability was characterized by means of its angle of repose, porosity, and compressibility. The rehydration behaviour was also studied throughout the powder wettability and the rheological properties of the rehydrated powder, the latter compared with four commercial orange juices.

1. Materials and methods

2.1. Raw materials

2.1.1. Fruit

Orange (*Citrus sinensis* cultivar Delta seedless) used in this study were bought from a local supermarket in the city of Valencia. The selection of fruit pieces was made by visual inspection based on the size homogeneity, colour, and good physical integrity.

2.1.2. Biopolymers

Carriers used to obtain the dehydrated orange samples were GA (Scharlab, Sentmenat, Spain), MD DE 19 (Roquette, Lestrem, France), OSA (Roquette, Lestrem, France), PF (Roquette, Lestrem, France), NC (Roquette, Lestrem, France), and BF (VITACEL®, Rosenberg, Germany). Some relevant physical properties and composition of each biopolymer according to the suppliers are shown in Table 1.

Table 1. Physical properties and composition of bamboo fibre (BF), starch modified with octenylsuccinic anhydride (OSA), gum Arabic (GA), native corn starch (NCS), pea fibre (PF), and maltodextrin (MD).

| Biopolymer | pH | Solubility (20 °C) | Appearance ⁽⁴⁾ | Concentration |
|------------|--------------------|--------------------|---------------------------|---|
| BF | 5-8 ⁽¹⁾ | Insoluble | White | 95% |
| OSA | 3.8 ⁽²⁾ | >500 g/L | White to off-white | ≥94% |
| GA | <7 ⁽²⁾ | 500 g/L | Slightly yellow | ≥85% |
| NCS | 5.1 ⁽²⁾ | Insoluble | Off white | ≥87% |
| PF | 7.1 ⁽¹⁾ | Not specified | Light brown | Fibre: 50% Protein: 10% Starch: 35% |
| MD | 4.7 ⁽³⁾ | ~600 g/L | White | ≥96% |

⁽¹⁾ at 10%; ⁽²⁾ at 20%; ⁽³⁾ at 50%; ⁽⁴⁾ all fine powders.

2.1.3. Commercial juices

Four commercial orange juices were purchased in a local supermarket in the city of Valencia, which will be called A, B, C, and D. The compositional information according to its label is: A: orange juice and vitamin C; B: orange juice, pulp, and vitamin C; C: orange juice; and D: orange juice from concentrated and vitamin C.

2.2. Obtaining the different samples

To obtain orange puree, fruit pieces were washed, peeled, cut, and triturated in a bench top electrical food processor for 40 s at 2000 rpm followed by 40 s at 9200 rpm (Thermomix TM 21, Vorwerk, Spain). Different biopolymers were added to the orange puree and mixed for 10 min at 1000 rpm to obtain a homogeneous blend. Five different samples were formulated according to Table 2.

2.3. Obtaining and characterization of the powdered products

2.3.1. Freeze-drying

The formulated orange purees (FOP) (Table 2) were placed in aluminium plates of 250 mm diameter, with a thickness of 0.5 cm per plate, and were immediately frozen at -45 °C (Liebherr LGT 2325, Baden-Wurtemberg, Germany) for at least 48 hours. The frozen samples were dried (Telstar Lioalfa-6, Spain), at 0.05 mbar, -45 °C in condenser and 40 °C in the shelves for 20 hours, obtaining the corresponding freeze-dried puree (FDP).

Table 2. Sample codes assigned to the orange puree samples formulated with different amounts of biopolymers added.

| Formulated orange puree (FOP) | 5g/100 g orange puree | 1g/100 g orange puree |
|-------------------------------|--|-----------------------|
| GA+BF | Gum Arabic | Bamboo fibre |
| MD+PF | Maltodextrin | Pea fibre |
| MD+NCS | Maltodextrin | Native corn starch |
| OSA+PF | Starch modified with octenylsuccinic anhydride | Pea fibre |
| OSA+NCS | Starch modified with octenylsuccinic anhydride | Native corn starch |

2.3.2. Crushing and sieving

The obtained FDP were crushed in the Thermomix (section 2.2) at 3700 rpm for 20 s to obtain the corresponding powders. Before sieving, the water content was analysed (section 2.3.3). Different sieves (CISA 200/50, Spain) were used, with the corresponding top and bottom placed, and a vibrating drum (CISA, AMP0.40, Spain) working at 50 Hz for 5 min. The powder obtained after crushing each of the five formulations was passed through a sieve of 800 µm mesh as to discard not clearly enough crushed parts of the FDP. A part of the powder with smaller size to 800 µm was characterized in all the properties related to its flow behaviour (sections 2.3.5-2.3.7) and the rehydration behaviour (section 2.4.). In order to investigate a possible impact of the different biopolymers used in the mechanical resistance to crushing of the samples, another part of the powder < 800 µm was destined for the determination of the particle size distribution (section 2.3.4) using sieves of 500, 300, 200, 150, and 100 µm mesh.

2.3.3. Water content

The water content (x_w , g water/100 g product) of all the FOP and of their corresponding powders was obtained by drying in a vacuum oven (Vaciotem, J.P. Selecta) at 60°C ± 1°C under $p < 100$ mm Hg until constant weight (AOAC 2000,

method 934.06). To calculate the water content, weight difference before and after the drying to the initial sample weight (XS204 DeltaRange®, Mettler Toledo, Switzerland) was obtained. Three replicates were carried out on each of the five samples.

