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POMEGRANATE ARIL DRYING AND JUICE POWDER PRODUCTION

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IN

FOOD ENGINEERING

BY

ERHAN HORUZ

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Pomegranate aril drying and juice powder production

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Prof. Dr. Medeni MASKAN

by

Erhan HORUZ

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TO MY FAMILIY

NURHAN, PERİHAN, TAHİR

ABSTRACT

POMEGRANATE ARIL DRYING AND JUICE POWDER PRODUCTION

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M.Sc. in Food Eng.

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In this study, Hicaz type pomegranate (*Punica granatum*) juice powder and dry arils were obtained by spray drying, and hot air and microwave drying processes, respectively. In order to prevent stickiness problem in spray drying of juice, maltodextrin DE6 was used as a drying agent. Feed flow rate, feed temperature and air flow rate were kept constant. Air inlet temperature (110-140 °C), maltodextrin ratio in the feed mixture (39.08-64.12 %), and feed mixture concentration (19.61-44.11 °Brix) were the independent process variables.

As it was aimed, the products with high yield (86 %) and high antioxidant activity (77 %) were produced. It was found that the most important variables were maltodextrin ratio and drying temperature in production of pomegranate juice powders. Total color change (ΔE), bulk density, antioxidant capacity and powder yield were strongly affected from independent variables. Therefore, these parameters were used in optimization process of independent variables. The optimum temperature, feed mixture concentration and maltodextrin ratio were estimated as 100 °C, 30.8 °Brix and 53.5 % maltodextrin, respectively.

In the pomegranate aril drying part of this study drying behavior, shrinkage, rehydration, bulk density and color changes of the products were investigated. Hot air drying process was carried out at three different temperatures; 50, 60 and 70 °C. Also, three microwave power levels (210, 350, and 490 W) were studied as another drying technique. Increasing drying temperature and power intensity shortened the drying time of arils. Rate of drying was higher when microwave was used compared to air drying process. It was observed that shrinkage of air dried samples was higher than microwave dried sample. Rate of rehydration of microwave dried aril samples was relatively faster than air dried samples, but not significant. However, bulk density of air dried samples was higher than microwave dried sample.

Key Words: Pomegranate juice powder, maltodextrin, spray drying, optimization, shrinkage, rehydration, air drying, microwave drying

ÖZET

NAR TANESİ KURUTULMASI VE NAR SUYU TOZU ÜRETİMİ

HORUZ, Erhan

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Bu çalışmada, Hicaz tip nardan (*Punica granatum*) meyve suyu tozu ve nar tanesi kurusu, sırasıyla püskürtmeli kurutucu ve sıcak hava ve mikrodalga kurutucular kullanılarak elde edildi. Püskürtmeli kurutucudaki yapışma problemini önlemek için kurutma ajanı olarak maltodextrin DE6 kullanıldı. Besleme hızı, besleme sıcaklığı ve kurutucu hava akış hızı sabit tutuldu. Hava giriş sıcaklığı (110-140 °C), besleme karışımındaki maltodekstrin oranı (39.08-64.12 %) ve besleme karışımı konsantrasyonu (19.61-44.11 °Brix) bağımsız proses değişkenleri olarak alındı.

Amaçlandığı gibi, yüksek verimli (% 86) ve antioksidan içerikli (% 77) ürünler elde edildi. Nar suyu tozu üretiminde en önemli değişkenlerin maltodekstrin oranı ve kurutma sıcaklığı olduğu bulundu. Toplam renk farkı (ΔE), yığın yoğunluğu, antioksidan kapasitesi ve toz ürün verimi bağımsız değişkenlerden oldukça etkilendiler. Bu nedenle, bu parametreler bağımsız değişkenlerin optimizasyon işleminde kullanıldı. Optimum sıcaklık, besleme karışımı konsantrasyonu ve maltodekstrin oranı sırasıyla 100°C, 30.8°Brix % 53.5 olarak bulundu.

Nar tanesi kurutulması çalışması kısmında ürünün kuruma davranışı, büzüşme, rehidrasyon, yığın yoğunluğu ve renk değişimi araştırıldı. Sıcak havayla kurutma işlemi üç farklı sıcaklık 50, 60 ve 70°C'de yapıldı. Diğer bir kurutma tekniği olan mikrodalganın üç farklı güç seviyesinde (210, 350 ve 490 W) çalışma yapıldı. Hava sıcaklığı ve mikrodalga yoğunluğunun arttırılması kurutma zamanını kısalttı. Mikrodalga ile yapılan kurutma işleminde, kuruma hızının havalı kurutmaya göre daha yüksek olduğu görüldü. Sıcak havayla yapılan kurutmada büzüşmenin mikrodalgaya göre daha şiddetli olduğu gözlendi. Anlamlı olmamasına rağmen, mikrodalga ile kurutulan nar tanesi örneklerinin rehidrasyon hızı daha yüksek oldu. Ancak, hava ile kurutulan nar tanesi örneklerinin yığın yoğunluğunun mikrodalga örneklerininkine göre daha yüksek olduğu bulundu.

Anahtar Kelimeler: nar suyu tozu, maltodekstrin, püskürtmeli kurutma, optimizasyon, büzüşme, rehidrasyon, sıcak havalı kurutma, mikrodalga kurutma

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NOMENCLATURE

A	Absorbance
AC	Antioxidant capacity
AOAC	Association of Official Analytical Chemists
ANOVA	Analysis of variance
a^*	Redness-greenness value
b^*	Yellowness-blueness value
Brix	Total soluble solids
CIE	Commission internationale de l'éclairage
CMC	Carboxy methyl cellulose
D.B.	Dry bulb
DE	Dextrose equivalent
DF	Dilution factor
ΔE	Total color differences
DPPH	2,2-diphenyl-1-picrylhydrazyl
EA	Ellagic acid
ETs	Ellagitannins
F	Feed
FCR	Folin-Ciocalteu reagent
G	Gas
Ghz	Gigahertz
Ha	Hectare
HMF	Hydroxymethylfurfural
k	Drying rate constant (min^{-n}) in Page equation
L^*	Lightness-darkness value
LDL	Low density lipoprotein

M_e	Equilibrium moisture content
M_0	Moisture content at time t=0
M_t	Moisture content at time t=t
MA	Molar absorptivity
MDC	Maltodextrin concentration
MR	Moisture ratio
MHz	Megahertz
MW	Molecular weight
n	Page model constant
P	Product
PPO	Polyphenol oxidase
PJ	Pomegranate juice
PJC	Pomegranate juice concentration
PJs	Pomegranate juices
% A.C	Percent antioxidant capacity
%S	Percent shrinkage
RH	Relative humidity
RSM	Response surface methodology
R^2	Correlation coefficient
S	Spray
SPSS	Statistical package for social sciences
t	Time (min)
TAC	Total anthocyanin content
Tg	Glass transition temperature
$Tg^{'}$	Maximally freeze concentrated glass transition temperature
TSE	Türk standartları enstitüsü
V_0	Initial volume of sample (mL)
V	Volume of sample (mL) at any time

W	Watt
W_d	Weight of dried sample (g)
W_t	Weight of rehydrated sample (g) at any time
w.b	wet bulb
X_1	Air inlet temperature
X_2	Feed mixture concentration
X_3	Maltodextrin in feed dry solids

CHAPTER I

INTRODUCTION

1.1.Pomegranate

The pomegranate (*Punica granatum* L.) is an ancient fruit; it has been widely consumed in various cultures for thousands of years. The use of pomegranate fruit dates back to Biblical times and reports of its therapeutic qualities have echoed throughout the millennia (Longtin, 2003). The Babylonians regarded pomegranate seeds as an agent of resurrection; the Persians believed the seeds conferred invincibility on the battle fields, while for the ancient Chinese the seeds symbolized longevity and immortality (Aviram et al., 2000).

The pomegranate belongs to the family Punicaceae. It is native from the area of Iran to the Himalayas in northern India, and has been cultivated and naturalized over the entire Mediterranean region since ancient times (Meerts et al., 2009). Actually, the pomegranate is widely cultivated throughout Iran, India, Mediterranean countries, the drier parts of Southeast Asia, Malaysia, the East Indies, and tropical Africa and, to some extent, in the United States (drier parts of California and Arizona), China, Japan, and Russia (Fadavi et al., 2006).

The edible parts of pomegranate fruits are consumed fresh or used for the preparation of fresh juice, canned beverages, jelly, jam, and paste and also for flavouring and coloring beverage products (Fadavi et al., 2005; Mousavinejad et al., 2009). In addition, it is widely used in therapeutic formulas, cosmetics, and food seasonings. Since ancient times, the pomegranate has been regarded as a “healing food” with numerous beneficial effects in several diseases (Vidal et al., 2003). Indeed, the pomegranate has been commonly used in folk medicine, for eliminating parasites, as an antihelmintic and vermifuge, and to treat and cure aphtae, ulcers, diarrhea, acidosis, dysentery, hemorrhage, microbial infections, and respiratory pathologies. It was also used as an antipyretic (Larrosa et al., 2010; Lee et al., 2010).

Recent years foods have seen increased interest on the part of consumers, researchers, and the food industry into how food products can help maintain health; and the role that diet plays in the prevention and treatment of many illnesses has become widely accepted (Viuda-Martos et al., 2010a). At the present time, considerable importance is given to functional foods, which, in principle, apart from their basic nutritional functions, provide physiological benefits and play an important role in disease prevention or slow the progress of chronic diseases (Viuda-Martos et al., 2010b). There has been a virtual explosion of interest in the pomegranate as a medicinal and nutritional product because of its multifunctionality and its great benefit in the human diet as it contains several groups of substances that are useful in disease risk reduction (Martinez et al., 2006; Jaiswal et al., 2010).

1.2. Pomegranate growth and production

Pomegranate is a perennial plant usually grown in tropical and subtropical regions (Schubert et al., 1999). It is originated from Iran (Saleh et al., 1964) and widely produced by Iran, India, Turkey, America, the near and far eastern countries (Schubert et al., 1999). Current global production estimates for pomegranate are unavailable. However, it is widely grown in many countries where it is well adapted. In India more than 100,000 ha of pomegranate are produced. In Iran 65,000 ha of pomegranate produces 600,000 tons of fruit annually, with about 30% of yield exported (Stover and Mercure, 2007). Spain, with ~30,000 ha, is the largest European producer of pomegranate (Costa and Melgarejo, 2000). Pomegranate production has been increasing in Turkey. In 1990 annual production was 10,000 tons, 60,000 tons in 2002 and reached 80,000 tons in 2003. (Gölükcü and Tokgöz, 2008). Turkey contributes 150,000 tons of the world annual production of one million tons (Tezcan et al, 2009). Pomegranate is generally grown at Mediterranean, Aegean and South Eastern Anatolia regions in Turkey (Gölükcü and Tokgöz, 2008).

1.3. Chemical composition of pomegranates

The pomegranate fruit (Figure 1.1) has valuable compounds in different parts of the fruit. These can be divided into several anatomical origins: peel, seeds, and arils. Another important product obtained from pomegranate fruit is the juice that can be obtained from arils or from whole fruit.

The chemical composition of the fruits differs depending on the cultivar, growing region, climate, maturity, cultivation practice, and storage conditions (Poyrazoğlu et al., 2002; Barzegar et al., 2004; Fadavi et al., 2005). Significant variations in organic acids, phenolic compounds, sugars, water-soluble vitamins, and minerals of pomegranates have been reported over the years by various researchers (Aviram et al., 2000; Mirdehghan and Rahemi 2007; Çam et al., 2009; Davidson et al., 2009; Tezcan et al., 2009). About 50% of the total fruit weight corresponds to the peel, which is an important source of bioactive compounds such as phenolics, flavonoids, ellagitannins (ETs), and proanthocyanidin compounds (Li et al., 2006), minerals, mainly potassium, nitrogen, calcium, phosphorus, magnesium, and sodium (Mirdehghan and Rahemi 2007), and complex polysaccharides (Jahfar et al., 2003). The edible part of the pomegranate fruit (50% of whole fruit) consists of 80% arils and 20% seeds. Arils contain 85% water, 10% total sugars, mainly fructose and glucose, and 1.5% pectin, organic acid such as ascorbic acid, citric acid, and malic acid, and bioactive compounds such as phenolics and flavonoids, principally anthocyanins (Aviram et al., 2000; Tezcan et al., 2009). The seeds are a rich source of total lipids; pomegranate seed oil comprises 12% to 20% of total seed weight. The oil is characterized by a high content of polyunsaturated (n-3) fatty acids such as linolenic, linoleic, and other lipids such as punicic acid, oleic acid, stearic acid, and palmitic acid (Özgül-Yücel, 2005; Fadavi et al., 2006). The seeds also contain protein, crude fibers, vitamins, minerals, pectin, sugars, polyphenols, isoflavones (mainly genistein), the phytoestrogen coumestrol, and the sex steroid, estrone (El-Nemr et al., 2006; Syed et al., 2007).

Nowadays, it is widely accepted that the beneficial health effects of fruits and vegetables in the prevention of disease are due to the bioactive compounds they contain (Galaverna et al., 2008). The presence of significant amounts of bioactive compounds, such as phenolic acids, flavonoids, and tannins in pomegranate fruits assures them considerable nutritional value (Aviram et al., 2000).

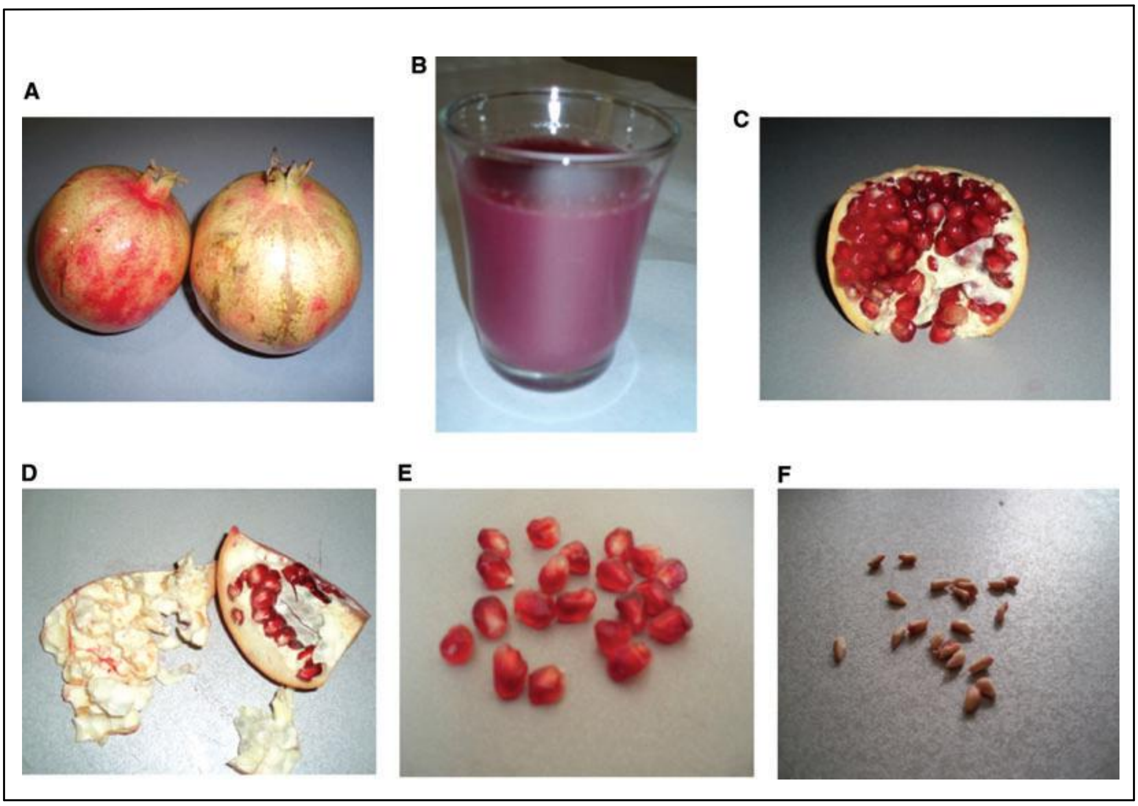

Figure 1.1. Different part of pomegranate fruit. A: whole fruit; B: pomegranate juice; C: Section of pomegranate; D: pomegranate peel; E: pomegranate arils; F: pomegranate seeds.

1.4. Phenolic compounds of pomegranate

One of the main compounds responsible for most of the functional properties of many foods, among them pomegranate fruit, are phenolic compounds in any of their forms. A range of phenolics had been found in pomegranate. Galloylglucose, punicalagin, punicalin, ellagic acid and gallic acid were main active compounds in pomegranate juice and husk (Viuda-Martos et al., 2010a; He et al., 2011). Natural polyphenols can range from simple molecules (phenolic acids, phenylpropanoids, flavonoids) to highly polymerized compounds (lignins, melanins, tannins), with flavonoids representing the most common and widely distributed subgroup (Soobrattee et al., 2005). Chemically, phenolic acids can be defined as substances that possess an aromatic ring bound to one or more hydrogenated substituents, including their functional derivatives (Marin et al., 2001).

Flavonoids are low-molecular-weight compounds consisting of 15 carbon atoms, arranged in a C_6-C_3-C_6 configuration. Essentially, the structure consists of 2 aromatic rings joined by a 3-carbon bridge, usually in the form of a heterocyclic ring (Balasundram et al., 2006).

Anthocyanins are the largest and most important group of flavonoids present in pomegranate arils, which are used to obtain the juice. These pigments give the fruit and juice its red color (Afaq et al., 2005). There is a great variety of anthocyanins present in pomegranate juice (Figure 1.2a) principally cyanidin-3-*O*-glucoside, cyanidin-3,5-di-*O*-glucoside, delphinidin-3-*O*-glucoside, delphinidin-3,5-di-*O*-glucoside, pelargonidin-3-*O*-glucoside, and pelargonidin-3,5-di-*O*-glucoside (Lansky and Newman 2007; Jaiswal et al., 2010). The main differences between them are the number of hydroxylated groups, the nature and the number of bonded sugars to their structure, the aliphatic or aromatic carboxylates bonded to the sugar in the molecule, and the position of these bonds (Kong et al., 2003). The phenolic acids present in pomegranate juice (Figure 1.2b) can be divided into 2 groups: (1) hydroxybenzoic acids, mainly gallic acid and ellagic acid (EA) (Amakura et al., 2000); and (2) hydroxycinnamic acids, principally caffeic acid, chlorogenic acid, and *p*-coumaric acid (Poyrazoğlu et al., 2002).

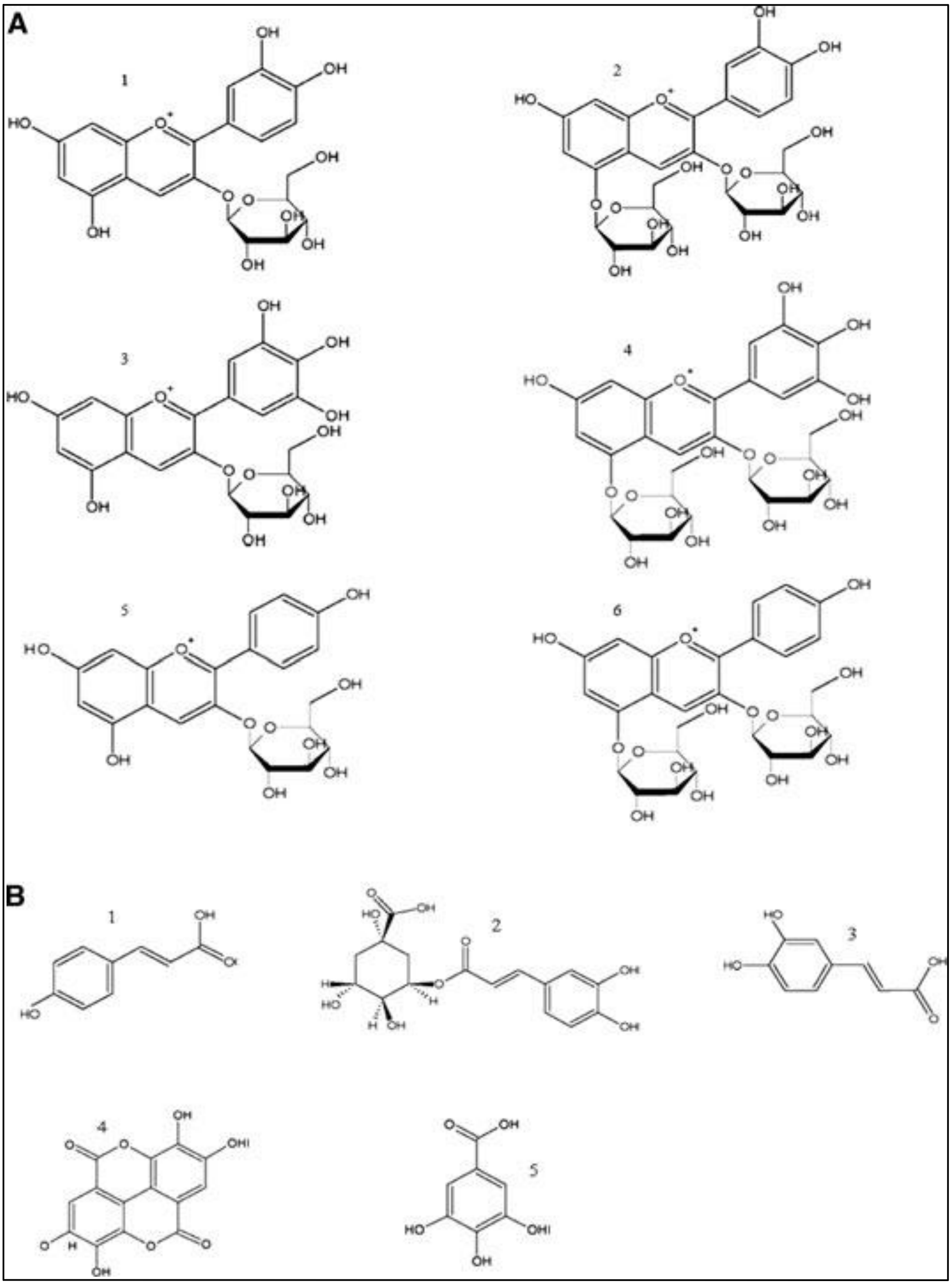

Figure 1.2 (a). Principal anthocyanins present in pomegranate juice. 1: cyanidin-3-*O*-glucoside; 2: cyaniding-3,5-di-*O*-glicoside; 3:delphinidin-3-*O*-glucoside; 4: delphinidin-3,5-di-*O*-glucoside; 5: pelargonidin-3-*O*-glucoside; 6: pelargonidin-3,5-di-*O*-glucoside. (b). Principal phenolic acids present in pomegranate juice: 1: *p*-coumaric acid; 2: chlorogenic acid; 3: caffeic acid; 4: ellagic acid; 5: gallic acid.

1.5. Antioxidant properties

Oxidative deterioration is one of the main factor of the reduction of the quality and acceptability of food products. This process is initiated by exposure to the enzyme lypoxygenase, heat, ionizing radiation, light, metal ions, and metallo-protein catalysts (Daker et al., 2008). Such oxidation leads to a significant loss of a food's nutritional value since it involves a loss of vitamins and essential fatty acids. It also affects the food's sensory quality—changes in color, texture, and taste—which shortens its shelf life and can result in rejection by consumers (Fernandez-Lopez et al., 2007).

The antioxidant activity of pomegranate components has been the subject of many studies (Naveena et al., 2008; Çam et al., 2009; Mousavinejad et al., 2009; Tezcan et al., 2009), most conducted *in vitro* and *in vivo.* All these activities may be related to the diverse phenolic compounds present in pomegranate, including punicalagin isomers, EA derivatives, and anthocyanins (delphinidin, cyanidin and pelargonidin 3-glucosides, and 3,5-diglucosides). These compounds are known for their properties to scavenge free radicals and to inhibit lipid oxidation *in vitro* (Gil et al., 2000; Noda et al., 2002). However, Tzulker et al. (2007) suggested that punicalagin originating from the peels is one of the major photochemical contributing to the total antioxidant capacity of pomegranate juice, whilst anthocyanins play only a minor role in this activity.

The action mechanism set in motion by the antioxidant activity of these compounds is still not clearly understood, although it is a known fact that antioxidant mechanisms involved in biological matrixes are quite complex and several different factors may play a role (Çam et al., 2009). Madrigal-Carballo et al. (2009) suggested that pomegranate polyphenolic molecules undergo redox reactions because phenolic hydroxyl groups readily donate hydrogen to reducing agents. Negi and Jayaprakasha (2003) have also reported a significant increase in the reducing power of pomegranate peel extracts with increases in concentration from 50 to 400 ppm. Reducing properties are generally associated with the presence of reductones (Pin-Der 1998). Gordon (1990) reported that the antioxidative action of reductones is based on the breaking of the free radical chain by the donation of a hydrogen atom. Reductones also react with certain precursors of peroxides, thus preventing peroxide formation (Naveena et al., 2008). However, Amarowicz et al. (2004) suggested that the antioxidant activity of phenolic compounds is due to their ability to scavenge free radicals or chelate metal cations.

The antioxidant and sensory qualities of pomegranates depend on several factors, such as cultivar and climatic conditions during fruit maturation and ripening and the part of the fruit used (Borochov-Neori et al., 2009). Thus, Singh et al. (2002) reported that a methanol extract of pomegranate peel had much higher antioxidant capacity than that of arils, as demonstrated by using the β-carotene linoleate and DPPH model systems. Tzulker et al. (2007) reported that the homogenates prepared from the whole fruit exhibited an approximately 20-fold higher antioxidant activity than the level found in the aril juice.

Gil et al. (2000) reported that pomegranate juice possessed a 3-fold higher antioxidant activity than that of red wine or green tea, and 2-, 6-, and 8-fold higher levels than those detected in grape/cranberry, grapefruit, and orange juices, respectively (Azadzoi et al., 2005; Rosenblat and Aviram 2006).

In another study, Seeram et al., (2008) observed that pomegranate juice had the greatest antioxidant potency composite index among such beverages as apple juice, açai juice, black cherry juice, blueberry juice, cranberry juice, grape juice, orange juice, red wines, and iced tea; and the antioxidant activity was at least 20% greater than any of the other beverages tested.

1.6. Evaluation of pomegranate

The fruit is consumed directly as fresh seeds or processed into fresh juice, concentrates, and syrup which can also be used in beverages for jellies, and flavouring and coloring agents. Pomegranate contains richly colored kernels that give a delicious juice. The kernels are also used as garnish for desserts and salads. (Cemeroğlu, 1977; Saxena et al., 1987; Onur, 1988; Rege and Pai, 1999; Vardin and Abbasoğlu; 2004).

