Processing of Orange (Citrus unshiu) Powder by Micro Wet Milling and Vacuum Spray Drying

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ABBREVIATION

BET	Brunauer-Emmett-Teller
CD	Degree of Caking
CI	Carr Index
COJ	Concentrated Orange Juice
DE	Dextrose Equivalent
DPPH	2, 2-diphenyl 1-picrylhydrazyl
DSC	Differential Scanning Calorimetry
EMC	Equilibrium Moisture Content
FD	Freeze Drying
FRAP	Ferric Reducing Power Assay
GAB	Guggenheim-Anderson-de Bore
GAE	Gallic Acid Equivalent
HR	Hausner Ratio
MD	Maltodextrin
MWM	Micro Wet Milling
OJ	Orange Juice
QE	Quercetin Equivalent
SD	Spray Drying
SEM	Scanning Electron Microscopy
TE	Trolox Equivalent
TFC	Total Flavonoid Content
TPC	Total Polyphenol Content
TS	Total Solids
TSS	Total Soluble Solids
VSD	Vacuum Spray Drying
WSI	Water Solubility Index

Chapter 1. Introduction

1.1 Background

Orange is one of the important citrus fruits grown all over the world. The consumption of fruit juice specially citrus increasing worldwide, due to the public perception of juice to favor a healthy quality of life. Citrus fruits are the major sources of health-promoting constituents such as flavanones, polyphenols, ascorbic acid and antioxidants (Gil-Izquierdo et al., 2002; Stinco et al., 2012). Particularly intake of orange juice shows positive effects on cardiovascular diseases (Elzbieta M. Kurowska et al., 2000). Narirtutin, hesperdin and didymin belong to the flavanone glycoside group and the most available flavonoid present in the orange juice. According to So et al. (1996), hisperdin in citrus juice can inhibit the chemically induced breast cancer. On the other hand, orange juice is the most popular fruit juice in the world market due its attractive color, appealing sensory properties. Moreover, orange juice contains a higher amount of ascorbic acid around 30-50 mg/100 mL (Johnston and Hale, 2005; Meléndez-Martínez et al., 2007) and the health-related properties of polyphenol contained in orange juice (Meyer et al., 1998) are based on their antioxidant activity. The physicochemical, functional and antioxidant properties of orange juice were studied by Kelebek et al. (2009) as shown in Table 1-1.

Orange juice (OJ) also a good source of carotenoid which is responsible for the attractive color of the juice (Meléndez-Martínez et al., 2007; Stinco et al., 2012). The fruits are consumed either fresh and juice. Due to the presence of higher amount of water (85-90%) (Kelebek et al., 2009), the fruits are easily deteriorated. The fruits are usually preserved as juice or powders for extending the shelf life and ensure the availability through the year. There are different methods used to process and preserve the juice or as a powder. During the processing and preservation, OJ undergoes changes in their chemical and functional components. According to Johnston and Hale (2005) and REKHA et al. (2012), it was found that 30-40% of initial ascorbic acid in some

fruit juice could be lost on dehydration. It is very important, therefore, to preserve or enhance the functional components of the processed OJ or OJ products, particularly high level of ascorbic acid, total polyphenol, flavonoid, carotenoid and the associated antioxidant activity.

Table 1-1. General composition of Kozan orange juice

Juice composition	Amount
Yield (%)	40.0±0.71
Density (g/mL)	1.052±0.01
Total acidity (g/L)	9.11±0.01
pH	3.35±0.01
Brix	11.8±0.00
Ash (g/L)	3.7±0.08
Sugar (g/L)	120.19±3.84
Citric acid (g/L)	12.66±0.16
Ascorbic acid (g/L)	0.49±0.01
Malic acid (g/L)	1.06±0.01
Hydroxybenzoic acid (mg/L)	4.28±0.29
Hydroxycinnamic acid (mg/L)	60.38±3.87
Flavanones (mg/L)	252.7±12.38

Source: (Kelebek et al., 2009)

In addition, OJ with pulp containing high nutritional content, because of the higher flavonoid content in OJ pulp. Flavonoid neutralizes the oxidative and inflammatory stress generated by the unhealthy food and helps to prevent blood vessel damage (So et al., 1996). OJ with pulp is an important source of dietary fiber to favor a healthy digestive system (Uichard, 2004). During the industrial processing of OJ or OJ powders, the pulp and seed are usually discarded or use another purpose. The percentages of different parts varied depend on species. Usually Japanese orange (*citrus unshiu*) doesn't contain any seed and skin are thin comparatively other oranges.

Therefore, developments of suitable processing methods for utilization of orange pulp and necessary to reduce the wastes, to increase the functional constituents and also enhance the economic value of the fruit.

1.2 Processing of orange juice

Fresh OJ has limited shelf life and the most common method for extending the shelf life of fresh OJ is inactivation of microorganisms and enzymes by thermal processing (Fellers, 1988). Orange juice is subjected to a process by industrially or in domestic. Orange juice is commonly marketed in three forms: Fresh juice or single strength juice or ready to drink juice, as a frozen concentrated OJ, which is diluted with water after purchases, as a reconstituted liquid, which has been concentrated and diluted before sale (Gil-Izquierdo et al., 2002; Stinco et al., 2012). There are only very few studies which reported the effects of processing technique that affects the compounds of orange juice (Tomás-Barberán and Clifford, 2000). According to Gil-Izquierdo et al. (2002) reported the steps of processing technique at an industrial scale on orange juice as shown in Figure 1-2. During the industrial processing of orange juice, pulp, seed, and skin are commonly discarded then undergoes some thermal treatment like pasteurization or evaporation, that extend the shelf-life but deteriorate the color, flavor, ascorbic acid or other heat-sensitive functional constituents (Escudero-gilete et al., 2012).

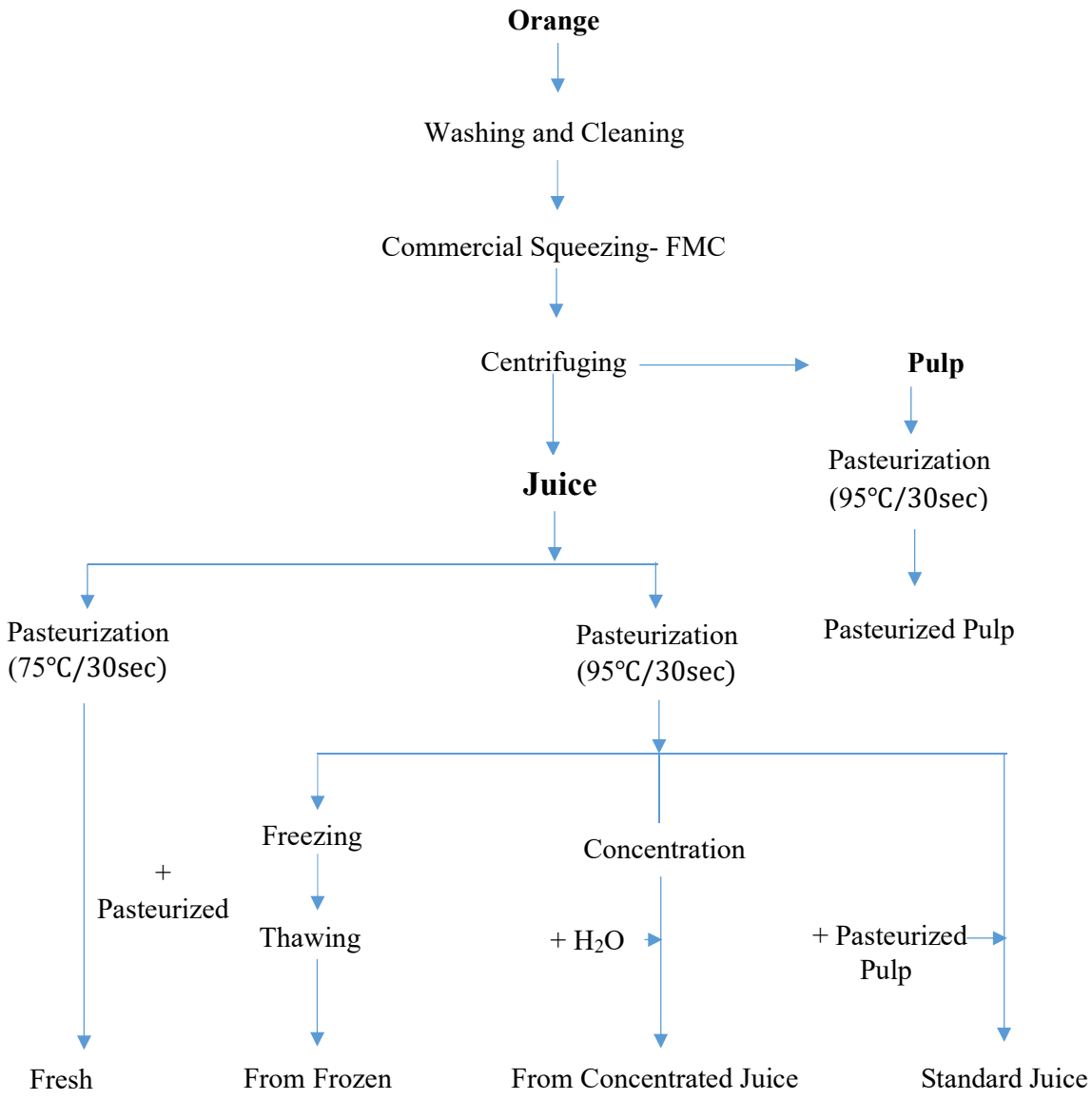


Figure 1-1. Scheme of the commercial orange juice processing (Gil-Izquierdo et al., 2002)

Pasteurization of OJ caused a loss of 58% of the initial ascorbic acid, 20-35% of total phenol, 25-30% of total flavanones, and OJ processed from concentrated and frozen also responsible for the loss of total phenols and flavanones (Gil-Izquierdo et al., 2002). Nonthermal processing such as High pressure and plus electric field are the new technique can minimize the loss of nutrient during processing of fruit juice. High-pressure treatment led to an increased the carotenoid

release (53.88%) and vitamin A value of 38.74% during OJ processing (Sánchez-Moreno et al., 2005), but their industrial use quite limited (Gil-Izquierdo et al., 2002).

A new technique of processing of high dietary fiber containing juice with minimum particle size is wet milling. Processing of juice pulp or peel in micro level particle size is known as Miro Wet Milling. This technique is quite known for processing of cereal milling or developing new beverage from rice (Koyama and Kitamura, 2014). The application of MWW for processing of concentrated juice from whole fruit will minimize the loss of fruit pulp and enhanced the nutritional content of the juice. Thus in a study of OJ processing, it is necessary to investigate the feasibility of MWM and its effects on the processing of OJ in relation to the retention of color and nutrient composition.