2.3.4. Particle size distribution (PSD)

The PSD was carried out by the sieving method (Ahmed et al., 2016; Barbosa-Cánovas et al., 2012). About 90 g of the powder obtained after crushing the FDP, was sieved in batches of 30 g. The sample retained on each sieve and on the bottom was weighed and both the particle size distribution, according to the relative frequency of each size (Eq. 1), and the average particle size (Eq. 2) was calculated.

$$\text{Relative frequency (\%)} = \frac{w_i}{w} * 100 \quad (1)$$

$$\text{Average particle size} = \frac{\sum_i(AD_i * w_i)}{w} \quad (2)$$

Being w_i the weight of powder retained on each sieve (g), w the sum of the weight of powder retained on the different sieves and on the bottom (g), and AD_i the average mesh diameter of the sieve in which the powder is retained and the previous one (μm).

2.3.5. Angle of repose (α°)

This is the angle shaped between the slope of the cone of product formed when the powder is dropped down onto a horizontal surface and the latter. Based on the method proposed by Gallo et al. (2011), to determine α° 15 g of powdered product in a funnel (top diameter= 80 mm, stem = 11 mm, stem length = 29 mm, approx. overall height = 85 mm), placed 5 cm height from the horizontal surface covered with 80 g/m² DIN-A4 (Apli Paper S.A.U., Barcelona, Spain), and measuring the diameter and height of the product cone formed (Eq. 3). Four replicates were carried out to each of the five formulated samples.

$$\alpha^\circ = \arctan * \left(\frac{2h}{d} \right) \quad (3)$$

Where h = height from the top of the formed product cone to the horizontal surface (cm); d = maximum cone product diameter (cm), taken as an average of at least 6 values.

2.3.6. Density and porosity

True and apparent densities were characterized. The former excludes the air present in the sample and the latter considers the air inter and intra (open and closed pores) powder particles. True density (ρ , g/cm³) was calculated based on the sample composition (Eq. 4). As apparent density, the density of the compacted powder (tapped density) was considered. To obtain the tapped density (ρ_T , g/ cm³), the powder was poured inside a graduated tube to a volume of 10 mL and submitted to a vibration process in a vortex (Advanced Vortex Mixer, ZX3, VELP® SCIENTIFICA, Italy, 1200 rpm, 10 s). The density was calculated from the weight of the powder and the volume

that it occupied. From the above data, the porosity (ϵ , Eq. 5), was calculated. Four replicates were carried out to each of the five powders.

$$\rho = \frac{1}{\frac{x_w}{\rho_w} + \frac{x_{CH}}{\rho_{CH}}} \quad (4)$$

Where x_w and x_{CH} are the mass fractions of the two main components of each sample (water and carbohydrates, respectively; x_w determined following section 2.3.3 and x_{CH} by difference); ρ_w and ρ_{CH} are their densities ($\rho_{CH} = 1,4246 \text{ g/cm}^3$, $\rho_w = 0,9976 \text{ g/cm}^3$, Okos, 1986).

$$\epsilon(\%) = 100 \frac{\rho - \rho_T}{\rho} \quad (5)$$

2.3.7. Compressibility

The compressibility of a product (b , Pa^{-1}) may be obtained from the variation of its apparent density when applying low stress levels ($\sigma < 9.807 \cdot 10^4 \text{ Pa}$) (Eq. 6, Peleg, 1977).

$$\frac{\rho_\sigma - \rho_0}{\rho_0} = \frac{v_0 - v_\sigma}{v_\sigma} = a + b \log \sigma \quad (6)$$

Where ρ_0 and v_0 are the apparent density and the volume of the initial poured sample, ρ_σ and v_σ are the apparent density and the volume of the sample under the normal stress applied at each moment (σ , Pa) and a and b are constants. Constant b represents, specifically, the compressibility of a powder (Pa^{-1}).

The variation of the apparent density was obtained from a mechanical compression test using a texture analyser TA-XT (Stable Micro Systems, Surrey, UK), using a cylindrical probe of 10 mm diameter. The powder was placed and filled up in a circular aluminium sample holder of 11 mm diameter and 4.5 mm height. The sample holder was placed on the corresponding support. The test was carried out at constant speed of 0.05 mm/s and total deformation of 3 mm. Force (F , N), distance (h' , m) and time (s) values were recorded. Five replicates of each sample were carried out. From the results, data of $\sigma < 9.807 \cdot 10^4 \text{ Pa}$ were selected and related to the volume of the sample at each moment (v_σ , m^3) to calculate the compressibility (Peleg, 1977). This volume was calculated considering the dimensions of the sample holder and the distance travelled by the cylindrical probe. The first part of this curve considered until the limit value of stress was linearized. Equations 7, 8 and 9 were used. This process was made with all replication of each formulation. Fig. 1 shows, as an example, the procedure used for one of the replicates (compressibility was 0.1311 Pa^{-1}).

$$\sigma = \frac{F}{A} \quad (7)$$

$$h = H - h' \quad (8)$$

$$V_{\sigma} = \left[\pi * \left(\frac{Di}{2} \right)^2 \right] * h_{\sigma} \quad (9)$$

Being σ applied stress (Pa); F: force (N); A: area of the cylindrical probe (m^2); h_{σ} : powder height at each σ (m); H: sample holder height (m); h' : distance travelled by the cylindrical probe (m), V_{σ} : powder volume at each σ (m^3); Di: sample holder diameter (m).