1.6.1. Pomegranate juice

The most common evaluation method of pomegranate is to produce its juice. It is known that especially in the countries doing pomegranate cultivation, production and consumption of pomegranate juice also reached huge dimension. On the other hand, importer countries has not got into the habit of consuming pomegranate concentrate or juice yet. A qualified pomegranate juice must be dark-colored, a bit harsh, and have appropriate equilibrium of acid and sugar. Because being dissolved in the juice, coloring matters in pomegranate are obtained with the juice in pressing without any separate process (Cemeroğlu, 1982).

The most important problem seen in clear fruit juice and its concentrates is haze formation event. Phenolic compounds do the most active role through these elements (Lea, 1984; Heatherbell, 1984; Dietrich et al., 1990; Alper and Acar, 2004). Phenolic compounds cause some technological problems while foods are being processed and stored. Pomegranate juice is generally produced as clear or semi-clear in line with the customers' expectations. Whereas, a large part of polyphenols and minerals which are very beneficial for health and have antioxidant function are removed from the pomegranate juice in the process of clarification. Because their phenolic compounds which have high-molecular-weight and in the form of tannin are able to produce sediment by losing their solubility (Harborne, 1993), in semi-clear and non-clear pomegranate juice, sedimentations and opacity occur in time, therefore, they are not preferred by customers. The reason of not consuming them is that they think the pomegranate juice spoils. The most suitable way to prevent this sediment in the pomegranate juice is to produce pomegranate juice concentrate first and then to store it under appropriate conditions for a while to separate the sediment. Formation of sediment in pomegranate juice can be prevented by bottling clear pomegranate juice concentrate which is produced in this way, reconstituted and then filtrated (Cemeroğlu, 1977).

1.6.2. Pomegranate juice concentration

Fruit juice concentrate is obtained by removal of water content of fruit juices and which are more stable than fruit juices. A research by Cemeroğlu (1977) focused on concentration of clarified and filtrated PJ from 15.2 to 46°Brix. He observed no color loss during evaporation. Also, the color intensity, anthocyanin content, ascorbic acid content, tannins of pomegranate juice concentrate decreased but soluble dry matter increased with increasing storage time. The most suitable packaging material has been reported as dark colored glass bottle for pomegranate juice concentrates.

1.6.3. Pomegranate sour

Pomegranate sour is produced by pressing the pomegranate and then filtering of juice and finally concentrating under vacuum or evaporating at atmospheric pressure. It is used for flavouring of foods (Anonymous, 2001). According to TSI (Anonymous, 2001) pomegranate sour must not contain residue, fruit pulp and sucrose. Some properties of pomegranate sour is given in Table 1.1.

Table 1.1 Some properties of pomegranate sour (TSE, 2001)

Brix, min	68
Titratable acidity (citric acid), %, min	7.5
pH	3.0
Hydroxymethylfurfural (HMF), mg/kg, max	50
Sucrose	Absent
Preservatives and colorant	Absent

The filtered pomegranate juice has a large amount of tannins content. Astringent taste in the juice is coming from polyphenolic substances and tannins. Also, this substances cause turbidity in the juice. Tannins can be removed by clarification process and makes the juice soft (Cemeroğlu, 1982). However, astringent taste is desirable for pomegranate sour. So that it is not advised to apply clarification technique in pomegranate sour production. Pomegranate sour is a stable product because it has low pH and high soluble dry matter and can be stored for a long time without pasteurization.

1.6.4. Pomegranate arils and dried arils

Pomegranate arils can be evaluated in modified atmosphere package (Paster et al., 1985). Also, pomegranate arils are exported to Middle East countries, by applying freezing technique, with suitable packaging material. It is ascertained that a product called 'anardana' is obtained by drying pomegranate aril in India and is used as acidifier in foods special to there for improving the taste.

1.6.5 Pomegranate seeds

Pomegranate seeds are valuable products which are waste material from production of pomegranate juice and concentrate. Seeds on the average have 20.8% lipid which is nearly the same as cotton seed oil. After oil process, waste of seeds is used as additive material in animal feed which improves milk yield (Onur, 1990). Lipid composition of pomegranate seeds has just started to investigate. Especially this lipid is healthier according to their essential fatty acid composition. These essential fatty acids naturally prevent the risk of cardiovascular diseases and decrease total and LDL (low density lipoprotein) cholesterol. In a research, essential fatty acids of seeds were determined by according to amount of high to low: linoleic, oleic, palmitic, stearic, linelonic, arashidonic, and palmitoleic acid (Malgarejo, 1995). Due to properties of lipid of pomegranate, seeds are being imported by some countries for using in drugs and cosmetics industry. Recently, dried and cleaned pomegranate seeds have been exported from Turkey (Vardin and Abbasoğlu, 2004).

1.6.6. Canned pomegranate arils

In recent years, pomegranate arils are evaluated by being canned as well as the other fruits. It is stated that besides of consuming as canned fruit, it is suitable for pastry sector if its sugar content is kept high (Saxena, 1987).

1.7. Evaporation

In many food processes water, or another solvent, needs to be removed from a dilute liquid to produce a concentrated or dried product. The processes that can be used to remove water include evaporation, freeze concentration, reverse osmosis (or other membrane separations), and drying. The choice depends on the extent of concentration required, the effect of the process on the product, available energy sources, and on the relative costs of the processes. Often a combination of processes is used.

Evaporation has several advantages over other water removal processes. Modern evaporation plants are very effective at utilizing small amounts of steam or electrical energy to generate large rates of evaporation. Techniques such as multiple-effect evaporation, thermal vapour recompression and mechanical vapour recompression greatly reduce the amount of energy required to give a certain degree of concentration. Another advantage of evaporation is the level of concentration attainable. Evaporation can concentrate most liquid feeds of any dilution up to 50% solids easily, while, in the extreme, sugar solutions for the production of hard candies (toffees) are evaporated to about 98% solids (Schwartzberg, 1989).

Usually the liquid being concentrated flows through a tube while heat is applied to the outside of the tube. The solvent boils and is separated from the concentrated liquid. Most foods are damaged by heat, so they are normally evaporated under vacuum conditions with a low boiling point. The latent heat of vaporization of water is high but by reusing energy in multiple stages or with vapour recycles; good energy efficiency can be obtained.

1.7.1. Rising film evaporator

The concept of film evaporator was first practically applied in rising film evaporation which was pioneered by Paul Kestner in the early 1900’s. The rising film concept overcomes the problem of liquor distribution among tubes by careful design of the bottom of the tube and an appropriate pre-heating of the liquor. However, the rising film is only established by heating the incoming liquid to above the equivalent boiling temperature of the liquid at the top of tube, this superheating being imposed by hydrostatic and velocity pressures present during the passage of the liquid up the tube (Karaaslan, 2010).

Schematic diagram of the rising film evaporator used in this study is shown in Figure 1.3. The liquid feed is processed by gravity from a stainless steel feed tank through a flow meter and manually operated control valve. Vapour, produced by boiling in the tube, rises and carries a film of more concentrated liquid up the tube and into a cyclone separator where the vapour and liquid separated. The vapour enters a water cooled condenser where it condenses and flows out into a condensate collecting tank. The liquid concentrate can be recycled back through the evaporator tube or collected as product in the product collecting tank. The evaporator ducting, cyclone and condenser, are all constructed from stainless steel, and a glass 'elbow' at the top of the evaporator allows the vapour/liquid film mechanism to be seen before entering the cyclone. Both concentrate and condensate collecting tanks are borosilicate glass to allow the condition and quantity of products to be observed (http://www.consultexim.hu/katalogus/armfield/pdf/ft/ft22.pdf).

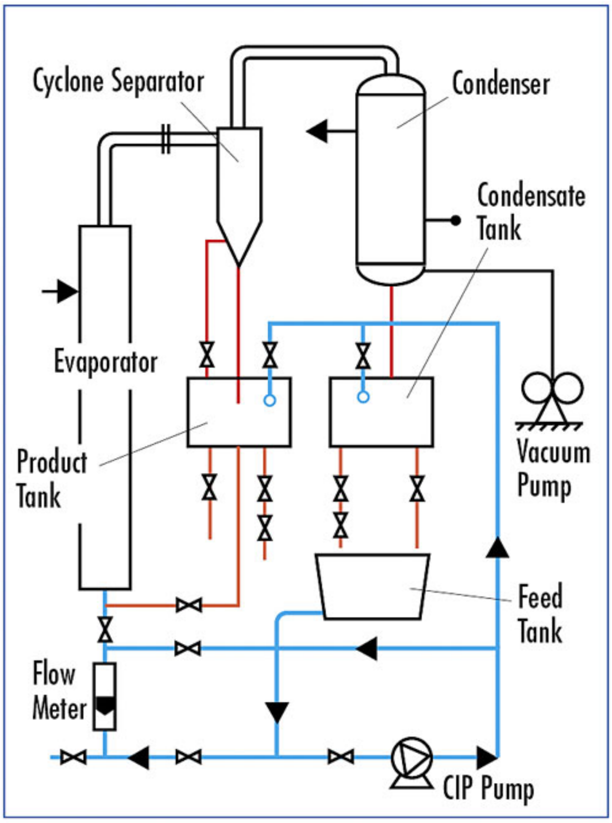

Figure 1.3. Schematic diagram of a rising film evaporator

1.8. Drying

The terms dried and dehydrated are not synonymous. The U.S. Department of Agriculture lists dehydrated foods as those with no more than 2.5% water (dry basis), while the term dried foods applies to any food product with more than 2.5% water (dry basis) (Vega-Mercado et al., 2001). Drying is one of the widely used methods of fruits and vegetables preservation. Water is removed to a final concentration, which assures microbial stability of the product and minimises chemical and physical changes of the material during storage. In most drying processes water is removed by convective evaporation, in which heat is supplied by hot air (Lewicki and Jakubczyk, 2004).

The preservation of foods by drying is the time-honoured and most common method used by humans and the food processing industry. Dehydration of food is one of the most important achievements in human history, making humans less dependent upon a daily food supply even under adverse environmental conditions (Erbersdobler, 1986; Rahman, 2007). Though in earlier times drying was dependent on the sun, nowadays many types of sophisticated equipment and methods are used to dehydrate foods. During the past few decades, considerable efforts have been made to understand some of the chemical and biochemical changes that occur during dehydration, and develop methods for preventing undesirable quality losses.

Drying reduces the water activity, thus preserving foods by avoiding microbial growth and deteriorative chemical reactions. The effects of heat on microorganisms and the activity of enzymes are also important in the drying of foods. In the case of foods to be preserved by drying, it is important to maximize microorganism and enzyme inactivation for preventing spoilage and enhancing safety, and reduce the components responsible for the deterioration of the dried foods. Also, in the case of drying bacterial cultures, enzymes, or vitamins, minimum inactivation of the microorganism and enzyme is required. Thus, detrimental effects of drying may be desirable or undesirable, depending on the purpose of the drying process.

1.8.1. Drying curves

Drying curve usually plots the drying rate versus drying time or moisture contents. Three major stages of drying can be observed in the drying curve (Figure 1.4):

1. Transient early stage, during which the product is heating up (transient period; A and A')
2. Constant or first period, in which moisture is comparatively easy to remove (constant rate period)
3. Falling or second period, in which moisture is bound or held within the solid matrix (falling rate period)

Typical drying rate curves are shown in Figure 1.4. The moisture content at which the change from the first to the second period occurs is known as the critical moisture content. Typically, two falling rate periods are observed for both hygroscopic and nonhygroscopic solids (Rahman, 2007). The first falling rate period is postulated to depend on both internal and external mass transfer rates; while the second period, during which drying is much slower, is postulated to depend entirely on internal mass transfer resistance. The slower rate may be due to the solid–water interaction or glass–rubber transition. The drying behaviours of food materials depend on the porosity, homogeneity, and hygroscopic properties. The immediate entrance into the falling rate is characteristic of hygroscopic food materials (Rahman, 2007).

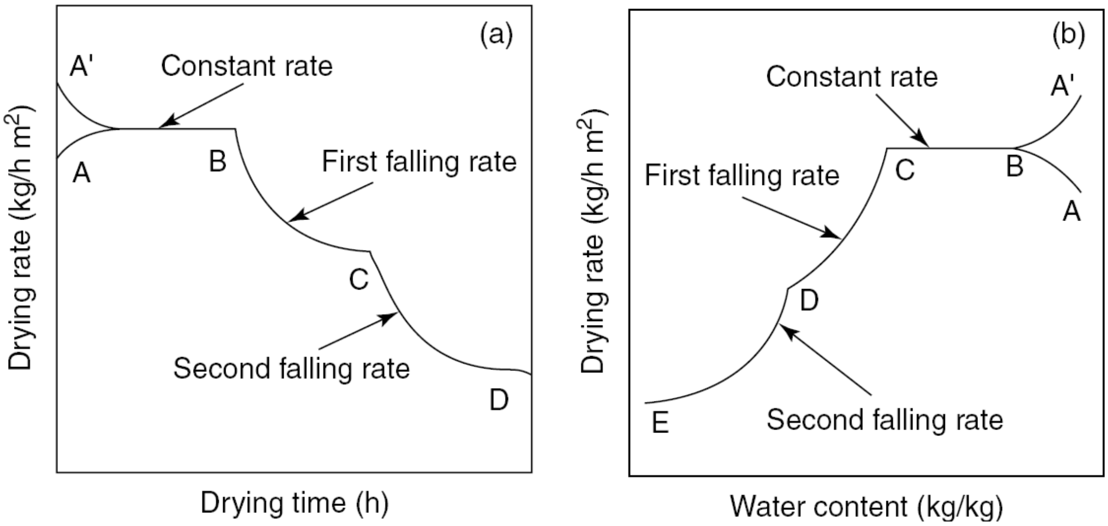

Figure 1.4. Typical drying curves(a): drying rate versus drying time, (b) drying rate versus water content.

1.9. Drying methods for fruits and vegetables

1.9.1. Convection air drying

Currently most of the dehydrated fruits and vegetables are produced by the technique of hot air drying which is the simplest and economical among the various methods. Air is generally used as the drying medium because it is plentiful, convenient, and overheating of the food can be controlled (Koçak, 2009).

Cabinet and bed-type dryers (i.e., kiln, tray, truck tray, rotary flow conveyor, and tunnel) fall into the first generation (Vega-Mercado et al., 2001). This is the simplest drying technique, which takes place in an enclosed and heated chamber. The drying medium, hot air, is allowed to pass over the product, which has been placed in open trays. Convection drying is often a continuous process and is mostly used for products that are relatively low in value. Air drying is usually accomplished by passing air at regulated temperature and humidity over or through the food in a dryer. Factors that affect the rate of drying are temperature, humidity, air velocity and distribution pattern, air exchange, product geometry and characteristics, and thickness. The sample is usually placed on mesh trays in one layer or in bulk on a bed or hung from a string for better air circulation over the product. Air circulation can be horizontal or vertical to the layer or bed. The structure and composition, such as fat content, of a product affects the drying rate. In general, the hotter is the air temperature, the faster is the drying rate; and similarly, the higher is the velocity, the higher is the drying rate; the lower is the air humidity, the higher is the drying rate. The relative humidity (a measure of dryness) is lower when air temperature is raised. A dryer must expel air to get rid of moisture, thereby allowing new, lower humidity air to enter the system. However, this process causes heat loss from the dryer. In many cases, two or multistage drying with different conditions could be used, for example, initial drying at 90 °C and then the second or final stage at 60 °C (Rahman, 2007).

Figure 1.5 is a diagrammatic sketch of a cabinet dryer (Maskan, 2000), which was used in this study, designed specifically for study of the air drying of cut pieces of fruit and vegetables, uniformly spread on shallow mesh-bottom trays.

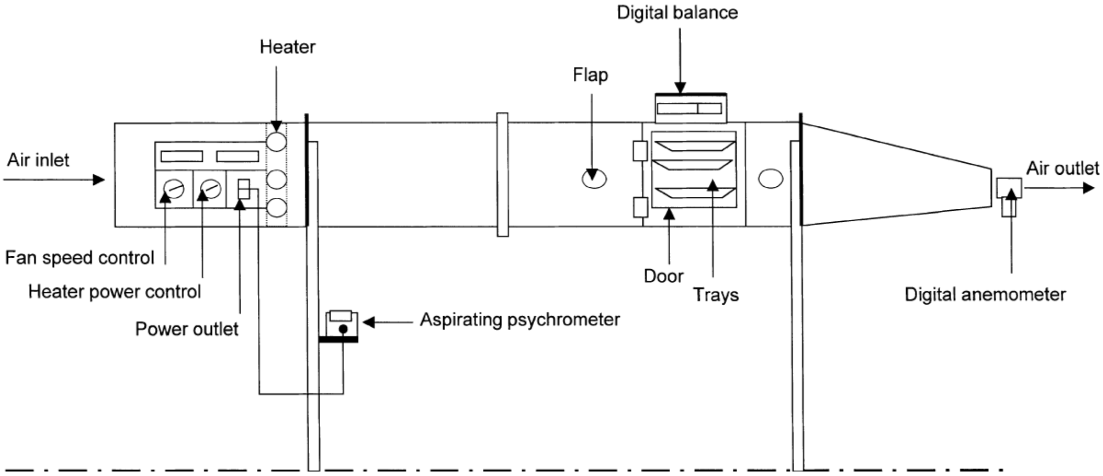

Figure 1.5. Schematic diagram of a laboratory tray type hot air drying equipment

Fresh air drawn in the past the adjustable recirculation damper is pulled through multiple fin-coil steam heater by the centrifugal fan. The fan discharges through a set of turning vanes and perforated screens intended to straighten out and equalize the air flow through the tray section. The entire tray section is supported on a balance arm so that its gross weight can be followed continuously during the drying. Usually, individual trays are easily removable for quick weighing on separate scales (Koçak, 2009).

1.9.2. Microwave drying

Microwaves are electromagnetic waves in the frequency range of 300 MHz to 300 GHz (equivalent to a wavelength of 1±0.01 m), generated by a magnetron-type vacuum tube. Electromagnetic energy at 915 and 2450 MHz can be absorbed by water containing materials or other ``lossy" substances, such as carbon and some organics, and converted to heat (Khraisheh et al., 1997; Maskan, 2000). Because the waves can penetrate directly into the material, heating is volumetric (from the inside out) and provides fast and uniform heating throughout the entire product. The quick energy absorption by water molecules causes rapid evaporation of water (results in higher drying rates of the food), creating an outward flux of rapidly escaping vapour. In addition to improving the rate of drying, this outward flux can help to prevent the shrinkage of tissue structure, which prevails in most conventional air drying techniques. Hence better rehydration characteristics may be expected in microwave dried products (Prabhanjan et al., 1995; Maskan, 2000).

The usual means of applying microwaves to a drying process is at the end of the falling rate period, which is referred to as finish drying (Schiffmann, 2001; Koçak, 2009). Generally, microwave drying of foods or foods components with high moisture content (over 20 % moisture) is not economical. Although water has a high dielectric constant and absorbs microwaves easily, it also has a very high specific heat capacity. Therefore, if the bulk of water is high, a considerable amount of microwave energy will be needed to raise significantly the temperature for drying (Owusu-Annah, 1991; Koçak, 2009). In general, microwave energy has been combined with hot air to shorten the drying times especially in the falling rate periods. In recent years, microwave drying has gained popularity as an alternative drying method for a wide variety of food products such as fruits, vegetables, snack foods and dairy products. Several food products have been successfully dried by the microwave-vacuum application and/or by a combined microwave assisted-convection process and reported by several authors. These authors included Kim and Bhowmik (1995) for plain yogurt, Yongsawatdigul and Gunasekaran (1996) for cranberries, Lin et al. (1998) for carrot slices, Drouzas et al. (1999) for model fruit gels, Al-Duri and McIntyre (1992) for skimmed milk, whole milk, casein powders, butter and fresh pasta, Bouraout et al. (1994) for potato slices, Prabhanjan et al. (1995) for carrots, Tulasidas et al. (1996) for grapes, Funebo and Ohlsson (1998) for apple and mushroom, Ren and Chen (1998) for American ginseng roots, Maskan (2000) for banana and Maskan (2001) for kiwi fruit.

1.9.3. Spray drying

Spray drying is by definition the transformation of feed from a fluid state into a dried form by spraying the feed into a hot drying medium. It is a one continuous suspended particle processing operation. The feed can be either a solution, suspension or paste. The resulting dried product conforms to powders, granules or agglomerates, the form of which depends upon the physical and chemical properties of the feed and the dryer design and operation (Masters, 1976)

Spray-drying is widely used both in food and pharmaceutical manufacturing processes. This technique offers short contact times (5–100 s) and relatively low temperatures, allowing some properties of foods such as flavor, color, and nutrients, to be retained in a high percentages (Masters, 1976; Kuts and Samsonyuk, 1989; Shiga et al. 2001). Schematic diagram of a typical spray dryer is shown in Figure 1.6.

Generally spray drying consists of four process stages (Figure 1.6):
-Atomization of feed into a spray.
-Spray-air contact (mixing and flow)
-Drying of spray (moisture evaporation)
-Separation of dried product from the air

Each stage carried out according to dryer design and operation, and, together with the physical and chemical properties of the feed, determines the characteristics of the dried product. The spray homogeneity fallowing atomization and the high rates of moisture evaporation (spray-air mixing and flow) enable the temperature of the dry product to be considerably lower than the drying air leaving the drying chamber. The product is thus not subjected to high temperatures, and when separated from the drying air is devoid of any heat degradation. The basic physical principle of “evaporation causes cooling” is very pertinent to the operation (Masters, 1976).

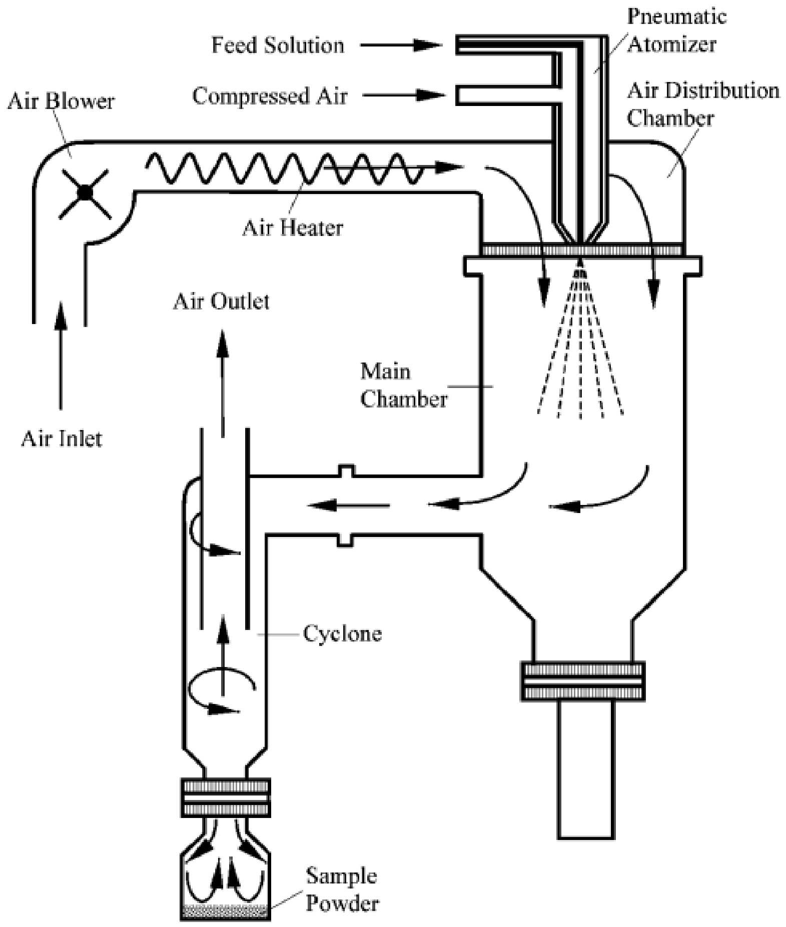

Figure 1.6 Schematic representation of the spray dryer

Atomization:

The formation of a spray (atomization) and the contacting of the spray with air are characteristic features of spray drying. The selection and operation of the atomizer is of supreme importance in achieving economic production of top quality products. The atomization stage must create a spray for optimum evaporation conditions leading to a dried product of required characteristics (Masters, 1976).

Rotary atomizers and nozzles are used to form sprays. With rotary atomizers centrifugal energy is utilized. There are two categories of rotary atomizers: (a) atomizer wheels, (b) atomizer discs. Wheel designs are available to handle feed rates up to tens of tons feed per hour. With nozzle atomization, pressure, kinetic or (less common) sonic energy is utilized. There is a wide range of nozzle sizes and design to meet spray drying needs. Feed capacities per nozzle are lower than per rotary atomizer, leading to nozzle duplications to meet high feed rate requirements (Masters, 1976).

The selection of atomizer depends upon the nature of the feed and the desired characteristics of the dried product. In all atomizer types, increased amounts of energy available for liquid atomization result in sprays having smaller droplet sizes. If the available atomization energy is held constant but the feed rate is increased spraying having larger droplet sizes will result. The degree of atomization depends also upon the fluid properties of the feed material, where higher values of viscosity and surface tension result in larger droplet sizes for the same amount of available energy of atomization (Masters, 1976).

Spray-air contact (mixing and flow):

There are three air flow patterns which are commonly used in spray drying: co current, counter-current, and mixed flow (Figure 1.7). The air pattern used most often with heat sensitive materials is co-current because the product temperature is lower than the inlet air temperature. When high density dried products of heat sensitive materials are required, counter-current flow is utilized. The drying air flows in the opposite direction of the falling particles. If the size of the dryer is limited, mixed flow patterns are used. The most economical spray drying systems have typically been mixed flow, but that flow pattern is not suitable for heat sensitive materials (Filkova and Mujumdar, 1987; Heldman and Lund, 2007). To size a chamber, pilot tests are run to determine the percentage of solids allowable in the feed, the dryer inlet and outlet temperatures, air flow rate, and the configuration of the dryer. The scale up from the pilot test is not precise and requires experience and extensive knowledge of drying operations. Usually, calculations with the air flow rate and temperatures are used to predict the heat load. From the air flow rate and exposure time, the volume of the chamber is calculated (Dittman and Cook, 1977; Heldman and Lund, 2007).