1.2.1 Current situation and limitation of drying of concentrated orange juice

The better preservation of fruit juice might be in powder form due to the fruit juice powder have many benefits and economic potential over their liquid counterparts such as reduced volume or weight, reduced packaging, easier handling and transportation and also enhanced the shelf-life of the product. Fruit juice powder is convenient for instant use in liquid preparation and also other uses like seasoning blends, confectionery, and pharmaceutical.

Drying is the unique method for producing fruit juice powder. There are different drying techniques widely used for producing fruit juice powder namely air, freeze, vacuum and spray drying. However, dehydration of fruit juice is complex, the technique of drying fruit juice is very specific for each drying method (Brennan et al., 2007). However, air drying is the simplest method for producing fruit powder, but the quality of the product is fairly poor, particularly as the product from the heat sensitive material.

Freeze-drying (FD) is the best method for producing high-quality fruit juice powder. Freeze drying is the process of free water separation by ice formation or the total solidification of the original solution and the ice sublimation at a very low temperature (-40 to -85℃) and very low

total gas pressure (high vacuum). The limitations of FD are the batch process, long processing time, low productivity, as a result, the operational cost is higher. According to Kitamura and Yanase (2011) the product cost of FD is 10 times higher than spray dried products.

Spray drying (SD) is widely used to the transformation of feed from a liquid or slurry to form a dry powder. During SD the liquid feed is atomized into a chamber where spray mixes with hot air which removes the moisture resulting in mostly amorphous (glassy) solid or powder produced (Shrestha et al., 2007a). The schematic diagram of spray drying process as shown in Figure 1-3. One of the main indices of spray dryer efficiency is the product recovery. Low productivity of SD is due to the adhesion of dried material into the wall. Long time retention of dried powder on the wall or frequent cleaning or collection is not cost effective or not able to get good quality of powder (Goula and Adamopoulos, 2010). A sticky product like fruit juice that contains higher sugar and acid is difficult to spray dry. During drying all the amorphous material change from the glassy to rubbery state at a glass transition temperature, that is specific for each material. The stickiness of the product occurred due to the low glass transition temperature or consequently higher drying temperature (Goula and Adamopoulos, 2010). To overcome the stickiness problems, various drying aids used for producing free-flowing powders such as maltodextrin, gum Arabic, soybean protein, dextrin (Chegini and Ghobadian, 2007, 2005a). The addition of carrier agent before atomizing is widely used to increase the glass transition temperature (Tonon et al., 2008). Another drawback of spray drying of orange juice is the loss of heat-sensitive components like ascorbic acid and antioxidant constituents, due to the high heat treatment (150-250℃) (Goula and Adamopoulos, 2006; Johnston and Hale, 2005; VILLOTA and KAREL, 1980). As a result of such investigations, depending on the quality of the final product and other considerations, a suitable and effective drying technique can be chosen.

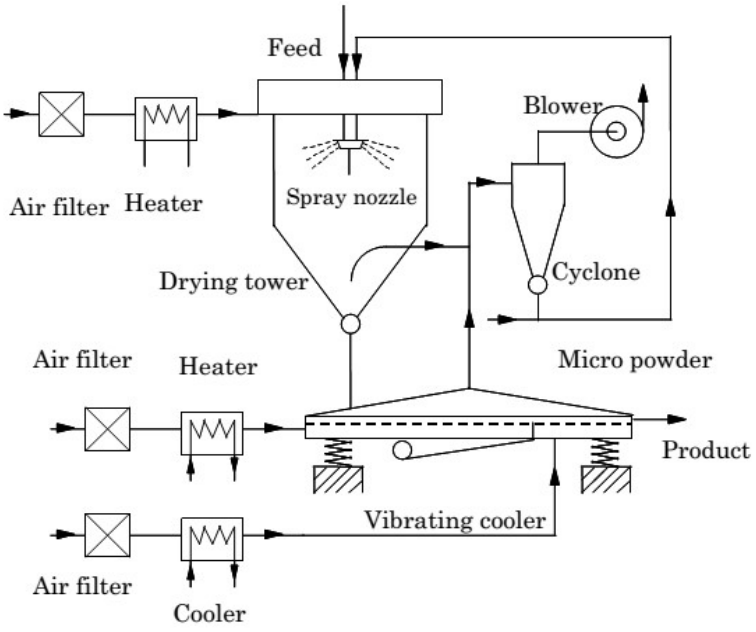


Figure 1-2. Schematic of spray drying process (Matsuno et. al. 1991)

Spray drying under low pressure or in a vacuum condition is a new technique. In addition, the superheated steam as a heating medium instead of hot air is another new approach. Superheated steam drying has been known for over 100 years (Pronyk et al., 2010), but it's application in spray drying under vacuum condition i.e. vacuum spray drying with superheated steam is unknown. The superheated steam that has additional sensible heat added so that its temperature is above the saturation temperature at a given pressure. The higher enthalpy of the superheated steam can transfer heat very quickly from the product to be processed. The benefits of superheated steam drying (SSD) are: superheated steam drying can lead to saving energy as high as 50 to 80% over the use of hot air (F., 2010). According to Alfy et al. (2016) the major advantages of superheated steam drying can produce a product with better quality in terms of color, flavor, shrinkage and rehydration characteristics. SSD can also reduce the oxidation, deodorization of the product, and higher energy efficiency.

Vacuum spray drying (VSD) is a new technique applied to powderization of liquid food containing thermosensitive ingredients under low temperature (40-60℃) and low pressure (3 to 5 kPa) as compared to the existing methods as shown in Figure 1-4.

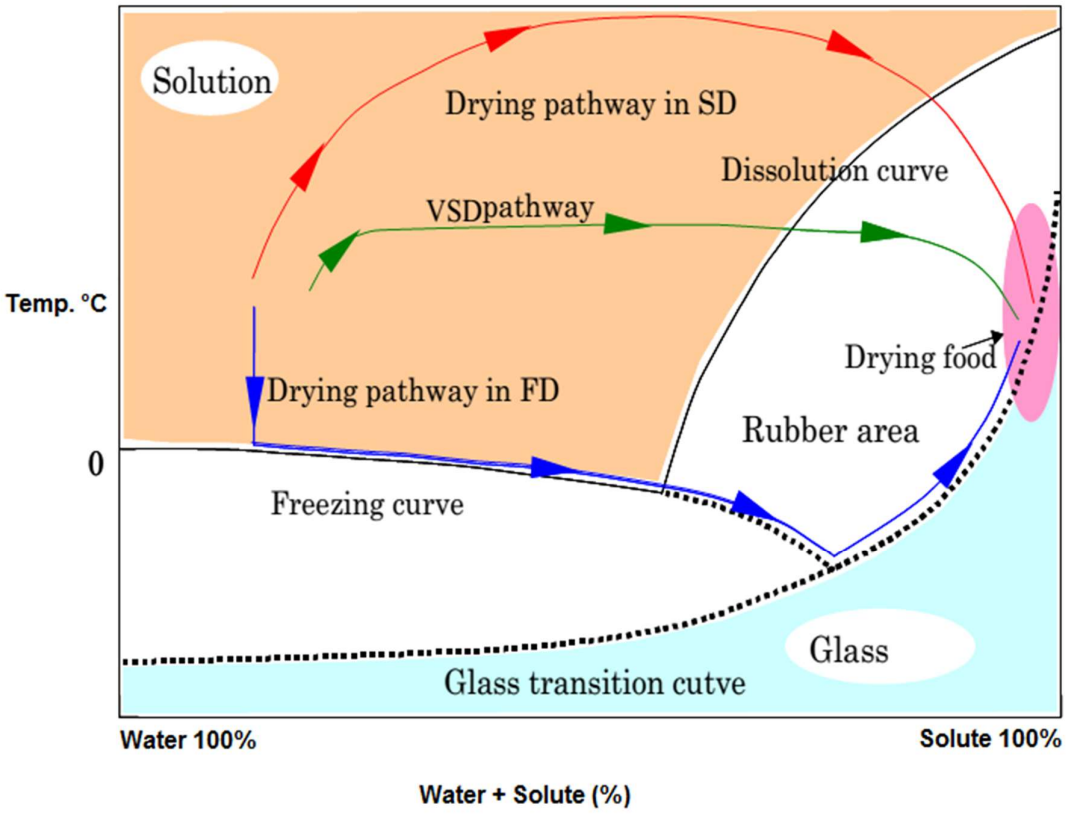


Figure 1-3. Model food state diagram (Takai, 2000)

The major food friendly drying methods with their features are shown in Table 1-2. Previously Kitamura and Yanase (2011) developed vacuum spray drying system for drying of probiotic powders, where the drying temperature was controlled by a far-infrared heater and a heating system with an outside jacket. The same year, Semyonov et al. (2011), conducted ultrasonic vacuum spray drying of probiotic powders, But the there is no literature of the application of superheated steam as a drying medium and producing heat-sensitive fruit juice powders.

Table 1-2. Features of the major food friendly dryer

	VSD	SD	FD
Heat Source	Superheated steam	Hot air	Freezing (cold air)
Temp. of heat source (℃)	150~250	200-400	-40 ~ -50
Product Temp. (℃)	33-48	<100	-40
Operating pressure	3~5 kPa	below atmospheric pressure	4 Pa
Driving method	continuous operation	continuous operation	batch operation
Thermal alteration	less	more	Less
Oxidation	less	more	Less
Processing volume	less	more	Less
Milling process	none	none	necessary
Energy efficiency	high	high	Low
Equipment price	minimum	cheaper	High

Source (Kitamura and Yanase, 2011)

Pre-treatment before drying of food powder is well known to reduce the color, flavor and higher retention of antioxidants. Similarly, the stability study of the product during storage plays an important role in the food processing industry. The quality of the fruit juice powders changes over time due to the shrinkage, caking or degradation of functional components when stored in undesirable conditions. As a result, the need for a stability study of OJ powder is important in establishing the effects of storage conditions.

1.3 Research objectives

The experiments designed for this study aims to process the concentrated orange juice powder by the application of micro wet milling and vacuum spray drying as a new technique.

In chapter 2, Moisture sorption isotherm and glass transition phenomenon of vacuum spray dried concentrated orange juice powder in detail. The feasibility investigation of vacuum spray drying of concentrated commercial orange juice with maltodextrin as a carrier agent.

In chapter 3, will discuss the process development for Micro Wet Milling (MWM) of orange juice with pulp and quality evaluation. Finally, a brief comparison with commercial orange juice is carried out.

In chapter 4, Analysis of physicochemical and antioxidant properties of MWM orange juice powder by vacuum spray drying and spray drying. A comparative study will be conducted in details. This chapter also discusses the suitable drying conditions for MWM orange juice with maltodextrin as drying aid.

In chapter 5, the investigation of the storage stability of the vacuum spray dried orange juice powders, in terms of degradation kinetics of ascorbic acid, color characteristics, and total antioxidant activity. Finally, the stability of MWM orange powders will be discussed based on the water activity and glass transition temperature.

In chapter 6, the conclusion and recommendation of our study will be summarized.