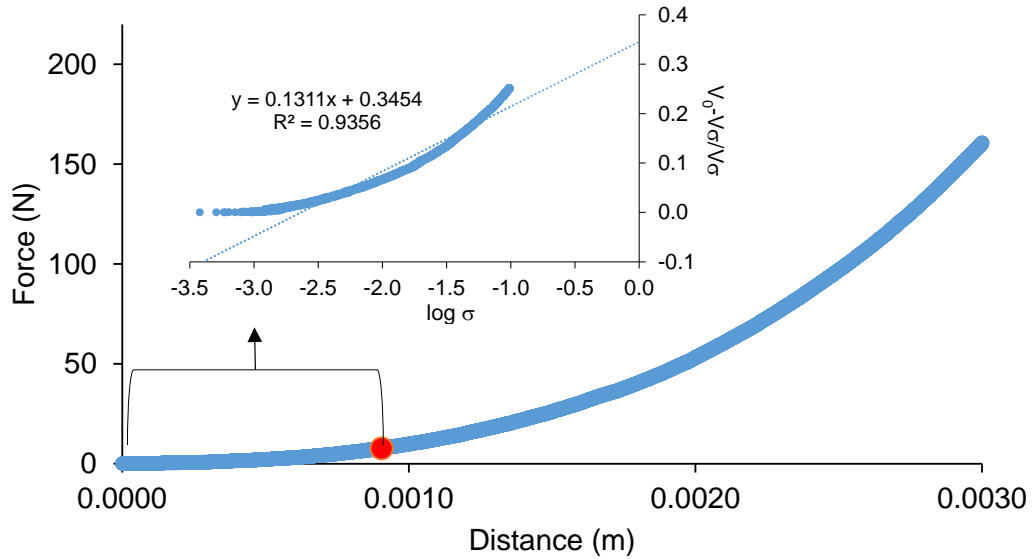


Figure 1. Example of force-distance curve corresponding to the compression test of the powder obtained from orange puree added with gum Arabic and bamboo fibre. Red point indicates the limit value of applied stress (σ) of $9.807 \cdot 10^4$ Pa for linearizing to get the relation between the variation of volume of the powder and the stress applied during the compression test.

2.4. Rehydration behaviour

2.4.1. Rehydration

Powder rehydration was carried out in order to obtain products with the same water content than the corresponding FOP. A mass balance, taking into account the water content of both the FOP and the powder, was applied to calculate the water content to be added to the powder (Eq. 10 and 11). Rehydration was carried out in jacketed beakers, connected to a thermostatic bath (Poly Science, Refrigerated Circulator 901, USA) at 20 °C, with constant magnetic stirring (800 rpm) during 5 min (Multi-Channer Stirrer MS-51M, JEIO TECH Lab Companion, Korea). Rehydrated product was left resting for 24 hours at 8 °C. Analysis of the rehydrated products was carried out in triplicate for the rheological behaviour.

$$m^{rp} = m^p + m^w \quad (10)$$

$$m^p \times (1 - x_w^p) = m^{rp} \times (1 - x_w^{FOP}) \quad (11)$$

Being m^p : final mass of the rehydrated product (g); m^w : water mass (g); m^p : powder mass (g); x_w^{FOP} : water content of the formulated orange puree (g water/g product); x_w^p : water content of the powder product (g water/g product).

2.4.2. Wettability

The wettability considers the time required for wetting all the particles of the powder when poured over water. This time is inversely related to the wettability. It was evaluated by the standard method 34-849-86 (UNE, 1986). Four replicates were made for each sample.

2.4.3. Rheological behaviour

The flow behaviour of the rehydrated products and the commercial samples was obtained at 8 °C by means of a controlled shear stress rheometer (Haake RheoStress 1, Thermo Scientific, Karlsruhe, Germany) with a coaxial cylinder sensor system (Z34 DIN), coupled to a thermostatic bath (Viscotherm VT 10, Physica). A relaxation time of 300 s was selected for the sample before running the test. Shear rate ($\dot{\gamma}$) was increased from 0 to 120 s⁻¹ and shear stress (σ , Pa) was recorded. Results were fitted to the Ostwald de Waele model (Eq. 12), to get the flow behaviour index (n) and the consistency index (K, Pa·sⁿ). Instead to calculate the corresponding apparent viscosity (η , Pa·s) at a concrete $\dot{\gamma}$, η was calculated applying the mean value theorem (Eq. 13, Mosquera, 2010). Taking into account that, for the samples considered in this study, η followed Eq. (14), Eq. (13) can be expressed as Eq. (15) to give the referred representative value.

$$\sigma = K(\dot{\gamma})^n \quad (12)$$

$$\eta = \frac{1}{\dot{\gamma}_{max}} \int_0^{\dot{\gamma}_{max}} \eta(\dot{\gamma}) d(\dot{\gamma}) \quad \dot{\gamma} \in [0, \dot{\gamma}_{max}] \quad (13)$$

$$\eta = K(\dot{\gamma})^{(n-1)} \quad (14)$$

$$\eta = \frac{K}{n} \dot{\gamma}_{max}^{(n-1)} \quad (15)$$

Where σ : shear stress (Pa), $\dot{\gamma}$: shear rate (s⁻¹), n: flow behaviour index, K: consistency index (Pa·sⁿ), $\dot{\gamma}_{max}$: maximum rate (s⁻¹) = 120 s⁻¹, η : apparent viscosity (Pa·s).

2.4.4. Particle morphology

Micrographs of rehydrated samples were acquired using an AMG EVO XL digital inverted microscope (ThermoFisher Scientific, USA).

2.5. Statistical analysis

An analysis of variance (ANOVA) using Tukey's HSD test was performed to establish the significant differences among the studied samples, which were considered when $p < 0.05$. Pearson correlation coefficient (r) between the different

studied properties was obtained. Statistical analysis was conducted using Statgraphics Centurion XVI.II.