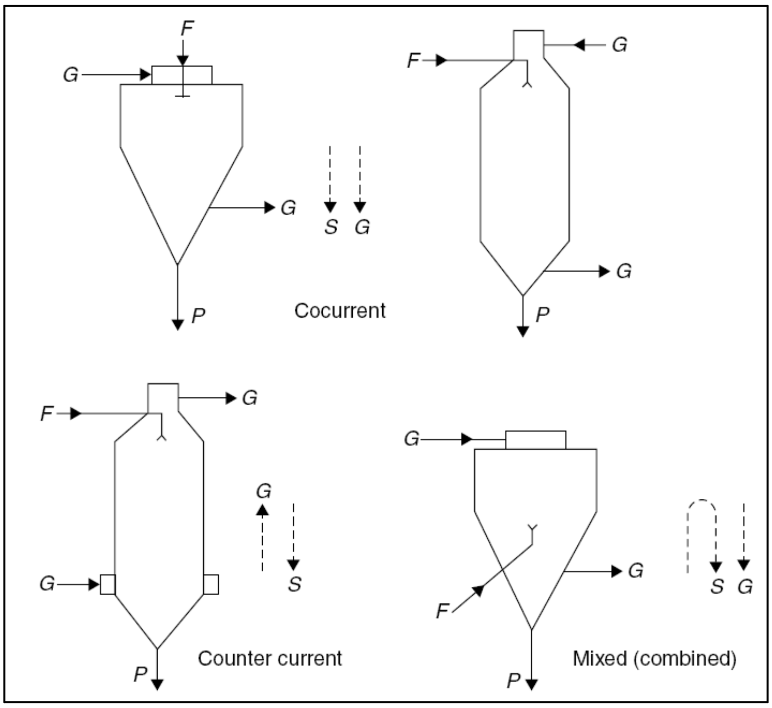

Figure 1.7. Airflow patterns in spray dryers; G: gas, F: feed, P: product, S: spray (Filkova and Mujumdar, 1987).

Drying of spray (moisture evaporation)

As soon as droplets of the spray come into contact with the drying air evaporation takes place from the saturated vapour film, which quickly established at the droplet surface. The temperature at the droplet surface approximates to the wet bulb temperature of the drying air. Evaporation takes place in two stages. At first there is sufficient moisture within the droplet to replenish that lost at the surface. Diffusion of moisture from within the droplet maintains saturated surface conditions and as long as this lasts, evaporation takes place at a constant rate. This is termed the first period of drying. When the moisture content is reduced at a level, where it is insufficient to maintain saturated conditions, the so-called critical point is reached and a dried shell forms at the droplet surface. Evaporation is now dependent upon the rate of moisture diffusion through the dried surface shell. The thickness of the dried shell increases with time causing a decrease in the rate of evaporation. This term is the falling rate period or second period of drying (Masters, 1976).

Separation of dried product from the air

There are many ways to collect the dried product. If the product separates from the air at the bottom of the conical chamber, it is continuously removed through a rotary valve or screw conveyor. It is common for much of the product to remain entrained in the air stream; cyclones, followed by bag filters or wet scrubbers, are used to recover the product. The efficiency of a cyclone is 98 to 99%. It is sometimes desirable to follow the first cyclone with another to collect more of the product (Williams-Gardner, 1971; Heldman and Lund, 2007).

Among these drying techniques, sun drying, solar drying, explosive puff drying, fluidized bed drying, vacuum drying, freeze drying technique are also used in fruits and vegetables drying industry. Since these techniques are not relevant to this study, the details about them will not be given.

1.10. Production of fruit and vegetable juice powders

Fruits and vegetables are seasonal food products so they are not available through a year. Drying is an alternative process for evaluating them through a year. Dried products are used mainly as convenient foods and have long storage life at normal temperatures.

Dehydration by spray drying is used extensively in food related industries for a wide range of products in dry particulate form as powders and agglomerates (Sagar and Suresh Kumar, 2010). Economic considerations include the maintenance of hygienic conditions during processing, operational costs, and short contact time. However, the high sugar content of fruit juice gives it great economical potential, (Cano-Chauca et al. 2005).

It results in powders with good quality, low water activity and easier transport and storage. The physicochemical properties of powders produced by spray drying depend on some process variables, such as the characteristics of the liquid feed (viscosity, particles size, flow rate) and of the drying air (temperature, pressure), as well as the type of atomizer. Therefore, it is important to optimize the drying process, in order to obtain products with better sensory and nutritional characteristics and better process yield (Tonon et al., 2008).

Spray-drying technique offers short contact times (5–100 s) and relatively low temperatures, allowing some properties of foods such as flavour, color, and nutrients, to be retained in a high percentages (Kuts and Samsonyuk, 1989; Shiga et al. 2001;

Rodriguez-Hernandez et al., 2005). However, the main problem during spray drying of sugar-rich foods such as fruit juices is their thermoplastic behaviour (Mani et al, 2002; Rodriguez-Hernandez, 2005). Fruit juices have simple sugars and acids (i.e., sucrose, glucose, fructose, and citric acid, malic acid, ascorbic acid) and their molecular weight are low. For fruit juice powder production two complex problems were available, stickiness of powder and its handling and the other was related to fruit juice natural characteristic that caused no powder production (Chegini and Ghobadian, 2004). The particles stick to one another and to the dryer and cyclone walls and remain there, forming thick wall deposits, while very little product comes out at the dryer's exit (Papadakis et al., 2006). Stickiness is a characteristic of the amorphous state and depends on temperature and moisture content. If particles from materials containing amorphous substances are at temperatures and/or moisture contents higher than what is called the powder sticky point and collide with each other, immobile liquid bridges are formed between them. The mechanism of bridge formation is viscous flow driven by surface energy (Dowton et al., 1982; Wallack and King, 1988; Papadakis et al., 2006).

The sticky-point temperature of amorphous materials has been related by many researchers, (Roos and Karel, 1991; Levine and Slade, 1992; Papadakis et al., 2006) to the glass transition temperature Tg. It has also has been recognized that, because of the rapid removal of water during drying, many spray-dried materials are either completely amorphous or with some crystalline regions dispersed in the amorphous mass. The amorphous form is a non-equilibrium metastable state and will tend eventually to convert to the crystalline. (Alexander and King, 1985; Papadakis et al., 2006). The stickiness behaviour of an amorphous powder depends on the difference between its temperature and the glass transition temperature. At temperatures equal to Tg+10°C, the product begins to show adhesion, and at Tg+20°C it shows stickiness (Bhandari et al., 1997a; Papadakis et al., 2006). Therefore, it seems probable that the problem of product stickiness in spray drying can be avoided if the surface temperature of the particles never exceeds Tg+20°C. Means to achieving this are the increase of Tg through product formulation and/or the prevention of exceeding Tg+20°C through dryer design and proper selection of operating conditions. During the course of drying, the glass transition temperature of the product increases as the moisture content is reduced. At the same time, the temperature of the product during the falling rate period is increased.

The glass transition temperature Tg of an amorphous dehydrated food depends mainly on its moisture and carbohydrate content. The most important plasticizer found in foods is water, with a glass transition temperature of -135°C. Simple sugars, like fructose, glucose, and sucrose, have low glass transition temperatures and their contribution to the depression of the glass transition temperature of sugar-rich foods is very significant. In Table 1.2 (Collares et al., 2004; Papadakis et al., 2006) the glass transition temperatures of certain carbohydrates are presented. As expected from its low value of Tg, fructose has the greatest tendency for stickiness; consequently, it is the most difficult sugar to spray dry successfully. The same applies to foods with high fructose content.

The most common approach to deal with the problem of stickiness in spray drying of sugar rich foods is the addition of drying aid agents (gums, pectins, maltodextrins etc.) to the product being atomized (Papadakis et al., 2006). The addition of high molecular weight additives to the product before atomizing is a widely-used alternative that increases Tg, (Bhandari and Howes 1999; Truong et al. 2005; Shrestha et al. 2007; Yousefi et al., 2010). Carrier agents such as maltodextrins, gum arabic, waxy starch, and microcrystalline cellulose, when introduced into the feed solution, influence the properties and stability of the powder. Crystalline and amorphous forms of the same material powder show differences in particle size, particle shape, bulk density, physicochemical properties, chemical stability, water solubility, hygroscopicity, flow properties and compatibility (Yousefi et al, 2010). The most frequently used additive is 5 to 30 DE (dextrose equivalent) maltodextrin. In Table 1.2, the Tg values of some maltodextrins are presented (estimated from a correlation given in Collares et al., 2004; Papadakis et al., 2006). A very significant observation is that the lower the DE of a maltodextrin, the higher its Tg value is. Therefore, in principle, the lower the DE of the maltodextrin used as drying aid, the lower the amount needed to be added to the food for successful spray drying because the low DE maltodextrin increases more the Tg of the mixture.

Table 1.2. Glass transition temperature of anhydrous sugars and carbohydrate polymers

Food materials	Molecular weight	T_g (° C)
Frucrose	180	5
Glucose	180	31
Galactose	180	32
Sucrose	342	62
Maltose	342	87
Lactose	342	101
Maltodextrin		
DE36	500	100
DE 25	720	121
DE 20	900	141
DE 10	1800	160
DE 5	3600	188
Starch		243

Another explanation of the mode of action of the high molecular weight drying aid agents is that they essentially increase the viscosity of the mixture and thus they decelerate the formation of liquid bridges between the particles and between the particles and the walls (Papadakis et al., 2006).

Besides maltodextrins, other drying aids that have been used are CMC (carboxy methyl cellulose), gum arabic, and pectin. However, none had better results than maltodextrins (Bhandari and Howes, 1999). In general, the addition of maltodextrin has now become common practice because maltodextrin is neutral in taste and color and relatively cheap (Papadakis et al., 2006).

1.11. Quality changes during drying

The primary objective of dehydration is the prevention of occurrence of dangerous or undesirable changes due to activity of microorganisms. This objective is attained by reducing the water activity below the threshold of relevant microbial activity. The reduction of water content and water activity may also have the beneficial effect of reducing the probability of nonmicrobial changes in quality, in particular those due to enzyme action, to nonenzymatic browning, and to hydrolytic reactions. Secondary objectives of dehydration my include (Karel and Lund, 2003);

- Reduction of weight, which facilities shipment
- Potential reduction in volume
- Engineering of desirable textures
- Generation of a food structure useful as a preparation for subsequent processing, such as frying and impregnation with desirable ingredients
- Encapsulation of desirable components

On the other hand, some physical and chemical changes take place during drying such as color degradation, aroma loss, nutritional values, texture, storage stability, etc. Some of the significant features of food dehydration process, which affect both drying rate and final product characteristics, are considered below:

Browning reactions

Browning reactions change color, decrease nutritional value and solubility, create off-flavours, and induce textural changes. Browning reactions can be classified as enzymatic or nonenzymatic, with the latter being more serious as far as the drying process is concerned. The two major types of nonenzymatic browning are caramelization and Maillard browning. In addition to moisture level, temperature, pH, and composition are the other parameters that affect the rate of nonenzymatic browning. The rate of browning is most rapid in the intermediate moisture range and decreases at very low and very high moistures. Browning tends to occur primarily at the center of the drying period. This may be due to the migration of soluble constituents toward the center. Browning is also more severe near the end of the drying period, when the moisture level of the sample is low and less evaporative cooling is taking place that causes the product temperature to rise. Several suggestions were found to help reduce browning during drying. In all the cases, it was emphasized that the product should not experience unnecessary heat when it is in its critical moisture content range (Okos, 1989; Rahman, 2007).

Case hardening

During drying, the concentration of moisture in the outer layers of foods is less than in the interior, since the outer layers necessarily lose moisture before the interior.

This surface shrinkage causes checking, cracking, and warping. This type of shrinkage causes moisture gradient and resistance near the surface. In extreme cases, shrinkage and drop in diffusivity may combine to yield a skin practically impervious to moisture, which encloses the volume of the material so that the interior moisture cannot be removed. This is called case hardening. In food processing, case hardening is also commonly known as crust formation. The extent of crust formation can be reduced by maintaining flattening moisture gradients in the solid, which is a function of drying rate. The faster the drying rate, the thinner the crust (Achanta and Okos, 1996; Rahman, 2007). Crust (or shell) formation may be either desirable or undesirable in dried food products. In microencapsulation of flavours, rapid crust formation is required to prevent flavour losses. Achanta and Okos (1996) pointed that crust formation may be inhibited by allowing the drying rate to be slow enough that moisture loss from the product surface is replenished by moisture from the inside. Crust formation is also important in explosion puffing. In this case, the high-moisture product is exposed to rapid drying conditions such as high temperature and vacuum, which create a crust. The impermeable crust, coupled with the extreme drying conditions, results in rapid moisture vaporization and causes large internal pressures to build up, resulting in product expansion/puffing. During the expansion stage, stress build up in the glassy surface may cause the surface to crack, allowing vapour to escape.

Shrinkage or collapse and pore formation

Shrinkage is an important phenomenon impacting dried food product quality by reducing product wettability, changing product texture, and decreasing product absorbency. Depending on the end use, crust and pore formation may be desirable or undesirable. If a long bowl life is required for a cereal product, a crust product that prevents moisture reabsorption may be preferred. If a product (such as dried vegetables in instant noodles) with good rehydration capacity is required, high porosity with no crust is required. Rahman (2001) provides the latest on the mechanism of pore formation in foods during drying and related processes. Glass transition theory is one of the proposed concepts to explain the process of shrinkage and collapse during drying and other related processes. According to this concept, there is negligible collapse (more pores) in the material if processed below glass transition and higher the difference between the process temperature and the glass transition temperature, the higher the collapse. The methods of freeze drying and hot-air drying can be compared based on this theory. In freeze drying, since the temperature of drying is below Tg' (maximally freeze concentrated glass transition temperature), the material is in the glassy state. Hence shrinkage is negligible. As a result, the final product is very porous. In hot-air drying, since the temperature of drying is above Tg' or Tg, the material is in the rubbery state and substantial shrinkage occurs. Hence, the food produced from hot-air drying is dense and shrivelled (Achanta and Okos, 1996; Rahman, 2007). However, the glass transition theory does not hold good for all products. Other concepts such as surface tension, structure, environment pressure, and mechanisms of moisture transport also play important roles in explaining the formation of pores. Rahman (2001) hypothesized that as capillary force is the main force responsible for collapse, so counterbalancing of this force causes formation of pores and lowers shrinkage. The counterbalancing forces are due to the generation of internal pressure, variation in moisture transport mechanism, and environmental pressure. Other factors could be the strength of the solid matrix (i.e., ice formation, case hardening, and matrix reinforcement).

Rehydration

Rehydration is a process of moistening dry material. It is mostly done by applying an abundant amount of water. In most cases, dried foods are soaked in water before cooking or consumption, thus rehydration is one of the important quality criteria. In practice, most of the changes during drying are irreversible and rehydration cannot be considered simply as a process reversible to dehydration (Lewicki, 1998; Rahman, 2007). In general, absorption of water is fast at the beginning and thereafter slows down. This rapid moisture uptake is due to surface and capillary suction. Rahman and Perera (1999), and Lewicki (1998) reviewed the factors affecting the rehydration process. The factors are porosity, capillaries and cavities near the surface, temperature, trapped air bubbles, amorphous–crystalline state, soluble solids, dryness, anions, and pH of the soaking water. Porosity, and capillaries and cavities near the surface enhance the rehydration process, whereas the presence of trapped air bubbles is a major obstacle to the invasion of the fluid. Until the void or air cavities are filled with water, water penetrates to the material through its solid phase. In general, temperature strongly increases the early stages of water rehydration. There is a resistance of crystalline structures to salvation, whereas amorphous regions hydrate fast. The presence of anions in water affects volume increase during water absorption.

Caking and Stickiness

Caking and stickiness of powders, desirable or undesirable, occur in dried products. Caking is desirable for tablet formation and undesirable when a dry free-flowing material is required. To reduce caking during drying, a logical option is to dry rapidly so that the moisture content drops to a level where caking is inhibited. The rapid drying will form a crust, which may be undesirable, thus product optimization or solutes in product formulation may be considered. Tendencies to form surface folds on particles during spray drying are governed by the viscosity of the concentrated solution. Stickiness and agglomeration tendencies also depend upon the viscosity of the concentrated solution, surface tension, particle size, and exposure time (King, 1984; Rahman, 2007). For viscosities below the critical value, stickiness usually occurs. The predicted critical viscosity was within the range of 108–1010 Pa.s. The mechanism of sticking and agglomeration was postulated through viscous flow driven by surface tension and forming bridges between particles (Dowton et al., 1982; Rahman, 2007). Adhikari et al. (2001) presented a complete review on stickiness in foods, including mechanisms and factors controlling the process. The main factors affecting stickiness are temperature, viscosity, and water, followed by low-molecular sugars, organic acids, and compaction or pressure. The use of a glass transition temperature-based model provides a rational basis for understanding and characterizing the stickiness of many foods.

Vitamin Retention

In general, losses of B vitamins are usually less than 10% in dried foods. Dried foods do not greatly contribute to dietary requirements for thiamin, folic acid, and vitamin B-6. Vitamin C is largely destroyed during drying due to heating (Chang et al., 1996; Rahman, 2007). From nonfatty vegetables as much water as possible should be removed, because this helps to conserve ascorbic acid. The loss of vitamin A and ascorbic acid in dried products could be avoided in the absence of oxygen. Even though most amino acids are fairly resistant to heating drying, lysine is quite heat labile and likely to be borderline or low in the diet of humans and especially so in developing countries where high quality animal proteins are scarce and expensive (Erbersdobler, 1986; Rahman, 2007).

Color retention or development

High temperature and long drying time degrade a product’s original color. Color in foods can be preserved by minimal heat exposure or applying high temperature and short time with pH adjustment. Water activity is one of the important factors degrading chlorophyll. Another cause of color degradation may be due to enzymatic browning causing rapid darkening, mainly of the leafy portions. The formation of dark pigments via enzymatic browning is initiated by the enzyme polyphenol oxidase (PPO). Another reason for discoloration is photooxidation of pigments, caused by light in combination with oxygen (Rahman, 2007).

1.12. Aim of the study

Fruits-vegetables and their derivatives play important roles in our diet. Therefore, many researches have been conducted from past to now. The major reason of this is that they contain natural antioxidant and that the importance of antioxidant for a healthy life is understood. Especially in developed western countries, the relationship between the functional feature of foods being consumed and general state of health have been starting to associate. In this point of view, between consumption patterns of societies and some cronical diseases' risk ratio are associated and are searched scientifically. In this study, two different products are produced from pomegranate since it contains invaluable components.

Many processes such as cold storage, concentration, or drying are used to conserve pomegranates or their juices. One of the main problems occurred in pomegranate juices (PJs) is haze formation. Natural antioxidant and phenolic components are the main factor for this problem. Commercially PJs are manufactured as clear or cloudy juice due to consumer’s expectations by undergoing clarification process. It is known that majority of the health benefit polyphenolics, acting as antioxidants, as well as minerals are removed during juice clarification. In the market, precipitation and haze formation occur in the bottle of cloudy PJs by the time and not preferred by consumers. The reason of not consuming them is that they think the pomegranate juice spoils.

Therefore, the objectives of this work were 1) to produce pomegranate juice in powder form by spray drying pomegranate juice concentrate from unclarified juice, to determine the optimum drying conditions for the production process and the final product quality and to investigate physical, chemical and sensory properties of the powders produced, 2) to dry pomegranate arils by two drying methods; hot air and microwave energy, to model drying kinetics of pomegranate arils. The powder and dry arils to be produced is expected to contain health benefit polyphenols and minerals mentioned and can be used for various purposes such as by dissolving the powder in water to reconstitute a fruit juice or by adding to foods to add the taste, flavour, nutritive value, etc. of the original fruit. The dry aril can be used in different areas; grinding and using as ingredient in many food formulations, consuming as a snack food or rehydrating for compote making.

CHAPTER II

MATERIALS AND METHODS

2.1. Materials

The fresh pomegranates (Hicaz type) were obtained from Gaziantep (Turkey). Chemicals were obtained from Merck (Darmstadt, Germany) and maltodextrin DE 6 was kindly obtained from Cargill (İstanbul, Turkey). Maltodextrin was added to pomegranate juice as carrier agent to prevent stickiness of product by increasing the Tg of the products during spray-drying.

2.2. Methods

2.2.1. Preparation of fresh and concentrated pomegranate juice

Fruits were washed in cold tap water and drained, manually cut-up and the outer leathery skin which encloses hundreds of fleshy arils was removed. The juice that is localized in the arils was manually pressed and extracted and filtered through multi-layer cheesecloth. It was immediately concentrated to 46 °Brix by a rising film evaporator (Armfield FT22-A, England) at 60 °C and 540 mmHg vacuum. Concentrated pomegranate juices were stored at -23 °C in 500 mL bags until used.

2.2.2. Preparation of fresh arils

Fruits were washed in cold tap water and drained. They were manually cut-up and the outer leathery skin which encloses hundreds of fleshy arils was removed and used in tray and microwave drying processes.

2.2.3. Experimental design and data analysis of pomegranate juice powder

The central composite design for three independent variables was performed. The independent variables considered were drying air inlet temperature (°C) (X_1), feed mixture concentration (°Brix) (X_2) and maltodextrin in feed dry solids (%) (X_3). The independent variables and variation levels are shown in Table 2.1. The levels of each variable were established according to literature data and preliminary trials. The outline of experimental design with the coded and actual levels is presented in Table 2.2. Dependent variables were moisture content, hygroscopicity, total anthocyanin content, color, solubility, bulk density, total phenolics content, antioxidant capacity, glass transition temperature, pH, titratable acidity and yield as product responses. Response surface methodology (RSM) was applied for experimental data using a commercial statistical package, Design-Expert version 6.0 (Statease Inc., Minneapolis, MN, USA) for the generation of response surface mathematical models and plots. The same software was used for statistical analysis of experimental data.

RSM has important applications in the design development and formulation of new products, as well as in the improvement of existing product design. It defines the effect of independent variables, alone or in combination, on the processes. In addition to analyzing the effects of independent variables, this experimental methodology generates a mathematical model which describes the chemical or biochemical processes. Mathematical models generated in current study include main parameters and only significant interaction terms.

2.2.4. Drying

2.2.4.1. Spray drying

The spray drying process was performed in a laboratory scale spray dryer LabPlant SD-04 (Huddersfield, England). The spray dryer inlet air temperature was between 100-150 °C and feed mixture concentration (pomegranate juice + maltodextrin DE 6) was between 19.61-44.11 °Brix according to experimental design shown in the Tables 2.1 and 2.2. The proportion of maltodextrin with respect to total solids in feed mixture was 39.08 to 64.12 %. This concentration was selected in a preliminary study, as the lowest concentration without excessive powder stickiness on the chamber wall. Below this concentration, the high powder stickiness resulted in an insignificant process yield. The feed mixture was kept under magnetic agitation, at 40 °C, being fed into the main drying chamber through a peristaltic pump, with drying air flow rate of 0.353 m^3/min. The feed flow rate used was 7 ml/min. The drying air inlet flow rate, feed flow rate and feed temperature were kept constant throughout the experiment.

2.2.4.2. Convection air drying

The convection air drying experiments were performed in a pilot plant tray dryer (UOP 8 tray dryer, Armfield, UK) shown in Figure 1.5.

Fruits were washed in cold tap water and drained. They were manually cut-up and the outer leathery skin which encloses hundreds of fleshy arils were removed. 20 g of arils were put into aluminium dish as a thin layer and replaced in the dryer. The dryer was operated at a constant air velocity of 1.0 m/s and 50, 60, 70 °C. The ambient air was used at 17.8±0.3 °C and 49.5±0.82 % relative humidity. Air was blown into the dryer by means of centrifugal fan with adjustable flow rate parallel to the drying surface of the sample. Moisture loss was recorded by taking out and weighing the dish at 30 minutes interval by a digital balance. Pomegranate arils were dried until equilibrium was reached.

2.2.4.3. Microwave drying

A programmable domestic microwave oven (Arçelik ARMD 580, TURKEY) with maximum output of 700 W at 2450 MHz was used. The oven has adjustable power and time controllers and was fitted with a turntable.

20 g of arils were put into a glass petri dish as a thin layer and replaced in the microwave dryer. The device operated at 210, 350, and 490 W power intensities. The drying was performed according to present powers and pre-determined time schedule. Moisture loss was recorded by taking out and weighing the dish at 5 minutes interval for 210 W, 3 minutes interval for 350 W and 2 minutes interval for 490 W by a digital balance. Pomegranate arils were dried until equilibrium was reached.

Table 2.1. Process variables used in central composite design for three independent variables

		Independent variable levels				
Independent variables	Code	-1.68	-1	0	1	1.68
Air inlet temperature (°C)	X_1	100	110	125	140	150
Feed mixture concentration (°Brix)	X_2	19.61	24.58	31.86	39.10	44.11
Maltodextrin in feed dry solids (%)	X_3	39.08	44.05	51.54	59.08	64.12

Table 2.2. Experimental design for spray drying experiment with coded and actual variable levels

	Coded levels			Actual levels		
Run	X_1	X_2	X_3	Air inlet temperature (°C)	Feed mixture concentration (°Brix)	Maltodextrin in feed dry solids (%)
1	0	0	0	125	31.86	51.54
2	0	0	0	125	31.86	51.54
3	1	1	-1	140	39.10	44.05
4	0	-1.68	0	125	19.61	51.54
5	-1	1	-1	110	39.10	44.05
6	0	0	0	125	31.86	51.54
7	0	0	0	125	31.86	51.54
8	-1	-1	1	110	24.58	59.08
9	-1	1	1	110	39.10	59.08
10	1.68	0	0	150	31.86	51.54
11	1	1	1	140	39.10	59.08
12	1	-1	-1	140	24.58	44.05
13	-1	-1	1	110	24.58	59.08
14	1	-1	1	140	24.58	59.08
15	0	0	-1.68	125	31.86	39.08
16	0	0	0	125	31.86	51.54
17	0	0	0	125	31.86	51.54
18	0	0	1.68	125	31.86	64.12
19	-1.68	0	0	100	31.86	51.54
20	0	1.68	0	125	44.11	51.54

2.2.5. Analysis

2.2.5.1. Determination of total soluble solids

Total soluble solid, as ºBrix, of PJ, PJ concentrate and reconstituted PJ were measured by refractometer (PTR 46, Optical activity limited, UK).

2.2.5.2. Determination of moisture content

The moisture content of powders were determined by the oven method according to AOAC (1995).

2.2.5.3. Hygroscopicity

Hygroscopicity is defined as the mass of moisture (g) absorbed by 100 g of the powder as the equilibrium reached. The experiment was conducted at 25 °C and 95% RH in a desiccator containing barium chloride saturated solution (Moreira et al., 2009). Result was expressed as g water absorbed / g powder.

2.2.5.4. Determination of total anthocyanins content

Total anthocyanin content (TAC) of PJs was determined by pH differential method using two buffer systems: potassium chloride buffer, pH 1.0 (0.025 M) and sodium acetate buffer, pH 4.5 (0.4 M) (Lako et al., 2007). Briefly, 0.4 ml of PJ sample was mixed with 3.6 ml of corresponding buffers and read against water as a blank at 510 and 700 nm. Absorbance (A) was calculated as:

$$A = (A_{510}\text{-}A_{700})pH_{1.0} - (A_{510}\text{-}A_{700})pH_{4.5} \quad (2.1)$$

$$TAC = (A*MW*DF*100)/MA \quad (2.2)$$

where A is absorbance, MW is molecular weight (449.2), DF is dilution factor (10), MA molar absorptivity of cyanidin-3-glucoside (29,600). Total anthocyanin content was expressed as mg cyanidin-3-glucoside/g dry PJ powder (excluding the mass of maltodextrin).