Chapter 2. Moisture sorption isotherm and glass transition phenomenon of vacuum spray dried concentrated orange juice powder

Vacuum spray drying is a new technique used to produce concentrated orange juice powder using maltodextrin as a drying agent. The dryer was developed for the low-temperature (40-50°C) drying powderization of liquefied food using superheated steam (200°C) as a heating medium. The physical properties of orange juice powder with four different combinations of juice solids to maltodextrin solids at 60:40, 50:50, 40:60, and 30:70 were determined. The moisture content, hygroscopicity, water activity, particle size, particle morphology, were significantly affected by the maltodextrin concentration and drying conditions. Highest product recovery as cyclone recovery was obtained from OJ/MD 30:70 powders. Moisture sorption isothers were constructed by isopiestic method, while thermal transition of the powder at different water activity levels ($a_w = 0.11 - 0.86$) was predicted by DSC. The data obtained were well fitted to both BET and GAB models. A strong plasticizing effect of water on Tg was predicted by the Gordon-Taylor model, where Tg was greatly reduced by the increasing moisture content of the powder.

2.1 Introduction

For many years, water activity is a widely-accepted concept and important to evaluate the product quality and stability than the total amount of water. The available or the free water presence in food product influences the microbial growth and enzymatic/or non-enzymatic activities (Rahman and Labuza, 1999). Expressing the relation between water activity and equilibrium moisture in a graphical form is known as sorption isotherm. The sorption isotherm is an important thermodynamic tool, specially for low moisture foods (Goula et al., 2008) and provides useful information for design, calculation of the food processing operations such as drying, packaging, and storage. Sorption isotherm commonly expressed based on empirical and/or theoretical models, those models parameters determine how water strongly bound to the solids or the amount of water that a material hold if it is exposed to air a certain relative humidity and temperature (Edrisi Sormoli and Langrish, 2015). The glass transition temperature (Tg) is the temperature at which an amorphous system changes from glassy to rubbery state. For a given substance, the fraction of water content, molecular weight, and nature of dry matter can describe by the glass transition temperature (Roos, 1995; Slade and Levine, 1991).

Spray drying is widely used to transform liquids into shelf-stable products. Spray dried powder have many benefits and economic potential over the liquid counterparts. Fruit juice powder can easily handle and transfer due to its reduced weight or volume as well as a reduced packaging system and have a much longer shelf life (Shrestha et al., 2007a). Spray drying parameters affect the quality of spray dried powder and drying condition they were the best way to describe the quality change factors of orange products (Brennan et al., 2007). However, the major drawbacks and complex in spray drying of orange juices is the matter of stickiness and flow problems of the powder (Tonon et al., 2008;Goula and Adamopoulos, 2010). Fructose, glucose, and citric acid are the main components of orange juice with very low Tg values (5, 31 and 16°C, respectively) in a pure, dry state, which decreases drastically when moisture is absorbed. During spray drying, fruit juice forms an amorphous solid or a syrupy/sticky powder; sticky products

are typically produced because the low glass transition temperature (Tg), high viscosity, low melting point and high hygroscopicity (Adhikari et al., 2003) lead the juice components to adhere to the drying chamber and agglomerate on the conveying system (Bhandari and Howes, 2005).

To overcome the stickiness problem, various methods that are able to produce free-flowing fruit juice powder have been suggested: using an adjunct or a carrier agent (maltodextrin, gum, starch or gelatin) as an additive in the feed material during spray drying (Saénz et al., 2009), scrapping the drying surfaces, spray freezing or cooling the drying chamber wall (Chegini and Ghobadian, 2005b; Chegini et al., 2008). Carrier agents with higher molecular weights increase the glass transition temperature and reduce the hygroscopicity and stickiness of the powder, which may, in turn, increase the final product yield and efficiency (Lee et al., 2013). Goula and Adamopoulos (2010) produced spray dried concentrated orange juice powder by using dehumidified air as drying medium and maltodextrin DE 6, DE 12, DE 21 as a carrier agent. Shrestha et al. (2007a) conducted spray drying of orange juice with various levels of maltodextrin solids as a carrier agent. For this reason, maltodextrin is commonly used as a carrier agent because it is relatively inexpensive and highly soluble in water with low viscosity and a bland flavor (Carolina et al., 2007; Saénz et al., 2009); it is also more efficient at protecting bioactive compounds from adverse conditions (Ferrari et al., 2012).

In the present study, a vacuum spray drying (VSD) method is proposed as a new technique for vacuum pressure spray drying of concentrated orange juice. The dryer was developed to turn liquefied food into a powder at a low temperature (40-60°C) drying using superheated steam as the heat source. Ultrasonic vacuum spray drying has been used to produce dried probiotic powder (Semyonov et al., 2011). Kitamura et al. (2009) and Kitamura and Yanase (2011) who were developed and conducted vacuum spray drying of probiotic foods at 40-60^{o}C. However, very few studies on the sorption isotherm and glass transition phenomenon of orange juice powder produced with other technique such as spray drying associated higher temperature, freeze drying

(Edrisi Sormoli and Langrish, 2015; Goula and Adamopoulos, 2010) are available but no data exists regarding this vacuum spray drying technique at a low-temperature treatment for orange juice powder.

Therefore, the objectives of this study were to produce orange juice powder by VSD with different combinations of maltodextrin; to physical properties of orange juice powders and to provide experimental data on water sorption isotherms and glass transition temperatures of the concentrated orange juice powder with a carrier agent for insight on powder stability. In order to predict the sorption characteristics by widely used BET and GAB models, and the Gordon-Taylor model used to determine the glass transition temperature and water plasticizing effect on the concentrated orange juice powder.

2.2 Materials and Methods

2.2.1 Raw materials

Concentrated orange juice was obtained from Ehime beverage Ltd., Japan. The basic composition of the concentrated orange juice was total soluble solid (TSS) 62±0.45%, citric acid 4.8-5.7%. Maltodextrin (MD) 12DE (Showa Sangyo Co. Ltd., Japan) with a moisture content of 4.15±0.02% was used as a carrier agent. Considering the TSS contents of concentrated orange juice (COJ) and maltodextrin solids at 60:40, 50:50, 40:60, and 30:70 (concentrated orange juice: maltodextrin) by weight were chosen for vacuum spray drying. A total of 1000 g of COJ and MD were prepared with 33 Brix% solutions for VSD. Moreover, in a previous work, it was found that the proportions of orange juice : maltodextrin by weight at 40:60, 35:65, 30:70 and 25:75 were used during spray drying at 160^{o}C Shrestha et al. (2007a). and Goula and Adamopoulos (2010) conducted spray drying of concentrated orange juice with matodextrin as carrier agent at a solid ratio of 4, 2, 1, and 0.25. The present study considered these conditions and also other higher concentration of orange OJ/MD 60:40, 50:50 by weight for vacuum spray drying.

2.2.2 Vacuum Spray Drying (VSD) System

The experimental VSD (Tanabe Engineering Corporation, Japan) diagram with its working principles is shown in Fig 2-1. The COJ/MD mixture is sprayed at 300 mL/h through a metering pump with an upward flow and atomized into the drying chamber by two fluid nozzles. The nozzles use compressed air at a rate of 40 N-L/min to atomize the mixture into droplets with small diameters of 10-50 μm. Heat exchange occurs as the atomized droplets come into contact with steam from another steam nozzle that has been superheated to 200°C by an electric heater. The latent heat required to evaporate water in the droplet is supplied by the sensible heat of the superheated steam. Therefore, superheated steam, whose thermal capacity is higher than that of dry air, can supply more heat capacity per unit area. However, an average droplet is not completely dried instantly, so the product temperature does not reach the saturated steam

temperature, which is 40°C for a vacuum of approximately 5 kPa in the evaporator. In addition, the main body of the drying chamber is decompressed by a vacuum pump, and the evaporator jacket temperature is maintained at 50°C by a supply of hot water. The dry powder it is completely captured by the first or second cyclone. To prevent the formation of wall deposits and classification in the cyclone, a hot water-2, and a return hot water-2 at the end of the first cyclone were supplied. The low-pressure dry air was used to collect the powder in a receiver at 45 ºC. However, superheated steam can greatly reduce the capacity by cooling with a condenser by supplying cool water; the steam can then be collected as water in a water reservoir, and the vacuum pump can be downsized to more easily maintain the pressure.

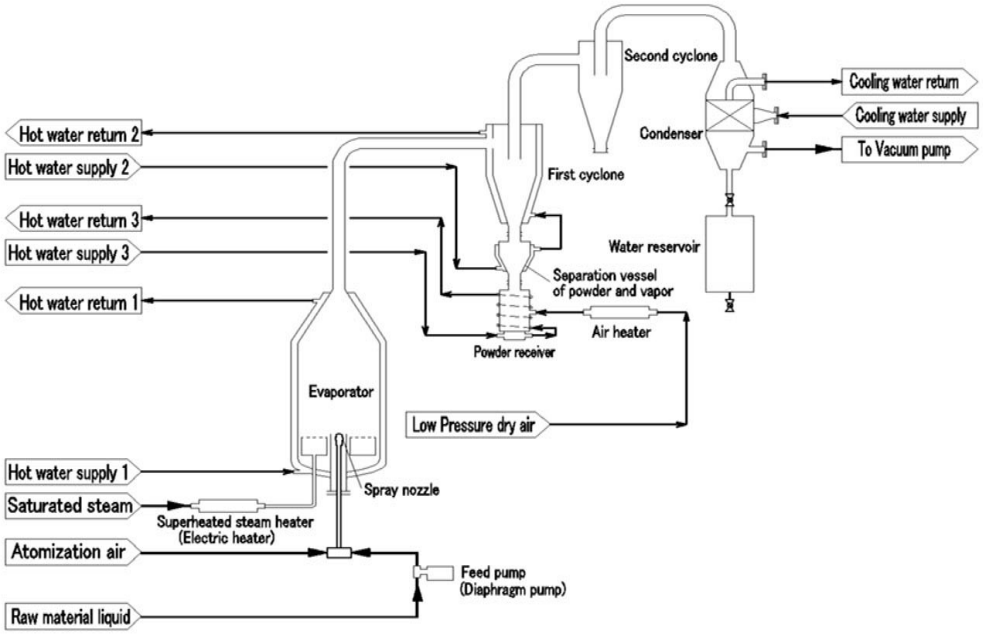


Figure 2-1. Schematic flow diagram of the vacuum spray drying

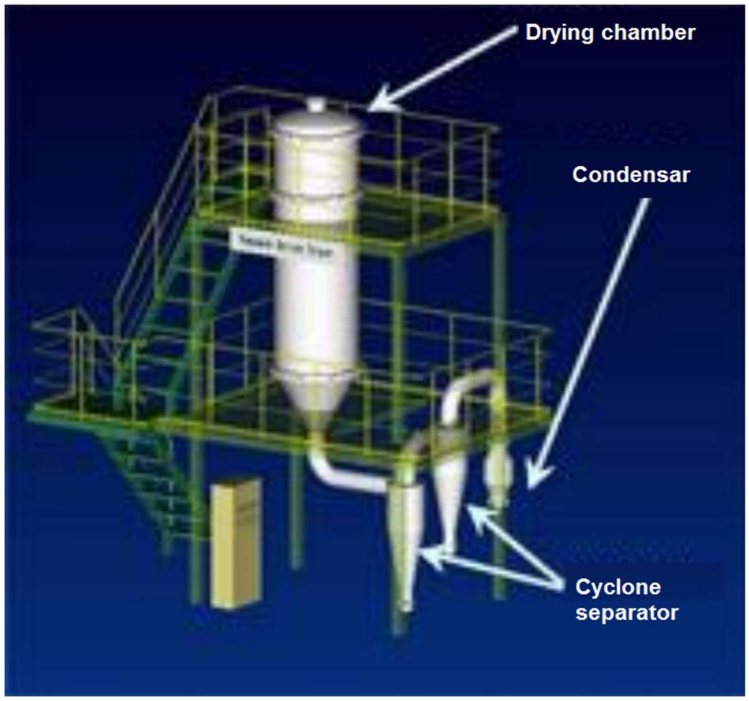


Figure 2-2. Vacuum spray dryer

Source: https://www.tanabe-ind.co.jp/en/VSD-development-en?cat=215 (2016)

Image 1. Vacuum Spray Dried Commercial Orange Juice powders

2.2.3 Assessment of the physical properties of orange juice powder

2.2.3.1 Moisture content

The moisture contents of the orange juice powder were determined by drying in an oven at 70°C until consecutive constant weights were obtained, followed by 2 h intervals and which gave variation less than 0.3%. Moisture content expressed as % of moisture in wet basis (Goula and Adamopoulos, 2010).

