Effects of Different Drying Processes on the Physicochemical and Antioxidant Properties of Gac Fruit Powder

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Declaration

I hereby certify that the work embodied in this thesis is the result of original research and has not been submitted for a higher degree to any other University or Institution.

Candidate’s signature: signed
Date: 20.02.2010
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<thead>
<tr>
<th>Abbreviation</th>
<th>Definition</th>
</tr>
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<tbody>
<tr>
<td>ABTS</td>
<td>2,2'-azino-bis (3-ethylbenzthiazoline-6-sulfonic acid)</td>
</tr>
<tr>
<td>AAD</td>
<td>Soaking in ascorbic acid solution prior to air drying</td>
</tr>
<tr>
<td>AD</td>
<td>Air drying or air-dried</td>
</tr>
<tr>
<td>AVD</td>
<td>Soaking in ascorbic acid solution prior to vacuum drying</td>
</tr>
<tr>
<td>BET</td>
<td>Brunauer Emmett Teller</td>
</tr>
<tr>
<td>BiAD</td>
<td>Soaking in bisulfite solution prior to air drying</td>
</tr>
<tr>
<td>BiVD</td>
<td>Soaking in bisulfite solution prior to vacuum drying</td>
</tr>
<tr>
<td>BIAD</td>
<td>Blanching prior to air drying</td>
</tr>
<tr>
<td>BIVD</td>
<td>Blanching prior to vacuum drying</td>
</tr>
<tr>
<td>CAD</td>
<td>Control sample air-dried</td>
</tr>
<tr>
<td>CVD</td>
<td>Control sample vacuum-dried</td>
</tr>
<tr>
<td>Aw</td>
<td>Water activity</td>
</tr>
<tr>
<td>dA/dt and dB/dt</td>
<td>change in concentration of A or B with time</td>
</tr>
<tr>
<td>–dC/dt</td>
<td>Rate of change of some index of deterioration C with time t</td>
</tr>
<tr>
<td>DE</td>
<td>Dextrose equivalent</td>
</tr>
<tr>
<td>DPPH</td>
<td>2,2 diphenyl-1-picrylhydrazyl</td>
</tr>
<tr>
<td>DT</td>
<td>Drying temperature</td>
</tr>
<tr>
<td>Ea</td>
<td>Activation energy</td>
</tr>
<tr>
<td>Ej</td>
<td>Extrinsic factors (j = 1…n)</td>
</tr>
<tr>
<td>EE</td>
<td>Encapsulation efficiency</td>
</tr>
<tr>
<td>EMCdb</td>
<td>Equilibrium moisture content on dry basis</td>
</tr>
<tr>
<td>ERH</td>
<td>Equilibrium relative humidity</td>
</tr>
<tr>
<td>FD</td>
<td>Freeze drying or freeze-dried</td>
</tr>
<tr>
<td>FW</td>
<td>Fresh weight</td>
</tr>
<tr>
<td>H0</td>
<td>Hue angle</td>
</tr>
<tr>
<td>HPLC</td>
<td>High performance liquid chromatography</td>
</tr>
<tr>
<td>HTST</td>
<td>High temperature/short time</td>
</tr>
<tr>
<td>Ii</td>
<td>Intrinsic factors (i = 1…m)</td>
</tr>
<tr>
<td>K</td>
<td>Rate constant for degradation reaction</td>
</tr>
<tr>
<td>L</td>
<td>Laminated bags</td>
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<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>$M_0$</td>
<td>Monolayer moisture content</td>
</tr>
<tr>
<td>MC</td>
<td>Moisture content</td>
</tr>
<tr>
<td>$MC_{wb}$</td>
<td>Moisture content on wet basis</td>
</tr>
<tr>
<td>$MC_{db}$</td>
<td>Moisture content on dry basis</td>
</tr>
<tr>
<td>MDC</td>
<td>Maltodextrin concentration</td>
</tr>
<tr>
<td>N</td>
<td>The order of the reaction</td>
</tr>
<tr>
<td>NL</td>
<td>Non-laminated bags</td>
</tr>
<tr>
<td>ORAC</td>
<td>Oxygen radical absorbing capacity</td>
</tr>
<tr>
<td>R</td>
<td>The universal gas constant</td>
</tr>
<tr>
<td>ROS</td>
<td>Reactive oxygen species</td>
</tr>
<tr>
<td>SD</td>
<td>Spray drying or spray-dried</td>
</tr>
<tr>
<td>T</td>
<td>Time</td>
</tr>
<tr>
<td>$t_{1/2}$</td>
<td>Half life</td>
</tr>
<tr>
<td>T</td>
<td>Temperature</td>
</tr>
<tr>
<td>TAA</td>
<td>Total antioxidant activity</td>
</tr>
<tr>
<td>TCC</td>
<td>Total carotenoid content</td>
</tr>
<tr>
<td>TE</td>
<td>Trolox equivalents</td>
</tr>
<tr>
<td>TEAC</td>
<td>Trolox Equivalent Antioxidant Capacity</td>
</tr>
<tr>
<td>Trolox</td>
<td>(S)-(-)-6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid</td>
</tr>
<tr>
<td>VD</td>
<td>Vacuum drying or vacuum-dried</td>
</tr>
<tr>
<td>WSI</td>
<td>Water solubility index</td>
</tr>
<tr>
<td>$\Delta E$</td>
<td>Total colour difference</td>
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**Units of Measure**

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<th>Description</th>
</tr>
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<tr>
<td>%</td>
<td>Percent</td>
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<tr>
<td>$^\circ C$</td>
<td>Degree Celsius</td>
</tr>
<tr>
<td>$^\circ K$</td>
<td>Degree Kelvin</td>
</tr>
<tr>
<td>day$^{-1}$</td>
<td>Per day</td>
</tr>
<tr>
<td>G</td>
<td>Gram</td>
</tr>
<tr>
<td>g/mL</td>
<td>Gram per milliliter</td>
</tr>
<tr>
<td>J mol$^{-1}$</td>
<td>Joules per mole</td>
</tr>
<tr>
<td>kcal mol$^{-1}$</td>
<td>Kilocalories per mole</td>
</tr>
<tr>
<td>kg/m$^2$</td>
<td>Kilogram per square meter</td>
</tr>
<tr>
<td>kPa</td>
<td>Kilopascal (unit of pressure)</td>
</tr>
<tr>
<td>m$^3$/h</td>
<td>Cubic meter per hour</td>
</tr>
<tr>
<td>Mbar</td>
<td>Millibar</td>
</tr>
<tr>
<td>Symbol</td>
<td>Abbreviation</td>
</tr>
<tr>
<td>--------</td>
<td>--------------</td>
</tr>
<tr>
<td>Mg</td>
<td>Milligram</td>
</tr>
<tr>
<td>mL</td>
<td>Milliliter</td>
</tr>
<tr>
<td>mL/min</td>
<td>Milliliter per minute</td>
</tr>
<tr>
<td>mM</td>
<td>Millimolar</td>
</tr>
<tr>
<td>Mmole</td>
<td>Millimole</td>
</tr>
<tr>
<td>MPa</td>
<td>Megapascal</td>
</tr>
<tr>
<td>Nm</td>
<td>Nanometer</td>
</tr>
<tr>
<td>Torr</td>
<td>Unit of pressure</td>
</tr>
<tr>
<td>w/v</td>
<td>Weight per volume</td>
</tr>
<tr>
<td>µg</td>
<td>Microgram</td>
</tr>
<tr>
<td>µmole</td>
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Abstract

Gac fruit, *Momordica cochinchinensis* Spreng contains extraordinarily high levels of carotenoids, especially β-carotene and lycopene, and a comparatively high content of α-tocopherol (vitamin E) and of polyunsaturated fatty acids.

The aim of this study was therefore to develop an understanding of suitable conditions for the processing of Gac fruit and the preparation of Gac fruit powder. The objectives of this study were to investigate the effect of 1) pre-treatments; blanching, ascorbic acid and bisulfite, and 2) drying techniques; air, vacuum, freeze and spray drying, on the physicochemical and antioxidant properties of powders produced from Gac arils. In addition, Gac arils (mixed with added maltodextrin) and untreated Gac skin and yellow pulp were air-dried and their properties were evaluated. The shelf life of a number of the Gac powder products was periodically evaluated during an extended storage period. The moisture sorption isotherms of various Gac powders were also constructed. Furthermore, the stability of three different types of Gac fruit powders was also tested when used in food and beverage products.

Results showed that freeze drying of fresh Gac aril without any pre-treatment produced powders of high quality as determined by colour (hue angle of 33.93, total carotenoid content (TCC) of 7.24 mg/g and total antioxidant activity (TAA) of 0.39 mmole TE/g). However, pre-treatment of fresh Gac fruit aril with 1% (w/v) ascorbic acid or bisulfite solution before vacuum drying at 40°C for 45 hours was just as effective as freeze drying in preserving colour (hue angle of 34.18 and 36.25, respectively), TCC (7.28 and 6.99 mg/g, respectively) and TAA of 0.36-0.40 mmole TE/g. Pre-treatment with 1% (w/v) ascorbic acid or bisulfite solution before air drying at 40°C for 48 hours was also effective (TCC of 6.36 and 6.11 mg/g, respectively and TAA of 0.33 mmole TE/g) but not to the extent of vacuum or freeze drying. In respect of the spray drying process, taking into account the dilution effect of the added maltodextrin, the addition of 10% maltodextrin to the feed mixture and drying at 120°C effectively preserved the physicochemical and antioxidant properties of the powder (hue angle of 66.85, TCC of 2.77 mg/g and TAA of 0.14 mmole TE/g).

In addition, in a comparison of fresh and frozen arils, both were found to be equally useful for production of Gac powder in terms of preservation of colour (hue angle of 33.93 and 31.28, respectively), TCC (7.24 and 6.27 mg/g, respectively) and TAA (0.39
and 0.33 mmole TE/g, respectively). However, the dried aril was found to be very
difficult to grind due to its stickiness. The addition of maltodextrin (0.5 or 1 g
maltodextrin/g of total fruit solids) prior to drying solved this problem and also
maintained the quality of the powder (hue angle of 28.04-30.55; TCC of 5.65-6.29 mg/g
and TAA of 0.29-0.31 mmole TE/g, respectively).

The storage study indicated that the degradation of TCC and TAA of freeze-dried and
spray-dried powders for up to 8 and 3 months, respectively, was lowest when samples
were packed into laminated bags and stored at temperature of 10°C (TCC loss of 11%
and 5% and TAA loss of 44% and 15%, respectively). The highest losses for freeze-
dried and spray-dried powders (TCC of 70% and 42%; and TAA of 88% and 42%,
respectively) occurred when stored at conditions of 37°C.

Isotherm curves of all the Gac powders have sigmoid shapes. By comparing different
drying methods for aril, the lowest hygroscopicity was observed in SD powder, followed
by VD, AD and FD powders.

Results also showed that the air-dried skin and yellow pulp powders contained low
levels of TCC and TAA compared to the aril powders, but these are still high levels of
TCC (0.90 and 0.42 mg/g, respectively) as compared to cherry tomatoes (0.36) and
pumpkin (0.14). The skin (18%) and yellow pulp (49%) account for 67% of the total
weight of the Gac fruit and as such comprise significantly high components. Therefore,
utilisation of these components can prevent environmental pollution due to waste
issues, and also enhances the overall value of Gac fruit.

Freeze-dried, vacuum-dried and spray-dried Gac fruit aril powders were found to be
easily incorporated into cooked glutinous rice, pasteurised Gac juice and pasteurised
Gac milk mixtures. The colour, TCC and TAA of the juice and the milk mixtures were
maintained after storage for 30 days under refrigeration temperature of 4°C.

Overall the study established that, the quality of Gac aril powders, in terms of colour,
TCC and TAA, is most effectively preserved by applying pre-treatments prior to
vacuum drying at 40°C. These powders should be packed in laminated bags and
stored at 10°C for any lengthy storage period. It was also found that the Gac powders
can be satisfactorily incorporated into “Xoi Gac”, juice and milk products. Finally, the
overall value of Gac fruit could be enhanced through utilisation of all the anatomical
components.
Chapter 1
INTRODUCTION

1.1 Introduction

Gac fruit, *Momordica cochinchinensis* Spreng, also known as baby jackfruit or sweet gourd, is one of the traditional fruits in Vietnam. Studies report that extraordinarily high levels of carotenoids, especially β-carotene and lycopene, are found in Gac fruit aril, the brightly coloured flesh covering the seeds (Bauernfeind, 1972; Vuong, 2000; Aoki et al., 2002). Gac fruit also contains significantly high levels of α-tocopherol (vitamin E) (Vuong et al., 2006) and of fatty acids (Vuong, 2000). It is very important therefore, to preserve or enhance these constituents in processed Gac fruit products, particularly the high levels of carotenoids and the associated antioxidant activity. Dried Gac powder is usually the dried aril component having the high concentration of nutrients and colour.

In addition to Gac aril containing a very high nutritional content, the total carotenoid content in the yellow pulp of the Gac fruit is relatively high as compared to many plant foods (Aoki et al., 2002; Ishida et al., 2004). Furthermore, the yellow pulp represents approximately half of the weight of an entire fresh fruit and is the largest anatomical component in quantity (Ishida et al., 2004). However, while the aril is traditionally used for food preparation due to its attractive colour and high nutrient levels, the pulp is often discarded. Similarly, Gac skin, which represents about 18% of the total weigh of the fruit, is not used. Therefore, identifying means of utilising these components is necessary to reduce the environmental problem of waste, and also to enhance the economic value of the fruit.

Carotenoids have been used for centuries as a natural food colorant due to the attractive yellow-orange-red colour, and as a nutrient supplement due to the health benefits, such as being vitamin A precursors and having strong antioxidant activity. Hence, it is very important to optimise factors such as solubility, physical form, composition, processing conditions, pH, packaging and storage conditions, in order to produce a stable final product colour (Henry, 1996; Delgado-Vargas & Paredes-López, 2003). Moreover, many studies have reported that using carotenoid-rich plants as nutrient supplements is one of the most effective solutions to preventing vitamin A
deficiency and for treating various diseases (Vuong, 2000; Vuong et al., 2002; Strobel et al., 2007; Schwarz et al., 2008; Tran et al., 2008). In particular, the daily consumption of lycopene-rich foods, such as fresh tomatoes and tomato products, has been linked with a lower risk of prostate cancer (Guns & Cowell, 2005; Chan et al., 2009) and coronary heart disease (Rao & Agarwal, 1999).

Drying is the unique method for producing powder forms of fruits and vegetables. The main benefits of powder forms, as compared with fresh fruits and vegetables, are the potential for long storage at ambient temperature, and a significant reduction in the costs for transportation and storage. This is particularly important for seasonal fresh fruits such as Gac fruit. Furthermore, the most important factor is that powder forms are very convenient food ingredients for use as flavours and colourings in food products, including juices and dairy products (Fellows, 2000; Tang & Yang, 2004).

At present, there are many drying techniques used for producing powder forms in the food industry; therefore, a suitable drying method for a particular food should be carefully selected. Many factors, such as the characteristics of the food material to be dried, the quality of the desired final product, and processing costs, that is, energy and space requirements, must be considered (Tang & Yang, 2004). As compared to other drying techniques, such as spray drying, vacuum drying and freeze drying, air drying is well known as the simplest and cheapest drying method. However, the quality of air-dried products is fairly poor, particularly as the resultant materials are sensitive to heat damage. Freeze drying is the best drying method for production of the highest quality food products in terms of excellent retention of nutrients, flavour and aroma, physical and sensory characteristics. However, the main disadvantage of this method of drying is the operational costs. Thus, in a study of Gac fruit, it is necessary to investigate the effects of various drying methods on the final quality of Gac powder products in relation to retention of colour and nutrient composition. As a result of such investigations, depending on the desired final product quality and other considerations, a suitable drying technique can be chosen.

Pre-treatment prior to air drying and vacuum drying is one of the most important factors that affect the quality of a final powder product produced by these drying methods. Many studies have shown that the colour, carotenoid content and antioxidant activity of dried fruits and vegetables can be successfully preserved when pre-treatments such as blanching, soaking in bisulfite, or soaking in ascorbic acid, were applied before drying (Mohamed & Hussein, 1994; Ramesh et al., 2001; Chen et al., 2007; Koca et al.,
Furthermore, the addition of maltodextrin before spray drying has also been reported to be effective in preserving carotenoids in fruits (Bhandari et al., 1993; Desobry et al., 1997; Chopda & Barrett, 2001; Abadio et al., 2004). Therefore, in this study these pre-treatments should be applied before drying the Gac fruit in order to test whether they increase the retention of carotenoids in the resulting powders.

Generally the stability of products during storage also plays an important role in the food industry. The quality of food products changes over time as a result of certain storage conditions. The environmental conditions for storage, such as exposure to light, temperature and oxygen, are well known as affecting the condition of the final products, especially carotenoid-rich powders and carotenoid-based processed products. As a result, the need for a storage study of Gac fruit powders is important in establishing the effects of storage conditions on Gac fruit powders.

Finally, the use of carotenoid-based foods as natural colorants and nutrient supplements is currently receiving considerable attention from food manufacturers and consumers. Therefore, an investigation of processing methods and storage conditions for Gac powder in several processed products is carried out in this study.

1.2 Aims of the study

The experiments designed for this study aim to determine the most suitable conditions for different drying methods used in Gac fruit powder processing, and the application of these powders as food colorants and nutrient supplement, as follows:

- To evaluate the processing parameters of different drying methods - hot air drying, vacuum drying, freeze drying and spray drying - for the production of Gac fruit powder.
- To evaluate and compare the physicochemical and antioxidant functional properties of powders produced from fresh and frozen Gac fruit by the methods of hot air drying, vacuum drying and freeze drying.
- To study the stability in different heat processed Gac powder products in relation to the colour, total carotenoid content and antioxidant activity.
- To monitor the shelf life of the Gac fruit powders and their products.
1.3 Hypotheses

The physicochemical and antioxidant properties, specifically colour characteristics, carotenoid content and antioxidant activity, of Gac fruit powder are affected by the conditions applied during drying processes and by storage conditions. Pretreatments and suitable drying conditions are expected to minimise the losses of colour, carotenoid content and antioxidant activity of resultant Gac powder.

1.4 Format of the thesis

The thesis is arranged in the following way. After this chapter of introduction, Chapter 2 contains a review of literature, including an overview of Gac fruit, an examination of carotenoids as natural colorants and in respect to their antioxidant properties, aspects of drying techniques for fruits and vegetables, and consideration of storage conditions for food products. The methodology will be explained and discussed in chapter 3. The experiments and their results will be shown in chapters 4, 5, 6 and 7. Specifically, chapter 4 will discuss the production of Gac fruit powders from fresh fruit and frozen fruit by three different drying techniques, being hot air drying, vacuum drying and freeze drying. Chapter 5 will give details of the experiments measuring the effects of varying added maltodextrin concentrations, and of spray drying temperatures, on the physicochemical and antioxidant properties of Gac fruit powder produced by this technique. In chapter 6, the investigation of the shelf life of Gac powder products, in terms of colour characteristics, total carotenoid content and total antioxidant activity, monitored under a variety of storage conditions will be discussed. Chapter 7 will discuss the stability of different Gac powders when incorporated into “Xoi Gac”, pasteurised milk, and juice. Finally, conclusions and recommendations will be given in chapter 8.
Chapter 2
LITERATURE REVIEW

2.1 Overview of Gac fruit
2.1.1 Characteristics and growth of Gac plants

Gac fruit, *Momordica cochinchinensis* Spreng, is botanically classified as Family *Cucurbitaceae*, Genus *Momordica*, and Species *Cochinchinensis*. It is also known as baby jackfruit, sweet gourd or cochinchin gourd in English. The fruit is one of the traditional fruits in Southeast Asia in general, and in Vietnam in particular. In Vietnam, Gac fruit is most commonly prepared as “Xoi Gac” (the Gac aril cooked in glutinous rice) for Tet (Vietnamese New Year) and wedding celebrations.

The Gac plant can be cultivated from seeds or root tubers, and grows as dioecious vines that are separate male and female plants (Figure 2.1). It can be commonly seen in Vietnam growing wild or in domestic settings with the vines growing on lattices in rural homes or in gardens. Two months after planting root tubers the plant will start flowering; flowering usually begins in April and continues until August or September. On average, one plant can produce 30 to 60 fruit in one season. The ripe fruit is then harvested from August to February (World Health Organization, 1990). The Gac fruit is typically round or ovoid in shape, with the exterior skin covered in short spines. Its green skin colour becomes red or dark orange when ripe.
2.1.2 Nutritional composition of Gac fruit

Several studies have reported that Gac fruit contains extraordinarily high levels of carotenoids, especially carotenes and lycopene, in comparison to other fruits and vegetables containing lycopene and β-carotene. According to Bauernfeind (1972) and Aoki et al. (2002), the lycopene concentration in Gac fruit is at least five times higher than in other fruits analysed (rosehip, pitanga (Brazil), tomato (USA), guava (pulp), watermelon, papaya and grapefruit) (Figure 2.2). Furthermore, when compared to a range of other fruits and vegetables the β-carotene level in Gac fruit is the highest. Vuong (2000) stated that Gac fruit has the highest β-carotene content of the edible plants of Northern Vietnam. For example it is eight times higher than the level in carrots, which are commonly recognised as being high in β-carotene (Figure 2.3).
Gac fruit aril also contains a significant amount of fatty acids, at 852 mg per 100 g of the edible portion. 70% of total fatty acids in the aril are unsaturated, and 50% of these are polyunsaturated (Vuong, 2000). Fatty acids are very well known as being beneficial for human health. The presence of fat in the Gac fruit aril plays an important role in the absorption of carotenes and other fat-soluble nutrients (Kuhnlein, 2004). Furthermore, it protects the trans- and cis- lycopene isomers against oxidation and retards the isomerisation reaction (Xianquan et al., 2005).
In addition to a significant carotenoid content the concentration of α-tocopherol (vitamin E) in Gac fruit, at 76 µg/g of wet weight, is also comparatively high (Vuong et al., 2006). Vitamin E, as a natural antioxidant, helps protect Gac oil produced from the aril from oxidation (Vuong & King, 2003).

It can be concluded that Gac fruit is an exceptional fruit whose aril contains excellent sources of carotenoids, essential fatty acids and α-tocopherol. Therefore, determination of the most suitable processing method is highly recommended in order to preserve these constituents in processed Gac fruit.

2.2 Carotenoids and their function

Since carotenoids are the major component in Gac fruit, it is important to review the carotenoid pigments in terms of their structure, classification and distribution. In addition, the natural functions of carotenoids are also reviewed in this section.

2.2.1 Overview of carotenoids

In general, carotenoids are isoprenoid compounds, containing eight isoprenoid units whose order is inverted at the molecule centre; these are widely distributed in nature as red, yellow and orange pigments. More than 600 different carotenoids have been identified from natural sources; however, approximately twenty-four carotenoids commonly occur in foods and fourteen carotenoids have been identified in human serum (Dutta et al., 2005; Xianquan et al., 2005). Carotenoids are chemically divided into two groups, carotenes and xanthophylls. The first group is the highly unsaturated hydrocarbons, known as carotenes, which contain no oxygen. Xanthophylls contain one or more oxygen functional group (most commonly hydroxyl, keto, epoxy, methoxy or carboxylic acid groups) at particular sites on the terminal rings. Additionally, carotenoids are also classified as primary and secondary. The primary carotenoid group includes compounds required for photosynthesis, such as β-carotene, violaxanthin, and neoxanthin. The secondary classification includes carotenoids that are localised in fruits and flowers; these are α-carotene, β-cryptoxanthin, zeaxanthin, antheraxanthin, capsanthin and capsorubin (Delgado-Vargas et al., 2000). The structures of common carotenoids are shown in Figure 2.4.
Colour characteristics of food items is one of the most important issues in the food industry, and colour is affected by carotenoid structures in relation to the molecular shape, chemical reactivity and light absorbing properties (Britton, 1995). For example, β-carotene and lycopene contain conjugated double bonds, so are usually orange and red in colour. The important properties of carotenoids are illustrated in Figure 2.5. Carotenoids are extremely susceptible to oxidative degradation because of their unique structure, containing a conjugated double bond system over the entire length of the polyene chain. Additionally, Britton (1995) states that each double bond in the polyene chain of a carotenoid exits in two forms, that is, trans- and cis- isomerisation. Furthermore, all trans- isomers of carotenoids are predominantly found in nature.
Carotenoids are not only found in plants but also in bacteria, fungi, algae and in animals. In plants, carotenoids are widely distributed in the chloroplasts of green tissues; however, they are masked by the chlorophylls. The proportional chemical composition of the main carotenoids is almost the same in the leaves of virtually all species. In fact, $\beta$-carotene (usually 25 to 30% of the total), lutein (around 45%), violaxanthin (15%), neoxanthin (15%), and small amounts of $\alpha$-carotene, $\alpha$- and $\beta$-cryptoxanthin, zeaxanthin, antheraxanthin and lutein-5,6-epoxide are generally found in the same proportions (Britton, 1996). While, the total carotenoid content in leaves of different species varies considerably, the carotenoid composition of the leaves is quantitatively similar. In addition, carotenoids, often located in chromoplasts, occur universally in non-photosynthetic tissues of plants. They are responsible for the yellow, orange and red colours of many flowers and fruits. Furthermore, large amounts of carotenoids, especially carotenes, are also found in roots such as carrots and sweet potato.