2.2.5.5. Color determination

Colour measurements of the juice samples were carried out using a HunterLab Colorflex (A-60-1010-615 Model Colorimeter, HunterLab, Reston, VA) according to CIELAB system. The PJ powders were reconstituted to 18 ºBrix and color parameters were measured. The colour values were expressed as L* (whiteness or brightness/darkness), a* (redness/greenness) and b* (yellowness/blueness) at any time, respectively. The instrument was standardized each time with a black and a white tile.

Color of pomegranate arils was measured during convectional and microwave drying. Data was taken 30 minutes interval for convectional drying and 5, 3 and 2 minutes interval for microwave drying according to power intensities of 210, 350, and 490 W, respectively. The total color difference (ΔE) was calculated equation 2.3.

$$\Delta E = \sqrt{(L_0^* - L^*)^2 + (a_0^* - a^*)^2 + (b_0^* - b^*)^2} \qquad (2.3)$$

where L_0^*, a_0^*, b_0^* were initial values of PJ; L^*, a^*, b^* were reconstituted PJ or dried aril sample values.

2.2.5.6. Solubility time determination

2 g of PJ powder was added to 50 mL distilled water and mixed with a magnetic stirrer and time was recorded when powder was fully dissolved.

2.2.5.7. Determination of bulk density

Bulk density of PJ powders was measured by weighting of 2 g of a powder sample and placing into a 10 ml graduated cylinder. The bulk density was calculated by dividing the mass of powder by the volume occupied in the cylinder (g/mL).

Bulk density of dried pomegranate arils was measured by weighing a known amount of sample in a 10 ml graduated cylinder. The bulk density was calculated by dividing the mass of dry arils by the volume occupied in the cylinder (g/mL).

2.2.5.8. Determination of total phenolics content

Total phenolic content was determined by Folin-Ciocalteu method. A 0.5 ml of sample was mixed with 2.5 ml of Folin–Ciocalteu's Reagent (FCR-1:10 dilution) and left to stand for 8 min at room temperature to allow for the FCR to react completely with the oxidizable substances or phenolates. A 2.0 ml of Na_2CO_3 (7.5% solution in water) was added to destroy the residual reagent. The absorbances were measured at 760 nm using Perkin Elmer UV-Vis spectrophotometer after incubating at room temperature for 2 h. Results were expressed as mg of gallic acid equivalents per mass of dry PJ powder (excluding the mass of maltodextrin) (Lako et al., 2007).

2.2.5.9. Antioxidant capacity determination

Antioxidant capacity was determined according to the method of Kulkarni and Aradhya (2005). A 0.1 ml of reconstituted PJ powder was mixed with 0.9 ml of 100 mM Tris–HCl buffer (pH 7.4) to which 1 ml of DPPH (0.500 mM in ethanol) was added. The control sample was prepared in a similar way by adding 0.1 ml of water instead of PJ. The mixtures were shaken vigorously and left to stand for 30 min. Absorbance of the resulting solution was measured at 517 nm by Perkin Elmer UV–vis spectrophotometer. The reaction mixture without DPPH was used for the background correction. The antioxidant capacity was calculated using the following equation as measure of antioxidant capacity (AC):

$$\%AC=[1-\frac{A\,sample(517\ nm)}{A\,control(517\ nm)}]*100 \qquad (2.4)$$

where, $A_{sample(517\ nm)}$ and $A_{control(517\ nm)}$ are the absorbance values of sample and control at 517 nm, respectively.

2.2.5.10. pH and titratable acidity determinations

pH measurements were performed using a pH meter (HANNA pH 211) at 25 °C. Total titratable acidity was determined potentiometrically using 0.1 N NaOH to the titration end point of pH 8.1 and expressed as g anhydrous citric acid per mass of dry PJ powder (excluding the mass of maltodextrin) (Poyrazoğlu et al., 2002).

2.2.5.11. Glass transition temperature (T_g) determination

Glass transition temperature (T_g) of powder samples were determined by using a Differential scanning calorimeter (DSC-6, Perkin Elmer, Netherlands) with using

Pyris manager software. The rate of thermal scanning was carried out in the following order:

Isothermal at -30 °C for 1 minute, and then temperature scanning from -30 °C to 20 °C by 10 °C/min rate, isothermal at 20 °C for 1 minute, then cooling from 20 °C to -30 °C by 20 °C/min, isothermal at -30 °C for 1 min, finally scanning from -30 °C to 220 °C by 10 °C/min.

2.2.5.12. Yield

The ratio of weight of the produced powder and consumed feed mixture on a dry basis were used to determine the spray drying yield and expressed as % yield.

2.2.5.13. Sensory analysis

Four PJ powders were selected among 20 PJ powders produced considering maximum antioxidant capacity, maximum solubility, maximum % yield and minimum color change of the samples. They were reconstituted to original PJ concentration (18 ºBrix). A semi-trained panel of 10 students and faculty from Food Engineering Department evaluated the reconstituted samples for color, flavour, aroma and general impression on a 5-point scale (0 = extremely different to 4 = extremely similar). Panellists rinsed their mouths with water after tasting each sample.

2.2.5.14. Optimization

Spray drying process parameters were optimized by using “desirability function” of response surface methodology in order to obtain pomegranate juice powders with maximum antioxidant activity, maximum product yield, minimum color change and acceptable bulk density values.

2.2.5.15. Rehydration capacity determination

Rehydration experiments were performed by soaking a weighed amount of dried aril samples into distilled hot water at 50 °C. At 15 min intervals the samples were withdrawn, drained over a mesh and placed onto two layers of paper towels, quickly blotted 4-5 times in order to eliminate the surface water by gently rolling the arils on the towel and then weighed to an accuracy of 0.0001 g. This procedure was established based on the preliminary test results and other reported studies.

The rehydration capacity, described as percent water gained, was calculated from equation 2.5 (Maskan, 2001).

$$\text{Weight gain (\%)} = \frac{Wt - Wd}{Wd} * 100 \tag{2.5}$$

where, Wt is weight of wet arils at any time and Wd is the initial weight of dry arils.

2.2.5.16. Measurement of shrinkage

For shrinkage studies, similar drying experiments given in Section 2.2.4.2 and 2.2.4.3 were conducted separately using the same hot air and microwave drying conditions. Measurement of shrinkage times were established from preliminary assays.

Volume changes due to sample shrinkage were measured by a water displacement method as described by (Sjöholm and Gekas, 1995; Maskan, 2001). Certain amount of arils whose initial volume is known was immersed in water and volume displayed was recorded immediately periodically. Measurements were made as quickly as possible to avoid water uptake by samples. The shrinkage/volume change of the samples was expressed as a percent shrinkage ratio of sample volume at any time to initial volume.

$$\%\ S = (V/V_0)*100 \tag{2.6}$$

where, S is shrinkage, V is volume of sample (ml) at any time during drying, V_0 is initial volume of sample (ml).

2.2.5.17. Statistical analyses

Spray drying experiments data were analyzed by using Design expert (Version 6.0.1, 2000, Stat-Ease, Inc., Minneapolis, USA) according to response surface methodology. The pomegranate arils drying results were compared by one-way analysis of variance (one-way ANOVA) to test for significant differences. Means of the groups were compared using Duncan’s multiple range test using a SPSS statistical packet (Version 16, 2007, Polar Engineering and Consulting, Nikiski, USA). Differences among sample means were reported to be significant when $p<0.05$.

CHAPTER III

RESULTS AND DISCUSSIONS

Studies with spray dryer aimed to produce pomegranate juice powder from unclarified juice. The effect of independent variables on physical, chemical and sensory properties of powder was investigated. The results were given in Appendix A (Table A1). In this part, multiple regression analyses were carried out using response surface analysis to 1) fit mathematical models to the experimental data, 2) define the relationship between three independent variables and the response variable. The results obtained were then analyzed by ANOVA to assess the "goodness of fit". Only terms found statistically significant ($P<0.05$) were included in the reduced model. Among the three independent variables, the effect of feed mixture concentration (°Brix) on the responses was found insignificant ($P>0.05$) statistically. Therefore, for the sake of simplicity, only plots of drying air temperature-maltodextrin in feed dry solids (%) vs responses were given at constant feed mixture concentration of 44.11 °Brix.

The second part of study involves drying of pomegranate arils by convectional and microwave drying processes. The kinetics of drying data was studied by using the well-known Page model. Also, some physical and chemical properties of dried arils were investigated and compared by one-way ANOVA with respect to drying methods. The results were given in Appendix B.

3.1. Initial properties of maltodextrin, fresh PJ and PJ-concentrate

The maltodextrin DE 6 used had the following specifications: moisture content; 3.76 % wet basis, bulk density (g/ml); 0.448, Hunter color L*, a* and b* values; 97.31, - 0.20 and 0.85, glass transition temperature (Tg); 160 °C and pH; 2.80. Hicaz type pomegranate was used as a raw material for juice powder production. According to analysis results the pomegranate juice has 18.16±1.01 ºBrix values where the pH value as 3.20, acidity content as citric acid 0.0816 g/g PJ dry solids, juice yield as

37.5±7.5 g juice/ 100 g whole fruit, anthocyanin content as 2.62±0.18 mg cyanidin-3-glucoside/g PJ dry solids, total phenolics content as 12.87±0.95 mg gallic acid/g PJ dry solids, antioxidant capacity (%) as 62.01±3.06 found. The color values of pomegranate juice was measured as L*, a* and b* values and found as 6.08, 23.20 and 4.57, respectively. The PJ used in this study has acidic character, as most fruit juices, with high soluble solids content, low juice yield and significant phenolics content. The PJ was concentrated to 45.49±0.96 °Brix and stored at – 23 °C to be used in spray drying experiments later. Before drying, the specifications of PJ concentrate were found follows: L*, a* and b* values were 1.74, 0.79 and 7.75, pH value was 2.80, anthocyanin content was 2.37±0.15 mg/g, total phenolics content was 17.54±2.23 mg/g and antioxidant capacity was 64.97±5.11 %.

3.2. Drying of pomegranate juice: Effect of independent variables on properties of spray dried PJ powder

In all experiments, PJ powder showed a noticeable tendency to stick to internal surfaces of the drying chamber, due to the nature of the low molecular weight sugars and acids present in such products. The results of moisture content, hygroscopicity, anthocyanin content, color, solubility, bulk density, total phenolics content, antioxidant capacity, glass transition temperature, pH-acidity and yield of powders produced with carrier agent (maltodextrin DE 6), sensory analysis of reconstituted PJ from selected powders and optimization of process variables were presented in figures and tables.

3.2.1. Moisture content

Effect of temperature and maltodextrin on the moisture content of the PJ powders is shown in Figure 3.1. The moisture content of powders varied from 3.44 to 9.13% (w.b). As it can be drawn from Figure 3.1, increases in air inlet temperature and maltodextrin lead to lower moisture contents. As shown in equation 3.1, the model obtained for predicting the moisture content of PJ powders explained the main linear effects of factors affecting the moisture content. Although it seems that temperature and maltodextrin effected moisture content, the model is not significant statistically ($p>0.05$). The lowest moisture content of powders was obtained at the maximum temperature and maltodextrin ratios. At low maltodextrin ratio, the increase in temperature resulted in a slight increase in moisture content, while at high maltodextrin ratio, the increase in temperature led to decrease in moisture content but not significant ($p>0.05$). On the other hand, at low and high temperature, the increase in maltodextrin ratio led to sharp decrease in moisture content. According to Figure 3.1, maltodextrin had higher effect on moisture content of the powder than temperature. The increase in maltodextrin caused decrease in moisture content of PJ powder significantly ($p<0.05$) due to increasing of solid content of mixture and loose binding character of water to maltodextrin solids. Quek et al. (2007) observed the same trend for spray drying of watermelon juice.

The previous workers (Kha et al., 2010; Chegini and Ghobadian, 2005; Goula and Adamopoulos 2004) showed that increasing the inlet air temperature increases the drying rate and as a result, the moisture content of the powder is reduced. Similarly, Tonon et al. (2008) reported that increasing temperature caused decrease of moisture content of açai powders during spray drying.

Moisture content $= -61.49 + 0.34*X_1 + 0.91*X_2 + 1.31*X_3$ (3.1)

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

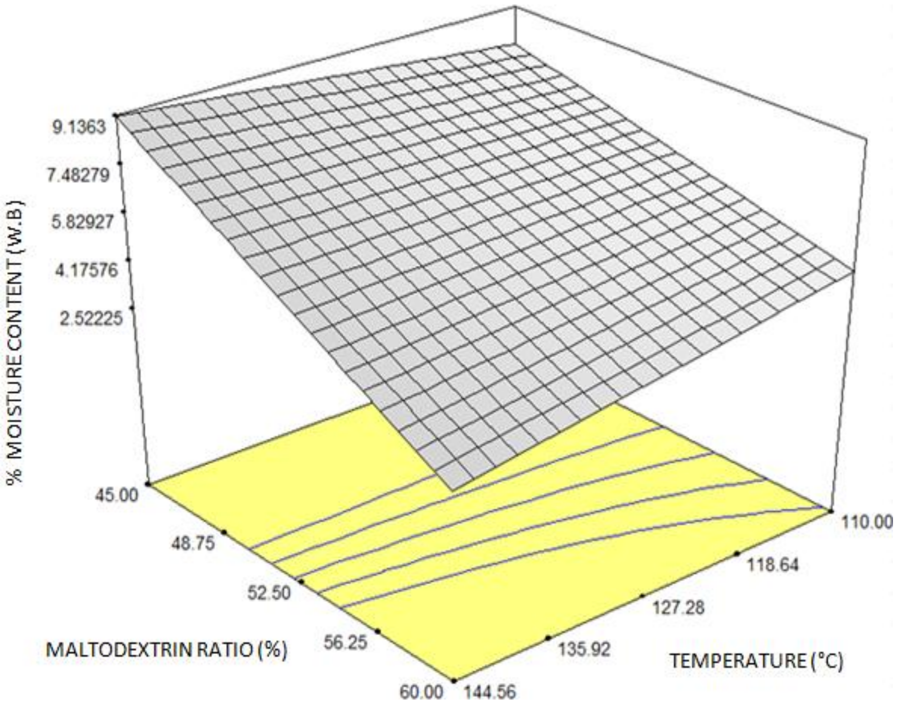

Figure 3.1. Effect of air inlet temperature and maltodextrin ratio on the moisture content of PJ powders

3.2.2. Hygroscopicity

Moisture absorption of the spray-dried powders at 25 ºC and 95 % relative humidity after equilibrium reached was shown in Figure 3.2. Temperature did not affect hygroscopicity of PJ powder significantly ($P>0.05$) but maltodextrin did. However, studies conducted by Moreira et al. (2009) and Goula and Adamopoulas (2008) on spray drying of acerola pomace and tomato puree, respectively, revealed that increase in temperature caused decreasing of hygroscopicity. Increasing amount of maltodextrin in feed mixture caused decrease in water holding capacity of powders (Figure 3.2). This result agrees well with the moisture content results (i.e., as increasing maltodextrin content decreases the moisture content of powder as shown in Figure 3.1). At low and high temperatures, the increase in maltodextrin ratio resulted in a sharp decrease in hygroscopicity of powders. This is in agreement with the literature findings. Rodriguez et al. (2005) reported that lower hygroscopicity was obtained when amount of maltodextrin increased in cactus pear juice during spray drying. Also, similar results were determined by other researchers (Moreira et al., 2009; Bhandari and Hartel, 2005; Bhandari et al., 1993, 1997a, 1997b; Silva et al., 2006). It is known that low molecular weight components (simple sugars and organic acids as seen in fruit juices) have more hydrophilic groups, hence, their hygroscopicity is higher. Maltodextrin have high molecular weights compared to sugars and the acids and relatively have lower hygroscopicity. Due to this reason, maltodextrin increases bulk molecular weight of pomegranate powder mixture and decreases water absorption capacity of powders (Kuruzowa et al., 2009). The model obtained for predicting the hygroscopicity of PJ powders explained the main linear effects of factors affecting the hygroscopicity (equation 3.2). A negative coefficient means a decrease in hygroscopicity when the level of the variable (temperature and maltodextrin ratio) is increased.

$$\textbf{Hygroscopicity} = 0.64 - 1.12*10^{-4}*X_1 - 6.18*10^{-5}*X_2 - 5.20*10^{-3}*X_3 \qquad (3.2)$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

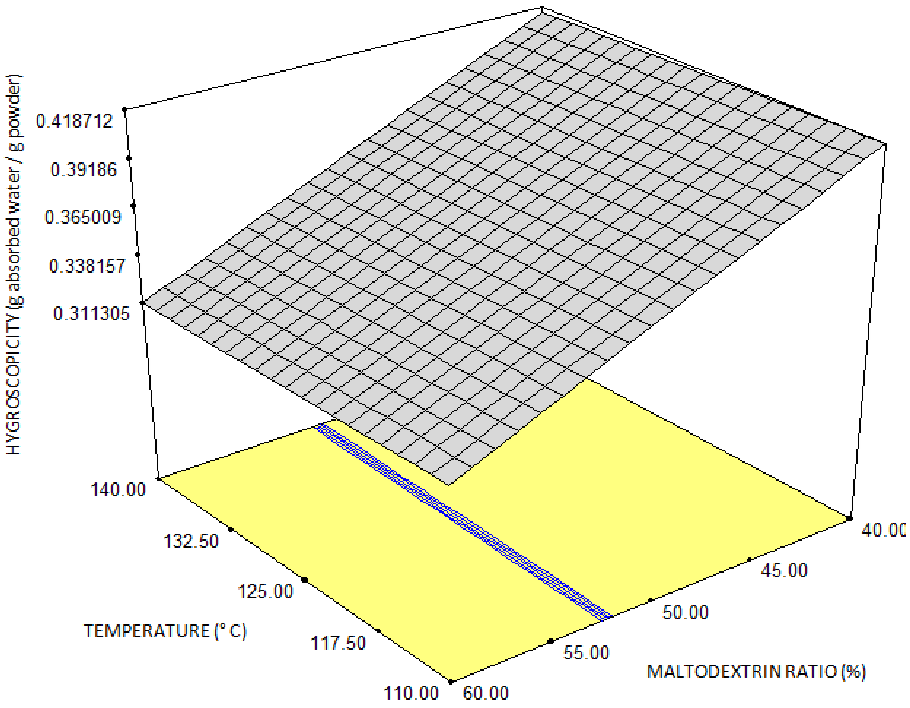

Figure 3.2. Effect of air inlet temperature and maltodextrin ratio on hygroscopic behaviour of PJ powders

3.2.3. Anthocyanin content

Anthocyanins provide the characteristic color of fruits and vegetables because contain color pigments. Unstable anthocyanins are negatively affected by heating and finally denaturated or polymerized with other phenolic compounds caused color loss (Maskan, 2006). Anthocyanin content of PJ powders was determined by pH differential method using UV-Vis spectrophotometer. As shown in Figure 3.3, at low maltodextrin ratio, the increase in temperature resulted in a sharp decrease in anthocyanin content. May be at this maltodextrin ratio, encapsulation of anthocyanins was not sufficient that caused sharp decrease in anthocyanin content with increase in temperature. Researches conducted by Tonon et al. (2008) investigating spray drying of a tropical fruit açai, Quek et al. (2007) spray drying watermelon, Cai and Corke (2000) spray drying amaranthus betacyanin pigment and Ersus and Yurdagel (2007) studying on drying behaviour of black carrot revealed that increasing temperature negatively affected anthocyanin content of the material studied. On the other hand, at high maltodextrin concentration, the increase in temperature resulted in a slight increase in anthocyanin content. This may be due to high encapsulation rate of excess maltodextrin present in the feed mixture, preventing anthocyanins from degradation as reported by Kha et al. (2010). Similar slight increases in anthocyanins with temperature had been observed by Ersus and Yurdagel (2007) in spray drying of black carrot study. At low temperature, the increase in maltodextrin ratio resulted in a slight decrease in anthocyanin content but not statistically significant ($p>0.05$). It is not in agreement with the results of Kha et al. (2010). At high temperature, the increase in maltodextrin ratio resulted in a sharp increase in anthocyanin content. It could be said that at high temperature and maltodextrin ratio there was an efficient encapsulation that protect anthocyanins against degradation anthocyanin content. Similar results have been reported by Cai and Corke (2000), Kha et al. (2010) and Robert et al. (2010). The model obtained (equation 3.3) for predicting the anthocyanins content of PJ powders explained the main linear, quadratic and interaction effects of factors affecting the anthocyanins content. A negative coefficient means a decrease in response when the level of the variable is increased, whereas a positive coefficient indicates an increase in response. The interaction suggests that the level of one of the interactive variables may increase while that of the other may decrease for a constant value of the response.

$$\begin{aligned}\textbf{Anthocyanin content} = {} & 14.87 - 0.07*X_1 - 5.18*10^{-3}*X_2 - 0.03*X_3 + 1.54*10^{-3}*X_3^2 \\ & + 1.19*10^{-3}*X_1*X_3\end{aligned} \tag{3.3}$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

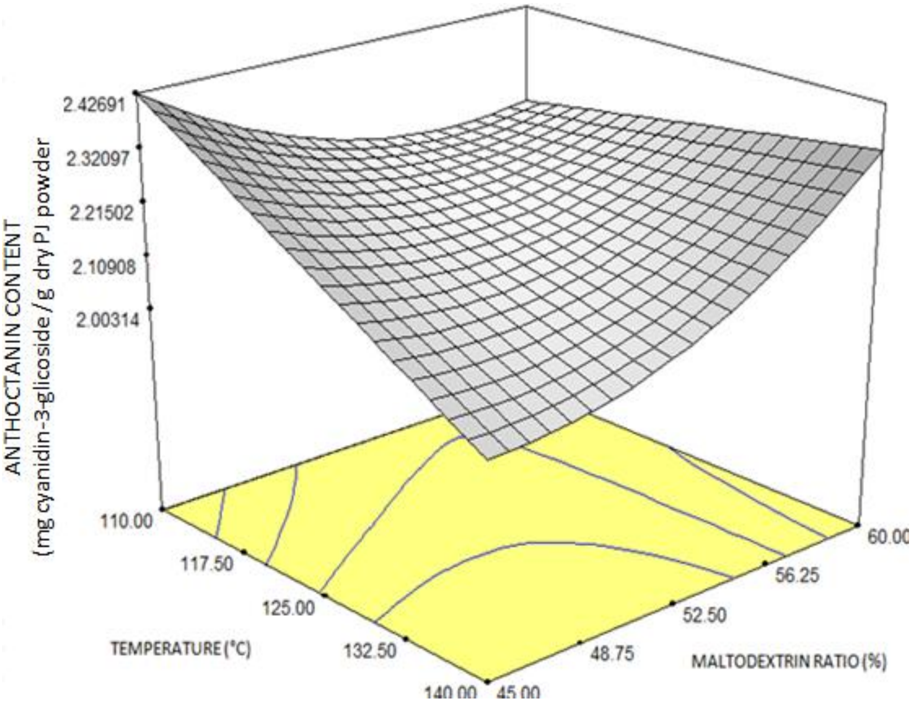

Figure 3.3. Effect of air inlet temperature and maltodextrin ratio on anthocyanin content of PJ powders

3.2.4. Color determination

Change in total color difference of reconstituted (18 ºBrix) powders was shown in Figure 3.4. It showed that as the temperature and ratio of maltodextrin in the mixture increase, change in total color difference also increases. At low and high temperatures, the increase in maltodextrin ratio resulted in large rise in ΔE value. However, at low and high maltodextrin ratios, the increase in temperature caused a slight increase in ΔE value. It can be argued that maltodextrin affects total color difference more than temperature. Maximum color difference was observed at the highest temperature and highest maltodextrin ratio. This may be due to the sensitivity of color pigments to heating process and diluting effect of maltodextrin (colorless) in color. A similar study by Altan and Maskan (2004) on spray drying of gilaboru *(Vibirnum Opulus L.)* juice found that addition of maltodextrin and increasing air inlet temperature to above 130 °C also increased the change in total color differences of the products. Also, Rodriguez et al. (2005) determined that as they increased the amount of maltodextrin in cactus pear juice during spray drying, the color of product changed significantly. Another reason for change in color at high temperature may be due to nonenzymatic browning of simple sugars present in pomegranate.