2.2.3.2 Bulk density

The bulk density of the orange powder was measured by gently adding 2 g of powder to an empty 10 mL graduated cylinder and holding the cylinder in a vibrator for one minute (Goula et al., 2004). The volume was then recorded and used to calculate the bulk density in g/mL.

2.2.3.3 Rehydration

Rehydration of orange powder was determined according to the method described by Goula and Adamopoulos (2010) with slight modifications. For measurement 2 g of powder were added in a 50-mL distilled water at 26°C in a 100 mL low form glass beaker. The mixture was agitated in a digital hot plate/stirrer at 900 rpm, using a magnetic stirrer bar with a size of 2 mm × 7 mm time required in a sec for the powder to be completely rehydrated was recorded.

Figure 2-3. Hot plate with magnetic stirrer for determination of rehydration time

2.2.3.4 Hygroscopicity

For hygroscopicity, 1.5g of the orange juice powder was kept in Conway unit (Model: 060310-02A, Shibata Co. Ltd., Tokyo, Japan) containing saturated salt solution of NaCl (75.29% RH). Samples were weighed after one week and hygroscopicity was expressed as the weight of the hygroscopic moisture per 100 g of dry solid (Cai and Corke, 2000).

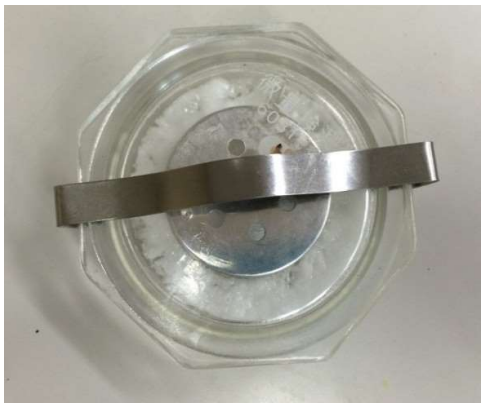


Figure 2-4. Conway unit (SHIBATA Co. Ltd, Japan)

2.2.3.5 Water activity

The water activity of the orange powder was determined with the standard water activity of the saturated salt solution such as Lithium Chloride (PubChem CID: 433294), Potassium acetate (PubChem CID:517044), Magnesium Chloride (PubChem CID: 5360315), Magnesium nitrate hexahydrate (PubChem CID: 202877), Strontium chloride hexahydrate (PubChem CID: 6101868), Sodium chloride (PubChem CID: 5234), and Potassium Chloride (PubChem CID: 4873) of 0.11 0.22, 0.33, 0.53, 0.71, 0.75 and 0.84water activities respectively at 25°C (A. ArabHosseini, 2005); Greenspan, 1977; (Young, 1967) by Conway unit (Model: 060310-02A, Shibata Co. Ltd., Tokyo, Japan) and the graph intersection method described by Kitamura and Yanase (2011).

One gram of powder was kept in an aluminium case with aluminium foil and weighed. Then 3 g of each salt placed into the Conway unit. After that, a few drops of water were added in the

salt and the sample containing aluminum case kept inside of the Conway unit and conditioned with the saturated salt solution. Finally, the lid of Conway unit was covered and kept in 25°C for 2h conditioned with the saturated salt solution. After 2 h, the weight of the sample containing aluminum case was measured. Then the differences of sample weight (Y-axis) were plotted against the known water activity of the saturated salt (X-axis). A straight line was drawn from each data set and intersection with the X-axis, which corresponds to zero sample weight variation, and which was defined as the water activity of the sample as shown in Figure 2-5. For each of the sample, triplicate measurements were conducted.

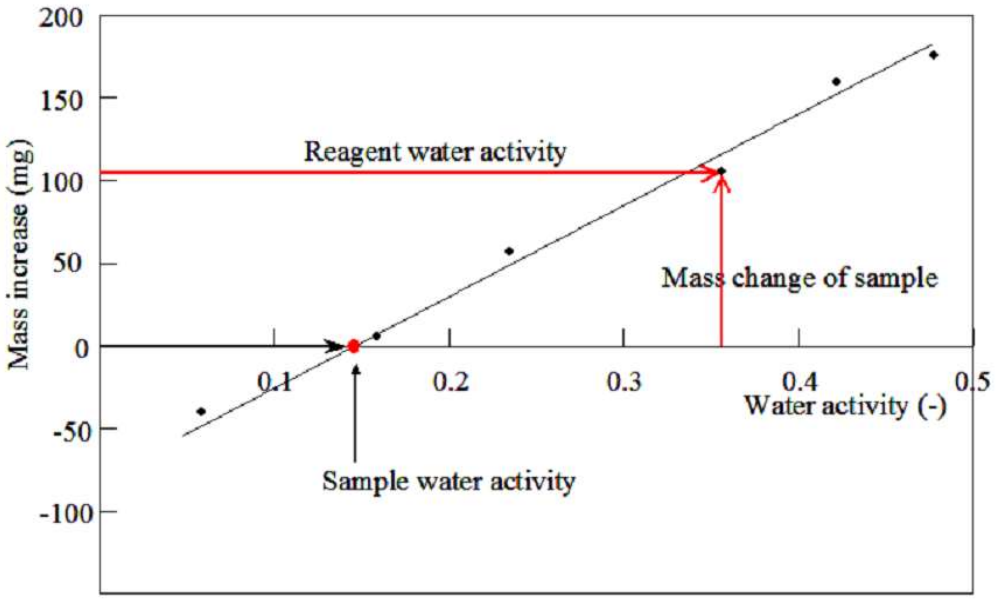


Figure 2-5. Extraction of water activity (Kitamura and Yanase, 2011)

2.2.3.6 Particle size and particle size distribution

Particle size (D50 and D75) and its size distribution were determined by a laser diffraction particle size analyzer (SALD-2200, Shimadzu Corporation, Japan) in dry measurement mode and according to (Koyama and Kitamura, 2014). Working conditions are as follows: sample suction type is hand shot; Pressure is at 0.6-0.8 kPa, filtering rate is 5μm or larger; Operating

temperature and the humidity are 25ºC and 60% respectively, and particle size was taken as D50 and D75 values expressed as μm. D50 is defined as the median diameter, and D75 is the particle size corresponding to 75% of the particles being under size by mass. The particle size distribution profile was constructed by the particle size and frequency of the distribution.

2.2.3.7 Particle morphology

Particle morphology was evaluated by field emission scanning electron microscopy (FE-SEM) according to (Mishra et al., 2014a) and slight modifications. The powders were mounted onto stubs with double-sided adhesive tape, and the samples were coated with a thin layer of gold under vacuum. Then, the samples were examined on an FE scanning electron microscope (SU8020, HITACHI, Japan) at 5 kV and 5000-fold magnification. The taking microphotographs were carried out with a camera coupled to the microscopic (Cano-Chauca et al., 2005)

2.2.3.8 Water sorption isotherm

The equilibrium moisture contents of the orange juice powder were evaluated at different values of water activity by isopiestic method Rockland (1987). Seven saturated salt solutions were prepared as mentioned before. One gram of each of the three orange powders were weighed in aluminum vials and equilibrated in the saturated salt solutions in a desiccator at 25°C with H_2SO_4 to maintain proper relative humidity. Sample equilibration took 3-4 weeks based on the change in weight, which did not exceed 0.01%. The equilibrium moisture content was determined by oven drying at 70°C unit a constant weight was obtained (AOAC, 1990).

Sorption isotherms are generally explained by the Brunauer–Emmett–Teller (BET) (Mrad et al., 2012) and Guggenheim–Anderson–de Boer (GAB) (Bizot, 1983) mathematical models based on the following empirical and theoretical parameters:

BET Model: $$X_e = \frac{X_m C_{BET} a_w}{(1-a_w)(1-a_w+C_{BET} a_w)} \quad (1)$$

GAB Model: $$X_e = \frac{X_m C_{GAB} K_{GAB} a_w}{(1-K_{GAB} a_w)(1-K_{GAB} a_w+C_{GAB} K_{GAB} a_w)} \quad (2)$$

In the present study, sorption isotherm data were fitted to the BET and GAB models and analyzed by Origin Pro 8.5 software. The goodness of fit was evaluated by determining the coefficient R^2, and mean deviation modulus was calculated by the following formula (Goula et al., 2008):

$$M_e = \frac{100}{N} \sum_{i=1}^{N} \left| \frac{V_e - V_p}{V_e} \right| \qquad (3)$$

2.2.3.9 Glass Transition Temperature

The glass transition temperatures, Tg, of the orange powder were determined by differential scanning calorimetry (DSC) (DSC-60, Shimadzu Corporation, Japan) according to Shrestha et al. (2007a) . Five to ten grams of the orange powder were scanned in a hermetically sealed 20 µL DSC aluminum pan. All samples had previously been equilibrated over a saturated salt solution with different relative humidities at 25°C. An empty aluminum pan was used as a reference. The rate of thermal scanning was carried out in the following order: (1) isothermal at -30°C for 1 min, (2) heating at a rate of 10°C/min from -30°C to a temperature just over the apparent or predetermined Tg, (3) rapidly cooling at 50°C/min to -30°C and (4) heat scanning at a rate of 10°C/min from -30°C to 200°C. All analyses were conducted in triplicate. The midpoint temperature of the DSC thermogram was considered the transition temperature (Goula et al., 2008).

