



Contents lists available at ScienceDirect

Food Research International

journal homepage: [www.elsevier.com/locate/foodres](http://www.elsevier.com/locate/foodres)

## Development and characterization of a new encapsulating agent from orange juice by-products

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### ARTICLE INFO

#### Keywords:

Encapsulation  
Fibers  
Glass transition  
Isothermic heat  
Orange wastes  
Sorption isotherm

### ABSTRACT

The replacement of maltodextrins as carriers for the spray drying of sticky and sugar based bioactives is an important development for the food industry. In this work, orange juice industry by-product was used to obtain a high dietary fiber powder to be used as carrier material. This powder was characterized with respect to its physical and chemical properties related to the process of encapsulation by spray drying. Adsorption isotherms of orange waste powder were determined at 30, 45, and 60 °C. The data were fitted to several models including two-parameter (BET, Halsey, Smith, and Oswin), three-parameter (GAB), and four-parameter (Peleg) relationships. The GAB model best fitted the experimental data. The isosteric heat of sorption was determined from the equilibrium sorption data using the Clausius-Clapeyron equation. Isothermic heats of sorption were found to decrease exponentially with increasing moisture content. The enthalpy-entropy compensation theory was applied to the sorption isotherms and indicated an enthalpy controlled sorption process. Glass transition temperatures ( $T_g$ ) of orange waste powder conditioned at various water activities were determined and a strong plasticizing effect of water on  $T_g$  was found. These data were satisfactorily correlated by the Gordon and Taylor model. The critical water activity and moisture content for the orange waste powder were 0.82 and 0.18 g water/g solids, respectively, at a storage temperature of 25 °C.

### 1. Introduction

The processing of fruits results in the production of byproducts that are rich sources of bioactive compounds, including phenolic compounds. The interest in natural phenolic antioxidants has been raised by suggestions that many of these compounds display antimutagenic, antimicrobial, chemopreventive, antitumour, antiinflammatory, apoptotic, neuroprotective, antisickling, anthelmintic, and many other activities (Shahidi & Ambigaipalan, 2015). Thus, utilization of fruit wastes as sources of bioactive compounds may be of considerable economic benefits and has become increasingly attractive.

However, because of the presence of unsaturated bonds in their molecular structure, polyphenols are vulnerable to oxidants, light, and heat (Zheng, Liu, Li, & Zhu, 2011). In addition, phenolic extracts may exhibit a very low water and lipid solubility, making their application into most food systems extremely difficult (Spigno et al., 2013). Encapsulation is the most applied technique to solve these problems. According to Fang and Bhandari (2010), phenolic compounds are usually encapsulated for the following reasons: (i) increase of their stability during storage and passage through the gastrointestinal tract; (ii) improvement of their color; (iii) masking of astringency; (iv)

suitability for use as an additive in functional foods. The most common encapsulation method is the spray drying technique, because is an economical and flexible method and produces microcapsules of high quality in a simple and continuous operation with good efficiency and yield (Kaderides, Goula, & Adamopoulos, 2015; Mahdavi, Jafari, Ghorbani, & Assadpoor, 2014).

However, the microcapsules properties depend not only on the encapsulation method and the process parameters, but also on the wall material. Wall materials are chosen so as to meet as closely as possible the following properties: effective emulsification, film forming characteristics, efficient drying properties, low viscosity even at highly concentrated solution, and low cost (Re, 1998). Among the available materials, the major encapsulating agents used for spray drying applications are gums, emulsifying starches, and hydrolyzed starches (maltodextrins, corn syrup solids). Kaderides et al. (2015) encapsulated phenolic compounds from pomegranate peels using maltodextrin, whey protein concentrate, and skim milk powder as wall materials. Maltodextrin has also been used by Ersus and Yurdagel (2007) as carrier for encapsulation of phenolics from carrots. A mixture of maltodextrin and starch has been used for encapsulation of phenolic extract from soybean (Georgetti, Casagrande, Souza, Oliveira, & Fonseca, 2008), whereas

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<http://dx.doi.org/10.1016/j.foodres.2017.07.057>

Received 12 June 2017; Received in revised form 24 July 2017; Accepted 26 July 2017  
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Betz et al. (2012) encapsulated bilberry phenolic extract with whey protein concentrate. However, according to Chiou and Langrish (2007), the properties of some of these wall materials, in terms of undesired taste alteration and also being unnatural additives, mean that a suitable alternative carrier for spray drying needs to be found. The development of alternative and inexpensive polymers that would be considered “natural” and could be used as improved carrier materials has been an active area of recent researches (Re, 1998). It is possible that natural fibers may be able to fulfil this role.

Soluble dietary fibers include pectin, mucilages, gums, and hemicelluloses, whereas insoluble dietary fibers include hemicelluloses, cellulose, and lignin (Dhingra, Michael, Rajput, & Patil, 2012). Increased consumption of dietary fibers in a balanced diet is beneficial for the prevention and treatment of many diseases, such as coronary heart disease, colonic cancer, diabetes, and gastrointestinal disorders (Crizel, Jablonski, Rios, Rech, & Flôres, 2013; Figuerola, Hurtado, Estévez, Chiffelle, & Asenjo, 2005). Soluble fiber is associated with decreased blood cholesterol and intestinal absorption of glucose and insoluble fiber contributes to proper functioning of the intestinal tract (Grigelmo-Miguel, Gorinstein, & Martin-Belloso, 1999). According to numerous health organizations, the recommended consumption of dietary fibers is 30 to 45 g per day (Grigelmo-Miguel et al., 1999). However, according to Lario et al. (2004), dietary fibers are desirable not only for their nutritional properties, but also for their functional and technological properties. Carbohydrates of insoluble dietary fibers are known to have strong absorption properties that make them excellent carriers for phenolic extracts (Chiou & Langrish, 2007). However, limited research has been achieved with regard to the use of natural food fibers as wall materials for encapsulation of phenolics.