In addition to their occurrence in plants, carotenoids are widely distributed in animals. In birds, carotenoids are responsible for yellow or red feathers, the skin colour of chickens and the colour of egg yolk. Similarly, in fish, carotenoids are responsible for the colour of the flesh of salmon and trout (astaxanthin and canthaxanthin). Additionally, significant amounts of astaxanthin and related carotenoids, such as carotenoprotein, are found in marine invertebrate animals (Britton, 1996).
2.2.2 Natural functions of carotenoids

Ecological functions, photosynthesis, the xanthophyll cycle and antioxidant activity are some of the remarkable processes involving carotenoids. Colour is one of the most important roles of carotenoids in terms of ecological functions due to the fact that colour can be related to reproduction. For example, animals and insects are attracted by the colour of seeds, pollen or spores of plants and are then involved in dispersing them. Also, carotenoids play a very important role in photosynthesis, in the light harvesting process and as photoprotectors against oxidative damage, and thus are necessary for the plant's survival. Moreover, to avoid cellular damage to sun-exposed leaves, it is very important to eliminate the excess absorbed energy, which is approximately 50 to 90%, since only 10 to 50% of the absorbed energy is required by the leaves in a fast-growing stage at midday. To eliminate this unwanted energy, the xanthophyll cycle, primarily involving the epoxy xanthophyll groups of carotenoids, carries out the process and protects the photosynthetic apparatus (Delgado-Vargas et al., 2000; Delgado-Vargas & Paredes-López, 2003).

The large number of in vivo and in vitro studies show that carotenoids are effective antioxidants owing to their conjugated double bond system. However, each carotenoid has a different level of antioxidant activity against radicals. According to Miller et al. (1996), the decreasing order of antioxidant activities, determined when assayed by the ABTS** (2,2'-azino-bis [3-ethylbenzthiazoline-6-sulfonic acid]) radical cation scavenging spectrophotometric method, is as follows: lycopene > β-cryptoxanthin > lutein = zeaxanthin > α-carotene > echineone > canthaxanthin = astaxanthin. Moreover, Jiménez-Escrig et al. (2000) found similar results by using the DPPH* (2,2 diphenyl-1-picrylhydrazyl) spectrophotometric method. Accordingly, antioxidant activity depends on the presence of functional groups in terminal rings and the number of conjugated double bonds (Miller et al., 1996).

In addition to the different chemical structures, the measurement of antioxidant activity relies on the specific analysis system, such as the radicals used in different assays, the type of carotenoid being examined, and the additional substances involved in the reaction environment. Therefore, individual carotenoids exhibit different levels of antioxidant activity depending on the system being used to study them. For example, zeaxanthin can quench radicals in the aqueous phase, whereas lipid peroxidation and methyl linoleate are effectively inhibited by β-carotene and canthaxanthin, respectively. As a result, it is recommended that diets that include carotenoid mixtures should be
maintained due to a great variability of radicals and microenvironments in vivo. Similarly, Paiva (1999) stated that antioxidant activity against lipid peroxidation can be significantly increased by the presence of carotenoid mixtures or in relation to others antioxidants.

Carotenoids (β-carotene, lycopene, zeaxanthin, and β-cryptoxanthin) and α-tocopherol, which are well known as antioxidants, protect against cancer and cardiovascular diseases by quenching singlet molecular oxygen (1O2) and scavenging peroxyl radicals. The number of conjugated double bonds of the carotenoid molecules plays an important role in antioxidant activities. Moreover, the antioxidant activity of a combination of hydrophobic carotenoids (such as β-carotene and lycopene) and α-tocopherol is more effective than that of individual compounds. The main reason for this is the synergism of different antioxidants being greater than the sum of the individual antioxidant activities (Arnao et al., 1998; Stahl & Sies, 2003). In this sense, Gac fruit is an excellent source of antioxidant activity because it contains significant amounts of both lipophilic carotenoid and α-tocopherol antioxidants. Therefore, it should be noted that Gac fruit is an antioxidant functional food because of its excellence source of carotenoid mixtures.

2.3 Antioxidants

It is clear that antioxidant activity is one of the most important properties of carotenoid compounds in food products. It is also well known that antioxidative carotenoids play a crucial role in human health. Therefore, determination of the level of antioxidant activity in food is important. Definition and classification of antioxidants are reviewed in this section. In addition, two common methods for the determination of total antioxidant activity in food products are also presented. The advantage and disadvantage of these methods are necessarily discussed.

2.3.1 Definition and classification of antioxidants

It is widely accepted that an antioxidant in biological systems can be defined as “any substance that, when present in food at low concentrations compared with those of an oxidisable substance, significantly delays or prevents oxidation of that substrate” (Halliwell, 1995, p.73). Antioxidants may help the human body to protect against functional damage caused by reactive oxygen species (ROS) (Shahidi, 2000) which are highly reactive pro-oxidants and toxins (Singh & Singh, 2008). The damage is
caused because ROS attack membrane lipids, intracellular proteins/enzymes, carbohydrates and nuclear DNA in cells and tissues (Singh & Singh, 2008). However, in food systems, antioxidants can be defined as small quantities of substances that can prevent or retard the oxidation of easily oxidisable materials (MacDonald-Wicks et al., 2006). Therefore, due to the importance of antioxidant potentials in foods and dietary supplements, there have recently been many studies focusing on the use and measurement of antioxidant activity, especially of natural antioxidants.

Antioxidants can be classified as substances which prevent or reduce the oxidation oxidisable substances (Halliwell, 1990). Furthermore, antioxidants can be chemically categorised as enzyme antioxidants, preventive antioxidants, and scavenging or chain-breaking antioxidants. Another classification of antioxidants is based on their sources as endogenous antioxidants (superoxide dismutases, catalase and glutathione peroxidase) and exogenous antioxidants (mainly from dietary sources, such as vitamin E, β-carotene and lycopene). Additionally, the antioxidants can be classified, based on their mode of action, as primary antioxidants and secondary antioxidants (or co-antioxidants). The compounds which donate a hydrogen atom to lipid radicals, then forming a new radical which is more stable, are known as primary antioxidants. The secondary antioxidants are the substances that can react with the initiating radicals or decrease the oxygen level to stop the formation of the radicals (Singh & Singh, 2008).

According to Arnao et al. (1998), the measurement aimed at quantifying the reactions of antioxidants is called total antioxidant activity (TAA). This is because the potential antioxidant compounds in foods would not exactly reflect their singular antioxidant activity. Moreover, the synergism, that is the cooperative effect of different antioxidants, results in a higher activity than the sum of the individual antioxidant activities. Therefore, total antioxidant activity measurement methods are very useful to obtain a global idea of the relative antioxidant activities in foods and how they change after processing and storage (Arnao et al., 1998; Halliwell, 2002). Furthermore, TAA is also a useful tool for the provision of quality standards and comparison of different food products (Shahidi & Ho, 2007).

There are many in vitro methods to assess TAA in foods; among them, ABTS and DPPH assays are two of the most common methods for the determination of TAA (Schlesier et al., 2002; Singh & Singh, 2008; Moon & Shibamoto, 2009). Furthermore, it is strongly recommended that at least two different methods are used to evaluate antioxidant activity in foods due to differences between the ways in which the test
systems investigate antioxidant activity (Schlesier et al., 2002; Moon & Shibamoto, 2009).

2.3.2 ABTS assay

The ABTS assay is widely used to determine total antioxidant activities in foods due to its applicability in both aqueous and lipid phases (MacDonald-Wicks et al., 2006). The assay is a decolorisation assay that measures the activity of an antioxidant to directly scavenge ABTS cation radicals generated by a chemical substance. The stable ABTS cation radical, a nitrogen centered radical with a characteristic blue-green colour, is produced by oxidation of ABTS with potassium persulfate prior to reacting with antioxidants (Figure 2.6). The stable ABTS cation radical will become colourless when it reacts with an antioxidant to form a non-radical.

![Image of ABTS radical formation](image)

Figure 2.6 Formation of stable ABTS radical from ABTS with potassium persulfate (Moon & Shibamoto, 2009)

The method quantifies scavenging capacity by spectrophotometrically measuring the absorbance of the antioxidant-radical mixture at 734 nm at a selected time point. Results are commonly expressed relative to a standard of Trolox (a commercial water soluble analogue of vitamin E). Therefore, this assay is also known as TEAC (Trolox Equivalent Antioxidant Capacity).
This method has been applied in the study of antioxidant activity due to its operational simplicity, analysis speed and economical cost per sample (MacDonald-Wicks et al., 2006; Moore & Yu, 2007). Moreover, this assay avoids unwanted reactions, does not require extreme conditions to generate radicals, and antioxidant activity can be studied over a pH range. Therefore, the result is considered to be highly accurate. However, the main disadvantage of this assay is that an ABTS radical is not found and is also not similar to radicals in biological systems (MacDonald-Wicks et al., 2006; Moore & Yu, 2007; Singh & Singh, 2008).

2.3.3 DPPH assay

The DPPH assay is also one of the most popular methods for establishing TAA in foods due to its simplicity and high sensitivity (Moon & Shibamoto, 2009). This assay is based on the principle that a hydrogen donor is an antioxidant. It measures the activity of an antioxidant to directly scavenge DPPH radicals by spectrophotometrically determining its absorbance at 515 nm. The DPPH radical is a stable organic nitrogen centered free radical with a dark purple colour which becomes colourless when it reacts with antioxidants to form non-radicals. The result is also expressed as a Trolox equivalent.

![Reaction between DPPH• and antioxidant to form DPPH](image)

Similarly to the ABTS assay, the DPPH assay is a valid, accurate, easy, sensitive and economic method for the determination of TAA in foods (Singh & Singh, 2008). As a result, almost 90% of antioxidant studies use the DPPH assay in combination with other methods (Moon & Shibamoto, 2009). The major disadvantage of this method, however, that it is not useful for the determination of antioxidant activity of plasma due to the precipitation of protein in the alcoholic solvent (Sánchez-Moreno, 2002).
As compared to the ORAC (oxygen radical absorbing capacity) assay, the ABTS and DPPH assays are shown to be simple and relatively cheap methods used for the determination of total antioxidant activity. In addition, the ORAC assay requires an expensive instrument, specifically, microplate readers. This assay is very temperature sensitive and results in more variance (Moore & Yu, 2007). However, many studies found high correlation among the total antioxidant activity in fruits and vegetables determined using ABTS, DPPH and ORAC methods (Awika et al., 2003; Thaipong et al., 2006; Dragović-Uzelac et al., 2007).

2.4 Drying techniques

Drying is one of the most effective methods used to preserve fruit and vegetables. This is due to the removal of moisture in food; hence, microorganism and other deterioration reactions can be prevented or reduced. Furthermore, other important advantages of dehydration are the reduction of transportation and storage costs, and the convenience of use. At present, in the food industry there are many drying methods used, including solar drying, hot air drying, vacuum or inert gas drying, freeze drying, drum drying and spray drying, refractance window drying, microwave drying and heat pump drying. A suitable drying method is chosen depending on the product to be dried, economics, the availability of equipment, and the desired quality of the final dehydrated product (Sablani, 2006). In addition, it is necessary to understand the mechanisms, the drawbacks and the advantages of the different drying methods that can be applied to dehydrating a specific food product. In this section, the processes and apparatus of several drying methods, specifically hot air drying, vacuum drying, spray drying and freeze drying, will be presented. Moreover, the fundamentals of drying procedures methodology are also reviewed.

2.4.1 Water activity (A\textsubscript{w})

The water activity of a food is defined as the ratio of water vapour pressure in the food to the saturated vapour pressure of water at the same temperature, and is expressed in decimal values between 0 and 1. In fact, water activity of a food equals the relative humidity of air (divided by 100) in equilibrium with the product.

The main purpose of any drying process is to remove the water necessary for the growth of microorganisms, for enzymatic activity and for other deterioration reactions. As a result, when water in foods is removed the water activity of dried products is low
enough to prevent spoilage during storage. The effect of water activity on the stability of product is shown in Figure 2.8. In general, microbial growth in food is prevented when water activity is below 0.6. Particularly, the inhibition of the growth of bacteria is found at $A_w$ of less than 0.9, yeast at $A_w$ of below 0.8, and moulds at $A_w$ of below 0.7. Therefore, an effective drying process is normally aimed at reaching water activity of less than 0.6 in the dried food product.

However, as can be seen in Figure 2.8, dried food products containing lipids and with a low $A_w$, of less than 0.2, are particularly sensitive to lipid oxidation due to the action of free radicals. Therefore, it is important that these products should be stored in good oxygen barrier packages, such as vacuum packaging, being flushed with nitrogen, or packed with oxygen scavengers (Tang & Yang, 2004).

![Figure 2.8 Effect of water activity on the stability of food (Labuza, 1977)](image)

**2.4.2 Drying curves**

A drying curve is normally plotted to indicate some forms of moisture change with respect to time. Two examples, plotting moisture content versus time, and plotting
drying rate versus moisture content are shown in Figure 2.9. A drying curve thus gives some useful information on the estimated time for foods to be dried under certain conditions, and on selecting or calculating the size of dryer. Therefore, to obtain a drying curve for a specific product, a known weight of the sample product is placed in a dryer at particular conditions of a constant temperature, humidity and air flow velocity, and the weight loss can be periodically determined over time.

Figure 2.9 Typical drying curves (Geankoplis, 2003)

In general, a drying curve is divided into two phases, that is, a constant-rate period and a falling-rate period in Figure 2.9. Point A indicates that the food material is initially at a cold temperature, while point A’ is for the material at a hot temperature. In the constant-rate period (line BC in Figure 2.9) the unbound water is removed from the food product. The evaporation is not dependent on the solid matrix and continues until water from the interior is no longer available at the food surface. Point C, called the critical moisture content point, distinguishes the constant-rate period from the falling-rate period. At this point, the surface of the material is no longer wet. In the falling-rate period, indicated by line CE in the figure, the surface of the food material becomes completely dry. The evaporation will continue by moving moisture from the centre of the material to the surface. Toward the end of this period, the rate of drying decreases slowly until it reaches zero at the equilibrium moisture content, that is when the material equilibrates with the drying air (Geankoplis, 2003). Dependent on the food being dried there can be one or more falling periods in the drying process. Non-hygroscopic foods generally have a single falling-rate period, whereas hygroscopic foods may show two or more distinct falling-rate periods (Fellows, 2000).

The drying rate is affected by many factors such as the size, composition, structure, and the amount of food to be dried. For example, in the constant-rate period smaller
pieces have a larger surface area available for evaporation whereas in the falling-rate period smaller pieces have a shorter distance for moisture to travel through the food. Furthermore, the effect of the composition and structure of the food on the mechanism of moisture removal is also observable. The rate of moisture movement in vegetables increases due to the orientation of the fibres, which allows more rapid moisture movement along their length than across the structure. Additionally, it is clear that the drying time is faster if smaller quantities of food are used in the dryer (Fellows, 2000).

2.4.3 Hot air drying method

Figure 2.10 shows a hot air dryer, which is the simplest form of air drying equipment. The dryer comprises perforated or shallow mesh trays fitted in an insulated cabinet. The material, from slurries to piece-form solids, is placed onto the trays. Air can be directly heated by a gas burner or an electric heater and is then circulated between the trays (cross-flow). Less commonly, the heated air can be direct to flow through the material on perforated trays (through-flow).

The main advantage of the tray dryer is its flexibility in operation for use with different types of foods, and that it has low capital and maintenance costs. However, the quality of the final dried products is variable and usually low as compared to material produced by other drying methods such as vacuum drying and spray drying. The tray dryer is commonly used for small scale production and in pilot plants for experimentation (Fellows, 2000).

Figure 2.10 Hot air dryer used in this study
2.4.4 Spray drying method

The spray drying technique is used to produce powder forms from pumpable liquid foods, concentrates or pulps. The spray dryer used in this study is shown in Figure 2.11.

In operation, according to Tang and Yang (2004), the liquid food is first pre-heated to reduce viscosity and to enhance drying economics as well. Then the liquid is pumped through a nozzle where it is atomised to form small droplets (10-20μm in diameter). The droplets are dried by hot air in a drying chamber. Finally, dried products are separated in cyclones and a bag filter.

In the spray drying process, due to the large surface area of the small droplets, drying takes place rapidly (1-10 seconds). As a result, it is highly recommended for heat sensitive foods (Fellows, 2000; Tang & Yang, 2004; Ramaswamy & Marcotte, 2006). Furthermore, several other advantages of spray drying can be found in that the drying process is continuous, easy and entirely automatically controlled. Importantly, the quality of final powders will not be variable from one batch to another when spray drying conditions remain unchanged (Masters, 1991). However, installation costs,
thermal efficiency, waste heat and exhaust-air handling are the key drawbacks of the spray drying process (Barbosa-Cánovas & Vega-Mercado, 1996).

2.4.5 Vacuum drying method

A vacuum dryer (Figure 2.12) consists of a vacuum chamber, a heat supply, a device for producing and maintaining the vacuum and components to collect water vapour evaporated from material. The vacuum drying process can be used for some fruit, vegetables, herbs, spices and fruit juice concentrates. Generally, vacuum drying has similarities to freeze drying; however, the material is not frozen and the vacuum pressure is not high (Ramaswamy & Marcotte, 2006).

![Figure 2.12 Vacuum dryer used in this study](image)

Vacuum drying is performed in the absence of oxygen, so this method of drying protects sensitive components of foods from oxidation. As such, it is evident that the vacuum drying has an advantage over hot air drying. Furthermore, since some materials are sensitive to heat damage, vacuum drying is the better choice compared to hot air drying; this is because the vacuum can reduce the necessary drying temperature (Fellows, 2000; Tang & Yang, 2004).

2.4.6 Freeze drying method

The principle of freeze drying, also known as lyophilization or sublimation, is that under high vacuum pressure (less than 4.58 torr or 0.61 kPa), frozen water in materials sublimes directly to vapour without melting. Figure 2.13 shows the conditions required for the sublimation of ice from water.
A freeze dryer is shown in Figure 2.14. In operation, the pre-frozen foods are placed on trays in an airtight drying chamber in which the vacuum pressure will reduce to between 0.1 to 2.0 torr. The heat is transferred to the sublimation front at which ice changes into vapour. The water vapour is continuously removed from material by keeping the pressure in the freeze dryer cabinet below the vapour pressure at the surface of the ice. Then the vapour is condensed onto condensing plates or coils with the low temperature of -35 to -50°C. This temperature changes the water vapour back to ice that is removed by melting (Fellows, 2000; Tang & Yang, 2004).
As compared to other drying methods, the freeze drying process offers some advantages such as very high retention of flavours and nutrients. This is because the product is dried at low temperature. Moreover, a porous structure is formed due to the removal of ice crystals; therefore, the sensory characteristics such as texture and appearance are still maintained. However, it is an expensive process for commercial use due to the lengthy time period for completion of the drying process (10 to 40 hours depending on material size and drying conditions) and the higher energy requirements to run compressor and refrigeration units. Generally, the costs of freeze drying are up to four times higher than those of conventional drying. An additional problem is that an oxidative reaction often occurs because of open porous structures of freeze-dried products. Therefore, to prevent this phenomenon, a good packaging is required (Fellows, 2000; Tang & Yang, 2004).

### 2.4.7 Quality changes in food during drying

In general, regardless of the drying method used, the quality of dried foods reduces when compared to the fresh materials. The quality changes that occur during drying are shown in Table 2.1.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Physical</th>
<th>Nutritional</th>
</tr>
</thead>
<tbody>
<tr>
<td>Browning reactions</td>
<td>Rehydration</td>
<td>Vitamin loss</td>
</tr>
<tr>
<td>Lipid oxidation</td>
<td>Solubility</td>
<td>Protein loss</td>
</tr>
<tr>
<td>Colour loss</td>
<td>Texture</td>
<td>Microbial survival</td>
</tr>
<tr>
<td></td>
<td>Aroma loss</td>
<td></td>
</tr>
</tbody>
</table>

(Okos et al., 2007)

In respect to chemical changes which result from the drying process, browning can occur due to the presence of soluble constituents such as sugar and as a result of high temperature. Browning reactions result in colour change, reduction of nutritional value and solubility, off-flavours, and texture changes. Furthermore, the quality of dried foods also decreases because of lipid oxidation, which creates development of off-flavours, loss of fat soluble vitamins and pigment loss. There are many factors contributing to lipid oxidation, such as moisture content, oxygen content, temperature, UV light,
presence of metals and other chemical reactions. Generally, a higher level of lipid oxidation is found in freeze-dried foods as compared to air-dried ones. This is because freeze-dried products are more susceptible to oxygen than air-dried products due to their highly porous structure (Okos et al., 2007). Additionally, carotenoids, being fat soluble pigments, are also easily degraded by heat and oxidation due to their unsaturated chemical structure. The loss of carotenoids will lead to colour changes in fruits and vegetables; this phenomenon can be prevented by blanching, or treatment with ascorbic acid or sulphur dioxide (Fellows, 2000). Moreover, according to Okos et al. (2007), carotene reduction is higher during storage than that occurring in drying process.

Generally, physical characteristics of food such as texture, rehydration potential, solubility and aroma will change during drying. The greatest changes of food texture are found when rapid drying and high temperatures are applied. This is because high temperatures will cause complex chemical and physical changes to solutes at the surface of fruits. As a result, a hard impermeable skin, also termed as “case hardening”, is formed. This can be reduced by preventing high moisture gradients between the interior and the surface of fruits (Fellows, 2000). Rehydration potential is one of the most important parameters for determining food quality. Under optimum drying conditions, the foods are less damaged and rehydrate more quickly and completely. Solubility is also another important physical characteristic that needs to be considered. There are many factors such as processing conditions, storage conditions, composition, pH, density and particle size that influence the solubility of dried foods (Okos et al., 2007). In terms of the aroma of dried foods, heat significantly causes loss of volatile organic compounds that are responsible for aroma and flavour (Fellows, 2000; Okos et al., 2007). Therefore, foods such as herbs and spices that have a high economic value due to their aromas and flavours should be dried at low temperatures (Fellows, 2000).

Nutritional values are very important parameters affected during the drying process; the resultant values greatly vary depending on the pre-treatments, drying temperature and drying time, and the storage conditions. According to Fellows (2000), heat and oxidation during drying are the most important causes of nutritional loss. When water is removed by heat during drying, the catalysts which dissolve in water will promote oxidation of oil soluble vitamins due to being more reactive. Furthermore, drying does not affect the biological value and digestibility of proteins in most foods. However, the biological value can be reduced after drying depending on drying temperature and
residence time. Finally, the microbiological quality of dried foods is the most critical factor that needs to be taken into account. This is because it affects not only the quality of the food product itself but also has consequences in terms of the human health when dried foods are consumed.

In this study, the physicochemical and antioxidant activity of Gac powder produced by air drying, vacuum drying, spray drying and freeze drying were evaluated. It was necessary to use several pretreatments before the drying processes to ensure prevention of carotenoid loss. In addition, it is clear that fat soluble carotenoids are easily degraded by light, temperature and oxygen during storage. Therefore, good packaging and storage conditions, such as high barrier vacuum and laminated bags, and low temperatures, should necessarily be used for the final Gac powders.

2.5 Encapsulation process by spray drying of carotenoids

Encapsulation by spray drying has been widely applied in relation to natural food colorants. Many studies examining means of protecting loss of active nutrients, such as carotenoids, during the encapsulation process have been carried out. Since Gac fruit contains a particularly high level of carotenoids, spray dried encapsulated Gac fruit carotenoids as a food colorant and nutrient supplement should be considered. Therefore, research to develop suitable drying conditions for this fruit needs to be investigated in terms of the encapsulating agent and spray drying conditions.

In fact, there are many encapsulating agents, or carriers, or wall (or coating) materials for encapsulating food ingredients, all of which are widely used in the food industry. Depending on the characteristics and constituents of materials containing “active” components, it is very important to understand the chemical and physical properties of the encapsulating agents and to select the best carrier for protecting the ingredients. The typical categories of encapsulating agents whose properties can be found in the literature are shown in Table 2.2. The selection of the coating material should completely meet various criteria such as good emulsifying properties, good wall forming characteristics, low viscosity at high solid concentration, effective protection of the active ingredients, stability during storage, and low associated costs (Shahidi & Han, 1993; Gharsallaoui et al., 2007).