The glass transition temperature of a binary water-solid mixture depends on the plasticizing effects of the water and is described by the Gordon-Taylor model (Gordon and Taylor, 1952).

$$Tg = \frac{(1-x_w)\cdot Tg_s + k\cdot x_w \cdot Tg_w}{(1-x_w)+k\cdot x_w} \qquad (4)$$

Where Tg, Tg_s, and Tg_w are the glass transition temperatures of the mixture, solids, and water, respectively, x_w is the mass fraction of water, and k is the Gordon-Taylor parameter.

2.3 Results and discussion

2.3.1 Physical properties of orange juice powder

To evaluate the physical conditions of the powder, water activity (a_w), moisture content, glass transition temperature (Tg) and product recovery were determined as shown in Table 2-1. The moisture content of the powder had a great impact on the flowability, stickiness and storage stability because of its plasticizing effects and crystallization behavior. The availability of free water in a food component is expressed as water activity, which is responsible for microbiological and biochemical reactions. All of the powder samples had water activity (a_w) values of 0.25±0.00-0.25±0.01 and moisture contents of 2.29 to 3.35%. whereas (Shrestha et al., 2007a) found the wetness of the powder was high as indicated the moisture contents and water activity of 4.3 - 4.5% and 0.30 to 0.40. The present research had the lower moisture content and water activity than spray dried of concentrated orange juice by Goula and Adamopoulos (2010). This was due to the higher moisture removal during vacuum spray drying, and where a greater the heat gradient occurred in VSD between the steam and the particles than hot air so that there was a higher heat transfer into the particles, which supplied the driving forces of the moisture removal. The moisture contents and other parameters of powder produced by a COJ/MD ratio of 60:40 were not evaluated because of the stickiness, low glass transition temperature of this powder. The moisture content and water activity (a_w) of powder decreased with increasing maltodextrin concentration likely because the sugar and acid in juice-rich powder are highly hygroscopic and maltodextrin increases the total solid of the feed and reduces the amount of water evaporation. This result agrees with the findings of Shrestha et al. (2007a) in spray-dried orange juice powder. The water activity values of all powder samples were below 0.30, which improves powder stability. The glass transition temperature Tg has a great impact on the surface stickiness of the amorphous powder during spray drying (Lloyd et al., 1996). The Tg values of orange juice/maltodextrin powder were investigated by DSC. The characteristic parameters Tg_{onset} and $Tg_{midpoint}$ (^oC) were observed from the DSC thermogram as shown in Table 2-1. The

glass transition temperature of the powder increased with increasing the amounts of maltodextrin solids because of the higher molecular weight of maltodextrin. Roos and Karel (1996) stated that the similar phenomenon, Tg value of binary mixture (sucrose:maltodextrin) increases with increasing the maltodextrin concentration. Bhandari and Howes (2005) mentioned that the recovery of spray dried sugar rich product was the direct functions of the temperature and moisture of the outlet air. The increases in the maltodextrin solids also increased the cyclone recovery because of low Tg sticks or collapses occur during drying, that's why in the present study OJ/MD 60:40 was failed due the low Tg (39.76°C) and higher moisture content ($\geq 4\%$) . In general, several authors stated that the recovery of feed solid in the final product increases with increasing the maltodextrin (Bhandari and Howes, 2005; Papadakis and Bahu, 1992). The pattern of the findings was similar to Shrestha et al. (2007a). The highest cyclone recovery, 63.3%, was obtained with 30:70 COJ/MD powders, which had the highest Tg of approximately 86.63°C, and the lowest recovery of 53.0% was observed with 50:50 powder at 61.78°C. These results differed from Shrestha et al. (2007a), who found that 50:50 orange juice/maltodextrin powder had Tg values of 66.4°C and no powder output in the cyclone. These differences are caused because the present study was conducted at a low temperature in a vacuum condition and superheated heated steam, as the drying medium, which has a higher heat capacity than dry air, as a result the COJ/MD 50:50 powders were produced at a low Tg (61.78°C).

Table 2-1. Physical properties of an orange juice powder

Types of Powder COJ/MD	Moisture %	Water activity	Glass Transition Temperature Tg °C		Cyclone recovery, %
			Tg_{Oneset}	$Tg_{midpoint}$	
50:50	3.35 ± 0.28^{a}	0.25 ± 0.00^{a}	56.42 ± 1.35^{c}	61.78 ± 0.97^{c}	53
40:60	2.57 ± 0.12^{b}	0.17 ± 0.02^{b}	68.01 ± 1.52^{b}	75.94 ± 0.92^{b}	60.5
30:70	2.29 ± 0.16^{bc}	0.15 ± 0.01^{b}	79.35 ± 1.28^{a}	86.63 ± 0.97^{a}	63.3

The values are mean ± S.D of three independent determinations. The means with different superscripts in a column differs significantly ($p\leq 0.05$)

Table 2-2 shows the hygroscopicity of the VSD powder in relations with the maltodextrin concentration. Hygroscopicity of powder decreased from 0.195±0.02 to 0.143±0.01 gH_2O/g with increasing maltodextrin concentration. This was in an agreement with Tonon et al. (2008). This trend was also similar to that of Cai and Corke (2000) and Rodríguez-Hernández et al. (2005) for spray-dried Amaranthus and cactus pear juice powder. According to Table 2-2, it is indicated that by increasing the maltodextrin, the solubility of the powder increases and rehydration index were greatly reduced. Because the maltodextrin has superior solubility in water (Cano-Chauca et al., 2005), the fast rehydration may be due to the low moisture contents of the powder (Goula et al., 2008).The solubility index of the potato flour was increased with increasing the maltodextrin solids (Grabowski et al., 2006). The bulk densities of the powder were not significantly different at $p \leq 0.05$. The determined bulk density of VSD orange juice powder agreed with that of Chegini and Ghobadian (2005b).

Table 2-2. Physical properties of orange juice powder (ii)

Types of Powder COJ/MD	Hygroscopicity, gH_2O/g	Rehydration, Sec	Bulk density g/mL	Particle size, µm	
				D50	D75
50:50	0.195±0.02[a]	253.65±1.05[a]	0.70±0.03[a]	7.75±0.25[a]	12.84±.62[a]
40:60	0.188±0.03[b]	237.40±1.25[b]	0.72±0.02[a]	6.36±0.45[b]	8.69±0.21[b]
30:70	0.143±0.01[c]	122.34±1.56[c]	0.73±0.02[a]	6.02±0.16[c]	7.68±0.26[b]

The values are mean ± S.D of three independent determinations. The means with different superscripts in a column differs significantly ($p \leq 0.05$)

Table 2-2 shows that the VSD orange juice powder particle diameters were significantly different at $p \leq 0.05$. The median (D50) values of COJ/MD 30:70 powders had the lowest peak with a smaller volume distribution and smaller particle diameter as shown in Figure 2-6. Powder containing a high amount of orange juice are rich in acid and sugar, which imparts a high hygroscopicity and stickiness to the liquid feed; as a result, larger particles are produced during spray drying ((Shrestha et al., 2007a). Powders with 50:50 COJ/MD had significantly (at $p \leq$

0.05) higher particle size and size distributions. These trends are similar to the findings described by (Shrestha et al., 2007a); however, powder particle sizes in the present study were much smaller because of the difference atomization at a lower temperature. Tonon et al. (2011) stated that when the inlet temperature is low, the particle remains shrunk and maintains a smaller diameter, whereas at higher temperatures, faster-drying rates, and higher swelling form larger particles. D75 powder particles with OJ/CMD ratios of 40:60 and 30:70 were not significantly different at $p \leq 0.05$.

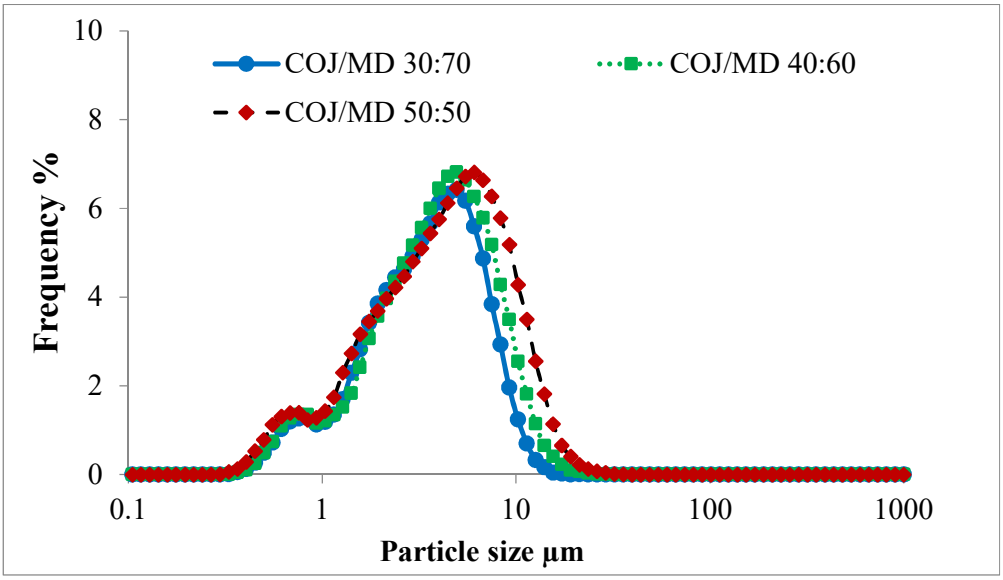
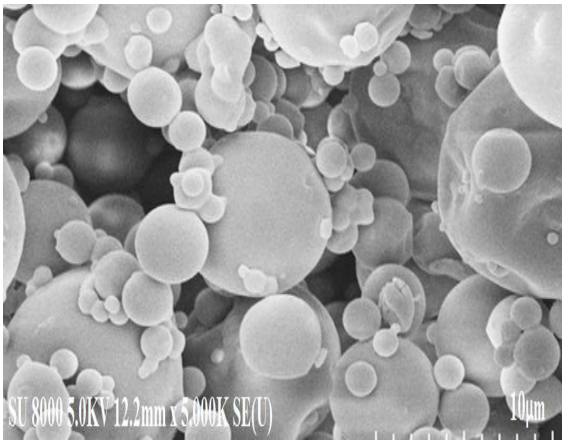


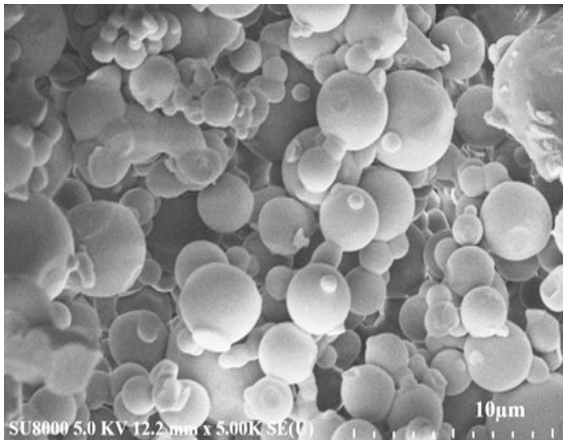
Figure 2-6. Particle size distributions of VSD orange juice powder.