Also, Hunter a^* value is an important parameter for many fruit juices because it is directly related to redness and it is investigated in many studies in order to measure the extent of red color degradation. Figure 3.5 shows change in Hunter a^* value of reconstituted pomegranate powder juice. The Hunter a^* value decreased with increasing inlet air temperature ($p<0.05$) and maltodextrin ratio in the mixture. This was expected because of thermal degradation of red color and diluting effect of maltodextrin. At low and high temperatures, the increase in maltodextrin resulted a sharp decrease ($p<0.05$) in Hunter a^* value, and at low and high maltodextrin ratios, there was slight decrease in this parameter when temperature was increased. It could be concluded that maltodextrin greatly affected Hunter a^* value than temperature. At highest process temperature and maltodextrin content the Hunter a^* had minimum value. This may be caused by oxidation and/or thermal degradation of the pigments during drying, caramelization of sugars in the pomegranate juice and occurrence of non-enzymatic browning reaction (Kha et al., 2010). Similar results have been observed by Tonon et al. (2008) and Quek et al. (2007) on spray drying of açai juice and watermelon, respectively. They reported that contents of the component that give redness (anthocyanin, β-carotene and lycopene) to these products had decreased significantly as drying temperature was increased. Also, maltodextrin had high L^* (97.31) and low a^* (-0.20) value. Therefore, the more maltodextrin in the mixture, the lower the bulk a* value of reconstituted pomegranate powder juice was. The models obtained for predicting the ΔE and Hunter a* values of reconstituted PJ explained only the main linear effects of factors affecting ΔE and a* values (equations 3.4 and 3.5).

$$\mathbf{\Delta E} = -6.27 + 0.04*X_1 - 8.21*10^{-3}*X_2 + 0.23*X_3 \tag{3.4}$$

$$\textbf{Hunter } \mathbf{a^*} = +31.35 - 0.04*X_1 + 1.45*10^{-3}*X_2 - 0.19*X_3 \tag{3.5}$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

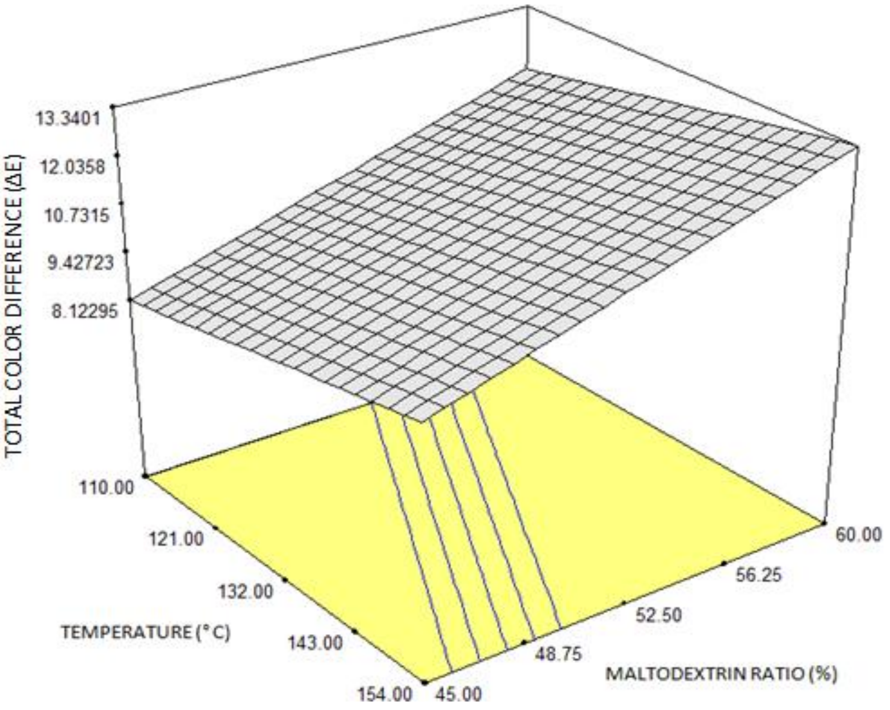

Figure 3.4. Effect of air inlet temperature and maltodextrin ratio on total color difference values of reconstituted PJ powder

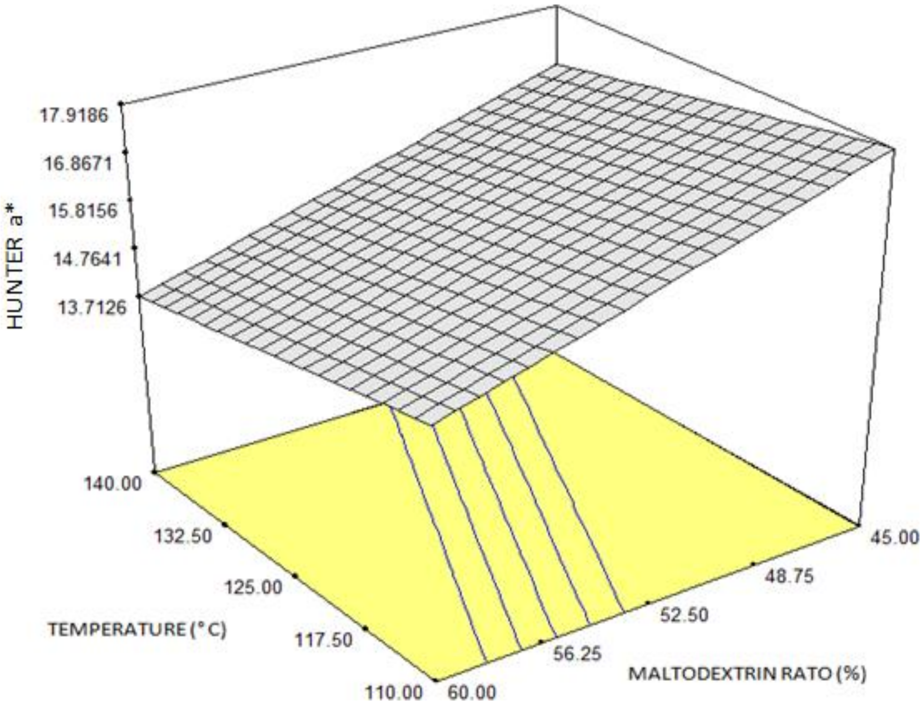

Figure 3.5. Effect of air inlet temperature and maltodextrin ratio on Hunter a* values of reconstituted PJ powders

3.2.5. Solubility

The solubility time of PJ powder against drying temperature and maltodextrin ratio is given in Figure 3.6. The little time was a measure of high solubility of powders. Solubility of powders decreased with increasing temperature and maltodextrin content of feed mixture. It is evident from equation 3.6 that positive coefficients indicate an increase in the response (solubility time). However effect of maltodextrin was more significant than that of temperature ($P<0.05$). The solubility of PJ powders was the lowest (high solubility time) at highest maltodextrin ratio (60 %) and drying temperature (140 ºC). Maltodextrin has lower solubility in cold water compared to PJ powder due to its high molecular weight. Adding more maltodextrin may increase bulk solubility time of the powder. Also, high drying temperature may damage water absorption sites of powder. This may be another reason decreasing the solubility as temperature increased. Quek et al. (2007) and Goula et al. (2004) obtained similar results from spray drying of watermelon juice and tomato juice.

Solubility $= + 34.13 + 0.13*X_1 + 0.49*X_2 + 0.17*X_3$ (3.6)

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

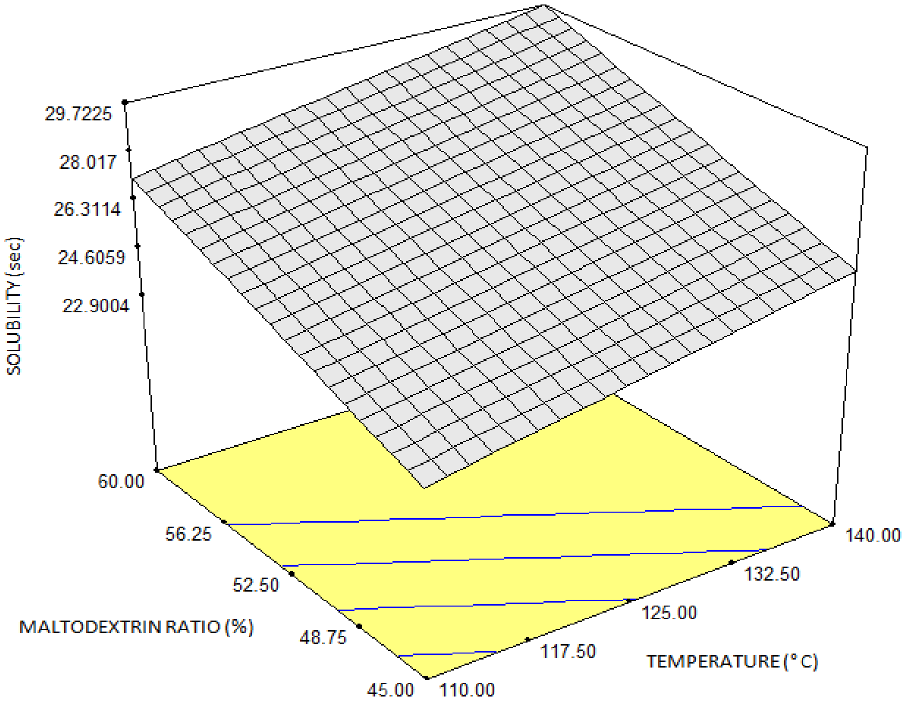

Figure 3.6. Effect of air inlet temperature and maltodextrin ratio on solubility of PJ powders

3.2.6. Bulk density

Bulk density was expressed as weight of powders (g) per unit volume (mL). Experimental bulk density values of powders changed between 0.3450 to 0.4720 g/mL. The relationship between bulk density-maltodextrin-drying temperature is shown in Figure 3.7. At high maltodextrin ratio, temperature had no effect on bulk density. However, at low maltodextrin ratio, temperature affected bulk density significantly ($P<0.05$), i.e., as temperature increased bulk density increased or vice versa. This behaviour can be related to moisture content of powder. PJ powder had high moisture content at low drying temperature. If the moisture content of powder is high, the more particles tend to stick together, leaving more interspaces between them and consequently resulting in a larger bulk volume dependently small bulk density. In contrast to our results, Kha et al. (2010) Cai and Corke (2000), Goula et al. (2004) reported that increasing residual moisture content increased bulk density of a dry product that they investigated. The increase in maltodextrin ratio at low drying temperature, resulted in an increase in bulk density due to poor water binding character of maltodextrin. The low moisture content decreases interspaces between particles, then, bulk density increases. Goula and Adomopoulos (2008) and Abadio et al. (2004) reported that increasing maltodextrin ratio decreases bulk density of a dry product. The model obtained (equation 3.7) for predicting the bulk density of PJ powders explained the main linear, quadratic (of maltodextrin) and interaction (of temperature and maltodextrin) effects of factors affecting bulk density. The interaction suggests that the level of one of the interactive variables may increase while that of the other may decrease for a constant value of bulk density.

$$\textbf{Bulk density} = -0.51 + 0.01*X_1 + 1.01*10^{-3}*X_2 - 2.50*10^{-3}*X_3 + 2.91*10^{-4}*X_3^2 - 2.19484*10^{-4}*X_1*X_3 \qquad (3.7)$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

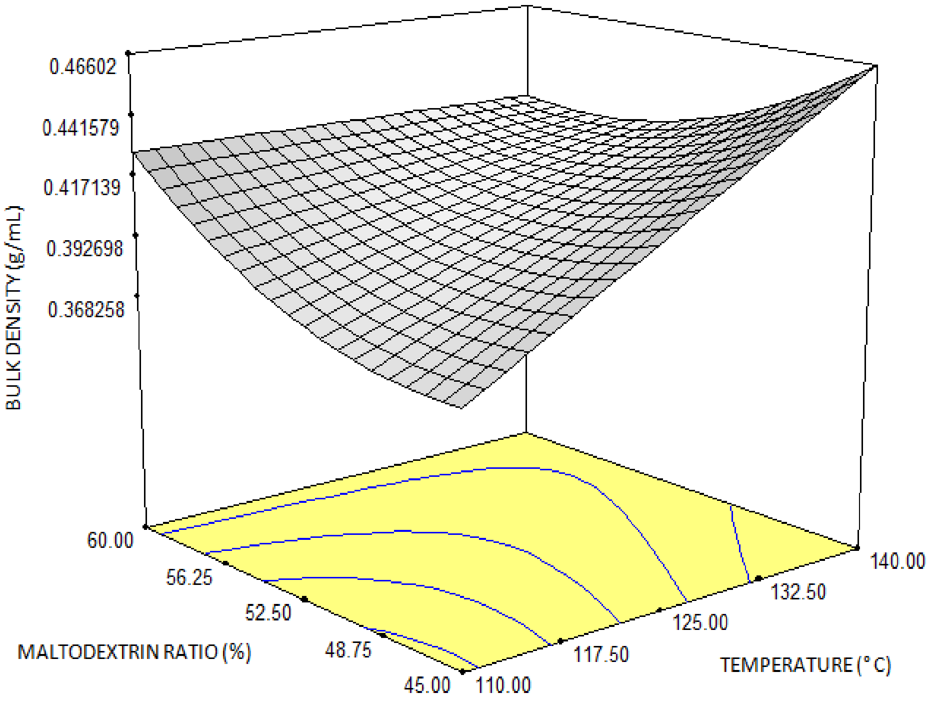

Figure 3.7. Effect of air inlet temperature and maltodextrin ratio on bulk density of PJ powders

3.2.7. Total phenolics content

The change in total phenolics content of spray dried PJ powders, on a dry basis, vs independent variables is shown in Figure 3.8. As shown, temperature and maltodextrin affected total phenolics content proportionally ($p>0.05$). The model obtained for predicting the phenolics content of PJ powders explained the main linear effects of factors (temperature and maltodextrin) affecting the total phenolic content (Equation 3.8). Positive coefficients indicated an increase in phenolics content when the level of the variable (temperature and maltodextrin) was increased. At low and high maltodextrin ratios, the increase in temperature resulted in an increase in total phenolics content. Similarly, at low and high temperatures, the increase in maltodextrin ratio resulted in an increase in total phenolics content of PJ powders. However, maltodextrin had greater effect ($P<0.05$) on total phenolics content than temperature statistically. This may be due to encapsulation effect of maltodextrin protecting phenolic compounds against external actions (Kha et al., 2010). Inactivation of polyphenol enzymes by heating process during spray drying may be another reason for increased amount of phenolics (Oludemi and Akanbi, 2010).

Türkmen et al. (2005) investigated the total phenolics and antioxidant activities of some vegetables during heating process. They observed that total phenolics content of squash, peas and leek decreased, but of green pepper, green beans, broccoli and spinach increased. Also, Stewart et al. (2000) found that free flavanol contents of vegetables increased with heating process. These were in agreement with our results.

Total phenolics content $= +1.19 + 0.05*X_1 + 5.88*10^{-3}*X_2 + 0.14*X_3$ (3.8)

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

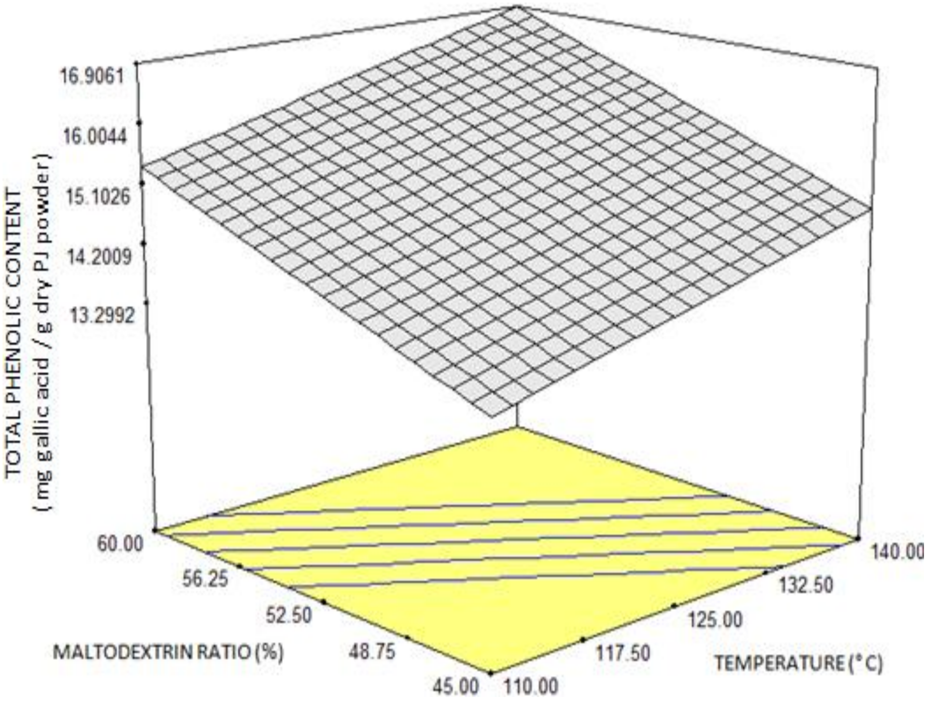

Figure 3.8. Effect of air inlet temperature and maltodextrin ratio on total phenolic content of PJ powders

3.2.8. Antioxidant capacity

Natural antioxidants in foods are very effective against free radicals that negatively affect human health. Pomegranate contains significant amount of phenolic compounds which was antioxidant property. These compounds play important role on human health by reacting with free radicals (Gil et al., 2000).

Antioxidant capacity of spray dried PJ powder against independent variables is shown in Figure 3.9. Drying temperature significantly increased antioxidant capacity of PJ powder ($P<0.05$). It increased with increasing drying air temperature. Similar results are present in the literature. Tezcan et al. (2009) indicated that commercially sent PJs have higher antioxidant capacity than fresh PJ. This situation could be explained that commercial PJs undergo heat treatment, and some chemical reaction can take place during heat treatment. Maillard reaction is one of these reactions. The product from Maillard reaction has very high antioxidant capacity (Morales and Jimenez-Perez, 2001). Oludemi and Akanbi (2010) reported that antioxidant capacity of tomato increased by heat treatment. Türkmen et al. (2005) investigated effect of heat treatment on antioxidant capacity of green vegetables and observed that antioxidant activity of green pepper, green beans, broccoli and spinach increased with increasing temperature, and antioxidant capacity of squash, peas and leek remain the same as fresh ones. They argued that the reason for increased antioxidant activity with heat treatment may be due to inactivation of peroxidase enzyme. Other authors; Manzocco et al. (2001) and Nicoli et al. (1999) investigated nonenzymatic browning and antioxidant capacity of heat treated fruits. They reported that heat treatment caused increase in antioxidant capacity of fruits because of Maillard product formation. For the same reason the antioxidant capacity of pomegranate juice powder could increase in the current study.

Figure 3.9 clearly shows that maltodextrin had no significant effect ($P>0.05$) on antioxidant capacity of PJ powder. Because maltodextrin doesn’t have any antioxidative property. Such results have been reported by several authors (Anese et al., 2003; Tezcan et al., 2009; Oludemi and Akanbi, 2010; Türkmen et al., 2005; Zhang ve Hamauzu, 2004; Vega-Galvez et al., 2008).

Higher antioxidant capacity of the powder is one of the main outputs of this research. But it is not known how much activity comes from pomegranate natural components and how much due to formation of Maillard products. The increase in antioxidant capacity results agrees well with the total phenolics content results discussed in Section 3.3.7. The model obtained for predicting the antioxidant capacity of PJ powders explained the main linear effects of factors (temperature and maltodextrin) affecting the antioxidant capacity (equation 3.9).

$$\textbf{Antioxidant capacity} = +203.99 - 1.03*X_1 - 5.52*X_2 - 0.09*X_3 \qquad (3.9)$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

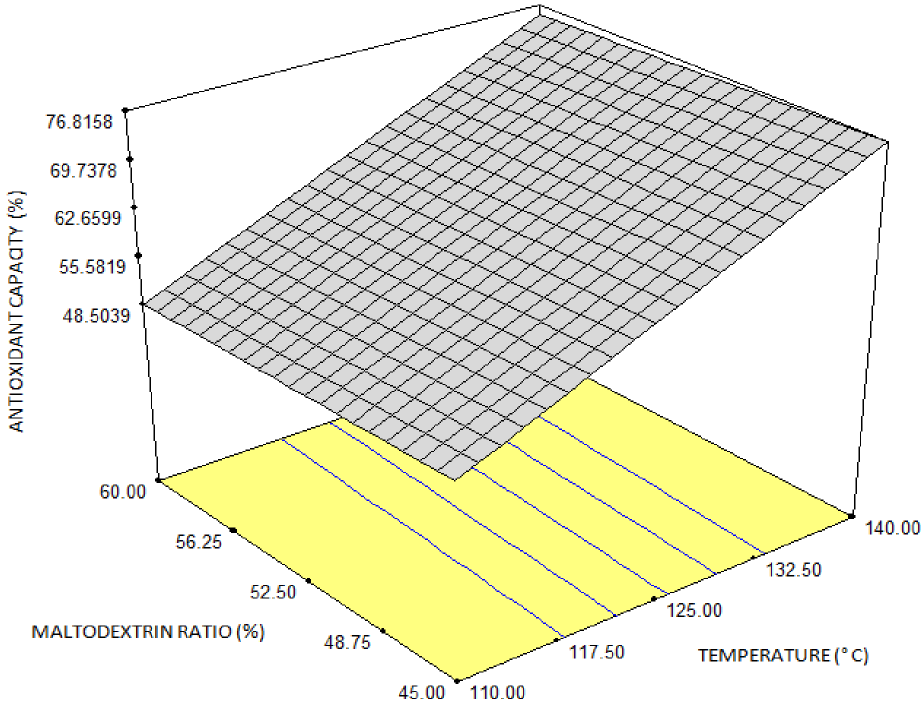

Figure 3.9. Effect of air inlet temperature and maltodextrin ratio on antioxidant capacity of PJ powders

3.2.9. Glass transition temperature

The sticky-point temperature of amorphous materials has been related by many researchers (Roos and Karel, 1991; Levine and Slade, 1992; Papadakis et al., 2006) to the glass transition temperature, Tg. It has also been recognized that, because of the rapid removal of water during drying, many spray-dried materials are either completely amorphous or with some crystalline regions dispersed in the amorphous mass. The amorphous form is a non-equilibrium metastable state and will tend eventually to convert to the crystalline (Alexander and King, 1985; Papadakis et al., 2006).

The glass transition temperature of pomegranate juice powders was measured by using DSC. The Tg values of pomegranate juice powders changed between 84.59 and 108.76 ºC. The highest and lowest Tg values were obtained from the samples with highest and lowest maltodextrin contents, respectively (Table 3.1). This was expected, because maltodextrin has high Tg value. Addition of maltodextrin to pomegranate juice increased the bulk Tg value of mixture. The model obtained (Equation 3.10) for predicting the Tg values of PJ powders explained the main linear, quadratic (of maltodextrin) and interaction effects of factors affecting glass transition temperature.

$$\textbf{Glass transition temperature} = -323.67 + 2.00*X_1 - 0.09*X_2 + 11.69*X_3 - 0.061547*X_3^2 - 0.041218*X_1*X_2 \tag{3.10}$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

Table 3.1. Glass transition (Tg) temperature of PJ powders

Run	Glass Transition Temperature (Tg), °C	Run	Glass Transition Temperature (Tg), °C
1	100.88	**11**	98.87
2	98.53	**12**	94.9
3	90.69	**13**	95.93
4	101.95	**14**	97.98
5	88.09	**15**	84.59
6	97.26	**16**	91.59
7	98.56	**17**	99.69
8	102.73	**18**	108.76
9	105.5	**19**	92.46
10	93.63	**20**	103.79

3.2.10. pH and acidity

The pH and acidity values of PJ powders are shown in Table 3.2. pH and acidity values changed between 3.0-3.1 and 0.0948-0.1351 g citric acid/g dry PJ powder, respectively. Gölükcü and Tokgöz (2008) investigated some properties of different pomegranates grown in Turkey. They reported that pH value of Hicaz type, which was used in this research, was 3.11. The change in these parameters were not significant statistically ($P>0.05$). These findings were in agreement with the result of Gonzalez-Palomares et al. (2009). Also, Kha et al. (2010) found that spray drying did not change the pH of Gac fruit aril significantly. The models obtained (Equation 3.11 and 3.12) for predicting the pH and acidity values of PJ powders explained only the main linear effects of factors affecting these responses.

$$\mathbf{pH} = +4.48 - 0.01*X_1 + 9.75*10^{-5}*X_2 - 0.02*X_3 \quad (3.11)$$

$$\mathbf{Acidity} = +0.10 - 7.35*10^{-5}*X_1 - 1.04*10^{-4}*X_2 + 4.15*10^{-4}*X_3 \quad (3.12)$$

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

Table 3.2. pH and acidity values of PJ powders

Run	pH	Acidity (g citric acid/g dry PJ powder)	Run	pH	Acidity (g citric acid/g dry PJ powder)
1	3.02	0.1097	**11**	3.07	0.1126
2	3.00	0.1351	**12**	3.07	0.0948
3	3.02	0.1082	**13**	3.07	0.1128
4	3.03	0.1087	**14**	3.06	0.1117
5	3.05	0.1030	**15**	3.07	0.1108
6	3.03	0.1227	**16**	3.06	0.1252
7	3.04	0.1218	**17**	3.08	0.1085
8	3.04	0.1265	**18**	3.09	0.1060
9	3.06	0.1186	**19**	3.10	0.1067
10	3.07	0.1179	**20**	3.06	0.1045

3.2.11. Yield

The yield of process is related to the mass ratio of dry product obtained after drying process. PJ powder yield results obtained at different experimental conditions were given in Figure 3.10. Product yield increased significantly ($P<0.05$) with increasing maltodextrin ratio in the mixture. Maltodextrin encapsulated organic acid and simple sugars present in pomegranate juice. This could help free flowing of PJ particles through the drying chamber. i.e., stickiness to the chamber wall was prevented. Temperature had no significant effect ($P>0.05$) on the yield. Only a slight decrease in yield was observed as temperature increased ($p>0.05$). The yield values of pomegranate juice powders changed between 86.0 and 19.67%. The highest and lowest yield values were obtained from the samples with the highest and lowest maltodextrin contents, respectively. In literature, it was reported that even 50 % yield could be acceptable. In our study, 8 runs resulted in more than 50 % yields. Papadakis et al. (2006) studied spray drying of raisin juice. They found that yield increased by increasing the maltodextrin ratio in the mixture. This was in agreement with our study. The mathematical model found (Equation 3.13) contains only the main linear effect of factors, as significant, affecting yield of PJ powder.

Yield = $-39.47 - 0.25*X_1 + 0.67*X_2 + 1.86*X_3$ (3.13)

Where X_1 is the air inlet temperature, X_2 is the feed mixture concentration and X_3 is the maltodextrin in feed dry solids.

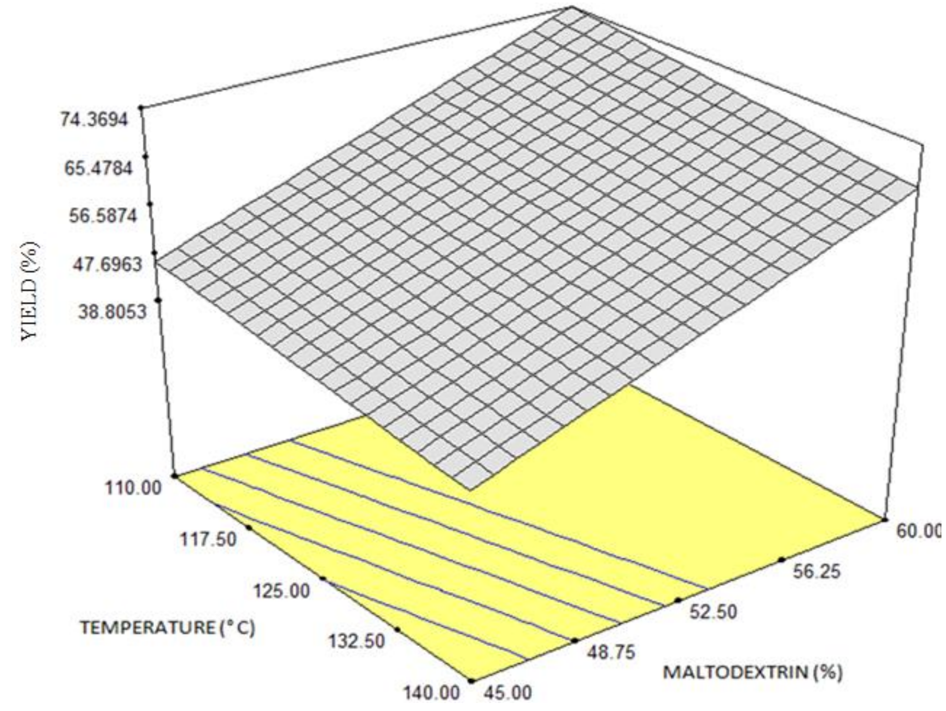

Figure 3.10. Effect of air inlet temperature and maltodextrin ratio on yield of PJ powders

3.2.12. Sensory analysis

Four PJ powders were selected among 20 PJ powders produced considering maximum antioxidant capacity (76.96 %), maximum solubility (16.20 sec), maximum yield (86.08 %) and minimum total color change (7.34) of the samples for sensory analysis. These are the runs numbered 2, 19, 18 and 15, respectively. The sensory analysis results were obtained from 10 panellists and averaged (Table 3.3). According to these results, it is seen that all features of the sample obtained from test condition number 15 is the most similar to the original sample in comparison with the other samples. This product has the lowest color change value according to experimental measurements. Also, it contains less maltodextrin. The other scores, aroma, flavour and overall acceptability got higher scores than the other runs. Besides, antioxidant activity value of the sample 15 is 68 % and water solubility value is 16.5 seconds. All these characteristics of PJ powder (run no 15) are acceptable, but its yield is very low (19.67 %). However, the product with the highest yield (run 18) got the lowest grade. This product contained the most maltodextrin. It is understood from those results that maltodextrin has an important effect on color, aroma, taste and general impression.