The encapsulating agent plays a very important role in the stability of active components, especially carotenoids. The encapsulating agent, or a combination of
them, is particularly suitable for a specific food ingredient due to its individual characteristics and constituents. In order to make a right choice of the agents for the encapsulation process, adequate tests should be performed. In fact, there are many studies that have reported success in maintaining the stability of carotenoids by utilising the encapsulation process. Wagner and Warthesen (1995) used four different dextrose equivalent (DE) hydrolysed starches (4, 15, 25 and 36.5 DE) as the encapsulating agent for spray drying to preserve the carotene content in carrots. The results indicated that the shelf life of spray dried encapsulated carrot powder using all four starches was 70 to 220 times longer than that of carrot juice without using wall material. Among the agents, hydrolysed starch of 36.5 DE had the highest retention of α- and β-carotenes in the resultant carrot powder. Moreover, use of cyclodextrins and cyclodextrin complexes as encapsulating agents has been widely applied in the food industry due to their favourable characteristics. These carriers have been scientifically proved as non-toxic, thermally stable and not hygroscopic, and therefore offer several advantages for the encapsulation process. One of these benefits is that it is an inexpensive means to protect against oxidation and decomposition by light or heat (Szente & Szejtli, 2004). According to Blanch et al., (2007), for example, all-trans-lycopene from tomatoes treated with the encapsulation process using β-cyclodextrin was stabilized for at least 6 months. Moreover, the authors pointed out that using α-cyclodextrin for encapsulated carotenoid preparations from canola oil could preserve the carotenoid and tocopherol content in the oil (Basu & Vecchio, 2001). Similarly, Szente et al., (1998) showed that the stability and solubility of carotenoids were effectively achieved by using α-cyclodextrin complexation and methylated β-cyclodextrin, respectively as encapsulating agents.

Table 2.2 Encapsulating agents for encapsulation of food ingredients

<table>
<thead>
<tr>
<th>Category</th>
<th>Encapsulating agents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbohydrate</td>
<td>Starch, maltodextrins, corn syrup, dextran, sucrose, cyclodextrins</td>
</tr>
<tr>
<td>Cellulose</td>
<td>Carboxy methylcellulose, methylcellulose, ethylcellulose, nitrocellulose, acetylcellulose, cellulose acetate-phthalate, cellulose acetate-butylate-phthalate</td>
</tr>
<tr>
<td>Gum</td>
<td>Gum acacia, agar, sodium alginate, carrageenan</td>
</tr>
<tr>
<td>Lipids</td>
<td>Wax, paraffin, beeswax, tristearic acid, diglycerides, monoglycerides, oils, fats, hardened oils</td>
</tr>
<tr>
<td>Protein</td>
<td>Gluten, casein, gelatine, albumin, hemoglobin, peptides.</td>
</tr>
</tbody>
</table>

(Shahidi & Han, 1993)
Furthermore, furcellaran, an anionic sulphated polysaccharide and co-polymer of β- and κ-carrageenan extracted from the red algae *Furcellaria lumbricalis*, is also used as an encapsulating agent for β-carotene (Laos et al., 2007). In addition, Loksuwan (2007) reported that acid-modified tapioca starch is the most effective carrier for β-carotene as compared to native tapioca starch and maltodextrin. Gelatine which is a water-soluble protein, has not only a good coating property for the encapsulation process but is also nontoxic, inexpensive and commercially available, in addition to being another effective wall material. Robert et al., (2003) reported that using a gelatine matrix is the best way to protect carotenoid pigments, especially *trans*-β-carotene, from *rosa mosqueta* oleoresin. Additionally, the stability of the lycopene microcapsule during storage was successfully achieved when using a ratio of gelatine to sucrose of 3:7 as the carrier (Shu et al., 2006).

According to Shahidi and Han (1993), there are many techniques that can be used in the encapsulation process. Generally, these can be divided into three groups, as physical, chemical and physicochemical methods (see Table 2.3). Among the numerous methods used to form microcapsules, spray drying has been widely used for the encapsulation process in the food industry due to the effective production costs and high quality of resultant dried food products.

### Table 2.3 Widely used methods for encapsulation process

<table>
<thead>
<tr>
<th>Category</th>
<th>Methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>Physical methods</td>
<td>Spray drying, spray chilling, spray cooling, fluid bed coating (spray coating in fluidized bed), extrusion, multiorifce centrifugal extrusion, co-crystallization, freeze drying</td>
</tr>
<tr>
<td>Chemical methods</td>
<td>Molecular inclusion (inclusion complexation), interfacial polymerization</td>
</tr>
<tr>
<td>Physicochemical methods</td>
<td>Coacervation (aqueous phase separation), organic phase separation, liposome entrapment</td>
</tr>
</tbody>
</table>

(Shahidi & Han, 1993)

To produce a good quality encapsulated ingredient powder in terms of physicochemical properties, spray drying conditions such as feed temperature, inlet temperature, outlet temperature and atomization pressure should be taken into account. This is very important since these factors directly affect a final product, so the suitable spray drying conditions should be fully utilised for specific materials containing sensitive food ingredients. For example, spray drying operating conditions set at an inlet temperature
of 200±5°C and outlet temperature of 100±5°C in combination with hydrolysed starch of 36.5 DE maintained the stability of encapsulated α- and β-carotenes from carrots (Wagner & Warthesen, 1995). However, to achieve the protective effect for carotenoid pigments from rosa mosqueta, Robert et al. (2003) reported that spray drying conditions of an inlet temperature of 100±5°C, outlet temperature of 65±5°C and atomisation pressure of 5 kg/cm² effectively prevented carotenoid degradation. Moreover, other results have shown that the suitable spray drying conditions for lycopene encapsulation are a feed temperature of 55°C and an inlet temperature of 190°C (Shu et al., 2006).

It can be concluded that encapsulation is a particular good potential method in the protection of carotenoids during drying processes. Therefore, the right selection of encapsulating agent and suitable spray drying should be applied for the process of effectively producing spray dried encapsulated carotenoid powder from Gac fruit for use as a food colorant and nutrient supplement. Since Gac fruit contains a high level of carotenoids research to develop the most suitable process for this particular fruit needs to be seriously investigated in terms of the appropriate encapsulating agent and spray drying conditions.

2.6 Stability of food during storage

2.6.1 Major modes of food degradation

In addition to quality loss through processing, the quality of foods may also change during the periods of storage and distribution. The major modes of the degradation of foods are generally classified as physical changes, chemical changes and microbiological changes. Therefore, it is very important to understand these modes of change in terms of the ways that they lead to food deterioration.

The physical changes in dried food products are frequently caused by storage in inappropriate conditions. As a result, the shelf life of the products may be significantly reduced. For example, when dehydrated foods are stored at high humidity, the process of moisture uptake will take place. As a result, the products become soggy, leading to degraded quality and shortened shelf life (Singh, 2000). Moreover, when food powders are exposed to moist atmospheres or elevated storage temperatures, the phenomenon of caking or spontaneous agglomeration will occur due to the sorption of moisture. The main reason for these occurrences is an inadequate barrier being provided by the packaging (Robertson, 2006).
The degradation of sensory quality and nutritional quality can be caused by chemical reactions. The main two causes of chemical changes during storage are the interaction between the internal food components and the external environmental factors. Environmental factors such as light, oxygen, temperature and $A_w$ affect the rate of lipid oxidation in foods during storage. The deterioration rate will accelerate in foods containing high unsaturated fatty acids when kept at inappropriate conditions, such as exposure to oxygen or high temperature. Additionally, chemical reactions are related to enzymic action, lipid oxidation and non-enzymic browning; these reactions are responsible for undesirable changes in colour, flavours and nutritive values (Singh, 2000; Robertson, 2006).

The deterioration of foods during storage is also influenced by the presence and activity of microorganisms. It is well known that the rate of microbiological growth is a function of various environmental factors such as temperature, moisture content, and oxygen. Generally, low moisture content in dried foods (fruits and vegetables) can prevent or minimise the growth of microbial organisms. Furthermore, according to Robertson (2006), the causes of spoilage in foods due to microorganisms can be divided into two factors, being intrinsic and extrinsic. The intrinsic factors are pH, $A_w$, and nutrient content, while storage temperature, relative humidity of the environment, and the concentration of gases in the environment are the extrinsic factors. Therefore, an understanding of these causes of the deterioration, and the appropriate storage conditions for food products is necessary.

2.6.2 The rate of deteriorative reactions

The quality change of foods during storage can be predicted by reaction kinetics. The index of degradation in terms of physical, chemical and sensory measurements can be used to quantitatively evaluate the quality changes. The changes can be predicted or simulated when the degradation index is expressed as a function of the conditions existing during storage (Robertson, 2006). The quality loss affected by intrinsic and extrinsic factors can be described by the equation:

$$- \frac{dC}{dt} = f(I_i, E_j)$$

where $-dC/dt$: rate of change of some index of deterioration $C$ with time $t$; a negative sign is used if the concentration of $C$ decreases with time. $I_i$: intrinsic factors ($i = 1 \ldots m$)
Ej: extrinsic factors (j = 1…n)

In fact, it is very difficult to determine the actual mechanisms of intermediate reactions which cause the quality changes due to the complex nature of foods. Therefore, the quality loss can be determined as being proportional to the power of the concentration of the reactant or product:

$$-\frac{dA}{dt} = kA^n \quad \text{or} \quad \frac{dB}{dt} = kB^n.$$  

where \(A\) and \(B\): concentration of quality factor measured

\(t\): time

\(k\): rate constant (dependent on extrinsic factors such as temperature, \(A_w\) and light intensity)

\(n\): a power factor called the order of the reaction. \(n\) can be a fraction or a whole number

\(\frac{dA}{dt}\) and \(\frac{dB}{dt}\): change in concentration of \(A\) or \(B\) with time.

The extrinsic factors are kept constant; otherwise, their effect on the rate constant \(k\) must be taken into account in evaluating the equation.

In general, the quality changes in foods follow the reaction order of 0 or 1. The zero order rate (also called pseudo zero order reaction) is useful in describing reactions such as non-enzymic browning (dry cereals and powdered dairy products), lipid oxidation (development of rancidity in dry foods) and enzymic degradation (fresh fruits and vegetables). However, the types of food degradation reactions that show a first order reaction include vitamin loss, protein losses and microbial growth. Figure 2.15 shows the quality changes of food following two different orders of reactions (Singh, 2000; Robertson, 2006).

![Figure 2.15 Quality change versus time showing the effect of order of the reaction on extent of change](image-url)
Furthermore, it is sometimes desirable to know the time for half life of a food degradation reaction. To obtain the half life time the following equation can be applied:

\[ t_{1/2} = \frac{0.693}{k}. \]

### 2.6.3 Effect of extrinsic parameters on the rates of degradation reactions

#### 2.6.3.1 Temperature

Temperature is the key parameter influencing the rate of degradation reactions. The most common and generally valid relationship for the effect of temperature on degradation reactions is that of Arrhenius. The relationship is expressed as

\[
\frac{d(\ln k)}{dT} = \frac{E_a}{RT^2}
\]

For practical reasons, the integrated form is used:

\[ k = Ae^{-\frac{E_a}{RT}} \]

Where
- \( k \): rate constant for degradation reaction
- \( A \): constant, dependent of temperature (also known as the Arrhenius, pre-exponential, collision or frequency factor)
- \( E_a \): activation energy (kcal mol\(^{-1}\))
- \( R \): the universal gas constant (1.987 kcal mol\(^{-1}\))
- \( T \): absolute temperature (\(^{\circ}\)K)

The integrated relationship assumes that the activation energy and the frequency factor do not change with temperature. The inherent assumption is generally, but not always true. As a result, predictions based on this model sometimes fail when applied over a temperature span of greater than 40\(^{\circ}\)C. The value of \( E_a \) indicates the temperature sensitivity of the reactions; that is, how much faster the reaction will proceed if the temperature is increased. Furthermore, \( E_a \) depends on factors such as moisture content, solid concentration, \( A_w \) and pH (Robertson, 2006).

#### 2.6.3.2 Water activity and isotherms

According to the definition of water activity given in section 2.4.1, the values of \( A_w \) are temperature dependent. Therefore, moisture sorption isotherms must exhibit temperature dependence. At any moisture content, \( A_w \) increases with increasing temperature and is expressed by the Clausius-Clapeyron equation as:
\[
\ln \frac{A_{w_2}}{A_{w_1}} = \frac{\Delta H}{R} \left[ \frac{1}{T_1} - \frac{1}{T_2} \right]
\]

Where \( A_{w_1} \) and \( A_{w_2} \): water activity at temperature \( T_1 \) and \( T_2 \) (\( ^{\circ}\)K)

\( \Delta H \): isosteric net heat of sorption at the moisture content of the food (J mol\(^{-1}\))

When a food is placed in an environment at a constant temperature and relative humidity, it will eventually equilibrate with that environment. The corresponding moisture content at steady-state is referred to as the equilibrium moisture content. Moisture sorption isotherms are plotted by measuring the moisture content and the corresponding relative humidity or \( A_w \) at constant temperature. This is a very useful way to evaluate the stability of foods and select appropriate packaging to minimise water activity (Robertson, 2006).

2.6.3.3 Gas atmosphere

It has been established that atmospheric oxygen impacts on the nutrient quality change of foods. For example, the loss of carotenoids is due to the oxidation of carotenoids in the presence of oxygen. Thus, it is very important to prevent the presence of oxygen or maintain low concentrations of oxygen during storage. There are several methods to modify the gas atmosphere inside food packages; these are creating a vacuum, removing most of the gases present, and flushing the headspace area inside the package with an inert gas such as nitrogen.

2.6.3.4 Light intensity

The light intensity and exposure time significantly affect the quality of foods in terms of discoloration and nutrient loss during storage and distribution. Losses of lipid soluble vitamins, particularly vitamin A and E, are caused by the catalytic effect of light on the lipid oxidation reaction. Furthermore, pigment compounds such as carotenoids in foods also reduce due to oxidation supported by light. Therefore, suitable packaging should be used to protect the packaged food from exposure to light (Robertson, 2006).

2.7 The role of carotenoids as a food colorant and nutrient supplement

Carotenoids have been used as a natural food colorant and nutrient supplement for centuries due to their attractive yellow-orange-red colour, and health benefits such as
the high level of vitamin A activity and strong antioxidant activity. However, to apply these successfully in the food industry, the colour and supplement applications of rich carotenoid fruits need to be further investigated.

2.7.1 Natural food colorants

Generally food colorants are divided into three groups, as synthetic colorants, nature-identical colorants and natural colorants. Colorants are called synthetic colours when they are obtained solely by chemical synthesis and are not found in nature. The second group are also produced by chemical synthesis, but they are chemically identical to natural colorants. Natural colorants, the third group, are extracted from natural edible materials using approved methods, for example lycopene extracted from tomatoes (Henry, 1996). Additionally, the classification of colorants is also based on legislation systems which state that colorants are divided into 2 groups, as certifiable or exempt from certification. However, gaining approval to use natural colorants as food additives is a complicated task, because it takes considerable time to meet the requirements laid down by a variety of legislations, organizations, and governments, for example, the European Union (EU), the U.S. Food and Drug Administration (FDA) and the World Health Organization (WHO) (Delgado-Vargas & Paredes-López, 2003). As a result, there are only 13 natural colorants approved in the EU and 26 natural colorants certificated in the USA (Downham & Collins, 2000). In addition, other drawbacks of developing new colorants are the high costs for manufacturers and the expense for consumers (Wissgott & Bortlik, 1996).

There are many factors affecting the choice and application of any particular natural colorant. The factors included are solubility, physical form, composition, processing conditions, pH, packaging and storage condition. Furthermore, each colorant will be differently suited with respect to a particular food matrix and will be influenced by several factors in varying conditions (Henry, 1996; Delgado-Vargas & Paredes-López, 2003). For example, the hue of carotenoids such as β-carotene, apo carotenal and canthaxanthin are often unchangeable over a pH range of 2 to 7 (Gordon et al., 1982). Therefore, it is very important to optimise the factors to produce stability in the final product’s colour. For instance, application forms of carotenoid food colorants should be maintained in the pH range described.

In reality, carotenoid extracts as food natural colorants are available in very many different application forms. They are necessary for use in oily products (margarines,
oils, fats and shortenings), fruit juice, beverages, dry soups, canned soups, dairy products, milk substitutes, coffee whiteners, dessert mixes, preserves, syrups, confectionery, salad dressings, meat products, pasta, egg products, baked goods and others (Francis, 2000; Delgado-Vargas & Paredes-López, 2003). Among these, the application of natural colorants is particularly evident in confectionery, soft drinks, alcoholic beverages, salad dressings and dairy products (Wissgott & Bortlik, 1996).

2.7.2 Nutrient supplements

It is well known that carotenoids from plant based foods play a crucial role in human health (Rao & Rao, 2007; Roberts et al., 2009). It has been reported that β-carotene supplements probably prevent cancer and may reduce the risk of several chronic conditions (Boonsiri et al., 2007). A number of studies found that consumption of foods containing lycopene are linked with a lower risk of prostate cancer (Guns & Cowell, 2005; Chan et al., 2009). Moreover, Mignone et al. (2009) found that the risk of premenopausal breast cancer may be reduced by a high consumption of carotenoids. Therefore, fruits and vegetables containing high carotenoid levels, such as Gac fruit, should be processed as nutrient supplements for the functional food industry. However, it is currently still difficult to assess the safety and effectiveness of nutrient supplements due to the associated time-consuming testing and measurement of outcome (Webb, 2006).

Usage of carotenoid-rich plants as a nutrient supplement is one of the most effective solutions to prevent vitamin A deficiency and in addition can assist in the treatment of various other diseases. Some health benefits of carotenoids are shown in Table 2.4. As a result, the nutrient supplement benefits of specific plant products should be scientifically investigated. Vuong et al. (2002) conducted a trial using Xoi Gac, rice cooked with Gac fruit containing 3.5mg of β-carotene, as a nutrient supplement. After 30 days of supplementation, results showed that plasma lycopene, β-carotene and retinol concentrations, and haemoglobin concentrations of the test subjects were significantly increased. In addition, Tran et al. (2008) reported that Gac fruit powder can be successfully used as food colorant and carotenoid supplement in Xoi Gac, yoghurt, fettuccine pasta, and creamy sauce. It is thus also recommended that further studies should be carried out to optimise processing parameters such as the appropriate dose of the powder, shelf life, and bioavailability of the final product.
Moreover, Strobel et al. (2007) reported that vitamin A plays a key role for pregnant and breastfeeding women. This is due to its important function in the maturation of the embryo, the lung development, and overall development of the baby. Hence, it is necessary to supply β-carotene as a source of vitamin A in the diet for the women in this period, especially as β-carotene-rich fruits. Bioavailability and conversion to vitamin A are two of the most crucial functions of β-carotene in food, and should thus be used as a precursor of vitamin A. The general conversion factor for fruits and vegetables is that 12 mg of β-carotene will produce 1 mg of vitamin A (12:1). In addition to β-carotene, lycopene is one of the most important nutritional components for preventing disease progression, particularly in prostate cancer. According to Schwarz et al. (2008), using a lycopene supplement at a dose of 15 mg/day for patients who had been diagnosed with benign prostate hyperplasia could inhibit progression of this disease. As a result, it is recommended that carotenoids-rich vegetables and fruits should be used as a food supplement for this specific purpose.

Table 2.4 Some biological activities of carotenoids

<table>
<thead>
<tr>
<th>Type of carotenoid</th>
<th>Biological function</th>
</tr>
</thead>
<tbody>
<tr>
<td>All</td>
<td>Effect in the immune response and in the intercellular communication, treatment of photosensitivity diseases. The use of algae (especially <em>Phaeophyta</em>) carotenoids diminishes the risks of being affected by certain types of cancer</td>
</tr>
<tr>
<td>β-carotene, canthaxanthin, 4-hydroxy-β-carotene, &amp; synthetic retro-dehydro-β-carotene</td>
<td>Induction of gap junctional communication (GJC) in murine fibroblasts; β-carotene and retro-dehydro-β-carotene are the most efficient inducers</td>
</tr>
<tr>
<td>β-carotene</td>
<td>Treatment of certain kinds of cancer (e.g., smoking-related cervical intraepithelial neoplasia and cervical and stomach cancer), affects the immune response in rats and by this mean tumor growth is inhibited, inhibits lipid peroxidation, suppresses the increase of hormones related to stress syndrome</td>
</tr>
<tr>
<td>Zeaxanthin and canthaxanthin</td>
<td>Quench radicals in the aqueous phase; inhibit methyl linoleate oxidation.</td>
</tr>
<tr>
<td>α-carotene, fucoxanthin and halocintiaxanthin</td>
<td>Higher inhibitory activity than β-carotene in the proliferation of human neuroblastoma cells.</td>
</tr>
<tr>
<td>Lutein, zeaxanthin</td>
<td><em>Capsicum annuum</em> is a rich source; these carotenoids have shown antimutagenic activity, protective effect against</td>
</tr>
<tr>
<td>Type of carotenoid</td>
<td>Biological function</td>
</tr>
<tr>
<td>-------------------</td>
<td>---------------------</td>
</tr>
<tr>
<td>Lutein</td>
<td>Aztec marigold (<em>Tagetes erecta</em>) is a rich source; has shown antimutagenic activity.</td>
</tr>
<tr>
<td>Retinoids</td>
<td>Treatment of certain kinds of cancers and some dermatological activity vision process, epithelial maintenance, mucous secretion, reproduction, morphogenesis and differentiation. Involved in aging; modulation of triiodothyronine receptor and of transglutaminase is mediated by retinoids. Retinoic acid is a modulator of the immune system that must be carefully controlled; regulates the γ-interferon gene, which has great influence in the immune system and in the inflammatory response; induces the response of protein associated with damage by ultraviolet lighting F9 and NIH3T3 cells; differentiation of F9 cells by an increase in of cellular communication.</td>
</tr>
</tbody>
</table>

*(Delgado-Vargas & Paredes-López, 2003)*
Chapter 3
MATERIALS AND METHODS

3.1 Materials
3.1.1 Chemicals and drying equipment

All chemicals used in this research, as listed in Table 3.1, were mainly purchased from Sigma-Aldrich Pty. Ltd., Australia.

Table 3.1 Chemicals used in the study

<table>
<thead>
<tr>
<th>Name and description of chemical</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABTS (2,2’-Azino-Bis(3-Ethylbenzthiazoline-6-Sulfonic acid) Diammonium)</td>
</tr>
<tr>
<td>acetone, ≥99.5%</td>
</tr>
<tr>
<td>carotene (approx. 2:1 of β:α) mixed isomers from carrots, ≥95% (HPLC) powder form</td>
</tr>
<tr>
<td>DPPH (2,2-Diphenyl-1-Picrylhydrazyl)</td>
</tr>
<tr>
<td>L-ascorbic acid, 99%</td>
</tr>
<tr>
<td>Maltodextrin, 12 DE, Glucidex®, Roquette, France</td>
</tr>
<tr>
<td>methanol, spectrophotometric grade</td>
</tr>
<tr>
<td>n-Hexane, 95%</td>
</tr>
<tr>
<td>potassium persulfate, 99.99% metal basis</td>
</tr>
<tr>
<td>sodium bisulfite, ≥99%</td>
</tr>
<tr>
<td>Trolox ((S)-(−)-6-Hydroxy-2,5,7,8-Tetramethylchroman-2-Carboxylic acid), 98%</td>
</tr>
</tbody>
</table>

As the drying processes for the study were carried out both in Vietnam and Australia, as detailed in the relevant sections in chapters 4 and 5, different apparatus were used at each location. The brands of the drying equipment used are listed in Table 3.2.

Table 3.2 Drying equipment used in the study

<table>
<thead>
<tr>
<th>Drying method</th>
<th>Location for experiments</th>
<th>Dryer Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>Australia</td>
<td>G.T.C. Pty Ltd., Australia</td>
</tr>
<tr>
<td></td>
<td>Vietnam</td>
<td>an oven made at Nong Lam University, Vietnam</td>
</tr>
<tr>
<td>Vacuum</td>
<td>Australia</td>
<td>Thermoline Scientific Equipment Pty. Ltd, Australia</td>
</tr>
<tr>
<td></td>
<td>Vietnam</td>
<td>Weiss Gallenkamp, UK</td>
</tr>
<tr>
<td>Freeze</td>
<td>Australia</td>
<td>Dynavac Engineering Pty. Ltd., Australia</td>
</tr>
<tr>
<td></td>
<td>Vietnam</td>
<td>VirTis, USA</td>
</tr>
<tr>
<td>Spray</td>
<td>Vietnam</td>
<td>LabPlant UK Ltd., England</td>
</tr>
</tbody>
</table>
3.1.2 Raw Gac fruit sources

Gac fruit comprises orange/yellow skin containing thorns, yellow pulp and aril (red flesh surrounding the seeds). The components are shown in Figure 3.1.