The micrograph shows that the morphology of the particles depends on the concentration of the carrier agent and the drying conditions Figure 2-7. The 30:70 COJ/MD powder produced with higher amounts of maltodextrin had the smoothest surface with smaller spherical shapes and no shrinkage. This agrees with the results published by Ravindra et al. 2013, who stated that a higher maltodextrin concentration was more susceptible to shrinkage during the spray drying of white-flowered *O. stamineus* plant extract and observed that the particle surface becomes smoother when the maltodextrin concentration increases from 0.53% to 10.67%. The 40:60 COJ/MD

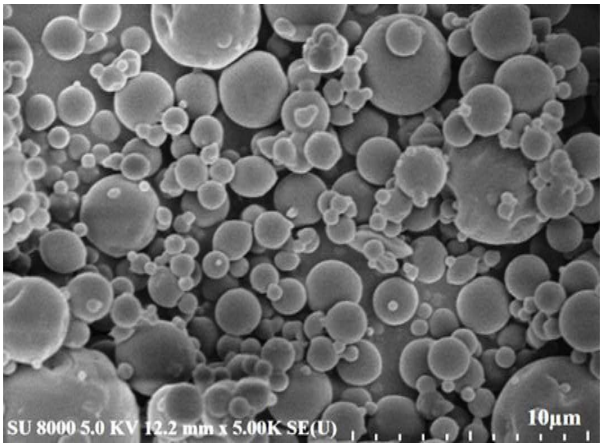
powder had smoother spherical shapes, but some smaller particles adhered to the larger ones, making the particle size larger than that of the 30:70 COJ/MD powder. The VSD powder with 50:50 COJ/MD had dented surfaces with wrinkles and deformation compared to the other powder.



COJ/MD 50:50



COJ/MD 40:60



COJ/MD 30:70

Figure 2-7. SEM micrograph of VSD orange juice powders

2.3.2 Moisture sorption isotherm of orange juice powder

Water sorption isotherms were constructed for the vacuum spray-dried orange juice powder, and the experimental moisture sorption data for the equilibrium moisture contents (EMC, X_e g H_2O/g of dry solid) are given in Table 2-3. With increases in water activity (a_w), the equilibrium moisture contents (X_e) of the powder also increased at a constant temperature. The multilayer

sorption region at low and intermediate water activity, the moisture content increase linearly, whereas at capillary condensation region at higher water activity levels, the moisture content increases rapidly with water activity. The nature of the sorption isotherm can be described by the phenomenon that the physical sorption of the active sites occurs by proteins, whereas the water can be sorbed only to surface OH sites of crystalline sugar (Falade and Aworh, 2004).

Table 2-3. Equilibrium moisture content (EMC) of orange juice powder at different water activity level.

Water activity (a_w)	Equilibrium moisture content, EMC (gH_2O/g of dry solid)		
	COJ/MD 50:50	COJ/MD 40:60	COJ/MD 30:70
0.11	0.0236 ±0.0002	0.0251±0.0003	0.0195±0.0008
0.22	0.0389±0.0003	0.0393±0.0001	0.0378±0.0011
0.33	0.0575±0.0001	0.0421±0.0012	0.0398±0.0017
0.53	0.0786±0.0011	0.0773±0.0001	0.0685±0.0019
0.71	0.1325±0.0013	0.1256±0.0015	0.1194±0.0004
0.75	0.178±0.0004	0.1585±0.00019	0.1507±0.0020
0.84	0.290±0.0012	0.2388±0.0010	0.2104±0.0013

The values are mean ± S.D of three independent determinations

Experimental data were also fitted to the GAB and BET models. A sharp increase in the equilibrium moisture content of orange powders observed in Figure 2-8 at higher water activity levels a_w > 0.52 at 25℃. The similar phenemonen reported by Kammoun Bejar et al. (2012) and Maroulis et al. (1988) for high sugar containing fruits such as orange peels and leaves, and raisins, figs, appricots, prunes, this was due to the dissolution of fruit sugar in the sorbed water vapour. The other authors mentioned that, the orange juice powder with high solid content consists more that 80% sugar with sugar content ratio of 2:1:1 sucrose:glusoce:fructose respectively showed the similar sorption behavior (Kelebek et al., 2009).

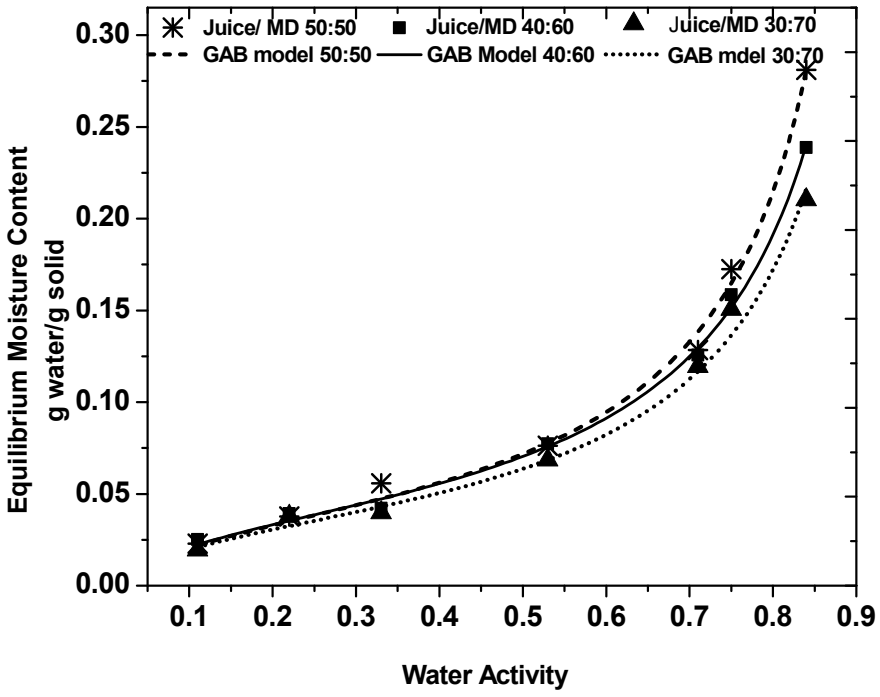


Figure 2-8. Sorption isotherm of VSD orange juice powder and the fitted GAB model

The regression data for the GAB and BET models are presented in Table 2-4. The coefficients of determination (R^2) and mean deviation modulus (M_e) were calculated to find the best fit model. The other model parameters such as X_m is known as the monolayer moisture content, is defined as the safest moisture content that strongly absorbs to active sites on the food surface at a given temperature. The strength of the binding for the water molecules to the primary binding sites on the product surface can be explained by the parameter c. The larger value of c represents the stronger bonds between the water molecules in the monolayer. The parameter k represents the values which determine the correction factors for multilayer molecules relative to the bulk liquid, such as when k =1, which describe the same characteristics of the molecules beyond monolayer and pure water (Quirijns et al., 2005). The GAB models resulted in X_m values of 3.54-4.03%. These results agree with (Tonon et al., 2009a) and (Caparino et al., 2013) for spray-dried Acai juice powder and freeze-dried mango powder, respectively. Whereas Edrisi Sormoli and

Langrish (2015) reported the molonayer moisture content of spary dried orange juice powders were in the ranges of 9.8 to 12.6 % at 20 to 50℃ temperature range. COJ/MD 50:50 powder exhibited the highest monolayer moisture water (4.03%) and also demonstrated the higher adsorption as shown in Figure 2-8. In respect to GAB parametes, COJ/MD 30:70 showed the highest c value (8.96) and lowest k value (0.98) whereas the powder 50:50 showed the higher k values and higher adsorption, this was due to the high hygroscopic nature of powder rich in orange juice solids. Conversely, powder with higher maltodextrin concentrations are less hydrolyzed and have fewer hydrophilic groups, causing them to absorb less moisture and which explains its lower hygroscopicity (Cai and Corke, 2000).

Table 2-4. Estimated BET and GAB model parameters for VSD orange juice powders

Model	Model Parameters	COJ/ MD 50:50	COJ/MD 40:60	COJ/MD 30:70
BET	X_m	0.0478	0.0402	0.0360
	C_{BET}	4.31	5.50	7.56
	R^2	0.99	0.99	0.98
	% Me	9.85	9.28	5.39
GAB	X_m	0.0403	0.0395	0.0354
	C_{GAB}	7.73	8.24	8.96
	K_{GAB}	1.02	0.99	0.98
	R^2	0.98	0.99	0.99
	% Me	6.63	6.92	8.41

The sorption data of the orange juice powders were also fitted to the BET model and typical curve as shown in Figure 2-11. The fitted parameters were presented in Table 2-11. The BET model consists of two parameters such as monolayer moisture X_m and the parameter c. The fitnes of the model was determined based on the parameter coefficients of determination (R^2) and mean deviation modulus (M_e). The X_m values of orange juice/maltodextrin powder range from 3.60 to 4.78% and c values showed in the ranges of 4.32 to 7.56. Whereas BET models don't have the third parameter and it's difficult to explain the sorption behavior of the product in the larger water activity levels ($0.05 < a_w < 0.9$) (Jonquières and Fane, 1998).

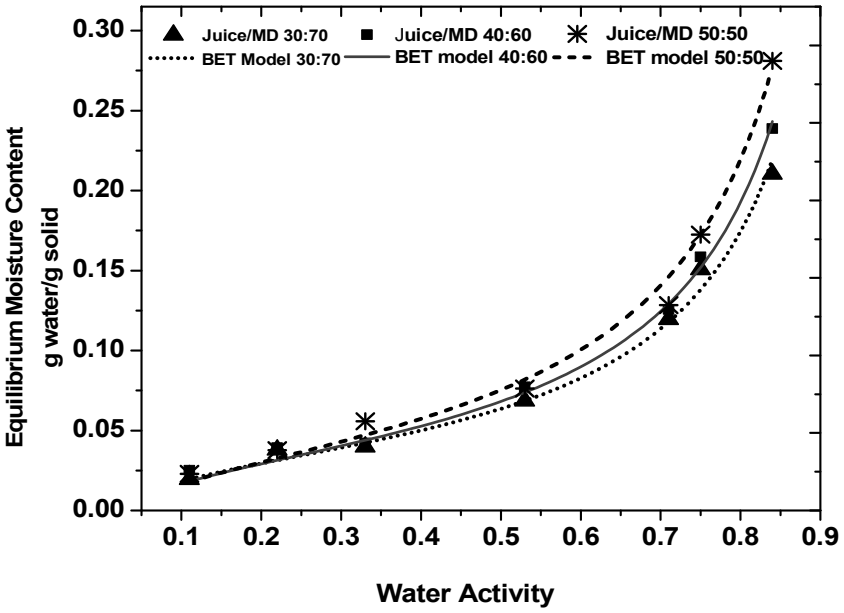


Figure 2-9. Sorption isotherm of VSD orange juice powder and the fitted BET model

The shape of the sorption isotherm Figure 2-10 and Figure 2-11, of the orange juice powder predicted by the BET and GAB models, showed a typical sigmoid curve and a type III (J shape) and typical of sugar-rich products according to Brunauer's classification. According to (Gabas et al. (2007) and Edrisi Sormoli and Langrish (2015) observed similar types of the curve for vacuum-dried pineapple juice powder with maltodextrin or gum Arabic as carrier agents and spray dried orange juice powder.