Table 3.3. Sensory analysis of selected PJ powders

Run No	Color	Aroma	Flavor	General Impression
2	1.60±0.667	1.70±0.737	2.00±0.667	1.70±0.471
15	3.00±0.667	2.20±0.629	2.11±0.737	2.22±0.786
18	1.11±0.314	1.30±0.471	1.30±0.471	1.11±0.314
19	2.00±0.667	1.33±0.471	1.80±0.786	1.50±0.500

3.2.13. Optimization

Optimization can be defined as the processing conditions that give the optimum (maximum or minimum) value of a function of certain decided variables subject to constraints that are imposed. Optimization may be the process maximizing a desired quantity or minimizing an undesired one. The values of the processing variables that produce the desired optimum value are called optimum conditions (Altan et al., 2008). Product responses such as total color change difference (ΔE), bulk density, antioxidant capacity and yield of powders were the most important major parameters determining quality of PJ powders. Therefore, optimum conditions for PJ powders were determined to obtain minimum color change and bulk density, maximum antioxidant capacity and product yield. To determine the optimum spray drying process conditions, response surface of desirability function was used for numerical optimization. The desirability function of the response surface is shown in Figure 3.11 for obtaining optimal conditions in spray drying of PJ. By applying the desirability function method, covering our criteria, one solution was obtained for the optimum conditions to produce PJ powder with the highest desirability value obtained. The optimum temperature, feed mixture concentration and maltodextrin ratio were 100 °C, 30.8 °Brix, and 53.5 % maltodextrin, respectively. By applying these optimal conditions, PJ powder with a 55 % dry solids yield, 9.78 total color change difference, bulk density 0.35 g/ml and antioxidant capacity of 57.8 % could be produced. The product with these specifications could be acceptable from consumer perspective. The estimated antioxidant capacity value (57.8 %) is close to 65.74 % which is the antioxidant capacity of natural pomegranate juice.

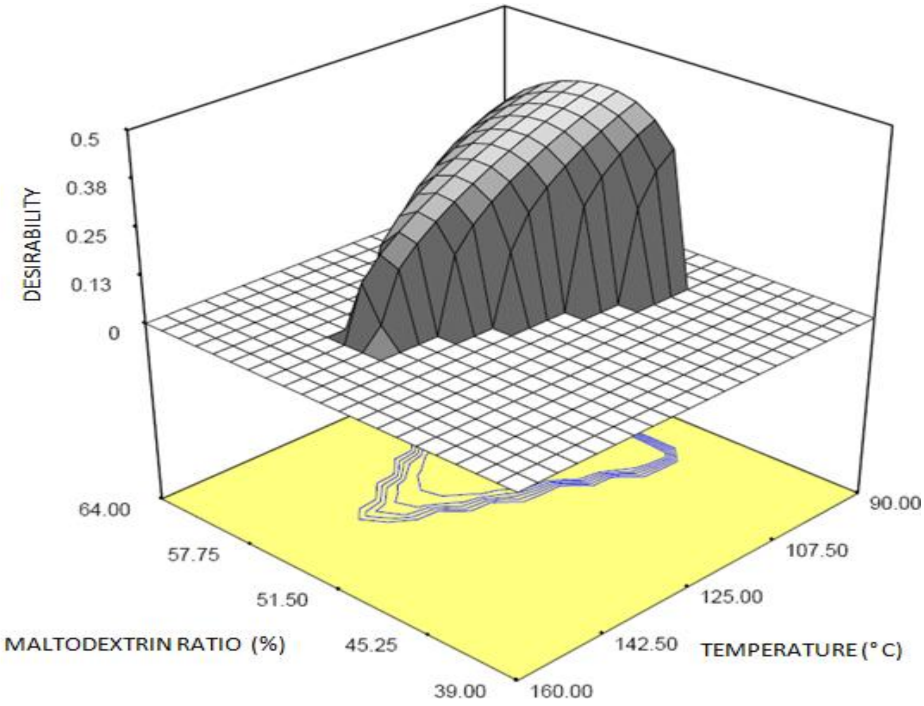

Figure 3.11. Effect of air inlet temperature and maltodextrin ratio on desirability function

3.3. Drying of pomegranate arils: Drying behaviour and physico-chemical properties of pomegranate arils

In this section, air drying and microwave drying curves of pomegranate arils are presented and compared. A well-known model, Page model, was used in order to numerically compare rate of moisture removal from arils by these two drying methods. Finally, some physical and chemical properties of dried arils were given. Initial properties of Fresh arils initially had 23.93±1.44 % moisture content, 0.5867 g/mL bulk density and L*, a*, b* values of 21.22±1.46, 18.06±2.69, 6.42±1.78, respectively.

3.3.1. Drying curves

The hot-air and microwave drying curves for pomegranate arils are shown in Figures 3.12 and 3.13, respectively. Experimental data are given in Appendix B (Tables B1, B2, B3 and B4). The time required to reduce the moisture content to any given level in microwave drying was dependent on the power level, being the highest at 210 W and lowest at 490 W. The times necessary to reach 0.222 g/g were 69.5, 35.0, and 23.8 min at microwave power level of 210, 350 and 490 W, respectively. However, for achieving the same moisture content of arils, hot air drying took 447, 237.5 and 121.5 min at drying temperatures of 50, 60 and 70 °C, respectively. Thus, it was clearly seen that compared with hot air drying, microwave greatly reduced the drying time of arils. ANOVA results showed that the drying temperatures and microwave power not significantly ($p>0.05$) influenced the moisture content vs time behaviour. Also, drying air temperature and microwave power had an important effect on drying. At higher temperature and higher microwave power, due to quick removal of moisture, the drying times were less. The decrease in drying times with increasing in drying temperature and microwave power may be due to increase in water vapour pressure within the arils, which increased the migration of moisture. Similar observations were observed by Kingsly and Singh (2007).

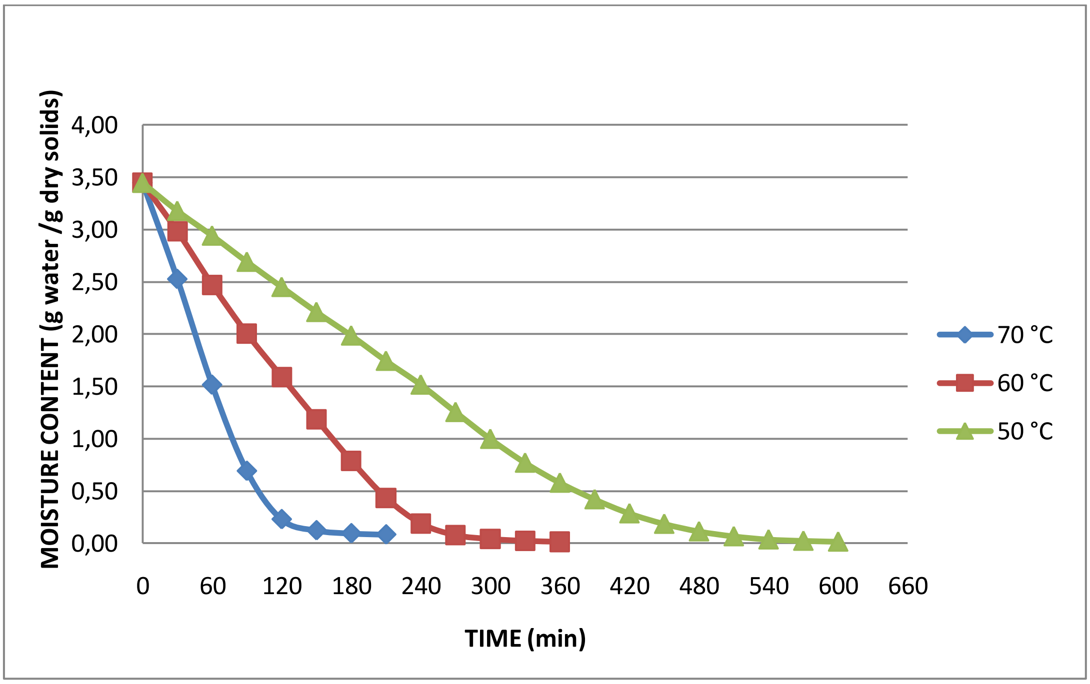

Figure 3.12. Air drying curves of pomegranate arils

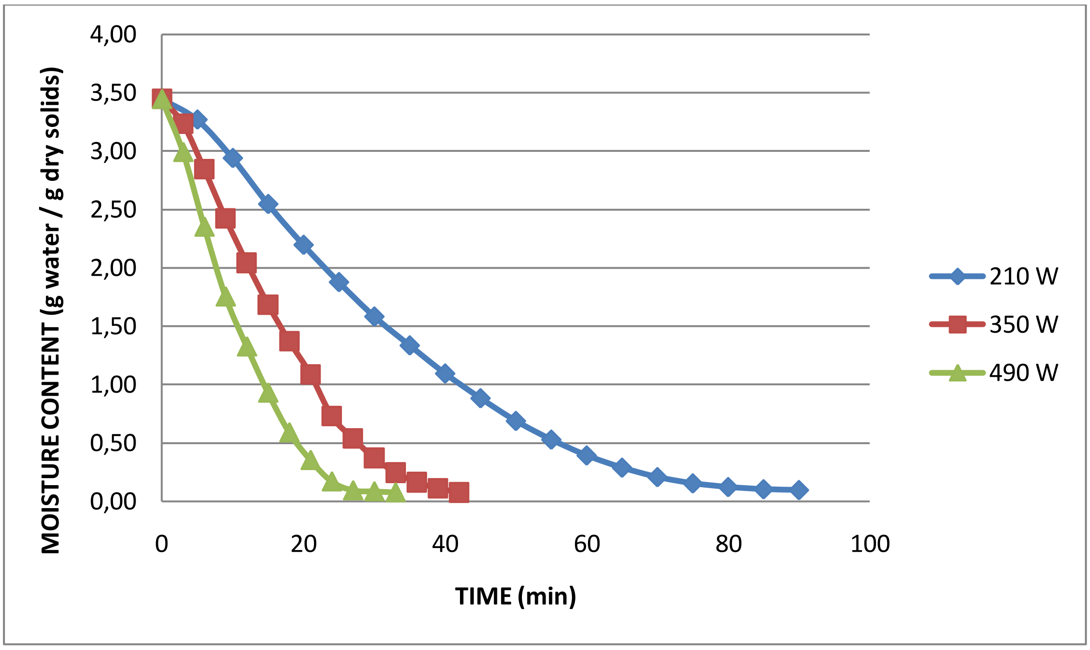

Figure 3.13. Drying curves of pomegranate arils dried by microwave method

The drying rate of each study was calculated at different times and plotted against average moisture content (Data are shown in Appendix B, Tables B5 and B7). Figures 3.14 and 3.15 showed drying rate against average moisture content of arils for hot air and microwave drying processes, respectively. From an examination of these figures it obvious that drying occurred in constant rate period at 50 and 60°C drying temperatures. For biological materials that contain high moisture a constant rate period is expected at low drying temperatures. Because of slow moisture diffusion at these temperatures, drying air is saturated and, by the time forms a thick film around the arils that prevents effective separation of the evaporated moisture from the arils. This may be the reason for existence of a constant rate period (Maskan, 2000). The entire drying process for the samples occurred in the range of falling rate period for microwave drying in this study. Although high moisture foods can be expected to have a period of constant rate drying, this was not observed in microwave drying process. The results indicated that mass transfer within the sample is rapid during microwave heating because heat is generated within the sample, creating a large vapour pressure differential between the centre and the surface of products. It accelerates removal of water from the tissues in the sample by microwave (Maskan, 2000; Maskan 2001). Increasing both air drying temperature and microwave power increased drying rates. Higher drying rates were obtained with higher drying temperature and microwave power. Microwave drying had the higher drying rates than hot air drying process (Figure 3.16). ANOVA results showed that the drying temperatures and microwave power significantly ($p<0.05$) influenced (Appendix B, Tables B6 and B8) the drying rate of pomegranate arils.

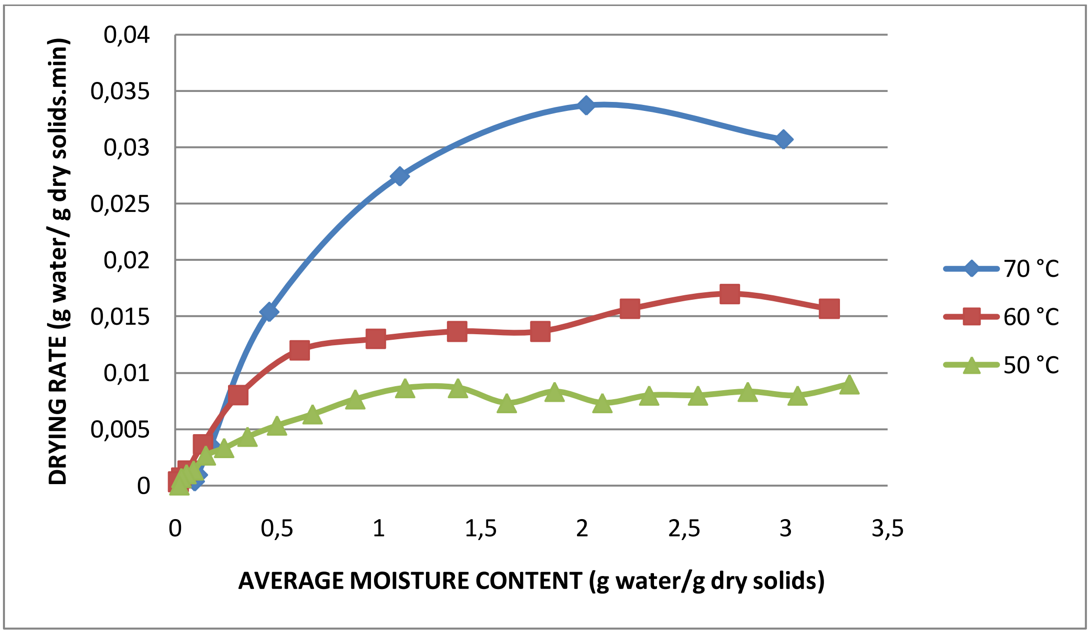

Figure 3.14. Drying rate curves for pomegranate arils dried by hot air

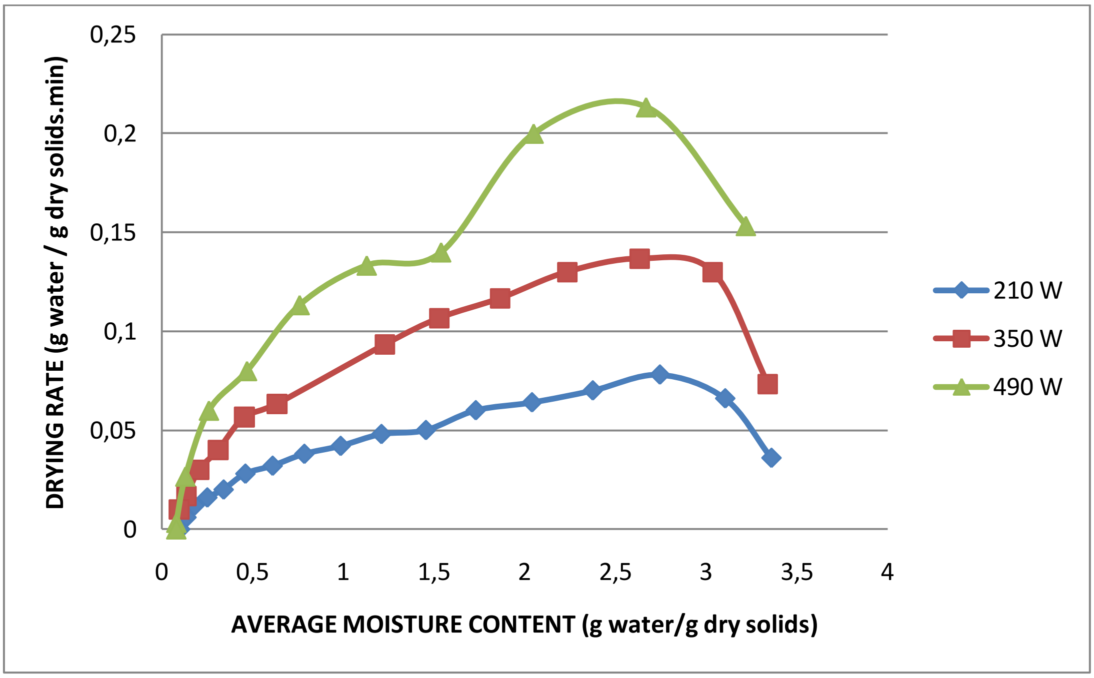

Figure 3.15. Drying rate curves of pomegranate arils dried by microwave drying.

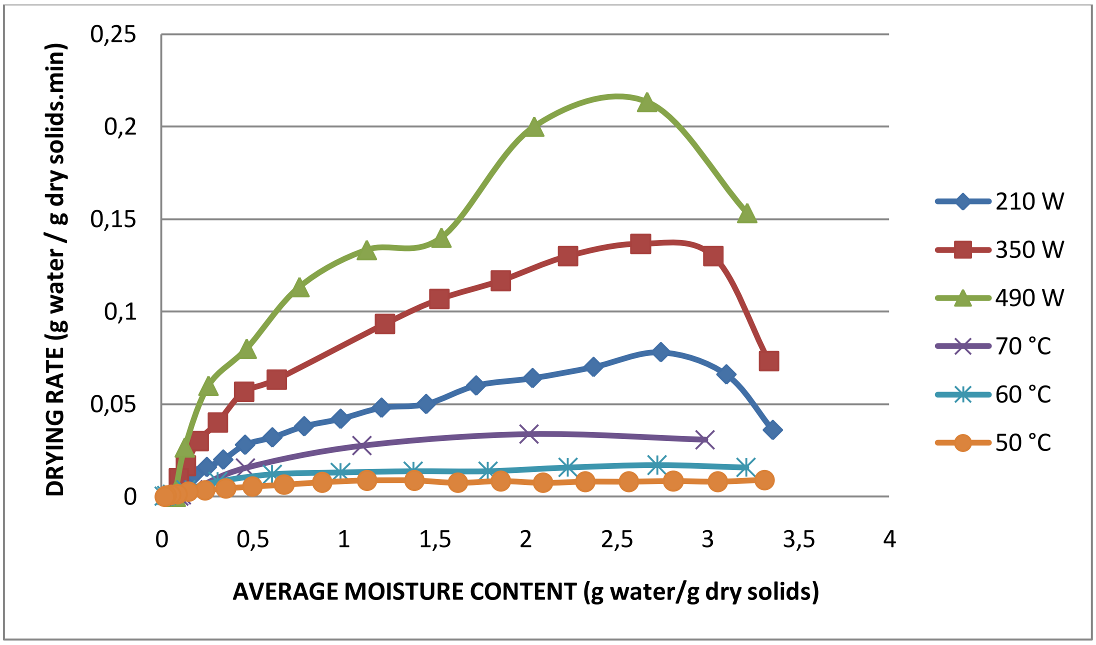

Figure 3.16. Comparison of drying rate curves of pomegranate arils dried by hot air and microwave drying processes

Curves of moisture ratio versus drying time for hot air drying and microwave drying of pomegranate arils were shown in Figures 3.17 and 3.18, respectively. The following thin layer drying model, Page model, (Equation 3.14) was used to describe drying kinetics of pomegranate arils rather than using Fick's diffusion model. The latter model could not be applied because of severe shrinkage in sample dimensions during drying. In this model, the moisture ratio (MR) was simplified to M_t/M_0 instead of $(M_t-M_e)/(M_0-M_e)$ as the value of M_e is relatively small compared to M_t or M_0. This model has been widely used to describe the drying kinetics of various agricultural materials in convective, microwave and microwave assisted convective drying (Sharma and Prasad, 2001; Kingsly and Singh, 2007; Contreras et al, 2008).

$$MR = M_t/M_0 = (M_t-M_e)/(M_0-M_e) = \exp(-kt^n) \qquad (3.14)$$

where MR is the moisture ratio ($M_t/(M_0)$ and dimensionless, M_t is the moisture content at time t = t in g water/g dry solids, M_0 is the moisture content at time t = 0 in g water/g dry solids, M_e is the equilibrium moisture content in g water/g dry solids, k is the drying rate constant in $\min^{-n}$, t is the time in minute and n is the Page model constant.

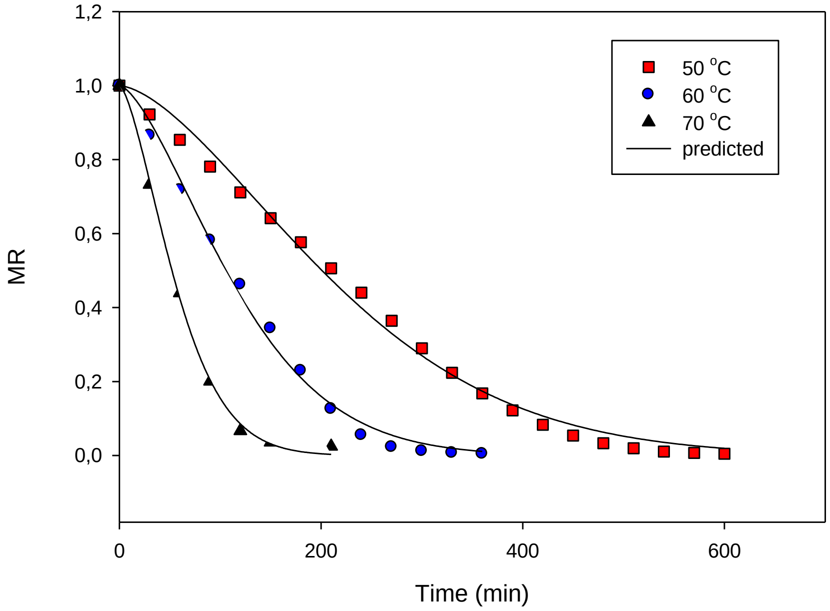

Figure 3.17. Moisture ratio vs time graph of air dried aril samples

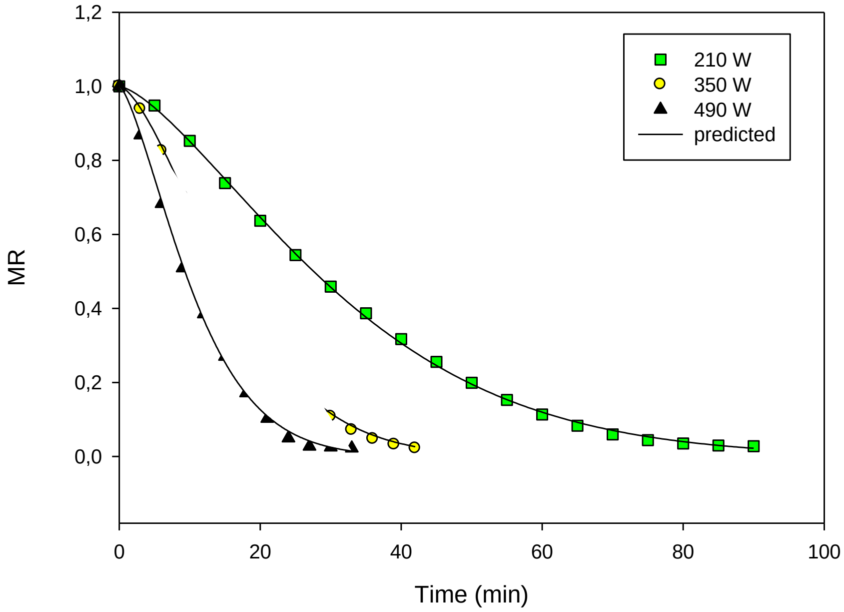

Figure 3.18. Moisture ratio vs time graph of microwave dried aril samples

Drying data (MR) were used to test the applicability of this model. The rate constant k was evaluated through non-linear regression analysis and the results were tabulated in Tables 3.4 and 3.5 for air and microwave aril drying data regression, respectively. The results of nonlinear regression analysis showed high R^2 values (>0.993) implying a good fit of model to the experimental data. As expected, the values of drying rate constant k were high for microwave drying compared to the air drying technique throughout the drying process. It has been reported that the higher the drying temperature and/or microwave power output, the faster the heating for a given mass (Schiffmann, 1986). The rate constant for conventional hot air drying was different (lower) from microwave drying process. The drying constant (k) changed from $1.52*10^{-4}$ to $1.70*10^{-3}$ g water/g dry solids.min and $5.83*10^{-3}$ to $2.85*10^{-2}$ g water/g dry solids.min for drying temperatures of 50 to 70 °C and microwave powers of 210 to 490 W, respectively. The k values for microwave drying were higher than hot-air drying. It was determined that increasing drying temperature from 50 to 70 °C increased drying rate 10.67 fold and increasing microwave power from 210 to 490 W increased the rate 4.88 fold. Also, the ratio of k value at 490 W to that at 70 °C drying temperature was 16.8 fold and of 210 W to that at 50 °C drying temperature was 38.38 fold.

Table 3.4. Nonlinear regression analysis of Page model for air drying of pomegranate arils

Parameters	Air drying temperatures		
	50 °C	60 °C	70 °C
k	$1.52*10^{-4}\pm5.24*10^{-5}$	$5.61*10^{-4}\pm2.05*10^{-4}$	$1.70*10^{-3}\pm4.34*10^{-4}$
n	$1.59\pm6.18*10^{-2}$	$1.53\pm7.35*10^{-2}$	$1.52\pm6.00*10^{-2}$
R^2	0.9930	0.9946	0.9985

Table 3.5. Nonlinear regression analysis of Page model for microwave drying of pomegranate arils

Parameters	Microwave drying powers		
	210 W	350 W	490 W
k	$5.83*10^{-3}\pm3*10^{-4}$	$1.09*10^{-2}\pm1*10^{-3}$	$2.85*10^{-2}\pm2.2*10^{-3}$
n	$1.44\pm1.6*10^{-2}$	$1.56\pm3*10^{-2}$	$1.43\pm3*10^{-2}$
R^2	0.9995	0.9989	0.9990

3.3.2. Rehydration

During reconstitution of dehydrated products the amount and rate of water absorption determines to a considerable extent the sensorial properties and preparation time. The rehydration characteristics of a dried product are used as a quality index and they indicate the physical and chemical changes during drying as influenced by processing conditions, sample pre-treatment and composition (Feng & Tang, 1998; Maskan, 2001). The rehydration capacities of pomegranate aril samples dried at different air temperatures and microwave powers were calculated from Eq. (2.5) and presented graphically. Experimental data are presented in Appendix B (Tables B9 and B11). The rehydration curves of dried pomegranate arils were shown in Figures 3.19 and 3.20 for air and microwave dried samples, respectively.