![Figure 3.1 Fresh Gac fruit components](image)

The frozen Gac arils with seeds used in this study were purchased from a local market in Sydney, Australia. The fruit was transported in an insulated container and stored frozen at -20°C until use within 2 days. The frozen arils with seeds were thawed in a refrigerator before drying. Due to limited availability of the frozen fruit in Australia, the experiments were carried out using small quantities.

Whole fresh Gac fruits were purchased from a local market in Hochiminh City, Vietnam. The fruit was transported in an insulated container and used on the same day. The fresh fruits were then pre-treated prior to drying treatment. Due to the almost year round availability of the fresh fruits, the drying experiments were carried out with large quantities of fruit using pilot plant equipment.

3.2 Analytical methods

All analytical methods in described in this chapter were those applied for the Gac powders or Gac products, in the experiments shown in the following chapters, as applicable.
3.2.1 Moisture content

The moisture content of Gac samples was determined by drying at the temperature of 105°C in the oven until a constant weight was obtained.

Moisture content indicates the total amount of water in food. The percentage of moisture content is normally expressed on a wet basis (wb) or a dry basis (db).

The moisture content on a wet basis is calculated as

\[ MC_{wb}(\%) = \frac{\text{mass of water}}{\text{total mass of food}} \times 100\% \]

\[ = \frac{\text{mass of water}}{\text{mass of dry solids + mass of water}} \times 100\% \]

The moisture content on a wet basis (a range between 0 and 100%) is usually used in the food industry.

The moisture content on a dry basis is defined as

\[ MC_{db}(\%) = \frac{\text{mass of water}}{\text{mass of dry solids}} \times 100\% \]

The moisture content on dry basis is often used in the drying engineering literature; this is because the mass of dry matter in food is constant during a drying process.

The conversion between \( MC_{wb} \) and \( MC_{db} \) is calculated as

\[ MC_{db}(\%) = \frac{MC_{wb}(\%)}{1 - MC_{wb}(\%)/100} \quad \text{or} \quad MC_{wb}(\%) = \frac{MC_{db}(\%)}{1 + MC_{db}(\%)/100} \]

3.2.2 Water activity (Aw)

The definition of Aw and relationship between Aw and deterioration reactions are reviewed in section 2.4.1. A water activity meter (AquaLab PawKit, Decagon Devices, USA) was used to measure \( A_w \) of the powders.

3.2.3 pH determination

The \( \text{pH} \) value of Gac aril powders treated with different maltodextrin ratios, yellow pulp powders, and skin powders was determined by using a \( \text{pH} \) meter calibrated with
standard buffers pH 7 and pH 4. Before measuring pH, Gac powders were reconstituted in distilled water to the same moisture content as the fresh Gac fruit.

3.2.4 Colour characteristics

The colour of Gac fruit powder samples was determined using a Minolta Chroma Meter calibrated with a white standard tile. The results were expressed as Hunter colour values of L*, a*, and b*, where L* was used to denote lightness, a* redness and greenness, and b* yellowness and blueness. Prior to colour measurement, the powder samples and “Xoi Gac” were packed into a polyethylene pouch and measured. For the reconstituted Gac powders and Gac beverage products, the samples were placed in clear Petri dishes and filled to the top before the measurement. Hunter values of the samples for each treatment method were measured in triplicate.

Chroma, representing colour intensity, was calculated by the formula \((a^{*2} + b^{*2})^{1/2}\). The hue angle \((H^{0})\) was calculated by the formula \(H^{0}=\arctan(b^{*}/a^{*})\). The hue angle values vary from 0\(^{0}\) (pure red colour), 90\(^{0}\) (pure yellow colour), 180\(^{0}\) (pure green colour) to 270\(^{0}\) (pure blue colour). The desirable hue angle is about 45\(^{0}\). The ratio of \(a^{*}/b^{*}\) was also used for the colour measurement. A lower hue angle and a higher ratio of \(a^{*}/b^{*}\) are desired, indicating a redder colour. Total colour difference or change between two samples was calculated by the following formula:

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

where \(L_0^{*}, a_0^{*}\) and \(b_0^{*}\) are the values of the samples at zero time, and \(L^{*}, a^{*}\) and \(b^{*}\) the measured values of each sample with time (Duangmal et al., 2008).

3.2.5 Water solubility index (WSI)

The WSI of the Gac powders was determined using the method described by Anderson et al., (1969). Spray-dried Gac fruit powder (2.5 g) and distilled water (30 mL) were vigorously mixed in a 100 mL centrifuge tube, incubated in a 37\(^{0}\)C water bath for 30 minutes, and then centrifuged for 20 minutes at 10,000 rpm in a J2-MC Centrifuge (Beckman, USA). The supernatant was carefully collected in a pre-weighed beaker and oven dried at a temperature of 103±2\(^{0}\)C. The WSI (%) was calculated as the percentage of dried supernatant with respect to the amount of the original 2.5 g Gac fruit powder.
3.2.6 Bulk density

Bulk density (g/mL) was determined by gently adding 2 g of Gac powder into an empty 10 mL graduated cylinder and holding the cylinder on a vortex vibrator for 1 minute. The ratio of the mass of the powder and the volume occupied in the cylinder determines the bulk density value (Goula et al., 2004).

3.2.7 Extraction and separation

A method described by Tran et al. (2008) was employed, with some modifications, to extract the carotenoid content from the Gac samples. A quantity of approximately 0.1 g of Gac powder, or 0.3 g of fresh Gac aril or “Xoi Gac”, or 1 mL of Gac products was weighed in a beaker, and then extracted with 10 mL of the solvent, which was a mixture of n-hexane to acetone (v/v 3:2). The residue was further extracted four times using a magnetic stirrer until colourless, each time with 5 mL of the solvent. The extracts were combined and washed twice to remove acetone, each time with 25 mL of distilled water in a separating funnel. A few drops of saturated NaCl solution were added to the funnel to facilitate phase separation. The upper part was collected to measure total carotenoid content and lipophilic antioxidant activity. The lower part was collected to measure hydrophilic antioxidant activity. The process was conducted under dim light and samples analysed within one day.

3.2.8 Determination of total carotenoid content

Tran (2006) found that the optimal wavelength to measure the total carotenoid content in the Gac arils and Gac products using spectrophotometer method is 473 nm. The technique was chosen to truly reflect the total carotenoids content rather than just lycopene as the total antioxidant activity came from all carotenoids. Carotene (from carrots) solution (0.0005–0.01 mg/mL) was used to construct the standard curve for the determination of total carotenoid content of the samples. Total carotenoid content of the Gac fruit powders, the fresh fruit and the Gac products was spectrophotometrically determined at 473 nm and expressed based on carotene equivalents (µg or mg/g of powder or fresh weight or “Xoi Gac”; or µg/mL of juice).

3.2.9 Determination of total antioxidant activity

3.2.9.1 ABTS assay

The procedure for determination of total antioxidant activity followed the method of Thaipong et al. (2006). A 7.4 mM ABTS solution and a 2.6 mM potassium persulfate
solution were used as the stock solutions. The equal quantities of the stock solutions were mixed as the working solution and reacted for 12-16 hours at room temperature in the dark. This solution was then diluted by mixing 1 mL ABTS solution with 60 mL methanol to obtain an absorbance of 1.1±0.02 units at 734 nm using the spectrophotometer. Fresh ABTS solution was prepared for each assay. Gac sample extracts (0.15 mL) were reacted with 2.85 mL of the ABTS solution for 2 hours in a dark situation. The absorbance was spectrophotometrically taken at 734 nm. The standard curve was linear between 0.025 and 0.8 mM Trolox. Results were expressed in mmole Trolox equivalents (TE)/g of powder or µmole TE/mL of juice or µmole TE/g of “Xoi Gac”.

3.2.9.2 DPPH assay

The DPPH assay was adapted from that used by Thaipong et al. (2006). The stock solution contained 24 mg DPPH and 100 mL methanol. The working solution was obtained by mixing 10 mL stock solution with 45 mL methanol to obtain an absorbance of 1.1±0.02 units at 515 nm using the spectrophotometer. Gac sample extracts (0.15 mL) were allowed to react with 2.85 mL of the DPPH solution for 24 hours in the dark. Then the absorbance was taken at 515 nm. The standard curve was linear between 0.025 and 0.8 mM Trolox. Results were expressed in mmole TE/g of powder.

3.2.10 Encapsulation efficiency (EE)

A method described by Shu et al. (2006) was employed, with some modifications, to calculate encapsulation efficiency. The EE (%) was determined as the ratio between the initial content of total carotenoids before spray drying and the content of the final powder product. The calculation for EE is based on the total carotenoid content (mg/g of total solids).

3.3 Calculation of kinetic parameters

The degradation of TCC and TAA in freeze-dried and spray-dried powders was calculated by using the standard equation for a first-order kinetic model as follows:

\[ \ln C = \ln C_0 - k(t) \]

where \( C \) is the concentration at time \( t \); \( C_0 \) is the concentration at time zero; \( k \), the degradation rate constant (day\(^{-1}\)) is obtained from the slope of a plot of the natural log of \( C/C_0 \) vs. time; and \( t \), being the storage time (days).
The half-life was calculated at a specific temperature by the equation: 
\[ t_{1/2} = \frac{\ln 2}{k} \]

The activation energy (\(E_a\), kcal mol\(^{-1}\)) was determined by Arrhenius equation:
\[ k = A e^{E_a/RT} \]

where \(A\) is the frequency factor; \(R\) is the universal gas constant (1.987 kcal mol\(^{-1}\)); and \(T\) is the absolute temperature (°K).

### 3.4 Moisture sorption isotherms

Various Gac fruit powders, including red Gac aril powder, yellow pulp powder, skin powder and spray-dried powders were used for constructing moisture sorption isotherms. The fresh Gac aril powders were freeze-dried at a condenser temperature of -46°C, air-dried and vacuum-dried at temperature of 60°C and spray-dried at inlet temperature of 120°C and maltodextrin of 10%, whereas the yellow pulp and skin powders were air-dried at temperature of 60°C. Aril, yellow pulp and skin powders were sieved using a lab sieve with a mesh opening of 0.0553 inches. About 2 g of powders were weighed in aluminium containers and then put into a series of hermetic glass desiccators containing saturated salt solutions of sodium hydroxide (NaOH), lithium chloride (LiCl), potassium acetate (CH\(_3\)COOK), potassium carbonate (K\(_2\)CO\(_3\)), magnesium nitrate (Mg(NO\(_3\))\(_2\)), sodium chloride (NaCl), and potassium chloride (KCl), respectively. The desiccators were tightly closed and placed at room temperature. The samples were then weighed every three days until they reached equilibrium. The final moisture contents of the samples were determined by standard oven drying methods (at a temperature of 103°C for 24 hours).

The monolayer moisture content \(M_0\) (db) was calculated using the Brunauer Emmett Teller (BET) equation as follows:
\[ \frac{A_w}{(1 - A_w)MC} = \frac{1}{M_0C} \]

where \(MC\) is moisture content of powders expressed in g per 100 g solids; \(M_0\) is g of water equivalent to monomolecular layer adsorbed per 100 g dry solids; \(A_w\) is water activity at moisture \(MC\); and \(C\) is BET constant.

### 3.5 Statistical analysis

The experiments were carried out either in triplicate or duplicate, and results were presented as mean values with standard deviations. Different mean values (one, two and three factors) were analysed by analysis of variance (ANOVA) and least significant difference (LSD) using SPSS software version 17.0. The graphs of mean values and error bar were created using Excel version 2003.
Chapter 4

PRODUCTION OF GAC FRUIT POWDERS BY AIR DRYING, VACUUM DRYING AND FREEZE DRYING METHODS

4.1 Introduction

It has been established that Gac fruit contains very high levels of carotenoids, vitamin E and polyunsaturated fatty acids (section 2.1.2). It is very important, therefore, to preserve these nutritional components in any powder forms produced from Gac fruit. Additionally, Gac fruit can be used as a food colorant and as a nutrient supplement and these properties must be maintained in the processed forms, as discussed in chapter 2.

The powder form of fruit is used in the food industry due to its convenience in relation to use, storage and transport. Pre-treatments, prior to drying are one of the most important factors that affect the quality of product, especially in terms of carotenoid compound and antioxidant activity. Moreover, different drying methods, such as hot air drying, vacuum drying and freeze drying, result in different effects on the colour, content of carotenoid compounds and antioxidant activity of dried fruit and vegetable products.

It has been generally agreed that the choice of pre-treatments and drying treatments plays an important role in effectively maintaining the highest content of carotenoids, colour and antioxidant activity. Therefore, the quality of the final powder products from fruit and vegetables could be preserved by utilising particular pre-treatments and drying treatments.

Since very little information is published about Gac fruit, there is a need to investigate the effects of different pre-treatments (ascorbic acid solution, sodium bisulfite solution, and blanching) and different drying processes on powders mostly produced from the aril of frozen and fresh Gac fruit in respect of the colour, total carotenoid content (TCC), and total antioxidant activity (TAA) of Gac fruit powder. The results of this work are reported in this chapter.
Furthermore, since maltodextrin has been found to be useful as a drying aid for many fruit and vegetables, additional investigations were carried out in this study into the effect on the colour, TCC and TAA of powders produced by mixing various ratios of maltodextrin with the aril, skin, and fruit pulp of Gac fruit prior to air drying.

4.2 Drying treatments
4.2.1 Pre-treatments used prior to drying

Experiments were designed for air drying and vacuum drying of Gac aril from frozen and fresh fruit, at a range of temperatures. Table 4.1 shows the combination of parameters for the designed experiments. The whole seed aril of the frozen fruit was untreated for each of the drying processes. The whole seed aril of fresh Gac fruit was scooped out and subjected to the following treatments before drying: the aril soaked in 0.1% w/v ascorbic acid solution (ratio of 1 to 4) for 1 hour, soaked in 0.1% w/v sodium bisulfite solution (ratio of 1 to 4) for 1 hour, blanched in steam water for 3 minutes, and untreated as a control.

<table>
<thead>
<tr>
<th>Drying conditions</th>
<th>Frozen aril</th>
<th>Fresh aril</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air drying or vacuum drying temperature (°C)</td>
<td>40</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>70</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>80</td>
<td>X</td>
</tr>
<tr>
<td>Pre-treatment prior to drying (AD or VD)</td>
<td>Control (untreated)</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>Ascorbic acid</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>Bisulfite</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>Blanching</td>
<td>X</td>
</tr>
</tbody>
</table>

X: chosen drying temperatures or pre-treatment

In a separate set of experiments, the untreated aril of fresh Gac fruit was thoroughly mixed with various ratios of maltodextrin (12 DE, Glucidex®, Roquette, France) in a lab mixer before air drying. The ratios of maltodextrin and total Gac fruit solid (g/g) were 0.5, 1.0 and 1.5. A one factor of maltodextrin ratio was randomly designed to investigate the effect of maltodextrin on physicochemical and antioxidant properties of Gac powder.
4.2.2 Drying processes for Gac fruit components

Before drying, the frozen whole seed Gac arils were thawed to the temperature of 4°C, and were then dehydrated by hot air drying (AD), vacuum drying (VD), and freeze drying (FD). The equipment used to produce powders by the different drying techniques is listed in Table 3.2. The loading of material for AD and VD processes was 5 kg/m². Different temperatures of 50, 60, 70 and 80°C were applied in the air drying process (forced air dryer) and in the vacuum drying process with the pressure at -80 kPa gauge. The freeze drying process was carried out at a condenser temperature of -35°C and pressure of 0.2 mbar absolute for 42 hours as the material dried. The drying for the AD and VD processes was terminated when the final moisture content of each sample was constant (approximately 6%). After the drying processes were completed the seeds were separated from the aril. The dried Gac fruit aril was then powdered using a lab blender, and packed in quantities of 2 g into high barrier vacuum bags and laminated aluminum vacuum bags, using a vacuum sealer.

The two factors (drying methods and drying temperature) were randomly designed to investigate the influence of the air and vacuum drying conditions on the colour, TCC and TAA of frozen Gac aril powder. The drying processes were all carried out in triplicate. A total of 24 runs was conducted.

The whole seed arils of the fresh Gac fruit were dehydrated by hot air drying or vacuum drying after pre-treatment by the methods shown in Table 4.1, while freeze drying of the whole seed arils of the fresh Gac fruit was carried out without any pre-treatment. The loading of material for AD and VD processes was 5 kg/m². The AD process was carried out in an oven at different temperatures of 40, 50, 60, 70 and 80°C with the input air velocity of 1.1±0.1 m/s. The oven comprised four trays (1.2 x 0.8 m) equipped with manual temperature and heated air flow devices. The VD process was conducted at different temperatures of 40, 50, 60, 70 and 80°C and with the pressure at -80 kPa gauge. The freeze drying (FD) process was carried out at a condenser temperature of -46°C and pressure of 0.3 mbar absolute for 40 hours in a freeze dryer. The drying for the AD and VD processes was terminated when the final moisture content of each sample was constant (approximately 6%). The seed separation was carried out after each drying process was completed. As for the frozen samples, the dried Gac fruit from the fresh fruit was powdered using a lab blender and sealed in 2 g quantities into high barrier vacuum bags and laminated aluminum vacuum bags.
The three factors (pretreatment, drying methods and drying temperature) were randomly designed to investigate the effect of the air and vacuum drying conditions on the colour, TCC and TAA of fresh Gac aril powder. The drying processes were all carried out in triplicate. A total of 120 runs was conducted.

Additionally, untreated samples of the skin, the yellow pulp and a paste of the untreated aril mixed with maltodextrin were air-dried at 60°C for 12, 17 and 18 hours, respectively. These dried samples were powdered using a MF 10 Basic Microfine Grinder (IKA, Germany), and then packed into high barrier vacuum bags using a vacuum sealer. All the drying processes were carried out in duplicate.

4.3 Results and discussion
4.3.1 The physicochemical characteristics and weight distribution of fresh Gac fruit

A fresh Gac fruit consists of skin, yellow pulp, red aril, and seeds as shown in Figure 3.1. It is necessary to determine the physicochemical properties and the weight distribution of Gac fruit components for food processing. The physicochemical characteristics and weight distribution of the fresh Gac fruit components are shown in Table 4.2 and Table 4.3, respectively.

Table 4.2 Physicochemical characteristics of fresh Gac fruit aril

<table>
<thead>
<tr>
<th>Analysed item</th>
<th>Mean value</th>
<th>Analysis method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content (MC%, FW)</td>
<td>82.33 ± 2.01</td>
<td>Standard method</td>
</tr>
<tr>
<td>pH</td>
<td>5.79 ± 0.03</td>
<td>pH meter</td>
</tr>
<tr>
<td>Colour</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L*</td>
<td>36.25 ± 1.77</td>
<td>Minolta Chroma Meter</td>
</tr>
<tr>
<td>a*</td>
<td>30.79 ± 3.09</td>
<td></td>
</tr>
<tr>
<td>b*</td>
<td>14.06 ± 2.00</td>
<td></td>
</tr>
<tr>
<td>Total carotenoid content (µg β-carotene equivalent/g, FW)</td>
<td>1051.40 ± 381.33</td>
<td>Spectrophotometric method</td>
</tr>
<tr>
<td>α-tocopherol (µg/ g, FW)</td>
<td>76</td>
<td>Vuong et al., 2006</td>
</tr>
<tr>
<td>Fatty acids (µg/g of edible portion)</td>
<td>852</td>
<td>Vuong et al., 2006</td>
</tr>
</tbody>
</table>

#: Values for these items from the study by Vuong et al. (2006)
Table 4.3 Weight distribution of fresh Gac fruit (10 fruits)

<table>
<thead>
<tr>
<th>Gac fruit</th>
<th>Fresh weight (g)</th>
<th>Percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole fruit</td>
<td>7527±475</td>
<td>100</td>
</tr>
<tr>
<td>Skin</td>
<td>1332±87</td>
<td>18</td>
</tr>
<tr>
<td>Yellow pulp</td>
<td>3700±195</td>
<td>49</td>
</tr>
<tr>
<td>Aril</td>
<td>1375±147</td>
<td>18</td>
</tr>
<tr>
<td>Seeds</td>
<td>1084±52</td>
<td>14</td>
</tr>
</tbody>
</table>

From the figures shown in Table 4.3, the highest anatomical component of a Gac fruit is the yellow pulp (49%), whereas the aril, that contains the high level of carotenoids, accounts for only 18%. Therefore, it is necessary to consider ways to utilise the yellow pulp, skin and seeds in order to reduce environmental waste and economic problems. This will be discussed in section 4.3.8.

4.3.2 Drying curves for air drying and vacuum drying of fresh Gac fruit aril

Samples of approximately 200g of the untreated fresh Gac aril, in which the moisture content (MC) varied from 79.6 to 85.7%, were dried at a range of temperatures from 40 to 80°C, using both air and vacuum drying techniques. The results for moisture content versus drying time are presented in Figure 4.1. Generally, both the drying temperature and the drying method had a direct effect on the moisture content and drying time.

The graphs show that while the MC of air-dried samples initially dropped quicker than that of vacuum-dried samples, the total time to reach MC of 6% or less was the same or longer in air-dried samples. For both drying methods, the increase in drying temperature resulted in a more rapid moisture drop to reach the MC of 6% or less. For example, it took 48 and 45 hours respectively for air-dried and vacuum-dried samples at 40°C to attain MC of 6%, while the drying time at 80°C for both air-dried and vacuum-dried samples was approximately 12 hours. Moreover, during the initial stage of drying (6 hours) at lower temperatures, from 40 to 50°C, the water evaporation rate in the air drying process was much higher than in vacuum drying. However, the water evaporation rate in the vacuum drying process increased more than twice at the temperatures of 60 and 70°C, whereas in air drying this rate increased slowly. Additionally, at the drying temperature of 80°C, this rate was nearly the same in both drying methods. This phenomenon could be explained by the fact that only a minute
amount of air is allowed to come gradually through the vacuum dryer to enable water removal. It is more likely that the heat transfer rate is lower due to conduction from the hot shelves.

As Figure 4.2 shows, by plotting the drying rate against moisture content the impact of drying temperatures and drying methods can be clearly observed. The highest drying rate was observed in the air drying process at 80°C, whereas the lowest was in vacuum drying at 50°C. It can be observed that there was no constant rate drying stage during either the air or the vacuum drying processes at the temperatures of 70 and 80°C, and that all the drying process at these temperatures took place in the falling rate period. Similar results for dried fruits and vegetables products were also found by Piga et al. (2004), Singh et al. (2006), Schultz et al. (2007) and Yadollahinia et al. (2009).
Figure 4.1 Drying curves for air drying and vacuum drying of untreated fresh aril
Figure 4.2 Drying rates for air drying and vacuum drying of untreated fresh aril
4.3.3 Effect of air drying and vacuum drying on the colour of the powder from frozen and fresh Gac fruit arils

4.3.3.1 The colour characteristics of powder from frozen Gac fruit aril

The colour characteristics of the powders produced from frozen Gac fruit aril, in terms of how they are affected by AD and VD methods and by drying temperatures are shown in Figures 4.3a and 4.3b. The results show that drying temperatures and the drying methods affected the lightness of samples significantly (P<0.001 and 0.01, respectively). Generally, the L* value of vacuum-dried powders was higher than that of air-dried samples. A decreasing L* value of the powders products was observed with increasing drying temperature of 50°C to 70°C, indicating a darker appearance of the samples. However, the L* value of samples slightly increased from the temperature of 70°C to 80°C, for both drying methods. In addition, there was no interaction between drying temperatures and drying methods on the lightness (P>0.05). The lightness value of the sample vacuum-dried at temperature of 50°C was the highest while the lowest lightness was the powder air-dried at 70°C.

The chroma, a*/b*, and hue angle of samples were not statistically impacted by the drying method used (air drying or vacuum drying). However, the effect of drying temperature on the chroma, a*/b*, and hue angle of powder samples was statistically significant (P<0.001). For the chroma, it is clear that increasing drying temperatures resulted in decreasing chroma value; the chroma of air-dried powder at 50°C was the highest while the lowest was observed in air-dried powder at 80°C.

There was significant interaction between drying temperature and drying method on the a*/b* value and the hue angle (P<0.001), but not on the chroma (P>0.05). In general, the a*/b* value of vacuum-dried powders was higher than that of air-dried samples for the temperatures of 60 to 80°C. In contrast, at the lower temperature of 50°C, the a*/b* value of the sample produced by air drying was higher than that produced by vacuum drying. Moreover, the highest hue angle of vacuum-dried product at temperature of 50°C was found to be significant, whereas the lowest hue value was found in powder vacuum-dried at 60°C. However, as drying temperature increased from 60 to 80°C, the hue angle of air-dried powders was higher than that of vacuum-dried ones. No statistical difference of hue was observed between the temperature of 60 and 70°C, or 70 and 80°C.
Figure 4.3a Lightness and chroma of powder from frozen fruit aril affected by AD and VD treatments
Figure 4.3b $a^*/b^*$ and hue angle of powder from frozen fruit aril affected by AD and VD treatments.
4.3.3.2 The colour characteristics of the powder from fresh Gac fruit aril

The colour characteristics of powder samples produced from the fresh fruit aril at various drying temperatures by means of air and vacuum drying are shown in Figures 4.4a, 4.4b, 4.4c and 4.4d. The statistical results indicated that pre-treatments (soaking in 1% w/v ascorbic acid solution, 1% w/v bisulfite solution, and blanching) prior to drying significantly affected the lightness of samples (P<0.05), however, no significant effect of drying method and drying temperature on the lightness of samples was observed (P>0.05). Moreover, the interaction between pre-treatments and drying temperatures and between pre-treatments and drying methods on the lightness were statistically established (P<0.001 and P<0.05, respectively).