2. Results and discussion

3.1. Characterization of the powder

3.1.1. Particle size distribution

Particle size distribution is directly related to the physical properties of a powdered product. Properties such as the bulk density, compressibility and flowability are highly dependent on the particle size and its distribution (Barbosa-Cánovas et al., 1987). But in the case of a powdered product obtained by freeze-drying, the particle size distribution may also be indicative of the mechanical resistance of the freeze-dried product to crushing, which may be of interest from a technological point of view. With this aim, the dry sieving size method was used as to characterize the particle size distribution of the different formulations (Section 2.3.2). PSD of each formulation is represented in Figure 2. The mode for all formulations was the particle size smaller than 100 μm . The cumulative relative frequency allows knowing the median of the distribution, this being 150-200 μm .

The average particle size of all the powders ranged between 201 and 221 μm . A greater particle size may be related to a greater mechanical resistance of FDP when crushed to obtain the powder, as promoted by the different biopolymers used in the formulation. In this sense, GA combined with BF, with a value of 221 μm , seems to promote a certain greater resistance while OSA+PF, with a value of 201 μm , leads to the more fragile FDP. As it can be observed in Fig. 2, these differences are mainly due to the higher frequency of particle size between 500 and 800 μm as opposed to the lower frequency of particle size between 100 and 150 μm found in the GA+BF sample. On the other hand, the combined use of PF instead of NCS contributed to the lower mechanical resistance. Nevertheless, significant differences ($p < 0.05$) were found only between GA+BF and the sample OSA+PF. From this point of view, differences among the powder properties described in the following sections may be attributed to the different composition of the samples and not to a different particle size resulting from the crushing process to which they have been subjected.

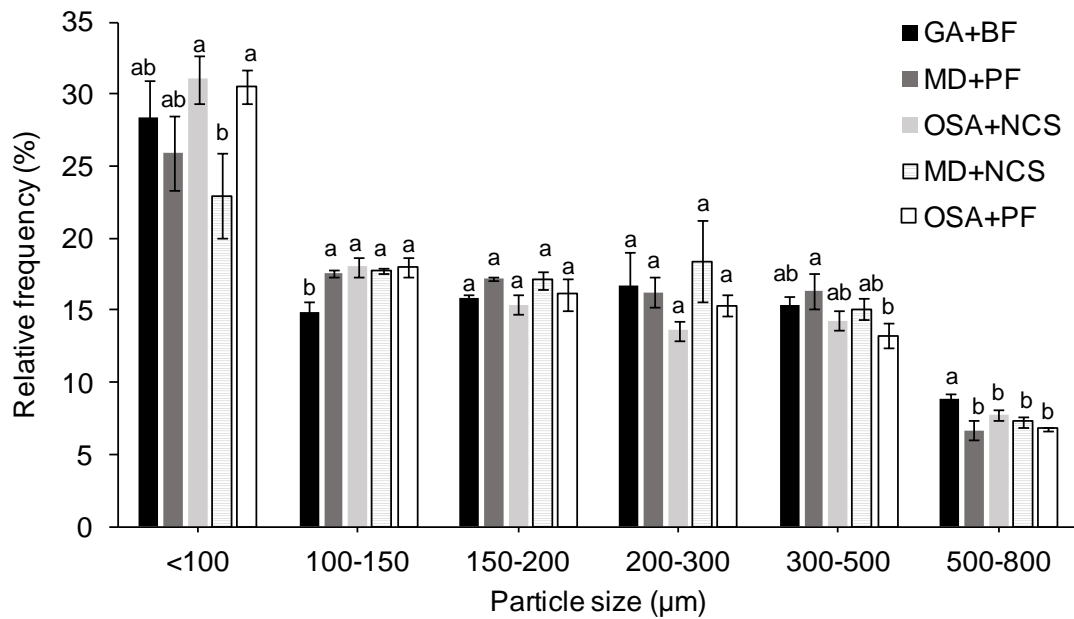


Figure 2. Particle size distribution according to the ratio weight of the retained sample in each sieve to total weight (relative frequency, %). Samples formulated with GA: gum Arabic, BF: bamboo fibre, MD: maltodextrin, NCS: native corn starch, PF: pea fibre, OSA: starch modified with octenylsuccinic anhydride.

3.1.2. Powder flowability

In this study, α° , ε and b were selected to evaluate the powder flowability. The α° depends on many variables, the fine particle content, density homogeneity, shape irregularities, shape of the particles themselves, particle-particle and particle-wall frictions being some of the most important (Ilari & Mekkaoui, 2005). In any case, the smaller the α° , the more easily the powder flows (Choi, Ryu, Kwak, & Ko, 2010). Regarding to b high values of this property are related with a worse flow behaviour (Schubert, 1987). The ε of the powder depends on the particles morphology and how this property is related to the powder flow depends on each product (Barbosa-Cánovas & Juliano, 2005; Ilari & Mekkaoui, 2005; Shenoy et al., 2015).

Three different homogeneous groups were observed (Table 3), the samples containing MD with the lower values of the three properties and GA+BF that with the highest ones ($p < 0.05$). According to the Royal Spanish Pharmacopoeia (RFE, 2015) powders with α° values between $31-35^\circ$ are considered with good flowability, $36-40^\circ$ with regular flowability and $41-45^\circ$ with acceptable flowability. In this case, samples formulated with MD, with smaller α° ($p < 0.05$), can be considered as “regular” in terms of flow, while the GA+BF, with the greatest α° , is classified as “acceptable” flow powder. The results indicated the good flowability provided by MD to the powders, related to a low α° and b and also, in the case of these samples, with a low ε . OSA samples showed an intermediate behaviour; OSA+PF could also be grouped with MD formulations while OSA+NCS could be with GA+BF.