GAB models resulted in the highest R^2 and lowest M_e values, similar to Goula et al. (2008) and Tonon et al., (2009a) for spray-dried Acai juice and tomato pulp powder, respectively. Both the BET and GAB models had satisfactorily fitted values for powder produced by COJ/MD 30:70 powders. According to Table 6, GAB model was highly fitted to the experimental data of the orange juice powder, because this model has the third parameter k and it can predict over range of water activity (0.05 ~ 0.9) than BET model fails over water activity 0.5 (Jonquières and Fane,

1998); (Goula et al., 2008). The similar results reported for the sorption behavior of the spray dried orange juice powder (Edrisi Sormoli and Langrish, 2015).

The physical characteristics of orange juice powder were investigated during storage at 25°C with different water activities. According to Labuza and Altunakar (2007) the safest monolayer moisture content in the ranges of 0.2 to 0.3, the dehydrated products within this range having the maximum shelf life. The VSD orange powders showed the monolayer moisture within the reported ranges. The free-flowing powder had water activities below 0.53, and stickiness or agglomeration was observed above a_w of 0.53. At a relative humidity above 75%, collapse and liquefaction were observed in the powder.

2.3.3 Glass transition temperature

Several studies have shown that stickiness problems of amorphous solids during spray drying occurs as a result of particle plasticization, and this phenomenon can be explained by the glass transition temperature (Lloyd et al., 1996). The glass transition temperature of VSD orange powders was investigated by DSC. The onset, midpoint, and end-set temperature were determined from the characteristic DSC thermograph. However, some authors have used the midpoint transition as a glass transition temperature (Goula and Adamopoulos, 2010, 2006). Based on the transitions occur at Tg_{onset}, but the present study considered midpoint temperature of the change in heat capacity was taken as the safest the glass transition temperature of the orange juice powder, furthermore it was used to establish the relationship between Tg, water activity and water content of the orange juice powder. The figure 2-10 shows the glass transition temperature of the vacuum spray dried orange juice powders. The results show that the addition of maltodextrin in orange juice significantly increased the glass transition temperature.

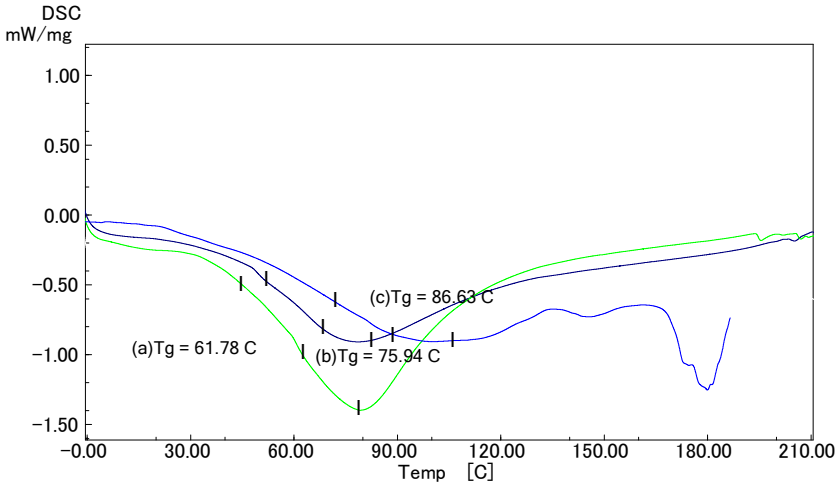


Figure 2-10. DSC profile of orange juice powders and conditioned at water activity (a_w = 0.33). where (a) COJ/MD 50:50, (b) COJ/MD 40:60 and (c) COJ/MD 30:70

Previously mentioned that OJ/MD 60:40 powder were unsuccessful, this was due to the stickiness of the powder. The OJ/MD 60:40 powders had the lower glass transition temperature (37.15 ℃) . The DSC thermograph of OJ/MD 60:40 powders as shown in Fig. 2-10.

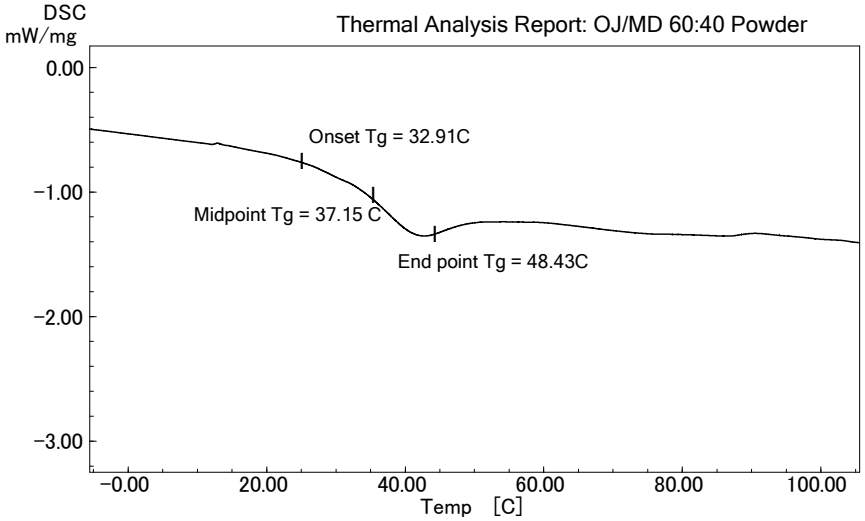


Figure 2-11. DSC profile for VSD orange juice powder (COJ/MD 60:40) and conditioned at water activity (a_w = 0.33)

Similar curves were also obtained for all other powders as presented in Figure 2-11. The DSC profile shows that with an increase of carrier agent, Tg of the powders increased. The similar phenomenon obtained by Goula et al. (2008); Shrestha et al. (2007a) and Tonon et al. (2009b) for spray dried orange juice, tomato pulp, and acai juice powders. The results show that there was a huge jump in Tg from 61.78 to 86.63 due to the increases of maltodextrin as 20% solids. Product with higher juice solid had lower glass transition temperature. The glass transition temperature of VSD orange juice powders was also determined at different relative humidity. The results are shown in Table 2-5. Glass transition temperature of VSD orange juice powders decreased with increased of moisture content, this behavior due to the plasticizing effect of water. The similar results were obtained for spray dried orange juice, tomato pulp, acai juice powders (Goula et al., 2008; Goula and Adamopoulos, 2010; Tonon et al., 2009b).

Table 2-5. Glass transition temperature of VSD orange juice powders at different water activity levels

Water activity (a_w)	Glass Transition Temperature, Tg, (°C)		
	COJ/MD 50:50	COJ/MD 40:60	COJ/MD 30:70
0.11	61.78±1.23	77.94±2.15	88.63±2.12
0.22	53.12±2.11	67.76±1.19	74.85±1.17
0.33	45.16±0.05	63.23±1.13	68.13±1.12
0.53	32.23±1.13	45.67±1.23	55.13±2.13
0.71	15.15±1.19	17.21±1.11	30.12±1.13
0.75	-2.32±0.03	4.14±0.92	15.19±1.11
0.84	-30.25±0.95	-23.96±1.21	-12.67±1.23

The values are mean ± S.D of three independent determinations.

The predicted values are given in Table 7, and the fitted curve is shown in Fig 5; satisfactory values for R^2 and M_e were obtained During storage and also in the processing orange juice powder becomes agglomerated and caking occur due to the plasticizing effects of water on the particle surface. These physical phenomena were successfully explained and predicted by the

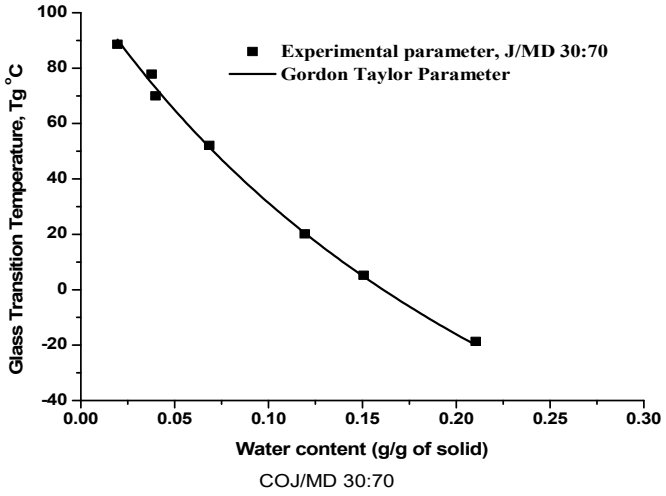
glass transition concept (Chuy and Labuza, 1994). The predicted values are given in Table 2-6, and the fitted curve is shown in Figure 2-11; satisfactory values for R^2 and M_e were obtained.

Table 2-6. Estimated Gordon Tailor Parameters for VSD concentrated orange powders

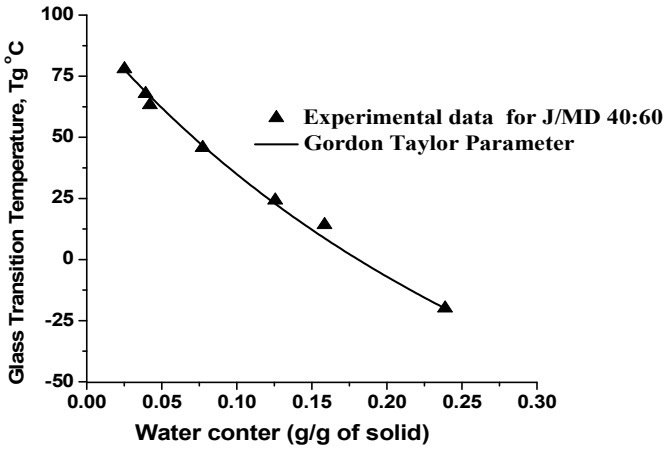
Parameter	COJ/MD 50:50	COJ/MD 40:60	COJ/MD 30:70
Tgs (°C)	71.89	95.18	103.82
K	2.498	3.194	3.52
R^2	0.99	0.98	0.99
E (%)	6.70	3.84	3.47

The Gordon Taylor parameters Tgs represents the glass transition temperature of the binary solid mixture, lower the Tgs mean higher plasticizing effects of water into solids. Due to the plasticizing effects of water, the Tg of the powder decreased with increasing water content.