In general, pre-treatments (soaking in 1% w/v ascorbic acid or 1% w/v bisulfite solution) prior to drying did not impact the lightness of the powders in comparison with the control (untreated). This is shown in that the lower lightness value was obtained in soaked samples as compared to the control. There was no significant difference in lightness between soaking in an ascorbic acid and a bisulfite solution (Figure 4.4a). However, the higher lightness value was obtained in blanched samples as compared to others. The highest lightness, indicated by the highest L* value, was recorded in the samples blanched in steam water for 3 minutes prior to drying at a temperature of 40°C. This is consistent with the results of Piga et al. (2004), who reported that, the lightness value of figs blanched before drying was higher than that of untreated fruits. However, Dandamrongrak et al. (2003) found that the L* value of samples of bananas undergoing all pre-treatments, such as blanching, prior to drying was significantly lower in comparison with untreated bananas. A similar result was also found by Perez and Schmalko (2009), who observed that a higher lightness of untreated pumpkin samples was obtained. Therefore, it can be concluded that the lightness of different materials is differently affected by the various pre-treatments which are applied before the drying process.

The chroma of the Gac fruit powders was significantly affected by different drying methods, pre-treatments and drying temperatures (P<0.001). The chroma of the sample blanched prior to air drying at 40°C was the highest, whilst the lowest value for chroma was observed in the sample soaked in bisulfite solution before air drying at 80°C (Figure 4.4b).
The results showed the ratio of $a^*/b^*$ of the samples was significantly influenced by the factors of pre-treatments before drying and drying temperatures ($P<0.001$). Moreover, the interaction among the three factors impacted the $a^*/b^*$ value of samples statistically ($P<0.001$). Generally, the samples soaked in a bisulfite solution before air drying at a temperature of $80^\circ C$ showed the highest $a^*/b^*$ value in comparison with the control, and with samples treated with ascorbic acid solution and samples undergoing blanching prior to drying (Figure 4.4c). However, samples soaked in bisulfite before vacuum drying at $70^\circ C$ had the lowest level of redness. A study measuring the colour of carrots by pre-soaking in bisulfite solution was carried out by Zhao and Chang (1995). Their results indicated that the redness of samples soaked prior to air drying was higher than that of untreated samples. Moreover, the effect of the addition of ascorbic acid on the colour of paprika pepper was studied by Carvajal et al. (1997). Their results indicated that the redness of ascorbic acid treated samples was higher than others.

As indicated by the hue angle, the tonality of samples was significantly influenced by pre-treatments and drying temperatures ($P<0.001$) and by drying methods ($P<0.05$). Generally, the samples soaked in an ascorbic acid or a bisulfite solution prior to either drying process had a lower hue angle value, indicating a redder colour (Figure 4.4d). This is also in agreement with the results of Latapi and Barrett (2006) who stated that tomatoes pre-treated in a sodium metabisulfite solution resulted in a redder colour after drying. Additionally, there was no significant difference in hue angle between the untreated and the blanched samples, or between soaking in an ascorbic acid solution and a bisulfite solution ($P>0.05$).
Figure 4.4a Lightness of powder from fresh fruit aril affected by AD and VD treatments
Figure 4.4b Chroma of powder from fresh fruit aril affected by AD and VD treatments.
Figure 4.4c $a^*/b^*$ of powder from fresh fruit aril affected by AD and VD treatments
Figure 4.4d Hue angle of powder from fresh fruit aril affected by AD and VD treatments
4.3.3.3 Comparison of colour characteristics of powders from frozen and fresh Gac fruit arils

The effect of air and vacuum drying methods and different kinds of Gac fruit forms (untreated frozen and fresh arils) on the colour of the resultant powders is shown in Figures 4.5a, 4.5b, 4.5c and 4.5d. In general, in this study, the colour characteristics of powder products were found to be significantly affected by different drying methods and drying temperatures.

For the lightness (Figure 4.5a), the drying method and drying temperature significantly impacted on the lightness of powders produced from the frozen fruit, but not on those produced from the fresh fruit. The powders produced from frozen fruit were slightly darker as drying temperatures increased from 50 to 80°C. A similar result found by Park and Kim (2007) who showed that the lightness value of paprika samples was decreased by a high drying temperature of 80°C. However, the lightness of products from the fresh fruit still remained unchanged. Similarly, Krokida et al. (1998) reported that there was no change of lightness in various fruits and vegetables dried by conventional and vacuum drying methods at temperatures of 50, 70 and 90°C. On the contrary, Perez and Schmalko (2009) found that drying temperature significantly affected the lightness of the pumpkin samples in their study. When the air drying temperature increased from 60 to 70°C a higher value of L* (indicating less darkness) was observed.
Figure 4.5a Comparison of lightness of AD and VD powders from frozen and fresh Gac arils
Figure 4.5b Comparison of chroma of AD and VD powders from frozen and fresh Gac arils
Figure 4.5c Comparison of a*/b* of AD and VD powders from frozen and fresh Gac arils.
Figure 4.5d Comparison of hue angle of AD and VD powders from frozen and fresh Gac arils
In this study, the drying method did not influence the ratio of $a^*/b^*$ (redness) of powders produced from the frozen and fresh fruit arils (Figure 4.5c). However, Krokida et al. (1998) reported that the redness of air-dried fruits and vegetables examined was higher than for vacuum-dried samples. This is due to the different effect of drying methods on different substances. However, in this study the redness ratio was significantly affected by drying temperature. The results showed that increasing drying temperature from 50 to 80°C resulted in higher or unchanged redness of the samples. The $a^*/b^*$ ratio of samples remained unchanged for those produced by air drying from both frozen and fresh fruit arils, and for vacuum-dried fresh aril samples.

However the $a^*/b^*$ ratio slowly increased with increase temperature for the vacuum-dried frozen samples. This is inconsistent with results reported by Shi et al. (1999) who studied the effects of different drying processes on the colour of tomatoes. The results of their study indicated that the ratio of $a^*/b^*$ was lower in the products conventionally air-dried at a temperature of 95°C for 6 to 10 hours in comparison with the ratio in products vacuum-dried at 55°C for 4 to 8 hours. This inconsistency could be explained by the fact that the gap between drying temperatures of 95°C and 55°C was quite large. In addition, the colour quality could have been destroyed by drying at a temperature as high as 95°C.

In terms of the colour parameters of chroma (Figure 4.5b) and hue angle (Figure 4.5d), a similar effect of the different drying methods and drying temperatures was observed. The chroma and hue angle of powders from both frozen and fresh samples were significantly affected by drying temperature. However, the drying method significantly influenced the chroma and hue angle of samples from fresh fruit, but not the samples from the frozen fruit. With respect to the drying method, both parameters of the air-dried powder products were significantly higher than those of the vacuum-dried ones. In addition, both colour values of products produced from fresh fruits were higher than those of the frozen ones. The trend of chroma of samples from frozen and fresh fruits declined as the drying temperature was increased from 50 to 80°C. This means that a high chroma value of samples could be obtained at lower drying temperature for both drying methods. A similar observation was made when drying the chestnut flours at temperatures of 40 to 70°C (Correia et al., 2009).

For air-dried materials, the trend of hue angle was unchanged; meanwhile, the trend very slightly increased for vacuum-dried fresh fruit materials and slightly diminished for vacuum-dried frozen ones from the temperatures of 50 to 80°C. Alibas (2006) reported
that the colour parameters of chard leaves were significantly influenced by different drying temperatures and methods. However, Beaudry et al. (2004) found that the colour parameters, of chroma and hue angle, of cranberries, that contain anthocyanins as main pigments and others such as carotenoids, were not affected by drying temperature and drying methods in their experiments.

It can be concluded that there are different resultant effects of drying conditions on the colour quality of different products. In this study, therefore, both frozen and fresh fruit arils can be considered for use for producing Gac powder because the colour characteristics are still preserved.

4.3.4 Effect of air and vacuum drying treatments on the total carotenoid content of the powder produced from frozen and fresh Gac fruit arils

The total carotenoid content (TCC) of powders of frozen fruit aril produced by air drying and by vacuum drying versus the drying temperatures is plotted in Figure 4.6.

![Figure 4.6 Effects of air and vacuum drying treatments on the total carotenoid content of powders from frozen Gac fruit arils](image-url)
The results show that the total carotenoid content of powders obtained from **frozen Gac fruit aril** was significantly influenced by different drying temperatures ($P<0.001$) and by drying methods ($P<0.01$). The highest carotenoid content (4.77 mg/g of powder) was for powder vacuum-dried at a temperature of 50°C whilst the powder that was air-dried at a temperature of 80°C had the lowest carotenoid content (2.11 mg/g of powder). There was no significant difference between samples drying at temperatures of 70°C and 80°C in terms of total carotenoid content ($P>0.05$). Moreover, there was no interaction between drying temperature and drying method ($P>0.05$).

For the **fresh fruit aril**, the total carotenoid content of the powder products versus the different drying treatments in combination with the pre-treatments is presented in Figure 4.7. Control samples are represented by CAD and CVD for air-dried and vacuum-dried, respectively; AAD, BiAD, AVD and BiVD represent samples pre-soaked in ascorbic acid and bisulfite solutions prior to air drying and vacuum drying, respectively; and, BIAD and BIVD represent the samples that were blanched prior to air drying and vacuum drying, respectively.

![Figure 4.7 Effects of air and vacuum drying treatments on total carotenoid content of powders produced from fresh Gac fruit arils](image)
In general, the total carotenoid content of samples was statistically impacted by different pre-treatments, drying temperatures and drying methods (P<0.001). The highest TCC, for the sample soaked in ascorbic acid prior to vacuum drying at 40°C, was significantly observed (7.28 mg/g of powder). The sample which was blanched before air-drying at 80°C recorded the lowest TCC (2.22 mg/g of powder).

The higher quality of food in terms of TCC was generally obtained by the vacuum drying method in comparison to air drying. In this study, under the range of drying temperatures and with the different pre-treatments, in a comparison of the total carotenoids of samples, it is evident that vacuum drying was more effective in the retention of total carotenoids than air drying. Additionally, increasing drying temperature resulted in a greater loss of carotenoid content. Therefore, it can be concluded that the main reason for carotenoid degradation is due to heat plus oxygen. Shi et al. (1999) stated that tomato tissue was broken down by heat treatment in conventional air drying and was easily exposed to oxygen, which caused the loss of lycopene.

In a comparison between frozen and fresh Gac fruit arils, the total carotenoid content of powders obtained from the fresh fruit aril was significantly higher than that of powders from the frozen fruit aril. The explanation is linked to the fact that the fresh Gac fruit was frozen in Vietnam and then shipped to Australia for the retail market. As such, the loss of total carotenoids could be presumed to be due to the freezing process and the following storage period; however, the storage time and conditions for Gac fruit in this case are unknown. In fact, studies have indicated that the loss of carotenoids in fruits and vegetables was found to be from 5% to 48% as a result of freezing (Rickman et al., 2007).

In a variety of studies, different pre-treatments before drying have been used to preserve the concentration of carotenoids in fruits and vegetables. In this study, three pre-treatments prior to the air and vacuum drying processes, namely soaking in an ascorbic acid solution, soaking in a bisulfite solution, and blanching, were used to maximise the retention of carotenoids. Results indicated that the highest content of total carotenoids was for the samples soaked in the 1% w/v ascorbic solution, followed by the samples soaked in the 1% w/v bisulfite solution, the untreated samples, and finally the blanched samples.
Contradictorily, the loss of total carotenoids in blanched spice paprika pericarp was significantly minimised during drying (Ramesh et al., 2001). Similarly, Koca et al. (2007) found that the β-carotene content in dehydrated carrots was enhanced by blanching before drying at 60°C. Furthermore, many studies have shown that the blanching process can be used to effectively maintain nutrient components in general, and carotenoid contents in particular. For instance, a study on lycopene retention of carrots, when blanched and then dried at temperatures of 50 to 90°C for 15 minutes and in oxygen-free condition, was performed; the results showed that the lycopene level was unchanged during the blanching process (Mayer-Miebach & Spieβ, 2003). Furthermore, Ramesh et al. (1999) reported that the total carotenoid content in blanched carrots and paprika increased by 12 and 22% respectively. These increases may be caused by the removal of soluble solids from the tissue matrix during blanching. However, Vedrina-Dragojevic et al. (1997) found that the significant loss of carotenoids in fruits, such as apricots, apples and plums, blanched prior to the drying process was higher than that in samples without blanching.

From these results, it is evident that pre-treatments such as soaking in ascorbic acid and bisulfite solution are effective in retention of total carotenoids. This is due to the beneficial role of antioxidant additives in preserving carotenoids in dehydrated plant foods. Vibhakara et al. (2006) reported that oxygen is removed by radical SO₂; hence, carotene deterioration caused by molecular oxygen is prevented by the presence of SO₂. This is in agreement with the result by Tai and Chen (2000). They determined that the carotenoid content of daylily flowers, when soaked in a 1% sodium sulfite solution (NaHSO₃) for 4 hours before hot air drying, was higher than in those that were not soaked. Similarly, Mohamed and Hussein (1994) found that the carotenoid content of dehydrated carrots was effectively preserved when pre-treated with 0.1% sodium metabisulfite. Yet another similar result was found when mangoes were soaked in a sodium hydrogen sulfite solution (1%) or an ascorbic acid solution (1%) before hot air drying (Chen et al., 2007).

4.3.5 Effect of air and vacuum drying treatments on the antioxidant activity of the powder from frozen and fresh Gac fruit arils

The total antioxidant activity (TAA) of samples of Gac fruit aril, affected by different pre-treatments (in the case of fresh fruit aril), drying temperatures and drying methods is illustrated in Figures 4.8a, 4.8b, 4.9a and 4.9b. A significant effect of drying temperatures on the total antioxidant activity (determined by ABTS and DPPH assays)
of samples from **frozen Gac fruit arils** was statistically established (P<0.001). However, in the ABTS assay, the TAA of powders was not affected by the drying methods (AD and VD). On the contrary, in the DPPH assay, the TAA of samples was significantly influenced by the drying methods (P<0.05). The statistical results indicated that an interactive effect of drying temperature and drying method was not significantly observed in either the ABTS or DPPH assays. Furthermore, in both assays, there was no significant difference of total antioxidant activity between samples drying at 50°C and 60°C.

In the ABTS assay (Figure 4.8a), the highest TAA was observed in the powder air-dried at 50°C whereas in the DPPH assay (Figure 4.8b), the highest TAA was in the powder vacuum-dried at 50°C (0.32 and 0.28 mmole TE/g of powder, respectively). The lowest TAA in both ABTS and DPPH assays was observed for powder air-dried at 80°C.

![Figure 4.8a](image-url)  
*Figure 4.8a Effect of air and vacuum drying treatments on total antioxidant activity (ABTS assay) of powders from frozen Gac fruit arils*
For the powder samples produced from fresh Gac fruit aril the results showed that the effects of different pre-treatments, drying temperatures and drying methods on TAA (both in ABTS and DPPH assays) were significantly observed (P<0.001). In the ABTS assay (Figure 4.9a), no significant difference of TAA was statistically observed in samples soaked in ascorbic acid and bisulfite solutions, or between blanched and control samples (P>0.05). A similar result, that is that there was also no significant difference between pre-treatments with ascorbic acid and bisulfite solutions, was also found in DPPH assay (Figure 4.9b); however, the TAA of untreated (control) samples was statistically higher than that of samples blanched before drying. Result shows that the highest TAA (in both ABTS and DPPH assays) was recorded for samples soaked in ascorbic acid or bisulfite solution before vacuum-drying at 40°C (that is, 0.37 and 0.40 mmole TE/g of powder, respectively), while the lowest was found for vacuum-dried and air-dried untreated samples at a drying temperature of 80°C (that is, 0.15 mmole TE/g of powder). Furthermore, the loss of TAA generally increased when increasing drying temperature from 40 to 80°C.
Figure 4.9a Effect of air and vacuum drying treatments on total antioxidant activity (ABTS assay) of powders from fresh Gac fruit arils.

Figure 4.9b Effect of air and vacuum drying treatments on total antioxidant activity (DPPH assay) of powders from fresh Gac fruit arils.
In this study, it is evident that pre-treatments, drying temperatures and drying methods significantly affected the TAA of the Gac fruit aril powder products. These results indicate that the TAA of samples blanched prior to drying was lower than that of the other treatments, being ascorbic acid and bisulfite solutions. This is also consistent with the report of Yen et al. (2008) who stated that the highest antioxidant level was observed in dried carrot samples treated with an ascorbic acid and sucrose solution.

It can be clearly seen that increasing drying temperature resulted in a significant loss of TAA (in both ABTS and DPPH assays) in powder samples. The explanation for this is that the thermal treatment destroyed antioxidant components, which led to a reduction of antioxidant activity. This is in agreement with the research of Katsube et al. (2009) who studied effects of various air drying temperatures on the antioxidant capacity of mulberry leaves. Their results indicated that an air drying temperature of 60°C or below could preserve antioxidant activity in the leaves whereas the antioxidant activity significantly reduced at temperatures of 70°C or higher.

A similar result was also reported by Mrkic et al. (2006) in relation to broccoli. Likewise, Miranda et al. (2009) showed that higher antioxidant activity of Aloe vera gel was observed at lower temperatures; however, there was no statistical difference among samples air-dried at different temperatures. In contrast, several studies have found that a low drying temperature decreases antioxidant activity in comparison with a high temperature (Kerkhofs et al., 2005; Toor & Savage, 2006; Obied et al., 2008; Madrau et al., 2009). These inconsistencies are likely to be due to the different raw plant materials and the varied drying process conditions examined in the various studies.

In comparison with frozen Gac fruits, the powders produced from the untreated fresh ones were lower in terms of TAA using the ABTS assay; however, there was no difference when using the DPPH assay. The explanation for this result is that the different assays show different effects. The principal mechanism of the ABTS assay is that the absorbance of the radical cation ABTS⁺⁺ is inhibited by antioxidants, while the DPPH assay is based on the measurement of the scavenging ability of antioxidants towards the stable radical DPPH⁺⁺. Thus, the effect of the different radicals will produce different results based on Trolox equivalent used as a standard.

Moreover, many studies have shown that the TAA of frozen fruits and vegetables is higher than that of the fresh commodities. Danesi and Bordoni (2008) proved that it is
not completely true that antioxidant activity of frozen vegetables (such as carrots and tomatoes) is lower than the fresh ones as many consumers expected; however, it depends on the commodities considered. Likewise, Ninfali and Bacchiocca (2003) stated that two of six frozen vegetables (beet green, spinach, broccoli, carrot, onion, and celery) tested are significantly high in antioxidant activity compared to fresh ones, whereas other vegetables showed lower activity than the fresh samples.

In contrast, the freezing process produced a decrease of the radical scavenging activity in four raspberries cultivars, but after long term storage (about 1 year) the TAA of the four raspberry samples still remained unchanged compared with the first day of storage (Ancos et al., 2000). Furthermore, some reports also indicate that TAA of fruit and vegetable samples is not influenced by the freezing process. For example, Mullen et al. (2002) showed that the TAA of frozen red raspberries was similar to the fresh commercial ones. Similarly, Hunter and Fletcher (2002) stated that total antioxidant activity of frozen vegetables is similar to that of fresh ones purchased from supermarkets. An additional explanation, the TAA of fresh plant foods is lost during the storage period after harvest (Hunter & Fletcher, 2002), could be considered.

4.3.6 Comparison of Gac aril powders produced by freeze drying with AD, VD and commercial Gac powders

To further investigate the effects of drying processes on the colour characteristics, total carotenoid content and total antioxidant activity of Gac fruit aril powders, experiments were carried using i) powders produced from frozen Gac aril by freeze drying, ii) powders produced from fresh Gac aril by freeze, and iii) commercial Gac aril powders purchased from supermarkets in Australia and America.

The conditions for the freeze drying processes for producing powders from both frozen and fresh aril are described in section 4.2.2, and the results for the parameters of colour characteristics, TCC and TAA of the FD and commercial samples are presented in Tables 4.4 and 4.5. This section discusses these results, and compares them with the results for the aril powders produced by air and vacuum drying (in sections 4.3.3 to 4.3.5).

In a comparison of the three drying methods, results show that the highest lightness and chroma of FD powders produced from aril of fresh fruits were significantly observed, followed by those from untreated vacuum-dried and air-dried samples. A
similar pattern was also found by Tran et al. (2008) who reported that freeze drying under high vacuum and at a low temperature produced Gac powder with higher lightness and chroma. Likewise, according to Hsu et al. (2003), freeze-dried yam flours showed the highest L* values indicating higher lightness. However, in this study the lightness of fresh Gac aril samples blanched prior to air drying or vacuum drying at a temperature of 40°C was also comparable with results from the freeze drying samples produced from fresh and frozen fruits arils, at 49.57±3.28, 47.44±4.07, 50.13±1.90 and 47.64±1.68, respectively (refer to Figure 4.4a and Table 4.4).

<table>
<thead>
<tr>
<th>Powder samples</th>
<th>Colour characteristics</th>
<th>Lightness</th>
<th>Chroma</th>
<th>a*/b*</th>
<th>Hue angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder from frozen aril</td>
<td>Lightness</td>
<td>47.64±1.68</td>
<td>33.01±1.38</td>
<td>1.65±0.11</td>
<td>31.28±1.66</td>
</tr>
<tr>
<td>FD powder from fresh aril</td>
<td>Lightness</td>
<td>50.13±1.90</td>
<td>39.47±1.57</td>
<td>1.22±0.07</td>
<td>33.93±1.12</td>
</tr>
<tr>
<td>Powder from Australia</td>
<td>Lightness</td>
<td>44.38±3.56</td>
<td>28.22±1.68</td>
<td>0.74±0.02</td>
<td>53.42±0.87</td>
</tr>
<tr>
<td>Powder from America</td>
<td>Lightness</td>
<td>43.48±2.17</td>
<td>27.72±0.78</td>
<td>0.81±0.04</td>
<td>51.03±1.28</td>
</tr>
</tbody>
</table>

In respect of the a*/b* value, vacuum-dried powders from frozen fruit arils showed the highest value, followed by air-dried and freeze-dried products from untreated fresh fruits, then commercial powders from America and Australia. Additionally, the highest hue angle was observed in commercial powders from Australia and America, whereas the hue angle of samples from vacuum drying was the lowest, indicating more redness. Comparing the preservation methods, the powders obtained from the frozen fruit arils showed higher redness values than did the powders from fresh fruit arils.

It is clear that the freeze drying process can substantially preserve the nutritional value of samples, in terms of TCC and TAA. From the results of the various tests in the study, the quality of freeze-dried powder products produced from fresh fruit aril is seen to be highest, followed by FD powder from frozen fruit, vacuum-dried powder, air-dried powder, and then commercial powders from America and Australia. Similar results were also found in the study of Gac powder by Tran et al. (2008), of carrot slices by Regier et al. (2005), and of paprika powder by Park and Kim (2007). However, Regier et al. (2005) determined that the freeze drying process did not show any advantage to convective air drying at below 70°C in terms of carotenoid retention. For instance, β-carotene and lycopene contents remained almost constant after the convection air drying.
Furthermore, Sharma and Le Maguer (1996) revealed that in the event that freeze drying and oven drying (at 25-75°C) were applied for tomato pulp solids, a loss in lycopene content was not significantly caused by the increase in temperature. However, freeze drying is generally seen as a very expensive preservation method; for example, freeze drying costs are 4 to 8 times higher than those of air drying (Ratti, 2001). In a contrary finding, Chang et al. (2006) stated that the amounts of lycopene in two tomato varieties after freeze drying were reduced to 33%-48% of the levels of the fresh ones. The specific reason for this is still unclear. Interestingly, however, the lycopene contents after air drying increased by 152%-197% compared to the fresh ones. This is due to the fact that the cell walls and the bonding force between lycopene and the tissue matrix are broken down by the heating process. In the current study, in addition, the TCC of samples pre-soaked in ascorbic solution or bisulfite prior to vacuum drying at a low temperature of 40°C was also highly comparable with the results for TCC of FD samples; 7.28, 6.99 and 7.24 mg/g of powder, respectively (refer to Figure 4.7 and Table 4.5).