Table 3. Values (mean \pm standard deviation) of the different studied properties of each formulation.

| Sample ^a | Angle of repose (°) | Porosity (%) | Compressibility (Pa ⁻¹) | Wetting time (s) |
|---------------------|------------------------------|-----------------------------|-------------------------------------|-----------------------------|
| GA+BF | 42 \pm 2 ^a | 76.9 \pm 0.6 ^a | 0.138 \pm 0.013 ^a | 570 \pm 54 ^{cd} |
| MD+PF | 37.0 \pm 1.7 ^c | 66.5 \pm 0.4 ^c | 0.0809 \pm 0.0102 ^c | 367 \pm 56 ^{de} |
| MD+NCS | 38.0 \pm 0.7 ^c | 67.0 \pm 0.3 ^c | 0.082 \pm 0.009 ^c | 175 \pm 19 ^e |
| OSA+PF | 39.4 \pm 0.9 ^{bc} | 72.4 \pm 0.4 ^b | 0.088 \pm 0.009 ^{bc} | 1887 \pm 284 ^a |
| OSA+NCS | 41.5 \pm 0.8 ^{ab} | 73.1 \pm 1.8 ^b | 0.108 \pm 0.004 ^b | 914 \pm 87 ^b |

^aGA: gum Arabic, BF: bamboo fibre, MD: maltodextrin, NCS: native corn starch, PF: pea fibre, OSA: starch modified with octenylsuccinic anhydride. The same lowercase letter within columns indicates homogeneous groups established by Tukey HSD ANOVA.

The surface composition of the powder particles is relevant in the flow behaviour as the possible interactions between the molecules that make up the powder would affect its flowability (Fitzpatrick, Iqbal, Delaney, Twomey, & Keogh, 2004). In the cases of OSA and GA, they both have some hydrophobic groups in their structure, providing an emulsifying character, which may cause the increase of cohesiveness between particles, and therefore decrease the flowability as compared with the MD. The existence of significant and positive linear correlation ($r=0.74$, $p<0.05$) between the compressibility and the angle of repose was verified. Furthermore, as observed by Ilari & Mekkaoui (2005), a significant and positive correlation ($r=0.79$, $p<0.05$) between angle of repose and the porosity of the powder was obtained, which indicates for this product the usefulness of porosity as a measurement of the flowability. The highest porosity of GA+BF may be due to the irregular shape of bamboo fibre (Fig 4a), that could be hindering the flowability of the powder. Similar results about the relation between the irregular shapes and porosity were found by Shenoy et al. (2015) and Barbosa-Canovas and Juliano (2005) who related the particle shape with the flowability. On the other hand, the PF used in this study is a blend of pea fibre (50%), starch (35%) and protein (10%), where the latter is also used as a drying aid. It has been reported that the surface morphology of the powders got indented and wrinkled when protein is used as a drying carrier. This fact helped to reduce the cohesiveness, and therefore improved the flowability of the powder (Muzaffar & Kumar, 2015).

3.2. Characterization of the rehydrated products

The water content of the orange puree was of 88.00 \pm 0.07 g water/ 100 g product, which decreased to between 82.8 and 83.1 g water/ 100 g product when biopolymers were added. The powders obtained after crushing FDP had values between 0.3 and 1.3 g water / 100 g product.

3.2.1. Wettability

Rehydratable powders require high wettability, which means a short wetting time. The powders with the lower wetting time were those containing maltodextrin (Table 3).

Sample GA+BF was statistically grouped with MD+PF, showing an intermediate behaviour. Finally, OSA+NCS and specially OSA+PF presented a significant greater wetting time ($p < 0.05$, Table 3). The worst wettability of samples containing OSA may be attributed to its physicochemical properties. As a result of the chemical modification with OSA, the hydrophobicity of modified starch is greatly increased. When dissolved in water, such macromolecules preferentially migrate to the air/water interface forming a boundary layer whereby hydrophobic groups are oriented toward the air and starch extending to the water. When the interface is saturated, amphiphilic macromolecules start to aggregate (Sweedman et al., 2013).

3.2.2. Rheological behaviour

The flow behaviour of each rehydrated formulation was compared with four commercial juices (Fig. 3). The flow curves were well fitted to the Ostwald-de Waele model (Table 4), with coefficient of determination ($R^2 \geq 0.85$ for the commercial juices and ≥ 0.93 for the different rehydrated products). All the samples showed a typical pseudoplastic behaviour, except the commercial juice D which showed a Newtonian behaviour. Although what characterises pseudoplastic products is the decrease in viscosity as the shear rate increases, in this case η was calculated applying the mean value theorem (Eq. 15) to give a representative value of this variable in all the shear rate range considered in the study (Table 4). All the rehydrated powders had much higher values of η than the commercial juices A and D ($p < 0.05$), which did not contain visible pulp and are the least viscous.