Figure 3.19 showed that the rehydration capacity (water holding capacity) of arils dried at 70 °C is less than those dried at 60 °C drying temperature. This was probably a result of the sever shrinkage and tissue collapse produced by higher air temperatures resulting in a decrease in rehydration capacity (Vega-Galvez, 2008). Figure 3.20 showed the effect of microwave power on rehydration capacity of pomegranate arils. Increasing microwave power favoured decrease in rehydration capacity. It can be explained that during drying at high microwave powers, irreversible cellular rupture and dislocation occurrs, resulting in loss of tissue integrity producing a dense structure of collapsed, greatly shrunken capillaries with reduced hydrophilic properties. The reduced hydrophilic properties gave lower values for the rehydration capacity, disallowing imbibitions of water, and leaving unfilled pores (Jayaraman et al., 1990; Krokida and Maroulis, 2001; Vega-Galvez, 2008). However, ANOVA results showed that the drying temperatures and microwave power did not influenced (p>0.05) the rehydration capacity of pomegranate arils (Appendix B, Tables B10 and B12).

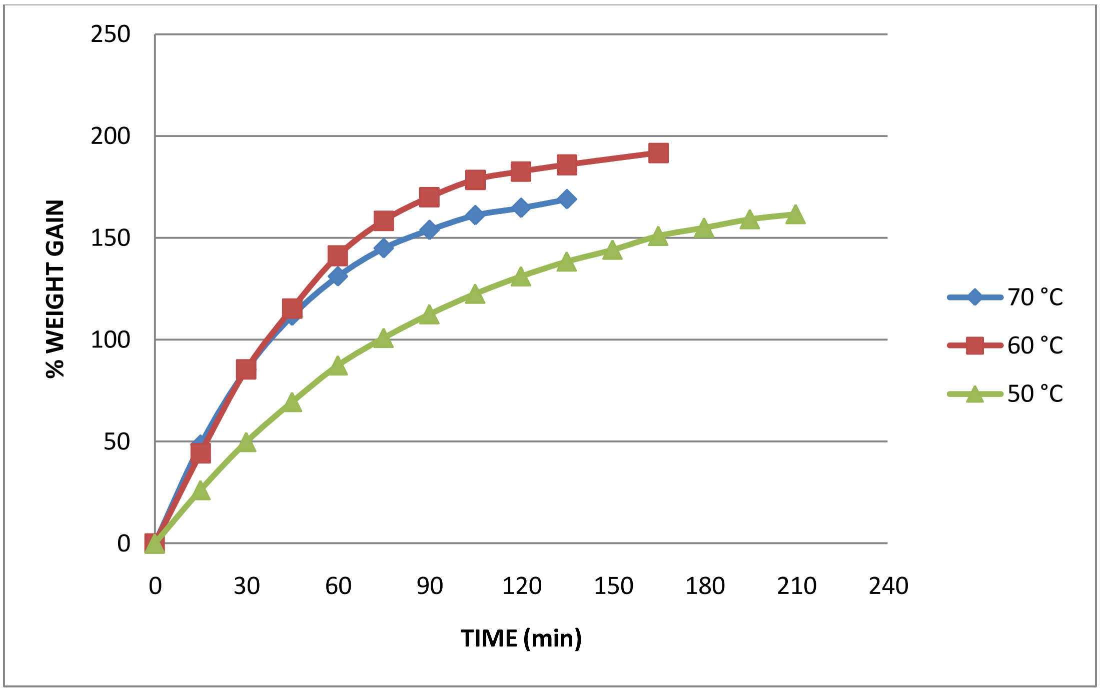

Figure 3.19. Rehydration behaviour of air dried pomegranate arils

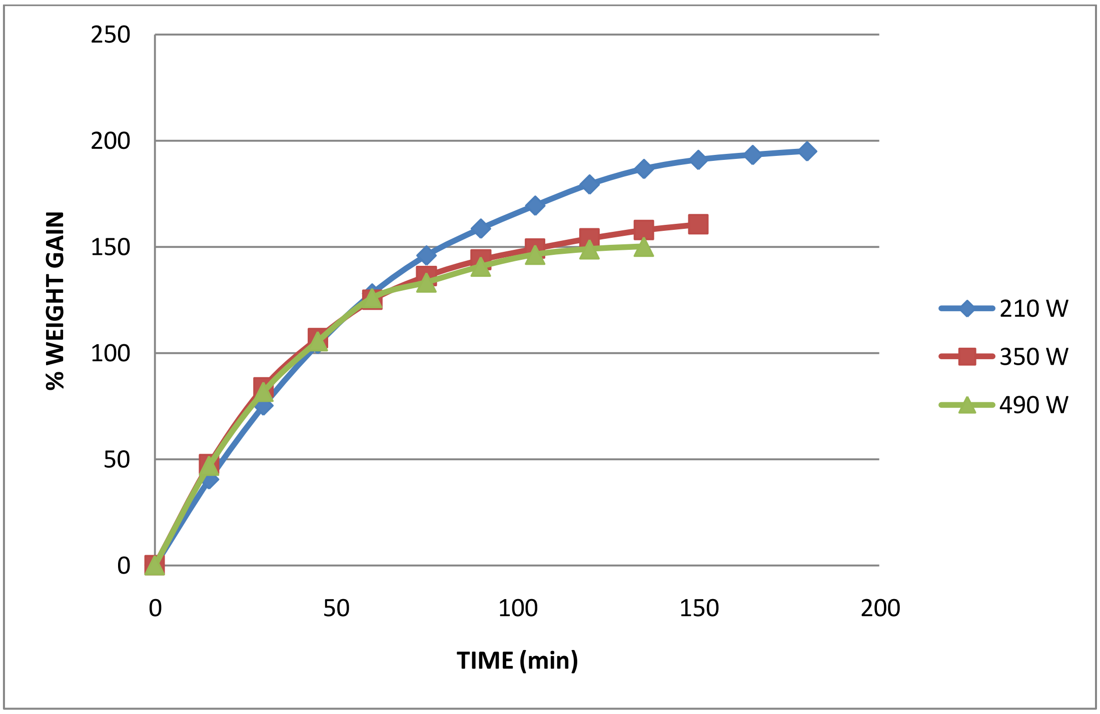

Figure 3.20. Rehydration behaviour of microwave dried pomegranate arils

3.3.3. Shrinkage

Shrinkage occurred during drying by both drying methods was calculated using Eq. 2.6. Shrinkage data were shown in Appendix B (Tables B13 and B15). The effects of temperatures and microwave power levels on shrinkage of pomegranate arils are shown in Figures 3.21 and 3.22. The shrinkage of air dried pomegranate arils samples were 78.57%, 77.06%, 75.62% for 50, 60 and 70 °C, respectively. This also showed that drying at 50 °C promoted relatively more shrinkage, which was explained by the fact that a long drying times gives more time for the product to shrink (Ratti, 1994; Koçak, 2009). The shrinkage of microwave dried pomegranate arils samples were 65.45%, 65.22% and 54.55% for 210, 350 and 490 W, respectively. This showed that drying at 210 W promoted more shrinkage because of low drying rate. Göğüş (1994) explained that the larger differences in moisture content that may exist within a single piece of material create shrinkage effects that are dependent upon the rate of drying. If a piece of highly shrinkable material is dried so slowly, like air dried at 50 °C, that its center is never very much wetter than the surface, the internal stresses are minimized and the material shrinks down fully onto a solid core. On the other hand, if it is dried rapidly, like microwave drying at high power intensity, the walls become more dry then the centre and are placed under sufficient tension to give them permanent set with similar dimensions to the original piece. Therefore, microwave dried samples shrunk less than air dried ones. On the other hand, Maskan (2001) found highest shrinkage value of microwave dried kiwifruits than hot air and hot air-microwave dried kiwifruits that is not in agreement with our results.

In all processes shrinkage followed the pattern of typical drying curves, with high shrinkage initially and gradual levelling off towards the end of drying so that the final size and shape of samples were fixed before drying was completed. Similar behaviour has been observed by Ratti (1994) on drying of potatoes, apples and carrots, Sjöholm and Gekas (1995) on apple drying and Maskan (2001) on drying of kiwifruits.

However, ANOVA results showed that there is no significant difference between air drying temperature and microwave powers on % shrinkage value of pomegranate arils ($p>0.05$) (Appendix B, Tables B14 and B16).

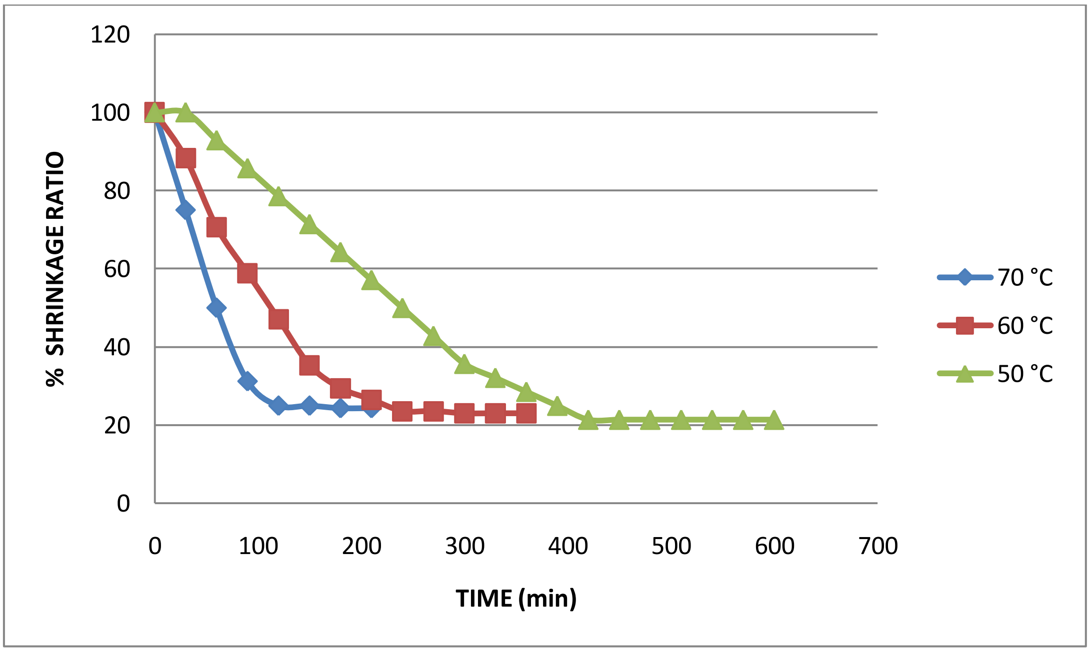

Figure 3.21. Shrinkage behaviour of air dried pomegranate arils

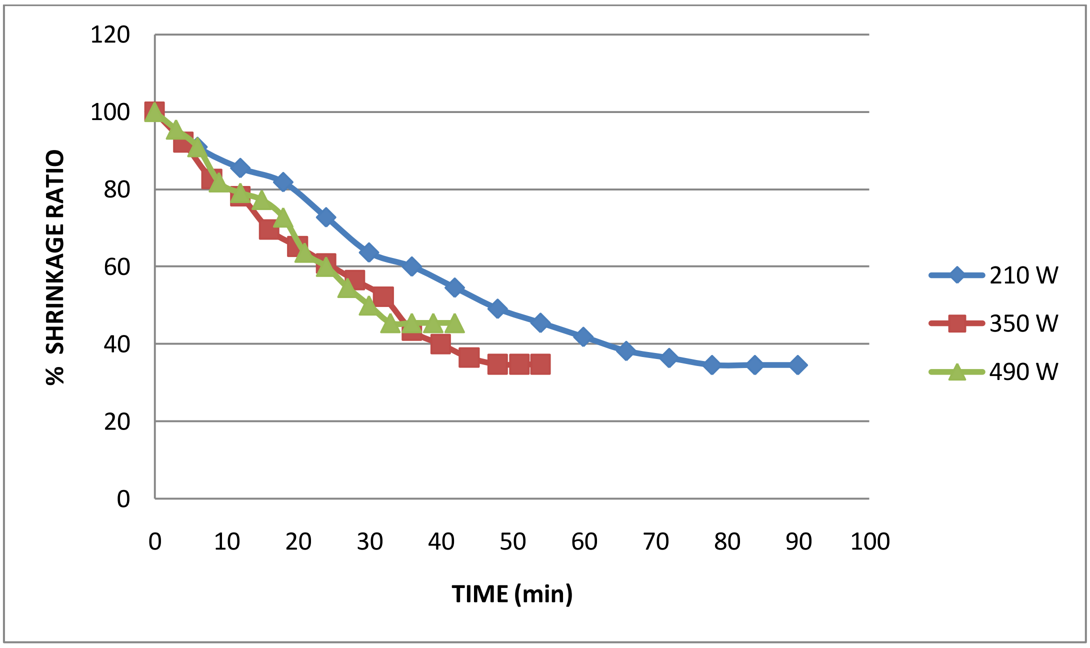

Figure 3.22. Shrinkage behaviour of microwave dried pomegranate arils

3.3.4. Color

The retention of color can be as a quality indicator to evaluate the extent of deterioration due to thermal processing (Avila and Silva, 1999; Akyıldız et al., 2004). The Hunter color parameters L^*, a^*, b^*, have been widely used to describe color changes during thermal processing of fruit and vegetable products.

Figure 3.23 shows that the ΔE values of air dried pomegranate arils was a function of the air inlet temperature used for drying process. The calculated total color difference data are given in Appendix B (Tables 17 and B19). Drying temperature and time are important parameters for color change during drying. The total color difference of pomegranate arils showed similar behaviour until 30 minutes of drying process. At that interval L^* values increased and a^* and b^* values decreased. The ΔE values increased with time. However, ΔE value for 50 °C drying temperature was higher than 60 and 70°C. At low drying temperatures material contacts with air for a long time to dry. During long drying time oxidation of pigments, enzymatic and non enzymatic browning may take place and material may change its color. ANOVA results showed that drying temperature significantly ($p<0.05$) affected ΔE values (Appendix B, Table B18).

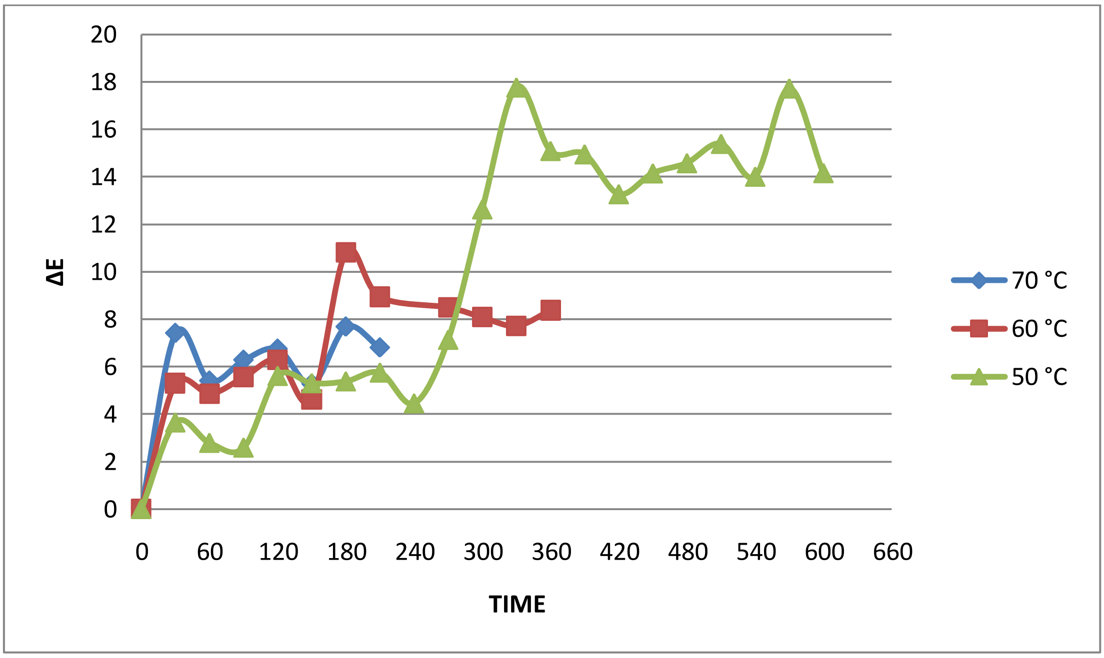

Figure 3.23. Total color difference behaviour of air dried pomegranate arils

Also, Figure 3.24 shows that the ΔE values of microwave dried pomegranate arils was a function of the microwave power intensity used for drying process. Firstly, ΔE values increased and then decreased.

The total color difference values increased with decrease in microwave power and increase in time. ΔE value of 210 W power level was higher than those of 350 and 490 W. It took longer time to dry at 210 W power compared to 350 and 490 W levels. So that oxidation may take place and material color may change during this long drying time. The lower color degradation of microwave dried arils at 490 W power level may, therefore, be due to the substantial reduction in drying time. ANOVA results showed that microwave power was not significantly ($p>0.05$) affected the total color difference of arils during drying (Appendix B, Table B20).

Microwave dried samples had higher final ΔE values than air dried one. i.e., microwave drying caused more color deterioration in terms of ΔE than air drying. It was observed that samples dried by microwave had brownish like color.

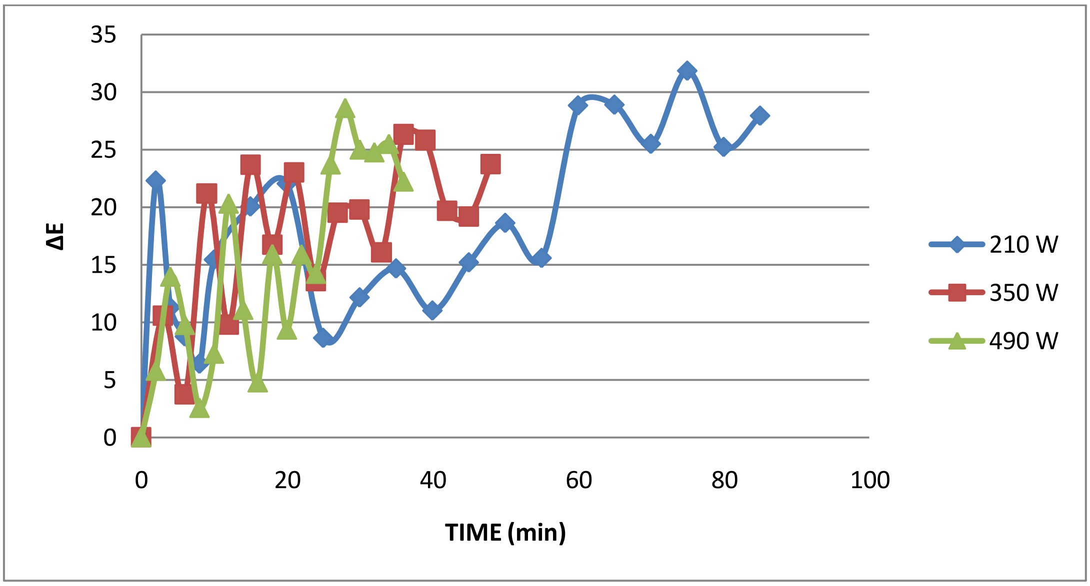

Figure 3.24. Total color difference behaviour of microwave dried pomegranate arils

Also Hunter a* is important parameter for many fruit juices because it is related to redness. Figures 3.25 and 3.26 show change in Hunter a* value of air and microwave dried pomegranate arils, respectively. Experimental data together with statistical analysis results are given in Appendix B (Tables B21 and B23). The Hunter a* values from both drying techniques exhibited decreasing trend generally. Pomegranates contain a variety of different anthocyanin pigments and they are sensitive to heat applications (Vega-Galvez, 2008). The decrease in Hunter a* value may be attributed to destruction of anthocyanins and occurrence of Maillard reactions during drying resulting in a color change from a natural red or purple to a more dull brownish color (Maskan, et. al., 2002).

Hunter a* had the lowest value at 50 °C drying temperature compared to the other drying temperatures. For microwave drying (Figure 3.24), highest Hunter a* value (less degradation in Hunter a* value) was observed at 490 W drying power. It may be due to short drying time at this power output, hence, preserving the red color of arils. In general, microwave dried samples had lower final Hunter a* values than those of air dried. That means microwave drying process caused more color darkening than air drying process.

ANOVA results of air dried aril samples showed that temperature significantly affected ($p<0.05$) the Hunter a* value (Appendix B, Tables B22). However, ANOVA results of microwave dried aril samples showed that effect of microwave intensity was not significant (p>0.05) on Hunter a* value (Appendix B, Tables B24). Similar results have been reported by Maskan (2000). He found change in color values was not dependent on microwave intensity during drying of banana samples.

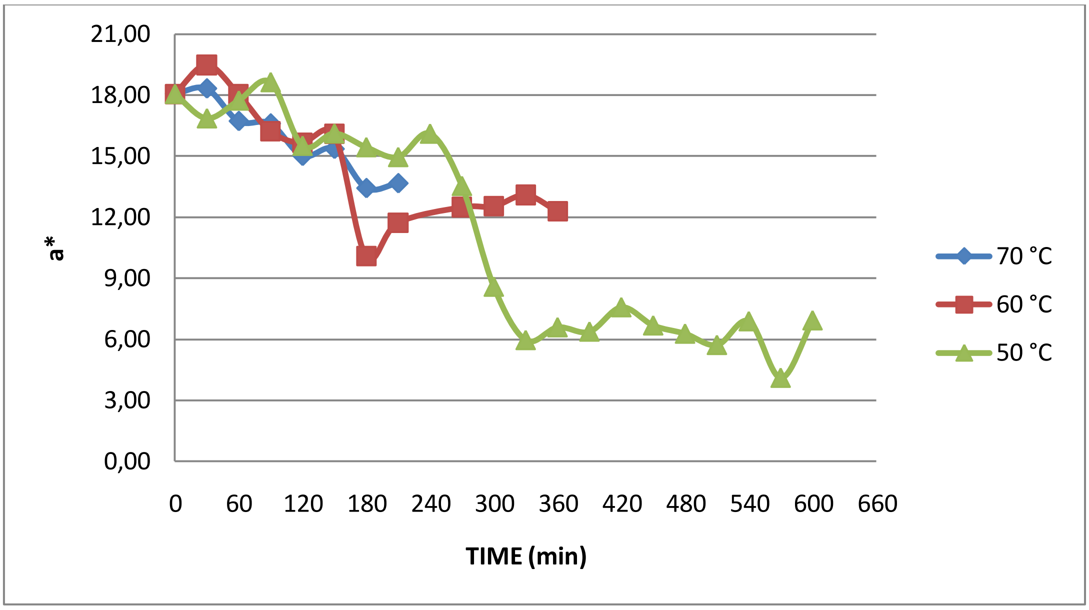

Figure 3.25. Behaviour of Hunter a* value of air dried pomegranate arils

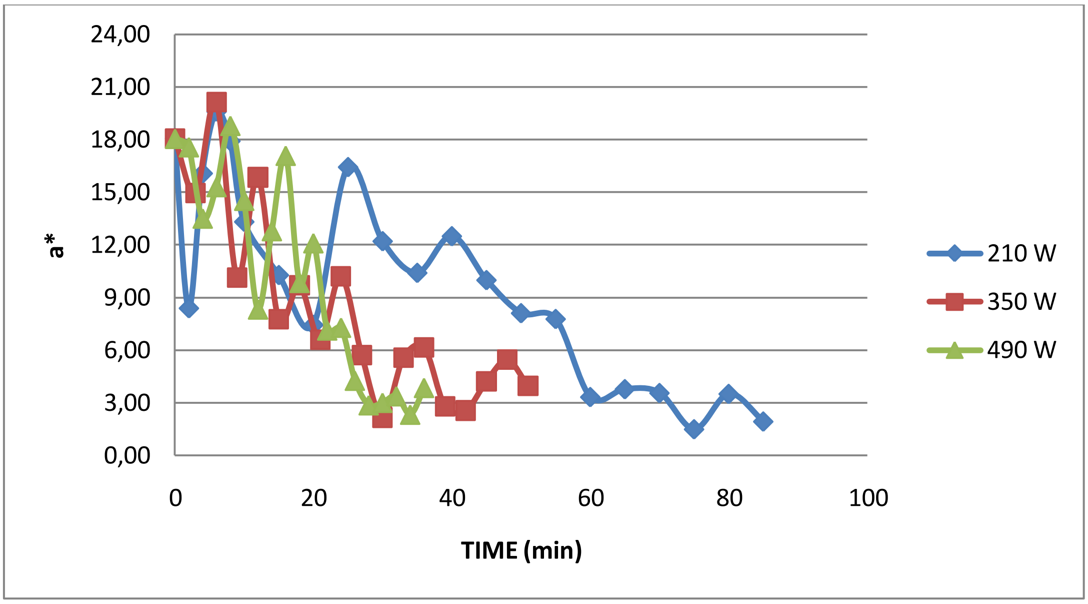

Figure 3.26. Behaviour of Hunter a* value of microwave dried pomegranate arils

3.3.5. Bulk density

The bulk density values of air and microwave dried pomegranate arils are shown in Figures 3.27 and 3.28, respectively and Appendix B (Tables B25 and 26). The bulk densities of air dried samples were between 0.4643 to 0.5239 g/mL and of microwave dried samples were 0.3739 to 0.4758 g/mL. The low/high bulk density values can be related to extent of shrinkage. The low bulk densities obtained from microwave drying correlate well with the low degree of shrinkage compared with hot air drying process (Figures 3.21 and 3.22). A negative influence of temperature and microwave power was found on bulk density of dried pomegranate arils which agrees with other investigations in literature. i.e., the bulk density of air and microwave dried samples decreased as the drying temperature and microwave power increased. The high bulk density from air drying at 50 °C can probably be explained by the longer drying time (10 hours) than the other temperatures. A long drying time promoted a higher bulk density that causes a severe shrink (Funebo and Ohlsson, 1998; Funebo et al., 2000). Similarly at low power intensity (210 W) bulk density was higher than other power intensities (350 and 490 W). Air dried samples have higher bulk density values than microwave dried samples because of long drying time for the former.