**Table 4.5 Total carotenoid content and total antioxidant activity of FD powders and commercial powders**

<table>
<thead>
<tr>
<th>Gac powder</th>
<th>TCC (mg/g of powder)</th>
<th>ABTS (mmole TE/g powder)</th>
<th>DPPH (mmole TE/g powder)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder from frozen aril</td>
<td>6.27±0.45</td>
<td>0.33±0.022</td>
<td>0.33±0.029</td>
</tr>
<tr>
<td>FD powder from fresh aril</td>
<td>7.24±0.16</td>
<td>0.39±0.009</td>
<td>0.38±0.005</td>
</tr>
<tr>
<td>Powder from Australia</td>
<td>0.53±0.09</td>
<td>0.03±0.003</td>
<td>0.05±0.003</td>
</tr>
<tr>
<td>Powder from America</td>
<td>0.71±0.03</td>
<td>0.07±0.001</td>
<td>0.08±0.002</td>
</tr>
</tbody>
</table>

Comparing the effect of the drying methods, the total carotenoid content of FD powders from fresh fruit arils was higher than from frozen ones, 6.27±0.45 and 7.24±0.16 mg/g of powder, respectively. This may be due to the fact that a loss of carotenoid content during the freezing process and during storage could be occurring. In this study, the TCC of commercial Gac powder products from Australia and America was very low compared to the experimental samples, about 4 to 13 times less for powders in Australia markets and 3 to 10 less times for powders in America markets. It is also quiet likely that a blend of Gac aril with yellow flesh and seed was involving. Moreover, the quality of packaging for the powders and the storage conditions also contribute for this situation. It was observed that commercial powders are packed in clear bags or containers of 25 to 100g, therefore, carotenoid content could be destroyed by light,
oxidation and isomerisation. Furthermore, the drying methods and the storage times for commercial powders are also unknown.

A trend of the TAA of the powders being compared was also similar to the TCC, in that the highest TAA (0.39 mmole TE/g of powder) of FD samples was significantly observed in the ABTS assay compared to the TAA of other samples. However, in the DPPH assay, the highest TAA (0.40 mmole TE/g of powder) was found for the sample soaked in bisulfite solution before vacuum drying at 40°C.

Studies have shown that not only the various drying methods but also different antioxidant assays affect the results for the total antioxidant activity of dried food samples. For example, with a reducing power assay, two varieties of freeze-dried tomatoes exhibited much better reducing powers than the hot-air-dried tomatoes. In contrast, the hot-air-dried tomatoes exhibited the highest ferrous ion chelating ability and DPPH radical scavenging activity in comparison with freeze-dried and fresh ones (Chang et al., 2006). Similarly, Hsu et al. (2003) demonstrated that different drying methods (freeze drying, hot air drying and drum drying) affected antioxidant activity; among them, the antioxidant activity of freeze-dried yam flours was the highest in the free radical scavenging effect, ferrous ion chelating capacity, reducing power, and total antioxidant activity assays.

Furthermore, the antioxidant activity of Korean red pepper was significantly influenced by a modified drying method (at 70°C for 6 hours in cut pods) and conventional drying method (at 80°C for 5 hours and then 60°C for 18 hours in whole pods). The results indicated that the antioxidant activity of the modified-process dried pepper was approximately three times higher than the conventionally dried one in the ABTS and DPPH assays (Kim et al., 2006). In contrast, other results have shown that the total antioxidant activity of three tomato cultivars was remarkably lower than that of the fresh ones as lower drying temperatures were applied, such as semi drying at 42°C for 18 hours or forced air drying at 42°C for 48 hours. Thus, the low drying temperature decreased the antioxidant content, but the color and nutrient contents were still maintained (Kerkhofs et al., 2005; Toor & Savage, 2006).

From these studies and their results mentioned above, it can be seen that different fruits and drying treatments will result in different outcomes. It justifies separate investigation into finding specific suitable drying treatment for particular fruit.
4.3.7 Air-drying process for aril of fresh Gac fruit mixed with various ratios of maltodextrin

As it is very difficult to grind dried Gac fruit after the drying process due to its sticky consistency, the addition of maltodextrin prior to air drying was used as a means to overcome this problem. Furthermore, this is an alternative pre-treatment to improve the retention of colour, carotenoid content and antioxidant activity of Gac powders. The pastes, comprised of the red aril pulp mixed with the various ratios of maltodextrin to total Gac fruit solids, were air-dried at a temperature of 60°C for 18 hours and then powdered using a MF 10 basic Microfine grinder. The physicochemical and total antioxidant properties of the resultant powders are shown in Table 4.6.

As can be seen in Table 4.6, statistical analyses showed that moisture content, lightness, chroma, pH, bulk density, water activity, TCC and TAA of Gac powders were not significantly affected by different ratios of maltodextrin and total Gac fruit solids (P>0.05). However, a significant effect of the ratios on the a*/b* value and hue angle of samples was statistically established (P<0.01).

<table>
<thead>
<tr>
<th>Analysed item</th>
<th>g maltodextrin/g total Gac fruit solids</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td>MCw.b (%, powder)</td>
<td></td>
</tr>
<tr>
<td>Colour Lightness (L*)</td>
<td></td>
</tr>
<tr>
<td>Chroma</td>
<td></td>
</tr>
<tr>
<td>a*/b*</td>
<td></td>
</tr>
<tr>
<td>Hue angle</td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td></td>
</tr>
<tr>
<td>Bulk density (g/ml)</td>
<td></td>
</tr>
<tr>
<td>A_w</td>
<td></td>
</tr>
<tr>
<td>TCC (mg/g of powder)</td>
<td></td>
</tr>
<tr>
<td>TAA (DPPH assay) (mmole TE/g of powder)</td>
<td></td>
</tr>
</tbody>
</table>

The values in the same row followed by different superscripts (a-c) were significantly different (P<0.05)

It is evident that using maltodextrin improved the quality of Gac fruit powder. For colour characteristics, the addition of maltodextrin prior to air drying at 60°C resulted in superior products in comparison to FD powders in terms of the a*/b* value and hue...
angle (refer to Table 4.4). The added-maltodextrin samples showed a higher $a^*/b^*$ value and lower hue angle, indicating a redder colour. Moreover, the lightness and chroma of the added-maltodextrin samples was also comparative with FD samples. In regard to the TCC and TAA, using various ratios of maltodextrin and Gac fruit solids could improve the loss of carotenoid content and antioxidant activity of the resultant powder products. From the results in this study, the highest TCC and TAA of an added-maltodextrin sample were observed at a ratio of 1, followed by those at ratios of 0.5 and 1.5. Additionally, it can be seen that the TCC and TAA of added-maltodextrin samples was higher than that of samples vacuum or air-dried at the same temperature of 60°C without maltodextrin (refer to Figure 4.7 and 4.9b).

Furthermore, moisture content is another parameter influencing the quality of the final product that should be considered. At the same drying time of 18 hours, the higher ratio of maltodextrin and Gac fruit solids resulted in a lower moisture content, and this was lower than that of the fresh fruit aril sample at the same air or vacuum drying temperature at about 6% MC$_{w.b.}$. In this study, it was found that dried samples having the addition of maltodextrin as a pre-treatment were easily powdered by a Microfine grinder. This could be explained by the fact that an increase in sticky point temperature, a decrease in hygroscopicity and the degree of caking are influenced by the addition of maltodextrin (Jaya et al., 2006). In conclusion, the addition of maltodextrin can be used as a pre-treatment prior to the drying of Gac fruit aril for the improvement of final quality of powders in terms of colour, carotenoid content and antioxidant activity. However, further experiments should be carried out for optimising the quality of the final powder product, for example using a lower ratio of maltodextrin as the pre-treatment.

In Table 4.6, results show that the bulk density of samples decreased from 0.63 to 0.57 as the level of maltodextrin and total fruit solids increased from 0.5 to 1; however, it was unchanged from the maltodextrin level of 1 to 1.5. Furthermore, it has been established that pH and water activity of dried food samples play an important role for preserving quality of product. Reducing water activity is one of the most important purposes of any drying process in order to inhibit the growth of microorganisms and other biochemical reactions. Tang and Yang (2004) stated that the water activity of dried products should be lower than 0.6 so that the deterioration caused by microorganisms and spoilage reactions will be prevented. The water activity of added-maltodextrin powders in the present study was 0.61 or below. Additionally, the pH values of the powder products after reconstituting ranged from 4.79 to 5.05, indicating
the high colour stability of samples. According to Gordon et al. (1982), the colour of carotenoids such as β-carotene, apo carotenal and canthaxanthin are relatively stable over a pH range of 2 to 7.

Table 4.7 shows the colour characteristics of freeze-dried and added-maltodextrin air-dried samples compared to fresh Gac fruits. For a comparison of colour characteristics the FD powders and added-maltodextrin samples were reconstituted to the sample moisture content of the fresh fruits.

<table>
<thead>
<tr>
<th>Sample</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>Chroma</th>
<th>a*/b*</th>
<th>H0</th>
<th>∆E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh fruit</td>
<td>38.79</td>
<td>25.51</td>
<td>14.34</td>
<td>29.26</td>
<td>1.78</td>
<td>29.34</td>
<td></td>
</tr>
<tr>
<td>FD</td>
<td>38.33</td>
<td>30.28</td>
<td>18.17</td>
<td>35.31</td>
<td>1.67</td>
<td>30.97</td>
<td>6.31</td>
</tr>
<tr>
<td>0.5</td>
<td>41.82</td>
<td>27.68</td>
<td>18.67</td>
<td>33.39</td>
<td>1.48</td>
<td>34.00</td>
<td>5.71</td>
</tr>
<tr>
<td>1</td>
<td>41.59</td>
<td>30.54</td>
<td>19.47</td>
<td>36.22</td>
<td>1.57</td>
<td>32.52</td>
<td>7.71</td>
</tr>
<tr>
<td>1.5</td>
<td>46.92</td>
<td>29.57</td>
<td>19.57</td>
<td>35.46</td>
<td>1.51</td>
<td>33.50</td>
<td>10.48</td>
</tr>
</tbody>
</table>

0.5, 1, 1.5: ratios of maltodextrin and total fruit solids (g/g)

The total colour difference (∆E) of powder with the ratio of maltodextrin to fruit solids of 0.5 is lowest, whereas the highest was observed in sample with the maltodextrin to fruit solids ratio of 1.5. This again confirms that the colour of dried Gac aril samples can be improved by the addition of maltodextrin prior to air drying. However, after reconstituting, the highest a*/b* value and the lowest hue angle, an indication of more redness, were obtained from the FD powder samples; this results occurred as the Gac fruit content of these samples had not been decreased by the presence of maltodextrin.

4.3.8 Air-drying of Gac fruit skin and yellow pulp

As shown previously in Table 4.3, the percentages of the skin and yellow pulp in Gac fruit are significantly high, at 18 and 49 respectively. Therefore, to utilise these by-products, the process of air drying at a temperature of 60°C was carried to produce powder samples. The untreated skin and yellow pulp of fresh Gac fruit were dried at 60°C for 12 and 17 hours respectively. The physicochemical and antioxidant properties of the skin and yellow pulp are shown in Table 4.8.
It can be seen that bulk density of skin powder was lower than that of yellow pulp powder, at 0.48 g/ml and 0.61 g/ml respectively. The values of pH and water activity of skin and yellow meat powders were low, indicating relatively high stability.

Table 4.8 Physicochemical and antioxidant properties of air-dried skin and yellow pulp powders

<table>
<thead>
<tr>
<th>Analysed item</th>
<th>Skin</th>
<th>Yellow pulp</th>
</tr>
</thead>
<tbody>
<tr>
<td>MCwb (% powder)</td>
<td>4.64 4.64</td>
<td>6.01 6.01</td>
</tr>
<tr>
<td>Colour Lightness (L*)</td>
<td>77.30 77.30</td>
<td>66.37 66.37</td>
</tr>
<tr>
<td>Chroma</td>
<td>51.20 51.20</td>
<td>28.04 28.04</td>
</tr>
<tr>
<td>b*/a* (yellowness)</td>
<td>9.31 9.31</td>
<td>2.75 2.75</td>
</tr>
<tr>
<td>Hue angle</td>
<td>83.72 83.72</td>
<td>69.68 69.68</td>
</tr>
<tr>
<td>pH</td>
<td>6.16 6.16</td>
<td>5.53 5.53</td>
</tr>
<tr>
<td>Bulk density (g/ml)</td>
<td>0.48 0.48</td>
<td>0.61 0.61</td>
</tr>
<tr>
<td>A_w</td>
<td>0.52 0.52</td>
<td>0.45 0.45</td>
</tr>
<tr>
<td>TCC (mg/g of powder)</td>
<td>0.90 0.90</td>
<td>0.42 0.42</td>
</tr>
<tr>
<td>TAA# (mmole TE/g of powder)</td>
<td>0.06 0.06</td>
<td>0.04 0.04</td>
</tr>
</tbody>
</table>

# using DPPH assay

Results showed that the quality of skin powder was better than that of yellow pulp powder in terms of colour, TCC and TAA. For the colour characteristics, the values of lightness, b*/a*, hue angle and chroma of skin powder was higher than that of yellow pulp powder, indicating more yellowness and a lighter colour. The hue angle of the skin powder (83.72) is closer to 90°, indicating a more yellow colour of this powder than yellow pulp powder (69.68). Moreover, the skin powder was also high in TCC and TAA in comparison with the yellow pulp powder. Interestingly, however, TCC of both of these powders is high compared to other fruits and vegetables. For example, Muratore et al. (2008) indicated that the total carotenoid content (including β-carotene and lycopene) of cherry tomatoes dried at a temperature of 60°C is about 0.36 mg/g. Furthermore, total carotenoid content of hot-air-dried pumpkin and carrot at 60°C were 0.14 and 1.1 mg/g respectively. In addition, according to Kerkhofs et al. (2005), total lycopene of several tomato cultivars air drying at 42°C for 48 hours ranged from 0.46 to 0.59 mg/g.

From the study results mentioned above, it can be concluded that it is very desirable to produce dried skin and yellow pulp powders from the Gac fruit due to their high carotenoid content. Furthermore, this utilisation also prevents environmental pollution due to waste problem, and enhances the overall value of Gac fruit.
4.4 Conclusions

In summary, the colour characteristics, carotenoid contents and antioxidant activities of the powder products produced from Gac fruits were significantly affected by pre-treatments, by drying methods and by drying temperatures. The colour of the Gac fruit powder samples shown in Figures 4.10a and 4.10b illustrates the range of colours of powders that are produced by some of the different drying methods and conditions that were investigated in the experiments described in this chapter.

The freeze drying process resulted in superior powder products in terms of colour parameters, TCC and TAA, followed in quality by vacuum-dried and air-dried products. However, pre-soaking in solutions of ascorbic acid or bisulfite prior to vacuum drying at temperature of 40°C was equally effective in preserving TCC and TAA as freeze drying. For colour characteristics, freezing drying produced powders with the highest lightness and intensity. In contrast, the powders produced by vacuum drying were high in a*/b* value and low in hue angle, indicating more redness than those produced by air drying and freeze drying. Moreover, loss of TCC and TAA was lowest in the powder dried at a temperature of 40°C, with increasing losses observed at increased drying temperatures (50, 60, 70, and 80°C). It is therefore suggested that pretreatment with ascorbic acid or bisulfite before air or vacuum drying at low temperature should be applied for Gac fruit processing.

In the comparison between powders from frozen and fresh arils, higher redness and lower TCC of powder from the frozen arils were observed. Total carotenoid content of powder from fresh Gac arils was significantly high in comparison with the powder from frozen fruits. Interestingly, although the powders produced from the fresh fruits were lower in TAA when tested using the ABTS assay, there was no difference recorded using the DPPH assay. Therefore, it is recommended that both frozen and fresh Gac fruit can be used for production of Gac powder.

Addition of different amounts of maltodextrin to the fresh Gac aril, with air drying at the same temperature of 60°C, could be seen to ease of milling and improve the quality of the powder in terms of the colour characteristics, TCC and TAA. Additionally, air-dried skin and yellow pulp powders produced from Gac fruit showed high levels of TCC compared to some other fruits and vegetables. To make use of all components of the whole Gac fruit not only resolves environmental problems but greatly enhances the overall value of the Gac fruit.
Fresh Gac aril powder
Pre-soaked in bisulfite and
Air-dried at 40°C

Fresh Gac aril powder
Pre-soaked in bisulfite and
Vacuum-dried at 40°C

Fresh Gac aril powder
Freeze-dried at pressure of 0.3 mbar and temperature of -46°C

Figure 4.10a Samples of Gac aril powder produced by air drying and freeze drying under specific conditions
Fresh Gac aril powder
Mixed with Maltodextrin/solids of 0.5 g/g and Air-dried at 60°C

Fresh Gac aril powder
Mixed with Maltodextrin/solids of 1 g/g and Air-dried at 60°C

Gac skin powder
Air-dried at 60°C

Gac yellow pulp powder
Air-dried at 60°C

Figure 4.10b Samples of Gac aril, skin, and yellow pulp powders produced by air drying at 60°C
Chapter 5
EFFECTS OF SPRAY DRYING CONDITIONS ON THE PHYSICOCHEMICAL AND ANTIOXIDANT PROPERTIES OF GAC ARIL POWDER

5.1 Introduction

This chapter focuses on spray drying of Gac aril because of the importance of the technology, being carried out for many food powders and here the Gac aril has to be converted into liquid form prior to processing, unlike the other drying methods in the previous chapter. The effects of the drying processes of air drying, vacuum drying and freeze drying on the physicochemical and antioxidant properties of Gac fruit powder were presented. Because of the high nutritional value of Gac fruit and the demand of consumers for different powder forms, it is necessary to develop alternative drying methods to produce functional and stable powder forms of Gac fruit. The spray drying method has been widely utilised for commercial production of dried fruit and vegetables. Various reports showed that spray drying is applied for study of carotenoid stability in plant foods such as carrots, tomato pulp, sweet potato and sea buckthorn (Wagner & Warthesen, 1995; Goula & Adamopoulos, 2005; Grabowski et al., 2006; Laos et al., 2007).

Earlier attempt by Tran et al. (2008) to spray dry enzyme treated Gac aril was not very successful, so a drying aid is considered for use in this study. Maltodextrin is one of the common drying aids for spray drying owing to its beneficial roles in increasing the stability of carotenoids and other nutritional components. Maltodextrin was used during the spray drying process in studies of β-carotene (Desobry et al., 1997); carrot carotenenes (Wagner & Warthesen, 1995); blackcurrant, apricot and raspberry juices (Bhandari et al., 1993); guava juice (Chopda & Barrett, 2001) and pineapple juice (Abadio et al., 2004). However, no studies on spray drying conditions using maltodextrin as the carrier/encapsulating agent have been reported in regard to producing Gac fruit powder.

In this chapter, the effects on the physicochemical and antioxidant properties of Gac fruit powder of adding varying maltodextrin concentrations and applying varying spray drying temperatures will be investigated.
5.2 Fresh Gac fruit preparation

Fresh Gac fruit were purchased from a local market in Hochiminh City, Vietnam. The fruit was transported in an insulated container and used on the same day. The whole Gac fruit was scooped out and the aril surrounding the seeds was completely separated. The red aril was blended with distilled water, in the ratio of 1 to 5, in a laboratory blender. The resulting juice was twice filtered using a filter screen of 100 µm mesh. Next, different ratios of commercial maltodextrin (12 DE, Glucidex®, Roquette, France) were added into the juice, which was blended and finally filtered before spray drying. The three ratios of maltodextrin 12 DE added to the juice were 10%, 20% and 30% (w/v).

5.3 Spray drying conditions for fresh Gac fruit solutions

The feed mixtures comprising added maltodextrin and red flesh Gac fruit juice were spray-dried in a Lab Plant SD-05 spray dryer. The inlet temperatures and measured outlet temperatures are presented in Table 5.1. The drying air flow rate, compressor air pressure and feed rate were constant, at 56±2 (m³/h), 0.06 MPa gauge and 12-14 mL/min, respectively. After the spraying process, the Gac fruit powder was collected in a glass collection vessel wrapped with aluminium foil, and immediately stored in a desiccator containing silica gel for equilibration to room temperature. The powder, in 5g amounts, was then packed into high barrier vacuum bags, using a vacuum sealer.

The two factors (maltodextrin concentration and inlet drying temperature) were randomly designed to investigate the influence of the drying conditions on the physicochemical and antioxidant properties of Gac powder and reconstituted product. The spray drying processes and the measurement of parameters were all carried out in duplicate. A total of 30 runs was conducted.

Table 5.1 Operating conditions for spray drying of Gac fruit

<table>
<thead>
<tr>
<th>Drying air temperature (°C)</th>
<th>Inlet</th>
<th>Outlet</th>
</tr>
</thead>
<tbody>
<tr>
<td>120</td>
<td>83</td>
<td></td>
</tr>
<tr>
<td>140</td>
<td>94</td>
<td></td>
</tr>
<tr>
<td>160</td>
<td>103</td>
<td></td>
</tr>
<tr>
<td>180</td>
<td>112</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>125</td>
<td></td>
</tr>
</tbody>
</table>
5.4 Results and discussion
5.4.1 Effects of spray drying conditions on the physicochemical properties of Gac powders

The effects of the three ratios of maltodextrin concentration and the different spray drying temperatures on the physicochemical properties of Gac fruit powder are shown in Table 5.2. Statistical analyses showed that the pH, water activity (Aw), and water solubility index (WSI, %) of spray-dried powders were not significantly affected by different maltodextrin concentrations and different drying temperatures (P>0.05). In the raw data the ranges of pH, Aw and WSI (%) of powder samples were 3.94 - 4.64, 0.38 - 0.54 and 35.94 - 39.07, respectively. However, the effects of maltodextrin concentration and drying temperature on moisture content (MC, %) of spray-dried powder products were statistically significant (P<0.001). Statistical interaction between drying temperature and maltodextrin concentration was significantly observed (P<0.01).

Increasing maltodextrin concentration and drying temperature significantly resulted in a decreasing moisture content of the samples. As the maltodextrin concentration increased from 10% to 30% the moisture content of samples reduced from 4.87% to 4.06%. A similar trend was observed while increasing drying temperatures from 120°C to 200°C, which resulted in a drop in moisture content from 5.29% to 3.88%. Furthermore, the bulk density of powder products was statistically influenced by drying temperature (P<0.01), but not by maltodextrin concentration (P>0.05). Generally, the bulk density of samples decreased as drying temperatures increased from 120°C to 200°C.

For spray drying in general, increasing the drying temperature results in a greater loss of the moisture content of the powder produced, due to the higher rate of heat transfer into particles, causing faster water removal. As shown in the results in this study, the moisture content of Gac powders reduced quickly when increasing the air inlet temperature from 120 to 200°C. Studies by Goula et al. (2004), Chegini and Ghobadian (2005), Rodríguez-Hernández et al. (2005), and Ersus and Yurdagel (2007) have reported that the moisture content in tomato powder, orange juice powder, cactus pear juice powder and black carrot powder, respectively, decreased as drying temperature increased.
Moreover, in this study a decrease in the moisture content of Gac powder was also obtained when the maltodextrin concentration increased from 10% to 30%. Similarly to this result, Abadio et al. (2004) found that an increased concentration of maltodextrin 10 DE, from 10% to 15%, reduced the moisture content of resultant pineapple juice powders. Another similar result was also reported by Grabowski et al. (2006) who carried out tests on sweet potato puree powder. These findings could be explained by the fact that additional concentrations of maltodextrin resulted in an increase in feed solids and a reduction in free water for evaporation.