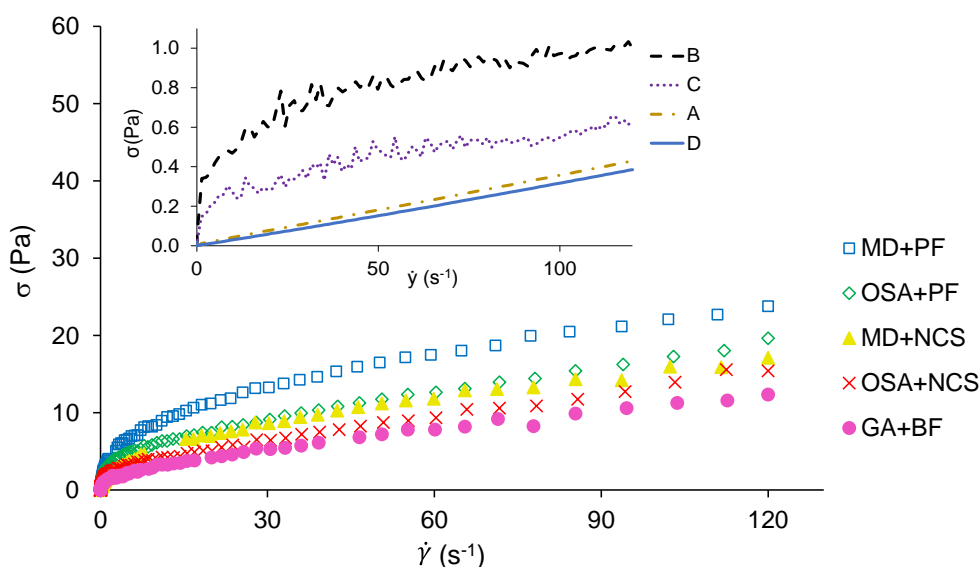


Figure 3. Flow shear stress (σ) vs. shear rate ($\dot{\gamma}$) curves of one of the replicates of the rehydrated products and the commercial juices. GA: gum Arabic, BF: bamboo fibre, MD: maltodextrin, NCS: native corn starch, PF: pea fibre, OSA: starch modified with octenylsuccinic anhydride. Commercial juices: A: orange juice with vitamin C; B: orange juice with pulp and vitamin C; C: orange juice; D: orange juice from concentrated with vitamin C.

Table 4. Mean values (and standard deviation) of flow behaviour index (n), consistency index (K, Pa.sⁿ) and apparent viscosity calculated throughout the mean value theorem (η , Pa.s) for the rehydrated products of each formulation and commercial juices.

| Sample ^a | n | K (Pa·s ⁿ) | η (Pa·s) |
|---------------------|------------------------------|------------------------------|------------------------------|
| GA+BF | 0.521 ± 0.008 ^{bc} | 1.027 ± 0.008 ^c | 0.199 ± 0.003 ^d |
| MD+PF | 0.589 ± 0.016 ^{bc} | 2.09 ± 0.15 ^a | 0.496 ± 0.016 ^a |
| MD+NCS | 0.6046 ± 0.0102 ^b | 1.20 ± 0.03 ^{bc} | 0.299 ± 0.002 ^b |
| OSA+PF | 0.47 ± 0.02 ^c | 1.97 ± 0.09 ^a | 0.334 ± 0.028 ^b |
| OSA+NCS | 0.49 ± 0.02 ^{bc} | 1.42 ± 0.08 ^b | 0.249 ± 0.019 ^c |
| A | 0.93 ± 0.05 ^a | 0.0047 ± 0.0012 ^e | 0.0035 ± 0.0003 ^e |
| B | 0.23 ± 0.04 ^d | 0.37 ± 0.09 ^d | 0.041 ± 0.009 ^e |
| C | 0.25 ± 0.12 ^d | 0.22 ± 0.13 ^{de} | 0.025 ± 0.013 ^e |
| D | 1.00 ± 0.03 ^a | 0.0032 ± 0.0006 ^e | 0.0032 ± 0.0002 ^e |

^aGA: gum Arabic, BF: bamboo fibre, MD: maltodextrin, NCS: native corn starch, PF: pea fibre, OSA: starch modified with octenylsuccinic anhydride. Commercial juices: A: orange juice with vitamin C; B: orange juice with pulp and vitamin C; C: orange juice; D: orange juice from concentrated with vitamin C. The same lowercase letter within columns indicates homogeneous groups established by Tukey HSD ANOVA.

Among the rehydrated products, formulation GA+BF showed the closest viscosity to the commercial juices, especially to B and C, even though there were significant differences between them ($p < 0.05$). Despite these commercial juices having added pulp, not all the pulp provided by the fruit puree used in the study is present. On the other hand, the rehydrated products containing PF showed the highest η . As commented above, the PF used in this study was composed for a blend of pea fibre, pea starch and pea protein where the pea starch corresponded to 35%. In this sense, a study carried out in extruded feeds showed that pea starch also offers a higher value of viscosity than other starches like wheat starch (Sorensen, Morken, Kosanovic, & Overland, 2011). Those samples formulated with NCS showed lower values of η than those with PF. Also, it was observed that those rehydrated products containing maltodextrin showed higher values of η .

The structure of some of the rehydrated formulations are shown in Figure 4. It can be clearly observed the presence of bamboo fibre in the rehydrated powder product (Figure 4a), which indicates the low solubility of this fibre in aqueous system. Native starches generally have limited solubility in water (Sweedman et al., 2013). In this sense, NCS was reported to be insoluble in water at 20 °C, where the insoluble starch granules can be observed in Figure 4b. However, with regards to the presence of pea fibre in the rehydrated products, this observation was not appreciated (Figure 4c and 4d).