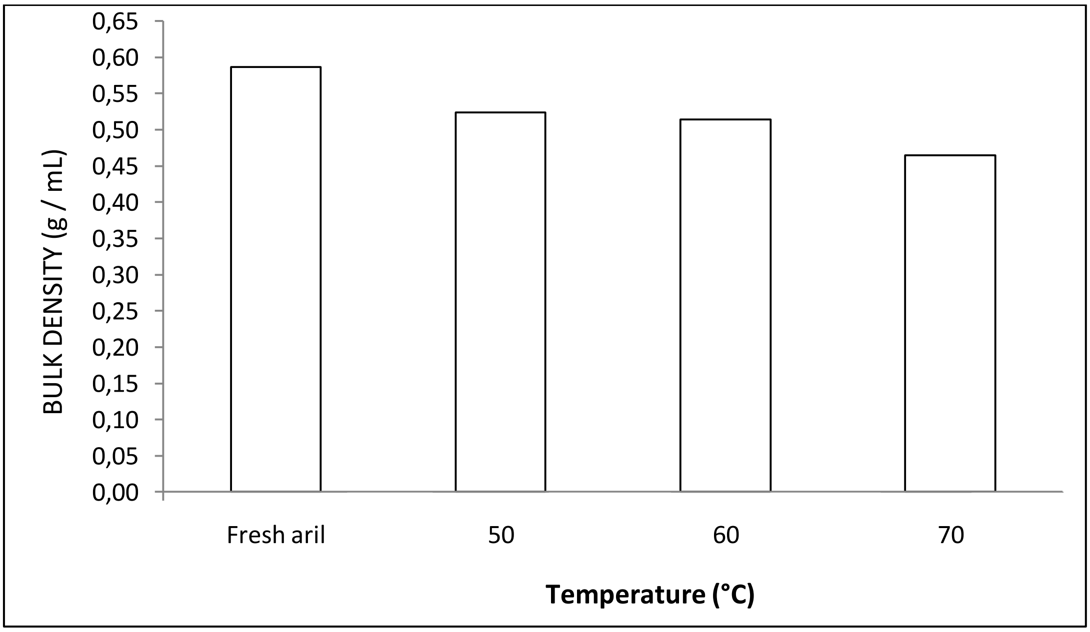

Figure 3.27. Bulk density values of air dried pomegranate arils

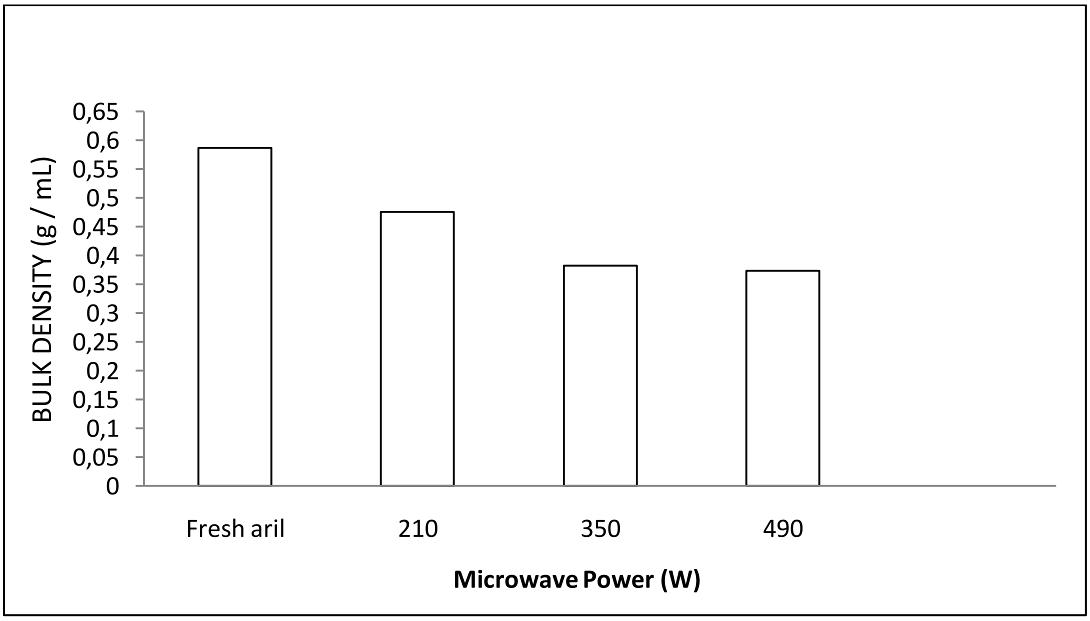

Figure 3.28. Bulk density values of microwave dried pomegranate arils

CHAPTER IV
CONCLUSION

The study of spray drying of pomegranate juice, and hot air and microwave drying of pomegranate arils revealed the following conclusions:

1. Pomegranate juice could be dried by spray dryer. Incorporation of maltodextrin DE6 to feed mixture reduced the stickiness of the products and altered the physicochemical properties of the spray-dried powders. The results showed that inlet temperature has great influence on the physicochemical properties of the spray-dried powders. As inlet temperature increased, the moisture content of the powder decreased meanwhile the time for reconstitution of PJ powder increased.
2. Color value of PJ powders was significantly affected by inlet air temperature and maltodextrin ratio. Total color difference increased with increasing maltodextrin ratio and temperature.
3. Maltodextrin significantly affected sensory parameters of reconstituted PJ powders.
4. Maltodextrin affected the solubility of powders. Solubility decreased with increasing maltodextrin ratio in the feed mixture.
5. Antioxidant capacity of sample increased with increasing air inlet temperature. However, total phenolics content of the samples was not affected from temperature.
6. Yield also increased with increasing maltodextrin ratio in feed mixture.
7. From optimization study it was estimated that the optimum temperature, feed mixture concentration and maltodextrin ratio were 100 °C, 30.8 °Brix 53.5 % maltodextrin, respectively in order to produce spray dried powder with acceptable physical and chemical properties.
8. Increasing drying air temperature and microwave power reduced the drying time of pomegranate arils.
9. Rate of moisture removal from arils was higher with microwave drying than air drying.

10. Rate of rehydration capacity of microwave dried aril samples was faster than air dried samples.
11. Low shrinkage values were obtained with microwave drying.
12. Air drying temperatures significantly affected color values of dried arils.
13. Among the drying parameters studied, 60 °C drying temperature and 350 W microwave power could be recommended for drying pomegranate arils with respect to better color, shrinkage, rehydration capacity and drying time.

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APPENDICES

APPENDIX A

Table A1. Experimental results for responses in spray drying of PJ powders

Runs	Moisture content ± St.deviation	Hygroscopicity	Anthocyanin content ± St. deviation	ΔE ± St.deviation	a^* ± St.deviation	Solubility ± St. deviation
1	7.73±0.10	0.4207	2.0351±0.0060	12.36±0.17	13.52±0.14	19±1.0
2	8.19±0.05	0.374	2.0916±0.0115	11.44±0.17	14.9±0.35	20±1.0
3	8.80±0.06	0.433	2.0107±0.0587	9.1±0.24	16.52±0.33	23.5±0.5
4	7.48±0.05	0.3654	2.0414±0.0549	9.79±0.77	16.38±1.07	17±1.0
5	9.14±0.33	0.3588	2.1078±0.0512	8.48±0.46	17.11±0.57	31.5±1.5
6	6.03±0.03	0.2865	1.999±0.0082	10.5±0.32	15.52±0.66	18.5±0.5
7	8.14±0.14	0.3624	2.1298±0.0043	10.45±0.16	15.43±0.21	20.5±0.5
8	6.64±0.04	0.3237	2.0422±0.0062	12.96±0.10	13.23±0.09	28±0.0
9	6.85±0.17	0.3602	2.2679±0.0778	12.66±0.20	13.48±0.32	22±1.0
10	7.90±0.03	0.3581	2.0388±0.0013	10.98±0.23	15.16±0.21	31.5±1.5
11	3.89±0.01	0.2665	1.894±0.0182	13.34±0.15	12.86±0.18	26±1.0
12	4.95±0.06	0.4093	1.708±0.0783	10.45±0.27	15.58±0.24	29±1.0
13	4.66±0.04	0.399	2.5498±0.0358	7.82±1.08	18.31±1.62	18.5±0.5
14	3.44±0.01	0.2899	2.4531±0.0193	11.94±0.74	14.96±1.26	22.5±0.5
15	4.81±0.06	0.4254	2.2416±0.0345	7.34±0.51	18.57±0.96	16.5±0.5
16	5.29±0.05	0.3456	1.7521±0.0031	9.36±0.69	17.35±1.24	28±1.0
17	4.88±0.03	0.342	1.7861±0.0217	8.65±0.45	18.69±1.43	24.5±0.5
18	3.72±0.02	0.3236	2.1754±0.0454	12.34±0.87	14.63±1.66	35.5±0.5
19	4.96±0.01	0.3462	2.1982±0.0264	7.8±0.48	19.22±1.67	16.20±0.0
20	3.58±0.03	0.3637	2.0434±0.0023	9.05±0.46	17.93±0.60	25±1.0

Runs	Glass transition temperature (Tg)	Bulk density ± St. deviation	Total phenolic content ± St. deviation	Antioxidant capacity ± St. deviation	pH	Acidity ± St. deviation	Yield
1	100.88	0,4210±0.0281	14.7±0.13	72.09±0.51	3.02	0.11±0.030	56.93
2	98.53	0,3796±0.0134	14.73±0.09	76.96±0.59	3.0.	0.135±0.024	52.29
3	90.69	0.4587±0.0154	14.29±0.03	72.04±0.01	3.02	0.108±0.001	25.02
4	101.95	0.3739±0.0077	16.31±0.21	67.92±0.50	3.03	0.109±0.001	35.37
5	88.09	0.3831±0.0112	15.38±0.28	62.32±0.74	3.05	0.103±0.002	26.33
6	97.26	0.3807±0.0082	16.87±0.03	60.90±0.25	3.03	0.123±0.002	44.72
7	98.56	0.3809±0.0025	15.79±0.13	59.21±0.84	3.04	0.122±0.001	42.99
8	102.73	0.3902±0.0039	15.60±0.23	71.94±0.61	3.04	0.127±0.001	37.24
9	105.5	0.4111±0.0181	19.93±0.45	46.07±0.70	3.06	0.119±0.003	71.24
10	93.63	0.4450±0.0005	18.33±0.02	72.91±0.50	3.07	0.118±0.000	42.74
11	98.87	0.3889±0.0072	16.50±0.03	71.47±0.71	3.07	0.113±0.001	57.40
12	94.90	0.4720±0.0055	14.63±0.01	64.74±0.55	3.07	0.095±0.001	30.96
13	95.93	0.3474±0.0070	10.78±0.02	58.60±0.48	3.07	0.113±0.001	42.36
14	97.98	0.4145±0.0174	14.13±0.18	63.01±0.45	3.06	0.112±0.002	36.86
15	84.59	0.4182±0.0018	13.97±0.04	68.20±0.10	3.07	0.111±0.001	19.67
16	91.59	0.3842±0.0143	14.96±0.08	59.15±0.95	3.06	0.125±0.004	54.32
17	99.69	0.3663±0.0081	14.32±0.06	72.09±0.09	3.08	0.109±0.000	44.51
18	108.76	0.4571±0.0045	15.77±0.07	65.76±0.23	3.09	0.106±0.002	86.08
19	92.46	0.3450±0.0300	10.82±0.18	51.57±0.39	3.10	0.107±0.006	57.64
20	103.79	0,4238±0.0142	10.08±0.11	61.75±0.98	3.06	0.104±0.001	56.00

APPENDIX B

Table B1. Experimental moisture content vs drying time for hot air drying process

50 °C		60 °C		70 °C	
Time (min)	Moisture content (g water/g dry solids)	Time (min)	Moisture content (g water/g dry solids)	Time (min)	Moisture content (g water/g dry solids)
0	3.45	0	3.45	0	3.45
30	3.18	30	2.98	30	2.53
60	2.94	60	2.47	60	1.51
90	2.69	90	2.00	90	0.69
120	2.45	120	1.59	120	0.23
150	2.21	150	1.18	150	0.12
180	1.99	180	0.79	180	0.10
210	1.74	210	0.43	210	0.09
240	1.52	240	0.19		
270	1.26	270	0.08		
300	1.00	300	0.04		
330	0.77	330	0.02		
360	0.58	360	0.01		
390	0.42				
420	0.29				
450	0.19				
480	0.11				
510	0.07				
540	0.04				
570	0.02				
600	0.02				

Table B2. ANOVA for moisture content at various temperatures

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	0.246	2	0.123	0.084	0.919
Within Groups	5.808	39	1.457		
Total	57.053	41			

DUNCAN'S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05
		1
70	8	1.0900
60	13	1.1715
50	21	1.2829
Sig.		0.715

Table B3. Experimental moisture content vs drying time for microwave drying process

210 W		350 W		490 W	
Time (min)	Moisture content (g water/g dry solids)	Time (min)	Moisture content (g water/g dry solids)	Time (min)	Moisture content (g water/g dry solids)
0	3.45	0	3.45	0	3.45
5	3.18	30	2.98	30	2.53
10	2.94	60	2.47	60	1.51
15	2.69	90	2.00	90	0.69
20	2.45	120	1.59	120	0.23
25	2.21	150	1.18	150	0.12
30	1.99	180	0.79	180	0.10
35	1.74	210	0.43	210	0.09
40	1.52	240	0.19		
45	1.26	270	0.08		
50	1.00	300	0.04		
55	0.77	330	0.02		
60	0.58	360	0.01		
65	0.42				
70	0.29				
75	0.19				
80	0.11				
85	0.07				
90	0.04				

Table B4. ANOVA for moisture content vs drying time at various microwave powers

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	0.221	2	0.110	0.080	0.923
Within Groups	59.368	43	1.381		
Total	59.589	45			

DUNCAN'S MULTIPLE RANGE TEST TABLE

MICROWAVE POWERS (W)	N	Subset for alpha = 0.05
		1
490	12	1.1808
210	19	1.2500
350	15	1.3587
Sig.		0.702

Table B5. Drying rates vs moisture content at different temperatures

50 °C		60 °C		70 °C	
Drying rate (g water/g dry solids.min)	Average moisture content (g water/g dry solids)	Drying rate (g water/g dry solids.min)	Average moisture content (g water/g dry solids)	Drying rate (g water/g dry solids.min)	Average moisture content(g water/g dry solids)
0.0090	3.32	0.0157	3.13	0.0307	2.99
0.0080	3.06	0.0170	2.73	0.0337	2.02
0.0083	2.82	0.0157	2.24	0.0274	1.11
0.0080	2.57	0.0137	1.80	0.0154	0.46
0.0080	2.33	0.0137	1.39	0.0035	0.18
0.0073	2.10	0.0130	0.99	0.0010	0.11
0.0083	1.87	0.0120	0.61	0.0004	0.1
0.0073	1.63	0.0080	0.31		
0.0087	1.39	0.0037	0.14		
0.0087	1.13	0.0013	0.06		
0.0077	0.89	0.0067	0.03		
0.0063	0.68	0.0003	0.02		
0.0053	0.50				
0.0043	0.36				
0.0033	0.24				
0.0027	0.15				
0.0013	0.09				
0.0010	0.06				
0.0007	0.03				
0.0000	0.02				

Table B6. ANOVA for Drying rate of air dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	0.001	2	0.000	5.254	0.010
Within Groups	0.002	36	0.000		
Total	0.002	38			

DUNCAN’S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05	
		1	2
50	20	0.00571667	
60	12	0.00955556	
70	7		0.01600662
Sig.		0.229	1.000

Table B7. Drying rates vs moisture content at different microwave powers

210 W		350 W		490 W	
Drying rate (g water/g dry solids.min)	Average moisture content (g water/g dry solids)	Drying rate (g water/g dry solids.min)	Average moisture content (g water/g dry solids)	Drying rate (g water/g dry solids.min)	Average moisture content(g water/g dry solids)
0.0360	3.36	0.0733	3.34	0.1533	2.99
0.0660	3.11	0.1300	3.05	0.2133	2.02
0.0780	2.75	0.1367	2.64	0.2000	1.11
0.0700	2.38	0.1300	2.24	0.1400	0.46
0.0640	2.04	0.1167	1.87	0.1333	0.18
0.0600	1.73	0.1067	1.53	0.1133	0.11
0.0500	1.46	0.0933	1.23	0.0800	0.1
0.0480	1.21	0.0633	0.64	0.0600	0.26
0.0420	0.99	0.0567	0.46	0.0267	0.13
0.0380	0.79	0.0400	0.31	0.0033	0.08
0.0320	0.61	0.0300	0.21	0.0000	0.08
0.0280	0.46	0.0167	0.16		
0.0200	0.34	0.0100	0.01		
0.0160	0.25				
0.0120	0.18				
0.0060	0.16				
0.0040	0.11				
0.0000	0.10				

Table B8. ANOVA for Drying rate of microwave dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	0.031	2	0.015	6.723	0.003
Within Groups	0.090	39	0.002		
Total	0.120	41			

DUNCAN'S MULTIPLE RANGE TEST TABLE

MICROWAVE POWER (W)	N	Subset for alpha = 0.05	
		1	2
210	18	0.0372222	
350	13		0.0771795
490	11		0.1021212
Sig.		1.000	0.185

Table B9. Experimental rehydration capacity data of air dried aril samples

50 °C		60 °C		70 °C	
Time (min)	Rehydration capacity(%)	Time (min)	Rehydration capacity (%)	Time (min)	Rehydration capacity (%)
0	0	0	0	0	0
15	26.23	15	44.29	15	48.45
30	49.81	30	85.43	30	85.31
45	69.44	45	115.21	45	111.68
60	87.40	60	141.20	60	131.03
75	100.86	75	158.42	75	144.83
90	112.49	90	169.88	90	153.78
105	122.55	105	178.55	105	161.01
120	131.09	120	182.54	120	164.62
135	138.35	135	185.84	135	168.85
150	144.14	165	191.72		
165	150.92				
180	154.96				
195	159.11				
210	161.62				

Table B10. ANOVA for rehydration capacity of air dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3951.532	2	1975.766	0.599	0.556
Within Groups	105606.665	32	3300.208		
Total	109558.197	34			

DUNCAN'S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05
		1
50	15	107.2647
70	9	115.3922
60	11	132.0982
Sig.		0.344

Table B11. Experimental rehydration capacity data of microwave dried aril samples

210 W		350 W		490 W	
Time (min)	Rehydration capacity(%)	Time (min)	Rehydration capacity (%)	Time (min)	Rehydration capacity (%)
0	0	0	0	0	0
15	40.41	15	47.58	15	46.92
30	75.20	30	83.76	30	81.77
45	104.20	45	106.97	45	105.61
60	128.03	60	125.05	60	125.96
75	145.95	75	136.26	75	133.29
90	158.61	90	143.93	90	140.76
105	169.43	105	149.20	105	146.37
120	179.41	120	153.98	120	148.91
135	186.72	135	157.90	135	150.15
150	190.98	150	160.58		
165	193.30				
180	195.06				

Table B12. ANOVA for rehydration capacity of microwave dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	4994.131	2	2497.065	0.788	0.464
Within Groups	98238.296	31	3168.977		
Total	103232.427	33			

DUNCAN'S MULTIPLE RANGE TEST TABLE

MICROWAVE POWER (W)	N	Subset for alpha = 0.05
		1
490	10	107.9740
350	11	115.0196
210	13	135.9462
Sig.		0.276

Table B13. Experimental shrinkage data of air dried aril samples

50 °C		60 °C		70 °C	
Time (min)	Shrinkage (%)	Time (min)	Shrinkage (%)	Time (min)	Shrinkage (%)
0	3.45	0	100	0	100.00
30	3.18	30	88.24	30	75.00
60	2.94	60	70.59	60	50.00
90	2.69	90	58.82	90	31.25
120	2.45	120	47.06	120	25.00
150	2.21	150	35.29	150	25.00
180	1.99	180	29.41	180	24.38
210	1.74	210	26.47	210	24.38
240	1.52	240	23.53		
270	1.26	270	23.53		
300	1.00	300	22.94		
330	0.77	330	22.94		
360	0.58	360	22.94		
390	0.42				
420	0.29				
450	0.19				
480	0.11				
510	0.07				
540	0.04				
570	0.02				
600	0.02				

Table B14. ANOVA for shrinkage of air dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	176.405	2	88.202	0.108	0.898
Within Groups	31736.738	39	813.763		
Total	31913.143	41			

DUNCAN'S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05
		1
2	13	44.0000
1	8	44.2500
3	21	48.1905
Sig.		0.738

Table B15. Experimental shrinkage data of microwave dried aril samples

210 W		350 W		490 W	
Time (min)	Shrinkage (%)	Time (min)	Shrinkage (%)	Time (min)	Shrinkage (%)
0	100	0	100	0	100.00
6	90.91	4	92.17	3	95.45
12	85.45	8	82.61	6	90.91
18	81.82	12	78.26	9	81.82
24	72.73	16	69.57	12	79.09
30	63.64	20	65.22	15	77.27
36	60.00	24	60.87	18	72.73
42	54.55	28	56.52	21	63.64
48	49.09	32	52.17	24	60.00
54	45.55	36	43.48	27	54.55
60	41.82	40	40.00	30	50.00
66	38.18	44	36.52	33	45.45
72	36.36	48	34.78	36	45.45
78	34.55	51	34.78	39	45.45
84	34.55	54	34.78	42	45.45
90	34.55				

Table B16. ANOVA for shrinkage of microwave dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	811.578	2	405.789	0.892	0.417
Within Groups	19559.408	43	454.870		
Total	20370.985	45			

DUNCAN’S MULTIPLE RANGE TEST TABLE

MICROWAVE POWER (W)	N	Subset for alpha = 0.05
		1
210	16	57.7281
350	15	58.7820
490	15	67.1507
Sig.		0.256

TABLE B17. Experimental total color difference(ΔE) data of air dried aril samples

50 °C		60 °C		70 °C	
Time (min)	ΔE	Time (min)	ΔE	Time (min)	ΔE
0	0	0	0	0	0
30	3.65	30	5.31	30	7.41
60	2.79	60	4.86	60	5.40
90	2.59	90	5.56	90	6.28
120	5.61	120	6.29	120	6.74
150	5.30	150	4.62	150	5.26
180	5.38	180	10.82	180	7.68
210	5.75	210	8.95	210	6.80
240	4.45	270	8.50		
270	7.13	300	8.09		
300	12.63	330	7.73		
330	17.77	360	8.38		
360	15.10				
390	14.95				
420	13.27				
450	14.15				
480	14.58				
510	15.40				
540	14.01				
570	17.73				
600	14.16				

Table B18. ANOVA for total color difference of air dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	136.217	2	68.108	3.299	0.048
Within Groups	784.600	38	20.647		
Total	920.817	40			

DUNCAN’S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05	
		1	2
70	8	5.6962	
60	12	6.5925	6.5925
50	21		9.8286
Sig.		0.636	0.093

TABLE B19. Experimental total color difference(ΔE) data of microwave dried aril samples

210 W		350 W		490 W	
Time (min)	ΔE	Time (min)	ΔE	Time (min)	ΔE
0	0	0	0	0	15.37
2	22.29	3	10.55	2	17.58
4	11.27	6	3.75	4	13.52
6	8.75	9	21.17	6	15.31
8	6.38	12	9.81	8	18.80
10	15.42	15	23.67	10	14.53
15	20.05	18	16.74	12	8.34
20	22.03	21	23.01	14	12.80
25	8.62	24	13.58	16	17.10
30	12.14	27	12.14	18	9.85
35	14.66	30	19.53	20	12.11
40	11.00	36	19.80	22	7.14
45	15.18	39	16.08	24	7.30
50	18.61	42	26.32	26	4.28
55	15.57	45	25.82	28	2.88
60	28.83	48	19.67	30	3.02
65	28.89	51	23.72	32	3.39
70	25.49			34	2.34
75	31.84			36	3.87
80	25.21				
85	27.93				

Table B20. ANOVA for total color difference (ΔE) of microwave dried samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	380.419	2	190.210	3.076	0.057
Within Groups	2597.312	42	61.841		
Total	2977.731	44			

DUNCAN’S MULTIPLE RANGE TEST TABLE

MICROWAVE POWER (W)	N	Subset for alpha = 0.05	
		1	2
490	13	10.0631	
350	13		16.1246
210	19		16.6853
Sig.		1.000	0.849

Table B21. Experimental Hunter a* data of air dried aril samples

50 °C		60 °C		70 °C	
Time (min)	a*	Time (min)	a*	Time (min)	a*
0	18.06	0	18.06	0	18.06
30	16.86	30	19.48	30	18.32
60	17.74	60	18.05	60	16.72
90	18.64	90	16.23	90	16.60
120	15.51	120	15.66	120	15.01
150	16.12	150	16.10	150	15.36
180	15.44	180	10.09	180	13.43
210	14.96	210	11.74	210	13.67
240	16.11	270	12.50		
270	13.54	300	12.54		
300	8.58	330	13.10		
330	5.96	360	12.30		
360	6.59				
390	6.37				
420	7.57				
450	6.68				
480	6.27				
510	5.27				
540	6.88				
570	4.10				
600	6.93				

Table B22. ANOVA for Hunter a* of air dried samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	192.706	2	96.353	4.943	0.012
Within Groups	740.653	38	19.491		
Total	933.359	40			

DUNCAN’S MULTIPLE RANGE TEST TABLE

TEMPERATURE (°C)	N	Subset for alpha = 0.05	
		1	2
50	21	11.2733	
60	12	14.7575	14.7575
70	8		16.4812
Sig.		0.064	0.351

Table B23. Experimental Hunter a* data of microwave dried aril samples

210 W		350 W		490 W	
Time (min)	a*	Time (min)	a*	Time (min)	a*
0	18.06	0	18.06	0	18.06
2	8.39	3	14.95	2	17.58
4	16.07	6	20.14	4	13.52
6	19.59	9	10.13	6	15.31
8	17.91	12	15.85	8	18.80
10	13.31	15	7.77	10	14.53
15	10.28	18	9.69	12	8.34
20	7.44	21	6.61	14	12.80
25	16.43	24	10.22	16	17.10
30	12.21	27	5.72	18	9.85
35	10.40	30	2.16	20	12.11
40	12.29	36	6.17	22	7.14
45	9.99	39	2.81	24	7.30
50	8.11	42	2.56	26	4.28
55	7.77	45	4.23	28	2.88
60	3.33	48	5.45	30	3.02
65	3.78	51	3.96	32	3.39
70	3.56			34	2.34
75	1.49			36	3.87
80	3.51				
85	1.93				

Table B24. ANOVA for Hunter a* of microwave dried aril samples

Source of Variation	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	59.251	2	29.625	1.154	0.325
Within Groups	1078.607	42	25.681		
Total	1137.858	44			

DUNCAN'S MULTIPLE RANGE TEST TABLE

MICROWAVE POWER (W)	N	Subset for alpha = 0.05
		1
350	13	9.8223
210	19	10.7579
490	13	12.7608
Sig.		0.147

Table B25. Experimental bulk density data of air dried aril samples

	50 °C	60 °C	70 °C
Bulk density (g/mL)	0.5239	0.5139	0.4643

Table B26. Experimental bulk density data of microwave dried aril samples

	210 W	350 W	490 W
Bulk density (g/mL)	0.4758	0.3831	0.3739