Table 5.2 Physicochemical properties of spray-dried Gac fruit powders

<table>
<thead>
<tr>
<th>Drying conditions</th>
<th>MC (%)</th>
<th>pH</th>
<th>Aw</th>
<th>Bulk density g/mL</th>
<th>WSI (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Maltodextrin concentration (MDC)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10%</td>
<td>4.87±0.71&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.40±0.18</td>
<td>0.50±0.03</td>
<td>0.72±0.05</td>
<td>37.49±1.01</td>
</tr>
<tr>
<td>20%</td>
<td>4.54±0.54&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.40±0.14</td>
<td>0.47±0.05</td>
<td>0.70±0.06</td>
<td>37.29±0.74</td>
</tr>
<tr>
<td>30%</td>
<td>4.06±0.47&lt;sup&gt;c&lt;/sup&gt;</td>
<td>4.32±0.12</td>
<td>0.46±0.05</td>
<td>0.73±0.07</td>
<td>37.46±1.05</td>
</tr>
<tr>
<td>120&lt;sup&gt;°&lt;/sup&gt;C</td>
<td>5.29±0.50&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.12±0.08</td>
<td>0.50±0.04</td>
<td>0.78±0.05&lt;sup&gt;a&lt;/sup&gt;</td>
<td>37.13±0.80</td>
</tr>
<tr>
<td>140&lt;sup&gt;°&lt;/sup&gt;C</td>
<td>4.81±0.49&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.41±0.10</td>
<td>0.49±0.04</td>
<td>0.74±0.05&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>37.62±1.00</td>
</tr>
<tr>
<td>160&lt;sup&gt;°&lt;/sup&gt;C</td>
<td>4.47±0.48&lt;sup&gt;c&lt;/sup&gt;</td>
<td>4.32±0.11</td>
<td>0.45±0.06</td>
<td>0.70±0.03&lt;sup&gt;bc&lt;/sup&gt;</td>
<td>37.39±0.92</td>
</tr>
<tr>
<td>180&lt;sup&gt;°&lt;/sup&gt;C</td>
<td>4.01±0.18&lt;sup&gt;d&lt;/sup&gt;</td>
<td>4.26±0.19</td>
<td>0.47±0.04</td>
<td>0.69±0.05&lt;sup&gt;bc&lt;/sup&gt;</td>
<td>37.34±1.00</td>
</tr>
<tr>
<td>200&lt;sup&gt;°&lt;/sup&gt;C</td>
<td>3.88±0.35&lt;sup&gt;d&lt;/sup&gt;</td>
<td>4.45±0.19</td>
<td>0.47±0.05</td>
<td>0.66±0.04&lt;sup&gt;c&lt;/sup&gt;</td>
<td>37.59±1.09</td>
</tr>
</tbody>
</table>

**Significant interaction**

<table>
<thead>
<tr>
<th>Significant interaction</th>
<th>Significance</th>
</tr>
</thead>
<tbody>
<tr>
<td>MDC</td>
<td>***</td>
</tr>
<tr>
<td>DT</td>
<td>***</td>
</tr>
<tr>
<td>MDC x DT</td>
<td>**</td>
</tr>
</tbody>
</table>

Values are mean ± SD (two replicates) after statistical analyses
N.S., *, ** and *** indicate not significant and significant at P = 0.05, 0.01 and 0.001, respectively
The values in the same column followed by different superscripts (a-e) were significantly different (P<0.05)

In this study, the bulk density of Gac powders was significantly affected by the drying temperature, with decreasing density observed with increased drying temperature. This is consistent with the findings of a number of studies, showing that increasing inlet air drying temperature results in reducing bulk density (Walton & Mumford, 1999; Cai & Corke, 2000; Goula et al., 2004; Chegini & Ghobadian, 2007). The reason for this phenomenon could be explained in that a large droplet surface, a large particle size,
and particles with a greater tendency to be hollow, were rapidly formed at higher temperatures. As a result, this leads to a decrease in bulk density due to particles with a larger diameter (Jumah et al., 2000; Walton, 2000; Chegini & Ghobadian, 2005).

The values of pH, Aw and WSI of the Gac powders in this study were not significantly affected by inlet air drying temperature and maltodextrin concentration. For the pH value, this finding is in agreement with the results of González-Palomares et al. (2009) who found that pH of the Roselle extract powders did not change with different air drying temperatures. Importantly, Aw is one of the key factors that significantly influence the shelf life of powder products. High water activity in products leads to shorter shelf life due to high free water for biochemical degradations. The deterioration of dried powder caused by microorganisms and biochemical reactions can be prevented at Aw lower than 0.6 (Tang & Yang, 2004). In the raw data the average Aw of powders in this study ranged from 0.38 to 0.54, and can thus be considered to be more microbiologically stable. Additionally, from the results shown in Table 5.2, the Aw of powders decreased with increasing maltodextrin concentration. The trend in the results for Aw of the powders was consistent with the findings of the study carried out by Quek et al. (2007). They stated that the water activity of spray-dried water melon powders was not significantly changed by inlet temperatures of between 145°C and 175°C. Furthermore, higher concentration of maltodextrin resulted in a decrease in the Aw of the powders.

The WSI of the samples in the study was not influenced by different drying conditions. A similar observation was also reported by De Sousa et al. (2008) who studied spray-dried tomato powders. In the raw data the range of WSI of Gac fruit samples in this study was 36.91% to 38.25%. These values were higher when compared to results for spray-dried tomato powders, which ranged from 17.65% to 26.73% (De Sousa et al., 2008). However, the Gac fruit WSI values were much lower compared to those of pineapple juice powder, with an average value of 81.56% (Abadio et al., 2004). This limitation for solubility of Gac fruit powders could be explained due to an extremely high content of liposoluble substances, such as carotenoids and tocopherol, a significant level of fatty acids, and a high level of insoluble pulp in the original aril. The level of screening in the filters still allows insoluble material to pass through.

Furthermore, the solubility of dried fruit and vegetable powders can be affected by many parameters, such as initial compositions of the raw material to be spray-dried, the carrier agents, compressed air flow rates, and low feed rates (Bhandari et al., 1993;
Al-Asheh et al., 2003; Goula et al., 2004). For example, a superior water solubility property of spray-dried cashew apple juice powder was obtained by using cashew tree gum as the drying aid agent (De Oliveira et al., 2009). Therefore, additional investigation may need to be carried out to identify methods for improving the water solubility of Gac fruit powders further, if desired.

5.4.2 Effects of spray drying conditions on the colour characteristics of Gac fruit powders

Figure 5.1a and Figure 5.1b show the effects of different maltodextrin concentrations and drying temperatures on the colour characteristics of spray-dried Gac aril powders. In general, the colour characteristics of spray-dried powders were significantly impacted by maltodextrin concentration and drying temperature.

For the lightness, the colour of products was significantly affected by maltodextrin concentration (P<0.01). An increase in the lightness of products was significantly obtained by increasing the maltodextrin concentration from 10% to 20%. However, there was no significant difference in lightness of samples when the concentration increased from 20% to 30%. A consistent result was also observed in terms of the colour characteristics of the a*/b* value and the hue angle. The highest value of a*/b* and the lowest hue angle were obtained in the sample with an added 10% concentration of maltodextrin, both indicating more redness. Moreover, the maltodextrin concentration also impacted on the chroma value, or colour intensity, of samples (P<0.001). A significant decrease in colour intensity was observed when increasing maltodextrin concentrations from 10% to 20% at all temperatures.

Spray drying temperature was another factor affecting the colour characteristics of the Gac powder products, in terms of the chroma, hue angle and a*/b* value, but not in relation to lightness. A significant effect of drying temperature on the a*/b* value and hue angle was statistically observed (P<0.01). Loss of redness of samples, resulting in lower a*/b* values and higher hue angle, increased when increasing temperatures from 120°C to 200°C; however, no statistical difference in the value of a*/b* and hue angle among the temperatures of 120°C, 140°C and 160°C, and no difference between these characteristics at 180°C and 200°C, was significantly observed. In contrast, the lightness of products was not significantly influenced by spray drying temperature (P>0.05).
Furthermore, statistical interaction between maltodextrin concentration and drying temperature was significantly observed in the lightness and chroma values of the powder products. For the a*/b* value and hue angle of products, however, there was no significant interaction between the two factors.
Figure 5.1a Lightness and chroma of Gac fruit powders as a result of different spray drying conditions
Figure 5.1b The $a^*/b^*$ and hue angle of Gac fruit powders as a result of different spray drying conditions.
Generally, an increase in the **lightness** value of the powders was observed with an increased maltodextrin concentration due to the effect of the maltodextrin. Because of white colour of maltodextrin, a greater lightness of powders, represented by a higher L* value, was obtained at higher concentrations of maltodextrin. Similar results were also found in spray-dried sweet potato powders (Grabowski et al., 2006) and in pineapple juice powders (Abadio et al., 2004). On the other hand, the lightness of Gac fruit powders in this study was not significantly influenced by the drying temperature. However, De Sousa et al. (2008) found that the highest value of lightness of spray-dried tomato powders was observed at the highest inlet drying temperature, indicating less darkness due to the pigment oxidation. In contrast, the lightness of water melon powders reduced when inlet drying temperature increased due to the high content of sugar causing browning of powders (Quek et al., 2007).

The **chroma** or colour intensity of Gac fruit powders was significantly affected by both drying conditions of maltodextrin level and the inlet drying temperature. High colour intensity of powders was observed at low maltodextrin concentration and at high temperatures. This could be due to significant interaction between the two factors investigated. This finding is in agreement with the results reported by Quek et al. (2007).

Lower values of a*/b* and higher **hue angles** were observed as a result of increasing maltodextrin concentration and increasing the inlet drying temperature. These results indicate that the loss of redness of powder products increased in these spray drying conditions. A similar result was observed by De Sousa et al. (2008) who reported that a decrease in the value of a*/b* in spray-dried tomato powder was found with increasing the inlet drying temperature. Further studies confirmed that increased drying temperatures resulted in low retention in the redness of carrot products (Chen et al., 1995), and of tomato products (Shi et al., 1999). The possible explanation for this phenomenon is that carrying out the spray drying process with a high ratio of surface area to volume of feed mixture caused rapid pigment oxidation (Desobry et al., 1997). In consequence, the spray drying conditions at high temperature resulted in a high loss of red colour due to thermal degradation of carotenoid pigment. Goula and Adamapoulos (2005) indicated that a higher loss of lycopene content in tomato powder was observed by increasing the air inlet temperature in spray drying.

In terms of effects of the maltodextrin concentration, moreover, the lesser redness of Gac fruit powders was due to the higher concentration of maltodextrin used in the
spray-drying process. Since maltodextrin has a white colour whereas Gac fruit juice is red, when mixing and spray drying, the red colour of powder products was reduced by increased concentrations of maltodextrin. As previously mentioned by Grabowski et al. (2006), increasing maltodextrin resulted in an increase in hue angle in sweet potato powders, indicating a loss of redness.

The total colour difference ($\Delta E$) of reconstituted powders compared to feed mixtures before the spray drying process is shown in Figure 5.2. The $\Delta E$ of reconstituted powders was not impacted by maltodextrin concentration ($P>0.05$); however, a significant effect of drying temperature on $\Delta E$ was statistically observed ($P<0.001$). Increasing drying temperature significantly resulted in an increase in $\Delta E$. Moreover, there was no significant interaction between maltodextrin concentration and drying temperature.

![Figure 5.2 Total colour differences of reconstituted Gac fruit powders after spray drying process](image-url)
The trend for total colour difference of reconstituted Gac powder products as a result of the spray drying process was similar to the results for redness, in terms of being significantly affected by the inlet drying temperature but not by the maltodextrin concentration. The reduction of redness, indicated by high hue angle and low a*/b* value, is the possible explanation for an increased total colour difference in reconstituted Gac powders due to high inlet temperature. Additionally, it can be clearly seen that ΔE is a function of value L*a*b*, therefore, increase in lightness with increased inlet temperature was also contribution to increasing ΔE.

Contradictorily, Rodríguez-Hernández et al. (2005) and Grabowski et al. (2006) indicated that the influence of maltodextrin concentration was found to be significant for the variation of ΔE in reconstituted cactus pear juice and sweet potato puree powders, respectively. However, as discussed above, a high addition of maltodextrin concentration significantly influenced the lightness and red colour of Gac samples. Although, it was observed that a significant increase in ΔE due to high maltodextrin concentration was not statistically found, the maltodextrin concentration, nevertheless contributed to increase in ΔE. This is due to resulting in higher lightness and hue angle, and lower a*/b* value.

5.4.3 Effect of spray drying conditions on the total carotenoid content and encapsulation efficiency of Gac powders

The total carotenoid content (TCC) and encapsulation efficiency (EE) of the Gac fruit powder products as a result of different spray drying conditions are presented in Figure 5.3 and Figure 5.4, respectively. Statistical results indicated that TCC and EE of products were significantly impacted by maltodextrin concentration and by spray drying temperatures (P<0.001). A significant interaction between the two factors was observed in TCC of the powders (P<0.01) but not in the EE of the powders (P>0.05).
The TCC in powder samples reduced from 1.95 mg/g to 0.61 mg/g of powder as the maltodextrin concentration increased from 10% to 30%. Furthermore, the spray drying temperature also affected TCC; significant loss of TCC in samples was observed as temperature increased from 120°C to 200°C. However, there was no statistical difference in TCC of samples between temperatures of 140°C and 160°C; between 160°C and 180°C; or between 180°C and 200°C.

The EE of the study samples was also significantly influenced by maltodextrin concentration and by drying temperature. Increasing maltodextrin concentration resulted in higher EE; however, no difference in EE between the concentrations of 20% and 30% was observed. Moreover, in general, EE of the samples reduced from 76.58% to 48.02% as the drying temperature increased from 120°C to 200°C, respectively.
According to Goula and Adamopoulos (2005), an increase in inlet drying temperature resulted in a greater loss of lycopene content in tomato powders. Similarly, Quek et al. (2007) observed that a decrease in the lycopene and β-carotene content of spray-dried watermelon powder occurred as a result of increasing the inlet air temperature. The main reason for these findings is due to thermal degradation and oxidation. In addition to the inlet temperature, the loss of carotenoids in the Gac fruit powder samples was also dependent on several factors, such as outlet temperatures, droplet moisture content, oxygen and exposure to light. These factors are governed by processing conditions such as feed rate, initial feed solid concentration, drying and compressed air flow rate. Moreover, as has been discussed previously (in section 5.4.1), a higher moisture content was obtained by spray drying at lower inlet air temperatures. Increasing moisture content caused a higher loss of lycopene, however, when the moisture content increased a greater degree of aggregation occurred because of the natural stickiness of the product. This leads to there being lower oxygen exposure.
resulting in lower lycopene loss (Goula et al., 2004; Goula & Adamopoulos, 2005). Moreover, carotenoids are particularly vulnerable to thermal treatment and oxidative processes due to their structure, which contains a conjugated double bond system over the entire length of the polyene chain (Britton, 1995; Quek et al., 2007).

In terms of the effect of drying temperature, a similar pattern to the TCC loss as a result of increasing the inlet temperatures was also observed in relation to EE. It was observed that increasing temperature resulted in a reduction of EE. The explanation for this phenomenon is that the degradation of carotenoids at higher temperature, as discussed above, leads to reduced EE. Furthermore, according to Shu et al. (2006), the balance between the rate of water evaporation and film-formation may break down due to a high inlet temperature; therefore, wall systems of microcapsules are broken down. This phenomenon will cause a low percentage of encapsulation efficiency. Similar findings were also reported by other authors (Shu et al., 2006; Sua et al., 2008). However, Leach et al. (1998) stated that effect of inlet and outlet temperature on EE was not important at low feed solid levels. The higher EE was obtained when combinations of these temperatures were carried out.

In this study, the lower TCC was significantly observed when increasing the maltodextrin concentration from 10% to 30%. This is due to high maltodextrin concentration leading to lower TCC obtained since the feed juice flow rates were constant. On the other hand, an increase in EE was observed with increasing maltodextrin concentration. This is well known that carotenoid content in powder is effectively protected in a high initial feed solid. Similar observations were found in other studies (Wagner & Warthesen, 1995; Rodríguez-Huezo et al., 2004).

It is well established that carotenoids are widely distributed in nature as red, yellow and orange pigments. Therefore, the content of carotenoids could affect the colour of the spray-dried Gac powders. In this study, the relationship between carotenoid content and colour characteristics in spray-dried powders was also considered. According to a Pearson correlation test, there was a high positive correlation between TCC and a*/b* value (r=0.65, p<0.01) and a high negative correlation between TCC and hue angle (r=-0.64, p<0.01).

However, as compared to spray-dried powder, a better relationship between TCC and colour was found in reconstituted powders. The strong positive and negative correlations between TCC and a*/b* value (r=0.75, p<0.01) and between TCC and hue.
angle \( r=-0.74, \ p<0.01 \), respectively, were significantly found. Similarly, Quek et al. (2007) also observed that an inverse relationship between hue angle and content of lycopene and \( \beta \)-carotene was significantly found in their study of spray-dried watermelon powders. Therefore, it can be concluded that the higher TCC indeed resulted in a redder colour in spray-dried Gac powder.

5.4.4 Effects of spray drying conditions on total antioxidant activity of Gac powders

Figure 5.5 shows total antioxidant activity (ABTS assay) of Gac powder samples as a result of different spray drying conditions. Generally, the two factors investigated, that is the maltodextrin concentration and the drying temperature, significantly affected TAA of powders \( \ p<0.001 \). There was no significant difference in TAA between the samples when adding maltodextrin at the concentrations of 10\% and 20\%. However, when the concentration of maltodextrin was increased from 20\% to 30\% a loss of TAA was observed. Overall, in increasing the spray drying temperature from 120\°C to 200\°C significant loss of TAA was observed, from 0.14 to 0.08 mmole TE/g of powder. However, there was no statistical difference in TAA of samples spray-dried at temperatures of 140\°C and 160\°C.

In a similar pattern to the results for the TCC of the samples, increasing maltodextrin concentration and drying temperature resulted in lower TAA of powder samples. The possible explanation is that at the higher drying temperatures the loss of TCC, a major antioxidant compound in spray-dried Gac powder, leads to TAA degradation. The other reason for the decrease of TAA is simply due to the increased concentration of maltodextrin.
Figure 5.5 plots the values of TCC and TAA of powder products at three levels of maltodextrin concentration and at five inlet air temperatures. It is clear that there was strong correlation between TCC and TAA ($R^2 = 0.915$ to 0.948). The highest correlation was found at the lowest maltodextrin concentration. Moreover, according to a Pearson correlation test between TCC and TAA results for different drying conditions, the Pearson correlation coefficient was 0.482 and significant at the level 0.01 level (2-tailed). The positive correlation means that with an increasing TCC the TAA in the spray-dried Gac fruit powders also increases. Likewise, Chanwitheesuk et al. (2005) reported that a high correlation between total carotenes and the antioxidant activity was found in *Piperaceae* ($R=0.99$) and in *Cucurbitaceae* ($R=0.87$), however, a low correlation was found in *Umbelliferae* ($R=0.46$). In contrast to this result, a negative correlation between total carotenoids and antioxidant activity in guava fruit extract was observed by Thaipong et al. (2006). Moreover, total colour difference of reconstituted powder in correspondence to the feed juice was strongly negatively correlated to TAA ($r=-0.74$, $p<0.01$) and negatively correlated to TCC ($r=-0.47$, $p<0.01$). It can be concluded that a high TAA was obtained with less colour difference in Gac powders during spray drying process.
5.5 Conclusions

In summary, the effects of the spray drying conditions of maltodextrin concentration and drying temperature on the physicochemical and antioxidant properties of Gac fruit powders were investigated. The colour of the Gac fruit powder samples shown in Figure 5.7 illustrates the differences resulting from the addition of different concentrations of maltodextrin at a specific drying temperature.

Moisture content, bulk density, colour characteristics, TCC, EE and TAA were significantly affected by maltodextrin concentration and by the inlet air temperatures. However, pH, Aw and WSI were not significantly influenced by the different spray drying conditions in this study. The Gac powder spray-dried at inlet temperature of 120°C and maltodextrin concentration of 10% was adequately effective in preserving colour, TCC and TAA. Strong positive correlation among TCC, TAA and colour characteristics was also confirmed.
Spray-dried Gac powder
Mixed with 10% Maltodextrin, and dried at 120°C

Spray-dried Gac powder
Mixed with 20% Maltodextrin, and dried at 120°C

Spray-dried Gac powder
Mixed with 30% Maltodextrin, and dried at 120°C

Figure 5.7 Samples of spray-dried Gac powder produced with varying concentrations of maltodextrin
Chapter 6
STORAGE STUDY OF GAC FRUIT POWDER

6.1 Introduction

It is well established that the quality of various fruit and vegetable powder products is generally affected not only by the processes of the drying method used but also by storage conditions. Many factors such as light, temperature and oxygen are considered to influence the quality of powder. In the previous Chapters, 4 and 5, the effects of several parameters of different drying processes on the quality of Gac fruit powders were investigated. It is also necessary to study the effect of storage conditions on the quality of powders, because several deteriorative reactions such as physical and chemical reactions impacting the colour and nutrient properties of the powders occur continually during storage.

It is also important to construct moisture sorption isotherms for calculating the moisture changes and predicting the stability of product during storage. The moisture uptake behaviour, which describes the hygroscopic properties of a product, can be obtained based on moisture sorption characteristics. Furthermore, many studies report the sorption characteristics for a variety of fruit powders, but not for Gac fruit powder. Therefore, the need to add to the limited information on sorption characteristics for Gac fruit powders is evident.

The objectives of this chapter are to examine the shelf life of the Gac fruit powder products, in terms of colour characteristics, total carotenoid content and total antioxidant activity, monitored under a variety of storage conditions. The correlations among these parameters are also discussed. Furthermore, kinetic parameters and moisture sorption isotherms are examined for predicting the shelf life of Gac fruit powders.

6.2 Storage conditions of powders

Freeze-dried fresh Gac fruit aril powder and powders produced from fresh aril spray-dried under drying conditions of air inlet temperature of 120°C with 20% maltodextrin concentration were used for this storage study. These powders were packed in
quantities of 5 g into laminated aluminium high barrier vacuum bags and non-laminated high barrier vacuum bags.

The laminated and non-laminated packages of the powders were stored at different temperatures of 10°C, 20°C and 37°C in lab refrigerators and an oven (Memmert, Germany). Duplicate samples were periodically withdrawn, during an 8 month period for freeze-dried powders and during a 3 month period for spray-dried powders, in order to measure the total colour difference, total carotenoid content and total antioxidant activity (ABTS assay).

6.3 Results and Discussion

6.3.1 Effect of storage conditions on total colour difference of powders

6.3.1.1 Freeze-dried powder

The effects of storage conditions on the total colour difference of freeze-dried powders are shown in Figure 6.1. Results indicated that total colour difference was significantly influenced by storage temperature (P<0.001), but not by packaging (P>0.05). Furthermore, under multiple comparisons for total colour difference, there was no significant difference in total colour difference of the samples between storage at temperature of 10°C and 20°C.
6.3.1.2 *Spray-dried powder*

As shown in Figure 6.2, the total colour difference of spray-dried powders under the different storage conditions was significantly influenced by storage temperature ($P<0.05$), but not by packaging ($P>0.05$). It was observed that greater total colour difference was caused by increasing storage temperature. Furthermore, no significant difference in total colour difference of samples was observed in storage temperature at $10^\circ$C and $20^\circ$C.

![Figure 6.2 Total colour difference of SD powder under different storage conditions](image)

**Figure 6.2 Total colour difference of SD powder under different storage conditions**

6.3.1.3 *Discussion*

As anticipated, a higher storage temperature and longer storage time significantly resulted in the largest difference in total colour of both the freeze-dried and spray-dried Gac fruit powders. It was also indicated by the results that higher lightness and lower
redness of powders were observed at the higher storage temperature, resulting in the greater total colour difference. This is in agreement with the study results reported by Duangmal et al. (2008). They stated that total colour difference of freeze-dried rosella extract increased in relation to storage time. Similarly, Chen and Tang (1998) reported that total colour difference of carrot pulp waste powder was greater as storage temperature and time were increased. Furthermore, in this study, the overall colour of the Gac fruit powders was not significantly changed at the low storage temperatures of 10°C and 20°C, as indicated by measurements lower than 10 units. However, Sagar et al. (2000) reported that the colour of ripe mango powder was not lost when stored at a low temperature of 7°C for up to 6 months, and at room temperature of 33-33.5°C for up to 4 months.

Studies on total colour difference of fruit and vegetable powders under varied storage conditions are very limited in the available literature. From the data in Figure 6.1 and Figure 6.2, a comparison between spray-dried and freeze-dried powders can be made in terms of total colour difference. A similar pattern was observed in total colour difference of the freeze-dried and the spray-dried powders. Total colour difference increased more rapidly with the increase in storage temperature. For powder samples stored at a temperature of 10°C, the total colour of spray-dried powder changed less than the total colour of freeze-dried powder in the first month of storage; however, in the periods of the second and third months, the colour of spray-dried powder changed more quickly. Moreover, at higher storage temperatures of 20°C and 37°C the colour change of spray-dried powder samples was more rapid compared to that of freeze-dried ones. It could be concluded that at the low storage temperature of 10°C the spray drying process was more effective than freeze drying in producing powders with minimised colour change.

At higher storage temperatures of 20°C and 37°C, in contrast, freeze drying was the more effective method for producing Gac powders which maintained their colour. According to Çinar (2004), freeze drying could retard the colour loss of plant samples, however, some contrary findings were also seen due to the natural unsaturated pigments and presence of active enzyme in some tissue samples. In this study of Gac fruit, it can be speculated that the presence of maltodextrin must have caused some reactions at the higher storage temperature.
6.3.2 Effect of storage conditions on total carotenoid content and total antioxidant activity of powders

6.3.2.1 Freeze-dried powder

Figure 6.3 and Figure 6.4 show the effects of storage conditions on the total carotenoid content and antioxidant activity of freeze-dried Gac powders. Storage temperature significantly affected TCC and TAA of powders over the storage period up to 8 months (P<0.001). An increase in storage temperature significantly resulted in loss of carotenoid content and antioxidant activity of the powders. Furthermore, the type of packaging also influenced TAA of samples during storage (P<0.05), but did not influence TCC (P>0.05). Loss of TAA was lower for powder packed in laminated aluminium high barrier bags as compared to non-laminated bags, except at 37°C.