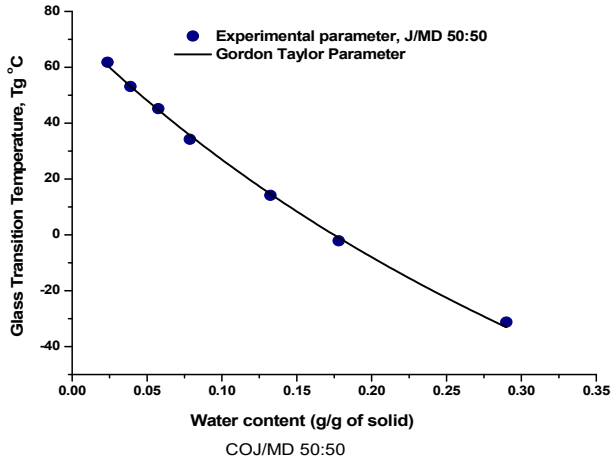
The present study shows that the Tg_s values changed from 71.89 to 103.82°C with an increasing maltodextrin solid content from 50 to 70%. The other parameters k presents the heat capacity of water around its glass transition to the heat capacity of the dry solids.The parameter k also known as the adjustable parameter, which determined the degree of curvature of the Tg-moisture curve. Incorporating of maltodextrin could increase the k value of the VSD orange juice powders from 2.50 to 3.35. The similar results reported by (Wang and Zhou, 2012) for spray dried soy sauce powder with maltodextrin as a carrier. In contrast, higher k values (0.83-0.85) obtained by (Goula and Adamopoulos, 2010) for spray-dried concentrated orange juice with maltodextrin. These differences in the Gordon-Taylor parameter may result from the different feed compositions and drying conditions of the orange juice/maltodextrin powder. Another possible reason reported by Wang and Zhou (2012), the predicted results of Tgs and k showed higher due to the lower DE value of maltodextrin.



COJ/MD 30:70



COJ/MD 40:60



COJ/MD 50:50

Figure 2-12. Relationship between the glass transition temperature and water content of VSD orange juice powders with curves fitted to the Gordon-Taylor model

Our present study used maltodextrin DE 12 whereas Goula and Adamopoulos (2010) used DE 6 as a carrier for spray drying. Lower the molecular weight of maltodextrin showed the higher glass transition temperature (Goula and Adamopoulos, 2010; Tonon et al., 2008). The higher R^2 values and lower percent modulus values indicated that Gordon Taylor model was satisfactorily fitted to the experimental data. Truong et al. (2005) stated that glass transition temperature is normally 10-23℃ lower than the sticky point temperature and in spray drying, particles which are above this temperature stick to the dryer wall and adversely affecting the free-flowing property. In the case of VSD orange juice powder COJ/MD 60:40, a sticky temperature much lower than outlet temperature of the dryer (<60℃) and that would result in high degree of stickiness and thus in an insignificant powder yield, whereas other combinations had successfully produced a powder.

2.4 Conclusions

Vacuum spray drying was successfully produced a concentrated orange juice powder with maltodextrin as a carrier. The produced VSD orange powders were stable due to their low moisture content (2.29-3.49%), low water activity (0.15-0.25) and higher glass transition temperature. An optimum concentration of drying aid as maltodextrin to VSD of concentrated orange juice and their relationship with glass transition temperature was established. Due to the stickiness problem, a COJ /MD 60:40 combinations was unsuccessful. Cyclone recovery increased up to 63.3% with increasing as Tg and maltodextrin. The particle sizes of the VSD orange powder were smaller, smooth and spherical in morphology. Overall, the sorption behavior of the orange juice powder exhibited a type III sigmoid curve, and the highest and lowest water adsorption occurred at a_w values above and below 0.53, respectively. The experimental water adsorption data were satisfactorily correlated by both the BET and GAB models. Based on the stability and product recovery the present study concluded that COJ/MD 30:70 by weight can be used in industrially to produce orange juice powder.

Chapter 6. Conclusion and Recommendation

The effects of micro wet milling and two different spray drying methods on the processing of fresh and commercial orange juice powders have been investigated in this study. The micro wet milled and commercial orange juice were also studied. In a comparison of micro wet milled and commercial orange juice, micro wet milled resulted in superior products in terms of color, nutrients, and antioxidant potentials. The result demonstrated that micro wet milled orange juice had lower particle sizes of 55 μm (median) and higher glass transition temperature of -21^{o}C whereas commercial orange juice particle sizes of 115 μm (median) and glass transition temperature of -42^{o}C. In terms of antioxidants, micro wet milled orange juice contained higher ascorbic acid, total polyphenol, total flavonoid and also showed the higher antioxidant activity as well.

The present study investigated the effects of vacuum spray drying on the water sorption and glass transition temperature of commercial orange juice powders with maltodextrin (DE 12) as a carrier agent. Orange powders were produced in the weight ratios of orange juice solids to maltodextrin solids of 60:40, 50:50, 40:60 and 30:70. Vacuum spray drying was successfully produced commercial orange powders at 50 % or higher combinations of maltodextrin solid ratios. Due to the lower glass transition phenomenon of the commercial orange juice sticky powder produced and vacuum spray drying was not successful at 60:40 combinations. Overall, the sorption behavior of the orange juice powder exhibited a type III sigmoid curve, and the highest and lowest water adsorption occurred at a_w values above and below 0.53, respectively. The experimental water adsorption data were satisfactorily correlated by both the BET and GAB models. Based on the stability and product recovery the present study concluded that OJ/MD 30:70 and 40:60 by weight can be used in industrially to produce orange juice powder.

In a comparison between vacuum spray drying and spray drying of micro wet milled orange juice powders were also determined. Powders were produced with above mention combinations of maltodextrin to micro wet milled orange juice solids. Vacuum spray drying was able to produce powders at higher combinations of juice solids (60:40) whereas spray drying was not successful when juice to maltodextrin solid ratios of 50:50 or more. The powder quality in terms of physicochemical and antioxidant properties was investigated. Vacuum spray dried powders had the superior quality in terms of color, physical properties and also the antioxidant properties. Vacuum spray drying was able to retain maximum ascorbic acid (74.0%) whereas spray drying retains only 39.7%. The product recovery of the vacuum spray drying and spray drying process were not significantly varied for OJ/MD 30:70 and 40:60 powders.

Finally, vacuum spray dried micro wet milled orange juice powders were also investigated for storage stability in terms of degradation kinetics, color and antioxidant activity of powders throughout the storage of 90 days. Powders were stored at 10, 25, 35℃ and relative humidity of 33%. Temperature and storage time negatively influenced the stability of ascorbic acid and color, whereas antioxidant activity increased at the beginning of storage at a higher temperature then decreased slightly after 60 days. For stability study, powders were stored at different water activities (0.11 to 0.84) in order to determine the plasticizing effects of water on glass transition temperature. Vacuum spray dried orange juice solids/maltodextrin solids (30:70) ratio powder considered as the most stable, once it showed the higher critical water activity ($a_w = 0.61$) and moisture content of 0.10 g water/g of dry solid.

We concluded that the combination of maltodextrin additions and use of vacuum spray drying of micro wet milled orange juice was proven to be an effective way of producing heat resistant fruit juice powders. Further study is needed to evaluate the biochemical changes during processing and storage of powders. It is also required to analyze the cost and feasibility study for industrial scale production.

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Abstract

Orange juice is the most appreciated juice beverage due to its pleasant aroma and healthful properties. Moreover, it is a good source of vitamin C, polyphenol, carotenoid, flavonoid and other antioxidant constituents. To lengthen the shelf life and for convenient uses, oranges are usually preserved in the form of juice and powder. During industrial processing, orange juice is passed through the finisher for separating juice from pulp and seeds, and undergoes some thermal treatments that extend its shelf life but may deteriorate the color and flavor, and substantially decrease vitamins and functional compounds. In addition, color, flavor and functional compounds also deteriorate during powder processing with high temperature. To overcome these limitations and reduce the loss of functional components, we exploited two new techniques namely micro wet milling (MWM) and vacuum spray drying (VSD) process for producing orange juice with pulp powders.

The present study includes 5 chapters. Chapter 1 describes the mentioned background of this study. Furthermore, the limitations and advantages of different methods for processing of orange juice and powders are illustrated in detail.

In Chapter 2, detailed application of VSD for processing of orange juice powders from the concentrated commercial orange juice is depicted. According to the experimental results, the physical properties of orange juice powder with four different combinations of juice solids to maltodextrin solids at 60:40, 50:50, 40:60, and 30:70 were determined. VSD was unable to produce powder from higher juice solids combinations such as 60:40, due to the lower glass transition temperature of the rich sugar. The experimental water adsorption data were satisfactorily correlated by both the Brunauer–Emmett–Teller and Guggenheim–Anderson–de Boer models. Based on the physical properties and product recovery, this study concluded that OJ/MD 30:70 by weight can be used industrially to produce concentrated orange juice powder.

In Chapter 3 a new technique for producing concentrated orange juice with pulp by MWM system was introduced. The suitable milling conditions were achieved by varying the feeding rate and rotational speed of the mill. Feeding rate of 15 mL/min and rotational speed of 50 rpm can able to produce orange juice with smaller particle size and better color quality than the industrially extracted orange juice. In case of antioxidants, MWM orange juice contained higher ascorbic acid, total polyphenol, total flavonoid contents and antioxidant activity than the commercial orange juice. We concluded that MWM can be used to minimize the losses in fruit juice processing and can also provide fiber enriched fruits juice with higher nutrient values.

The aim of the study in Chapter 4 was to produce concentrated MWM orange juice powders by the application of VSD and spray drying (SD) process. A comparative study based on the effects of VSD and SD on physicochemical and antioxidant properties of MWM orange juice powders produced with four different weight ratios of juice solids to maltodextrin solids; 60:40, 50:50, 40:60 and 30:70 were investigated. The experimental results demonstrated that spray drying (SD) was not successful in producing MWM orange juice powders at 50:50 solid ratios or higher, whereas VSD can produce them successfully. The analyzed physical properties of OJ/MD 40:60, 30:70 of both SD and VSD powders were not significantly different, but the ascorbic acid and other bioactive compounds differed significantly. VSD was able to retain maximum 73.97% ascorbic acid whereas SD retained only 39.72%. VSD with a low temperature of 50-60 °C using superheated steam as a heating medium was able to produce powders with higher quality than the SD.

Chapter 5 depicted the ascorbic acid degradation, color and antioxidant activity of VSD and MWM orange juice powders throughout the storage of 90 days. Powders were stored at 10, 25, 35°C and relative humidity of 33%. Orange powder with higher maltodextrin solids and lower storage temperature (at 10 °C) showed the lower degradation of physical properties as well as the antioxidants. Both water activity and glass transition temperatures were used to predict the

critical conditions for storage. The critical water activities of all the powders varied from 0.61 to 0.53 and moisture content from 0.10 to 0.08 g/g of dry solid. The results provide valuable information for predicting the stability and suitable storage conditions of VSD MWM orange juice powders.

The present study demonstrated that vacuum spray drying and micro wet milling was able to produce orange juice powders with better quality in terms of color, stability and antioxidant properties than the conventional spray drying process.

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