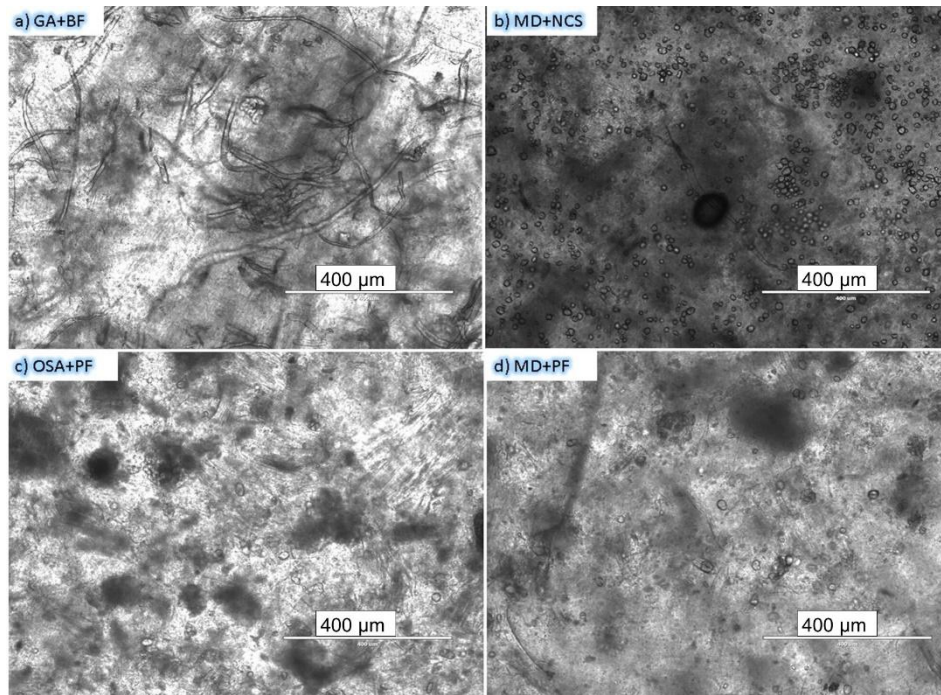


Figure 4. EVO's micrographs of the rehydrated products. Rehydrated and formulated with: a) GA: gum Arabic and BF: bamboo fibre; b) MD: maltodextrin and NCS: native corn starch; c) OSA: starch modified with octenylsuccinic anhydride and PF: pea fibre; d) MD: maltodextrin and PF: pea fibre.

This may be due to the lower amount of fibre in the samples formulated with PF than that with BF, because the previously mentioned PF composition or it is having a greater solubility. Both hypotheses may cause the increase of viscosity in those rehydrated products containing PF, while the presence of particles non-soluble, such as the BF and NCS may be causing the lower viscosities.

As regards the correlation with the other studied properties, the apparent viscosity showed a significant and negative linear correlation ($p < 0.05$) with angle of repose ($r = -0.67$), compressibility ($r = -0.75$) and porosity ($r = -0.77$). It seems to be that the worst product regards to flowability, GA+BF, gives a rehydrated product with the lowest viscosity, closest to those of the commercial juices.

3. Conclusions

The results confirm the influence of the different biopolymers studied on the characteristics of the freeze-dried orange puree powder. If what matters is to get a powder with good flow behaviour in air, it is preferable to formulate the puree with maltodextrin, either with pea fibre or with native corn starch. If the use of the powdered product is intended to be for rehydration and consumption as a juice, it is preferable those biopolymers that promote both a shorter wetting time and lower viscosities of the rehydrated products. In this sense, gum Arabic and bamboo fibre or those with maltodextrin should be selected. However, maltodextrin provides products of much higher viscosity after their rehydration. Then, the gum Arabic seems to be the

biopolymer more adequate in this case, thereby demonstrating that adding key biopolymers improve the physicochemical properties of the freeze-dried fruit powder.

Acknowledgments

The authors thank the Ministerio de Economía, Industria y Competitividad of Spain for the financial support given through the Project AGL 2017-89251-R (AEI/FEDER-UE) and the Ministerio de Universidades for the FPU grant (FPU14 / 02633) awarded to Ms. Andrea Silva.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.

References

- Agudelo, C., Igual, M., Camacho, M. M., & Martínez-Navarrete, N. (2017). Effect of process technology on the nutritional, functional, and physical quality of grapefruit powder. *Food Science and Technology International*, 23, 61–74. <https://doi.org/10.1177/1082013216658368>
- Ahmed, J., Taher, A., Mulla, M. Z., Al-Hazza, A., & Luciano, G. (2016). Effect of sieve particle size on functional, thermal, rheological and pasting properties of Indian and Turkish lentil flour. *Journal of Food Engineering*, 186, 34–41. <https://doi.org/10.1016/j.jfoodeng.2016.04.008>
- Barbosa-Cánovas, G. V., Malave-Lopez, J., & Peleg, M. (1987). Density and compressibility of selected food powder mixtures. *Journal of Food Processing Engineering*, 10, 1-19. <https://doi.org/10.1111/j.1745-4530.1987.tb00001.x>
- Barbosa-Cánovas, G. V., & Juliano, P. (2005). Physical and chemical properties of food powders. In: C. Onwulata (Ed.), *Encapsulated and powdered foods* (pp. 39-71). Taylor & Francis.
- Barbosa-Cánovas, G. V, Harte, F., & Yan, H. H. (2012). Particle size distribution in food powders. *Food Engineering*, 1, 303–328. <http://www.eolss.net/sample-chapters/c10/E5-10-01-06.pdf>
- Choi, K. O., Ryu, J., Kwak, H. S., & Ko, S. (2010). Spray-dried conjugated linoleic acid encapsulated with Maillard reaction products of whey proteins and maltodextrin. *Food Science and Biotechnology*, 19, 957–965. <https://doi.org/10.1007/s10068-010-0134-7>
- Dokić, L., Krstonošić, V., & Nikolić, I. (2012). Physicochemical characteristics and stability of oil-in-water emulsions stabilized by OSA starch. *Food Hydrocolloids*, 29(1), 185–192. <https://doi.org/10.1016/j.foodhyd.2012.02.008>
- Fang, Z., & Bhandari, B. (2012). Comparing the efficiency of protein and maltodextrin on spray drying of bayberry juice. *Food Research International*, 48(2), 478–483. <https://doi.org/10.1016/j.foodres.2012.05.025>
- FAO, Food and Agriculture Organization. (2003). *Increasing fruit and vegetable consumption becomes a global priority*. <http://www.fao.org/english/newsroom/focus/2003/fruitveg1.htm>
- Fitzpatrick, J. J., Iqbal, T., Delaney, C., Twomey, T., & Keogh, M. K. (2004). Effect of powder