Fruit wastes are an important source of natural fibers. Oranges are important commodities in terms of global agricultural production (Crizel et al., 2013). During orange juice production, only around the half of the fresh orange weight is transformed into juice (Braddock, 1995), generating great amounts of residue (peel, pulp, seeds), which accounts for the other 50% of the fruit weight (Garcia-Castello et al., 2011). This waste in most cases generates environmental problems or is valorized as animal feed and fertilizer, so it has no economic value for food industries, even though its composition is rich in soluble sugars, cellulose, hemicellulose, pectin, and essential oils that could form the basis of several industrial processes (Rezzadori, Benedetti, & Amante, 2012). According to Grigelmo-Miguel and Martin-Belloso (1999), by-products from orange juice extraction have a potential use as a dietary fiber source.

The objectives of the present work are 1) the preparation of a high dietary fiber powder from orange juice industry by-products to be used as a wall material for encapsulation and 2) its characterization with respect to its functional, physical, and chemical properties related to the process of encapsulation by spray drying, such as water sorption behavior and glass transition temperature, which are parameters employed in designing and optimizing drying, packaging, and storage of food powders (Sablani, Kasapis, & Rahman, 2007).

## 2. Materials and methods

### 2.1. Materials

Fresh oranges (*Citrus sinensis*) were procured from the local market. A juice extractor discharged the rind and the inner part of the fruit was pressed. After the oranges were thoroughly squeezed, the juice and the bagasse were passed through a prefinisher and a fine screen to separate juice from bagasse. This bagasse was washed and dried according to the process presented in Fig. 1. The process was based on Larrauri (1999) recommendations for orange fiber obtention: grinding orange waste to a high particle size and washing with hot water (90 °C) for 20 min to produce a fiber with high water holding capacity. The samples were dehydrated in a tray dryer at 60 °C until the weight remained constant.

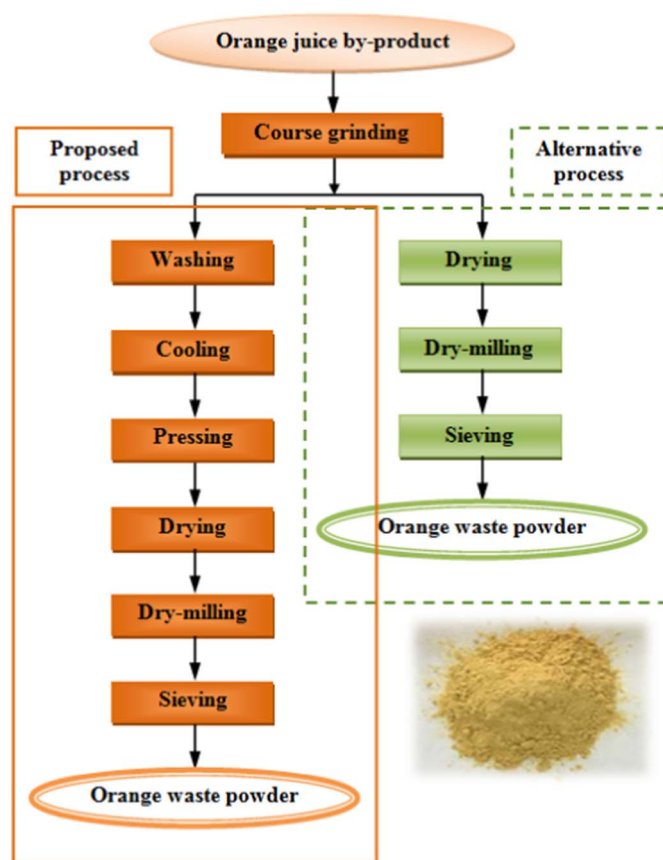


Fig. 1. Process for preparation of orange waste powder.

After cooling to room temperature, the dried product was ground in a mill. The milled fiber was separated using sieves for particle size analysis; the separated particles were smaller than 0.008 mm. An alternative process involves only drying and grinding as described in Fig. 1.

### 2.2. Trial encapsulation experiments

Pomegranate peels as a by-product of the fruit juice industry were provided by a local producer (Rodi Hellas, Greece). The peels were dried at 40 °C for 48 h and were ground in a laboratory mill (Type A10, Janke and Kunkel, IKA Labortechnik, Germany). The pomegranate peel extract was obtained by ultrasound-assisted extraction at optimum conditions according to a previous study (Kaderides et al., 2015).

The solution of coating matrix was prepared by reconstituting and dispersing orange waste powder in 40 °C deionized water and after cooling was mixed overnight to enhance hydration. Pomegranate peel extract was added into the hydrated coating material with a ratio of wall to core material of 9/1 g/g (Kaderides et al., 2015). The solution was spray dried in a pilot scale spray dryer (Buchi, B-191, Buchi Laboratoriums-Technik, Flawil, Switzerland) with concurrent regime and a two-fluid nozzle atomizer. In all experiments, the atomizer pressure and the feed rate and total solids concentration were kept at  $5.0 \pm 0.1$  bar,  $1.75 \pm 0.05$  g/min, and  $5 \pm 0.1\%$  respectively. The inlet air temperature, the drying air flow rate, and the atomizing agent flow rate were 150 °C,  $17.5 \text{ m}^3/\text{h}$ , and  $0.6 \text{ m}^3/\text{h}$ , respectively (Kaderides et al., 2015).

### 2.3. Determination of physicochemical properties of orange waste powder

#### 2.3.1. Chemical composition

The total dietary fiber (DF), soluble and insoluble, content was determined by the enzyme-gravimetric method described by AOAC

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