-
- properties and storage conditions on the flowability of milk powders with different fat contents. *Journal of Food Engineering*, 24, 435–444. <https://doi.org/10.1016/j.jfoodeng.2003.11.011>
- 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. <https://doi.org/10.1016/j.powtec.2010.12.021>
- Ilari, J. L., & Mekkaoui, L. (2005). Physical of constitutive size classes of spray-dried skim milk powder and their mixtures, *Lait*, 85, 279-294. <https://doi.org/10.1051/lait:2005029>
- Luallen, T.E. (1985). Starch as a functional ingredient. *Food Technology*, 39(1), 59-63.
- MAPA, Ministry of Agriculture, Fishing and Food. (2017). *Food Consumption Report in Spain*. https://www.mapa.gob.es/es/alimentacion/temas/consumo-tendencias/informeconsumoalimentacionenespana2017_prefinal_tcm30-456186.pdf
- Mosquera, L. H. (2010). *Influencia de la Humedad y de la adición de solutos (maltodextrina o goma arábica) en las propiedades fisicoquímicas de borjón y fresa en polvo* [Ph.D. Thesis, Universidad Politécnica de Valencia]. RiuNet Repositorio UPV. <https://riunet.upv.es/handle/10251/9035>
- Muzaffar, K., & Kumar, P. (2016). Effect of soya protein isolate as a complementary drying aid of maltodextrin on spray drying of tamarind pulp. *Drying Technology*, 34, 142-148. <https://doi.org/10.1080/07373937.2015.1042586>
- Okos, M. R. (1986). *Physical and chemical properties of food*. American Society of Agricultural Engineers.
- Peleg, M. (1977). Flowability of food powders and methods for its evaluation. *Journal of Food Process Engineering*, 1, 303 – 328. <https://doi.org/10.1111/j.1745-4530.1977.tb00188.x>
- Powers, S., Mirsky, E., Bandaranayake, A., Thavarajah, P., Shipe, E., Bridges, W., & Thavarajah, D. (2020). Field pea (*Pisum sativum* L.) shows genetic variation in phosphorus use efficiency in different P environments. *Scientific Reports*, 10(1), 18940. <https://doi.org/10.1038/s41598-020-75804-0>
- Ratti, C. (2013). Freeze drying for food powder production. In: B. Bhandari, N. Bansal, M. Zhang, & P. Shcuck (Eds.), *Handbook of food powders* (pp.57-84). Woodhead Publishing. <https://doi.org/10.1533/9780857098672.1.57>
- RFE, Real Farmacopea Española (2015). Ministerio de Sanidad, Servicios Sociales e Igualdad Website, fifth ed. http://tienda.boe.es/Farmacopea_index.html
- Schubert, H. (1987). Food particle technology part I: Properties of particles and particulate food systems. *Journal of Food Engineering*, 6, 1–32. [https://doi.org/10.1016/0260-8774\(87\)90019-7](https://doi.org/10.1016/0260-8774(87)90019-7)
- Shenoy, P., Viau, M., Tammel, K., Innings, F., Fitzpatrick, J., & Ahrné, L. (2015). Effect of powder densities, particle size and shape on mixture quality of binary food powder mixtures. *Powder Technology*, 272, 165-172. <https://doi.org/10.1016/j.powtec.2014.11.023>
- Silva-Espinoza, M. A., Camacho, M. M., & Martínez-Navarrete, N. (2020). Use of different biopolymers as carriers for purposes of obtaining a freeze-dried orange snack. *LWT-Food Science and Technology*, 109415. <https://doi.org/10.1016/j.lwt.2020.109415>
- Sorensen, M., Morken, T., Kosanovic, M., & Overland, M. (2011). Pea and wheat starch possess different processing characteristics and affect physical quality and viscosity

-
- extruded feed for Atlantic salmon. *Aquaculture Nutrition*, 17, e326-e336. <https://doi.org/10.1111/j.1365-2095.2010.00767.x>
- Stagnari, F., Maggio, A., Galieni, A., & Pisante, M. (2017). Multiple benefits of legumes for agriculture sustainability: an overview. *Chemical and Biological Technologies in Agriculture*, 4(1), 2. <https://doi.org/10.1186/s40538-016-0085-1>
- Sweedman, M.C., Tizzotti, M. J., Schäfer, C., & Gilbert, R.G. (2013). Structure and physicochemical properties of octenyl succinic anhydride modified starches: A review. *Carbohydrate Polymers*, 92, 905–920. <https://doi.org/10.1016/j.carbpol.2012.09.040>
- Tavarini, S., Degl'Innocenti, E., Remorini, D., Massai, R., & Guidi, L. (2008). Antioxidant capacity, ascorbic acid, total phenols and carotenoids changes during harvest and after storage of Hayward kiwifruit. *Food Chemistry*, 107, 282–288. <https://doi.org/10.1016/j.foodchem.2007.08.015>
- Telis, V. R. N., & Martínez-Navarrete, N. (2012). Biopolymers used as drying aids in spray-drying and freeze-drying of fruit juices and pulps. In: V.R.N., Telis (Ed.), *Biopolymer engineering in food processing* (pp.279-325). CRC Press.
- Tomás-Barberán, F. A., Gil, I., Cremin, P., Waterhouse, A. L., Hess-Pierce, B., & Kader, A. A. (2001). HPLC - DAD - ESIMS Analysis of Phenolic Compounds in Nectarines , Peaches, and Plums. *Journal of Agricultural and Food Chemistry*, 49, 4748–4760. <https://doi.org/10.1021/jf0104681>
- UNE 34849. (1983). *Instant dried milk. Determination of dispersibility and wettability*. The International Organization for Standardization