Figure 6.3 TCC of FD powder under different storage conditions
Total carotenoid content and antioxidant activity of spray-dried powders under different storage condition are presented in Figure 6.5 and Figure 6.6. Statistical results indicated that storage temperature significantly impacted both TCC and TAA of spray-dried powder (P<0.05 and 0.01, respectively). In general, increasing storage temperature resulted in higher degradation of TCC and TAA. However, TCC and TAA of spray-dried powders were not significantly affected by the packaging type (P>0.05).
Figure 6.5 TCC of SD powder under different storage conditions

Figure 6.6 TAA (ABTS assay) of SD powder under different storage conditions
6.3.2.3 Discussion

In this study, loss of TCC and TAA in spray-dried samples increased with higher storage temperatures and a longer period of time. Similar findings were established by various studies which confirmed that the main reasons for this phenomenon are oxidation and isomerisation (Desobry et al., 1997; Chen & Tang, 1998; Anguelova & Warthesen, 2000; Çinar, 2004; Dutta et al., 2005; Chiu et al., 2007; Davoodi et al., 2007). The possible mechanism of carotenoid degradation is that heat and available oxygen promote isomerisation of trans-carotenoids to the cis-forms, which have the lowest stability, during storage. As a result, low molecular mass compounds were formed, leading to significant loss of carotenoids. Furthermore, in this study, degradation of TCC and TAA in all samples during storage fitted a first-order reaction. This is similar to findings of several other studies (Wagner & Warthesen, 1995; Chen & Tang, 1998; Robert et al., 2003; Koca et al., 2007). It could be concluded that carotenoid reduction during storage was caused by increased temperature, which is more important than other storage factors.

Loss of TCC after eight months for freeze-dried powders stored at 10°C in laminated and non-laminated bags was just 10.81% and 18.49%, respectively. The possible explanation for this phenomenon is that reisomerisation from cis-isomers to all-tran forms may occur (Xianquan et al., 2005). Therefore, after the storage period of eight months, the degradation of TCC in freeze-dried powders stored at 10°C was lower than other powders stored at different temperatures. Comparatively, the degradation of the capsanthin (a carotenoid) content in red pepper powder stored at 0°C after six months was 11.4% (Kim et al., 2004). However, Anguelova and Warthesen (2000) reported a considerable loss of 30% of total lycopene in tomato spray-dried powders after 6 weeks stored at 6°C. The authors suggested that the lycopene degradation was due to isomerisation and autoxidation.

In a comparison with spray-dried powders, at the lower storage temperatures of 10°C and 20°C, the progressive loss of TCC in freeze-dried powders was lower over 3 months of storage, as shown in Figures 6.3 and 6.5. Furthermore, at the higher temperature of 37°C, the progressive degradation of TCC in freeze-dried powders was higher during the first two months. In contrast, the loss of TCC in spray-dried powders increased more rapidly during the third month. Inversely, in terms of TAA it can be seen that the progressive degradation in freeze-dried powders was much higher than that in
spray-dried powders; except for the sample with storage conditions of laminated packaging and a temperature of 10°C (refer to Figures 6.4 and 6.6).

For freeze-dried powder with both packaging types, TCC loss after 8 months at temperatures of 10°C, 20°C and 37°C was 11% - 19%, 25% - 41% and 67% - 70%, respectively. TAA reduction of freeze-dried powders after 8 months was 44% - 57%, 75% - 81% and 87% - 88%, at temperatures of 10°C, 20°C and 37°C, respectively. It can be seen that in freeze-dried powders the degradation rate of TAA was much higher than the rate of TCC reduction under the tested storage conditions. This is due to isomeric conversion of carotenoids, such as lycopene and β-carotene, to cis-isomers which are less stable and more susceptible to degradation (Deuel, 1951). Moreover, cis-isomers have generally higher antioxidant activity than trans-isomers (Levin & Mokady, 1994). As a result, the loss of cis-isomers may cause higher loss of TAA in the samples compared to TCC loss.

Interestingly, the two types of packaging used in this study did not significantly affect TCC of freeze-dried or spray-dried powders, or the TAA of spray-dried powder. This means that the effect of the light TCC and TAA of powders was not significant. Similarly, Çinar (2004) reported that the carotenoid loss of the freeze-dried plant samples such as orange peel, sweet potato and carrot was not affected by light and dark conditions when stored at 25°C for 45 days. This is also in agreement with data reported by Kopas-Lane and Warthesen (1995), who stated that the storage conditions of dark, light and cold did not significantly affect the loss of major carotenoid content in raw spinach and carrot during an 8 day period. Furthermore, Shi et al. (2008) also reported that the stability of lycopene in tomato puree was insignificantly changed by exposure to light. However, it was found that degradation and isomerisation of carotenoids in tomato pulp solid or tomato juice during storage were increased by exposure to light (Sharma & Le Maguer, 1996; Lin & Chen, 2005b). Although the reduction of TCC and TAA in the experimental Gac powders was insignificantly influenced by light, the losses were observed. Therefore, laminated packaging is highly recommended for Gac powder storage.

It is clear that degradation of TCC and TAA in Gac powders during storage in this study was best fitted by a first-order kinetic model. Many studies indicate that loss of carotenoids in samples during storage follow first-order reactions (Wagner & Warthesen, 1995; Tang & Chen, 2000; Robert et al., 2003; Koca et al., 2007). Table 6.1 and Table 6.2 show kinetic parameters of TCC and TAA degradations, respectively, of freeze-dried and spray-dried powders under the different storage conditions.
Generally, the higher degradation rates and lower half life values of TCC and TAA of the powders were obtained as a result of increasing storage temperatures from 10°C to 37°C. Results indicated that the breakdown of carotenoids and antioxidant activity in the stored powders was much faster at higher temperatures. It is therefore recommended that the Gac powders should be stored at a low temperature of 10°C to preserve the quality in terms of carotenoid content and antioxidant activity.

Table 6.1 Kinetic parameters of first-order total carotenoids degradation in FD and SD powders under different storage conditions

<table>
<thead>
<tr>
<th>Powder Samples</th>
<th>Degradation rate (day⁻¹)</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R²)</th>
<th>Activation energy (kcal mol⁻¹)</th>
<th>Arrhenius coefficient (R²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-FD-10</td>
<td>0.0005</td>
<td>1386</td>
<td>0.7831</td>
<td>14.28</td>
<td>0.9999</td>
</tr>
<tr>
<td>NL-FD-10</td>
<td>0.0010</td>
<td>693</td>
<td>0.9555</td>
<td>9.43</td>
<td>0.9970</td>
</tr>
<tr>
<td>20/120-L-10</td>
<td>0.0009</td>
<td>770</td>
<td>0.9738</td>
<td>13.11</td>
<td>0.9783</td>
</tr>
<tr>
<td>20/120-NL-10</td>
<td>0.0011</td>
<td>630</td>
<td>0.9437</td>
<td>10.06</td>
<td>0.7323</td>
</tr>
<tr>
<td>L-FD-20</td>
<td>0.0012</td>
<td>577</td>
<td>0.9576</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NL-FD-20</td>
<td>0.0019</td>
<td>365</td>
<td>0.9536</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-L-20</td>
<td>0.0015</td>
<td>462</td>
<td>0.8119</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-NL-20</td>
<td>0.0048</td>
<td>144</td>
<td>0.9996</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L-FD-37</td>
<td>0.0045</td>
<td>154</td>
<td>0.9790</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NL-FD-37</td>
<td>0.0043</td>
<td>161</td>
<td>0.9883</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-L-37</td>
<td>0.0065</td>
<td>107</td>
<td>0.8871</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-NL-37</td>
<td>0.0058</td>
<td>120</td>
<td>0.8698</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

L, NL = Laminated & Non-laminated bags; FD = Freeze-dried; 10, 20, 37 = storage temperature (°C) 20/120 = Spray drying conditions (20% maltodextrin / inlet temperature 120°C)

In a comparison between Gac fruit powders produced by the two different drying processes, lower degradation rates and longer half life values of TCC in freeze-dried powders were observed as compared to the spray-dried powders at the same storage conditions. Therefore, it can be concluded that during storage the stability of TCC in powders produced by freeze drying is higher than in powders produced by the spray drying process. The possible explanation is that physicochemical characteristics of powders may differ as a result of the different drying processes. According to Tang and Chen (2000) numerous tiny porous surfaces on the powder are formed by spray drying as a result of hot air penetration causing shrinkage of powder granules. However, shrinkage or deformation of the powder granule does not take place during freeze drying since water is sublimated from ice crystals. Furthermore, the amount of cis-
carotenoids formed in spray-dried powders is much higher than that in freeze-dried powders due to effect of heat promoting the geometric isomerisation of trans-carotenoids to cis-forms (Dutta et al., 2005). Similarly, Desobry et al. (1997) also stated that faster degradation rate of β-carotene was obtained in spray-dried powder in comparison with freeze-dried powder. The explanation is due to the smaller particle size and higher surface carotenoid content of spray-dried powders.

Table 6.2 Kinetic parameters of first-order total antioxidant activity degradation in FD and SD powders under different storage conditions

<table>
<thead>
<tr>
<th>Samples</th>
<th>Degradation rate (day⁻¹)</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R²)</th>
<th>Activation energy (kcal mol⁻¹)</th>
<th>Arrhenius coefficient (R²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-FD-10</td>
<td>0.0024</td>
<td>289</td>
<td>0.922</td>
<td>7.11</td>
<td>0.8964</td>
</tr>
<tr>
<td>NL-FD-10</td>
<td>0.0037</td>
<td>187</td>
<td>0.991</td>
<td>4.10</td>
<td>0.8245</td>
</tr>
<tr>
<td>20/120-L-10</td>
<td>0.0013</td>
<td>533</td>
<td>0.8835</td>
<td>8.37</td>
<td>0.9413</td>
</tr>
<tr>
<td>20/120-NL-10</td>
<td>0.0022</td>
<td>315</td>
<td>0.9975</td>
<td>5.098</td>
<td>0.9698</td>
</tr>
<tr>
<td>L-FD-20</td>
<td>0.0052</td>
<td>133</td>
<td>0.9799</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NL-FD-20</td>
<td>0.0062</td>
<td>112</td>
<td>0.9474</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-L-20</td>
<td>0.0029</td>
<td>239</td>
<td>0.9971</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-NL-20</td>
<td>0.0034</td>
<td>204</td>
<td>0.9666</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L-FD-37</td>
<td>0.0075</td>
<td>92</td>
<td>0.9343</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NL-FD-37</td>
<td>0.0072</td>
<td>96</td>
<td>0.9519</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-L-37</td>
<td>0.0049</td>
<td>141</td>
<td>0.994</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20/120-NL-37</td>
<td>0.0049</td>
<td>141</td>
<td>0.9999</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

L, NL = Laminated & Non-laminated bags; FD = Freeze-dried; 10, 20, 37 = storage temperature (⁰C) 20/120 = Spray drying conditions (20% maltodextrin / inlet temperature 120⁰C)

However, in an inverse trend to TCC, the study results show that faster degradation rates and shorter half life values for TAA of the Gac fruit powders were obtained in powders produced by freeze drying in comparison with those produced by spray drying. This means that spray drying was more efficient for TAA preservation under the tested storage conditions. It is possible that TAA of spray-dried powders is enhanced by components formed due to the temperature used. In addition, maltodextrin matrices could prevent the loss of antioxidant activity during storage. Therefore, although TCC is decreased, TAA is still good. Furthermore, because of the porous structure of freeze-dried powder, oxygen can easily be allowed to reside in the powder granules. As a result, oxidation of carotenoids may occur; and as a result the loss of TAA in freeze-
dried powder was much faster than in spray-dried powder. Wagner and Warthesen (1995) stated that retention of TCC could be improved by the addition of hydrolysed starches because of effective protection to oxidation of the physical barriers of the starches. Hence, this confirms the preservation of TAA by the addition of maltodextrin in the spray-dried samples.

In general, increasing the rate constants of total carotenoid and total antioxidant activity degradations at various storage temperatures of 10°C, 20°C and 37°C resulted in decrease in activation energy. Activation energies of total carotenoid and total antioxidant activity degradation in samples packed in non-laminated bags were lower than those in laminated bags. The results suggest that the progressive loss of TCC and TAA in the non-laminated bags was much higher than in the laminated bags under the same storage conditions. However, statistical results showed that packaging with laminated or non-laminated bags did not significantly influence the TCC and TAA of all samples, except for the TAA in freeze-dried powder samples. Generally, from the raw data, during storage the loss of TCC and TAA of the laminated bag powder samples was lower than losses in non-laminated bag samples. As such, it is recommended that the laminated packaging should be used during storage of carotenoid-rich powders, such as Gac fruit powders, to avoid the degradation in terms of TCC and TAA.

A high correlation (high Pearson coefficient and P<0.01) among TCC, TAA and total colour difference results during storage was also observed. Similarly, Chen and Tang (1998) also pointed out that a strong relationship between colour differences and carotenoid content in carrot pulp waste powder under various storage conditions was established. The results of this study suggest that a greater difference of total colour during storage at the temperatures examined will result in significant degradation of TCC and TAA. Moreover, preserving TCC of the Gac powder samples during storage could also maintain TAA.

6.3.3 Moisture sorption isotherms

The graphical relationship between the equilibrium moisture content (EMC_{db}, %) and the equilibrium relative humidity (ERH, %) at a constant temperature is described by moisture sorption isotherms. Figure 6.7 shows the sorption isotherm curves constructed for a variety of Gac fruit powder samples at room temperature. The conditions under which these Gac fruit powder samples were produced are described in section 3.4. The isotherms are extremely useful for comparing drying processes; optimising drying equipment; predicting shelf-life of product in terms of physical,
biochemical and microbial stability; determining packaging and storage conditions (Janjai et al., 2006; Yan et al., 2008). The isotherm curves of all the tested Gac powders exhibited a sigmoid shape and similar trends. Generally, EMC values at the constant temperature increased with increase in ERH. This is in agreement with several authors who stated that the moisture isotherms for dried plant food usually exhibit a sigmoid curve (Arogba, 2000; Janjai et al., 2006; Yan et al., 2008).

As can be seen clearly from Figure 6.7, at the ERH range of 51%-75% (region III), the powder samples exhibited an almost constant EMC, except for skin powder that showed a slight increase. This means that the other samples could be more stable than skin powder; therefore, these powders could be stored at this range. Furthermore, the moisture content and water activity were very low at the region I of the ERH range, indicating the very small amount of free water within the powders. Thus, the materials could be characteristically shrunken or brittle. Moreover, it is very difficult and costly to bring foods into this region of ERH by drying processes. In contrast to region I, deterioration of samples in region IV (ERH higher than 75%) occurs easily and rapidly due to high moisture uptake, a condition which supports chemical, biological and microbial reactions.

![Figure 6.7 Moisture sorption isotherms of Gac fruit powders](image)
Within region II, when ERH shifted from 22% to 51%, the greatest increase in water uptake behaviour was observed in the yellow pulp powder as displayed by the highest gradient slope in this section of the curves; this was followed by aril freeze-dried, aril air-dried, aril vacuum-dried, aril spray-dried and skin powders, respectively. These results indicated that the hygroscopicity of yellow pulp powder was higher as compared to the other powders. In the comparison of different drying methods for the aril, EMC of the freeze-dried powder was consistently higher than other aril powders at the same ERH. The lowest was for spray-dried powder, whereas air-dried and vacuum-dried showed no significant difference.

The possible reason for these results is due to the different physical characteristics of the aril powders produced by the different drying methods. Freeze-dried samples have a porous structure and little or no shrinkage, resulting in higher adsorptive capacity than powders produced by other drying methods, whilst marked shrinkage is produced during the spray drying process (Tang & Chen, 2000; Qing-guo et al., 2006). These results are consistent with findings of several other studies (Debnath et al., 2002; Qing-guo et al., 2006; Gong et al., 2007).

According to the BET equation, the highest monolayer moisture content, \( M_0 \) (\%, d.b.) was obtained in yellow pulp powder (10%), followed by the freeze-dried aril powder (8.16%), the air-dried aril powder (6.88%), the vacuum-dried aril powder (6.83%), skin powder (5.59%), and the spray-dried aril powder (4.53%). Generally, the \( M_0 \) of a dried food product is considered to be the safest condition in terms of good storage capability. This is because a higher rate of lipid oxidation is promoted at moisture below this level; at higher moisture content levels, deterioration of food occurs due to promotion of Maillard browning, enzymic and microbiological activities. Moreover, it is not necessary to dry food to a low \( M_0 \) value due to the increased extra heat of evaporation. Therefore, the initial moisture content of a product is preferred to be at or slightly above \( M_0 \) value for maximal shelf life with minimal spoilage (Fellows, 2000; Us et al., 2008). From the experimental results presented in Chapter 4 and Chapter 5, the MC of the Gac powder samples was close to \( M_0 \), indicating that these samples could be stored for a longer time.

6.4 Conclusions

As expected, a significant progressive loss of colour, TCC and TAA in FD and SD Gac aril powder samples increased as a result of increasing storage temperature from 10°C
to 37°C, and with a longer storage period. Degradation of TAA in FD powders was reduced when powders were packed in laminated aluminium high barrier bags as compared to non-laminated bags. Furthermore, the degradation rate of TCC and TAA in the powders was best fitted by a first-order reaction. In comparison, the stability of TCC in FD powders under the tested storage conditions was higher than the stability in SD powders. However, spray drying was shown to be more effective in preserving TAA of the samples than freeze drying. Moreover, there were high correlations between total colour difference, TCC and TAA in all powder samples under the various tested storage conditions. Therefore, preservation of colour, TCC and TAA of the Gac powders was more effective when samples of the Gac powders were packed into laminated aluminium high barrier vacuum bags and stored at a temperature of 10°C.

The isotherm curves of all of the Gac powder samples tested have sigmoid shapes and similar patterns to those which are usually observed in dried food products. EMC values at a constant temperature increased with increase in ERH. The highest hygroscopicity was observed in the yellow pulp powder. By comparing different drying methods for aril, the lowest EMC was obtained in SD powder, indicating the lowest hygroscopicity, followed by VD, AD and FD powders.
Chapter 7

STABILITY OF GAC FRUIT POWDER AS A FOOD COLORANT AND NUTRIENT SUPPLEMENT

7.1 Introduction

Foods containing carotenoids are currently receiving considerable attention from food manufactures and consumers. Carotenoids, namely β-carotene, lycopene, lutein and astaxanthin, are widely used within the food industry as colorants and nutrient supplements. For example, the European market for carotenoids was worth US $348.5M in 2003 and was estimated to annually grow by 2.7% to reach $419.6M in 2010 (Sullivan, 2004). Moreover, the potential market for beverages based on fruits is rapidly growing due to an increased awareness of consumers of the benefits of natural nutrient components. In previous chapters the Gac powder forms, containing high levels of carotenoids, produced by different drying treatments were discussed. In this chapter these powders are further considered in their role as natural food colorants and nutrient supplements.

Thermal processing such as pasteurisation is important to the stability of the fruit juice products during storage. Lee and Coates (2003) reported that the stability of food, based on carotenoids, greatly varies in different types of food and beverage products. Furthermore, choosing the appropriate pasteurisation temperature plays an important role in preserving the colour and nutrients. Generally, the intensity of the thermal process depends on a number of factors. A temperature below 100°C is normally chosen when the pH of the fresh sample is lower than 4.6; by contrast, for a product with a pH higher than 4.6, a higher temperature of above 100°C is used (Awuah et al., 2007). To maintain the nutritional value, however a temperature below 100°C together with refrigerated storage should be applied.

Since fresh Gac fruit is not always available throughout the year, it is very convenient to use the powder form of Gac in place of the fresh fruit for producing the rice dish “Xoi Gac” and beverages such as Gac powder juice and Gac powder milk beverage. There is presently little available information in the literature describing processing of Gac fruit in food and beverage products. Therefore, this research studies the stability in different
heat-processed Gac powder products in relation to the colour, total carotenoid content and antioxidant activity. Furthermore, a storage study of two beverages - Gac powder juice and Gac powder milk beverage - was carried out and samples were evaluated over a one month period.

7.2 “Xoi Gac” preparation

“Xoi Gac”, a Vietnamese dish of steamed glutinous rice, was prepared with the addition of Gac powder. The ingredients include glutinous rice (400g), coconut milk (300 mL), Gac powder, sugar and salt. The Gac powder, sugar and salt were added into coconut milk and then thoroughly blended to produce a homogenous mixture. The mixture was poured and mixed well into portions of previously steamed glutinous rice in a steamer, and then steamed for up to 30 minutes. In this study, three different fresh Gac aril powders (freeze-dried at condenser temperature of -46°C, soaked in ascorbic acid prior to vacuum drying at 40°C, and spray-dried at 120°C with 10% added maltodextrin) were used. The quantity of FD and of VD Gac powder used was 4 g, while about 10 g of SD powder was used, allowing for the presence of maltodextrin. All experiments were done in duplicate.

7.3 Gac fruit juice processing

Three different Gac powders (produced by the same drying conditions as those used for Xoi Gac preparation) were used for fruit juice processing. The ingredients for the Gac powder juice were drinking water (1 litre), sugar (70 g), citric acid (1.5 g), and Gac powder. The quantity of freeze-dried and of vacuum-dried powder used was 40 g, and the quantity of spray-dried powder was 100 g. The juices were thoroughly mixed using a lab blender, were filtered using a cloth filter, and then heated to temperature of 95°C for 30 seconds. After heating, the juices, with a pH of 4.18±0.12, were hot filled into glass bottles (330 mL) with 1.2 cm headspace. The heating pasteurisation process was performed using an autoclave (Korimat KA 160, Germany) with a thermocouple measuring a “cold region” temperature of the bottle. The two pasteurisation temperatures at the “cold region” that were used in this study were 90°C for 5 minutes and 80°C for 30 minutes. After thermal treatment, the juices were immediately cooled in an ice-water bath. Samples of the juice before pasteurisation (control juice samples) and samples of the two pasteurised juices, treated at different temperatures, were all analysed on the same day as processing for colour characteristics, total carotenoid content and total antioxidant activity (ABTS assay). For the storage study, the Gac
juices pasteurised at 90°C for 5 minutes were kept under refrigeration at 4±2°C, and then periodically withdrawn for testing, after 5, 10, 20 and 30 days.

The two factors (type of powder and pasteurisation temperature) were randomly designed to investigate the effect of processing and storage conditions on the colour, TCC and TAA of Gac juice samples. All experiments were carried out in duplicate. A total of 8 runs was conducted.

7.4 Pasteurised Gac milk beverage processing

Raw milk purchased from the morning milking at the experimental farm, University of Nong Lam, Hochiminh City, Vietnam, was heated at a temperature of 63°C for 20 seconds. Three different Gac powders (produced by the same drying conditions as those used for Xoi Gac preparation) were added into the milk (1 litre) and stirred until completely dissolved and then filtered using a cloth filter. The quantity of the FD and of VD Gac powders used was 10 g, while 50 g of SD powder was used. The finished beverages, with a pH of 5.32±0.22, were heated at a temperature of 95°C for 30 seconds and poured into glass bottles (330 mL) with 1.2 cm headspace. The thermal pasteurisation process, the storage conditions, the testing times and the experimental design were similar to those for the Gac fruit juices. All experiments were also performed in duplicate.

7.5 Results and Discussion

7.5.1 Stability of powders for use in production of “Xoi Gac”

The colour characteristics, TCC and TAA of “Xoi Gac” samples produced using FD, VD and SD Gac powders, respectively, are presented in Table 7.1.

Table 7.1 Colour characteristics, TCC and TAA (ABTS assay) of “Xoi Gac”

<table>
<thead>
<tr>
<th>Xoi Gac samples</th>
<th>Lightness</th>
<th>Chroma</th>
<th>Hue angle</th>
<th>TCC µg/g</th>
<th>TAA µmole TE/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>From FD powder</td>
<td>50.91±1.04a</td>
<td>26.65±0.85a</td>
<td>55.96±0.34a</td>
<td>28.54±2.87a</td>
<td>23.80±1.17a</td>
</tr>
<tr>
<td>From VD powder</td>
<td>50.07±1.56a</td>
<td>29.35±0.78a</td>
<td>58.44±3.47a</td>
<td>27.42±1.45a</td>
<td>21.27±0.99a</td>
</tr>
<tr>
<td>From SD powder</td>
<td>56.22±1.38b</td>
<td>26.21±0.79b</td>
<td>71.69±2.28b</td>
<td>15.94±1.31b</td>
<td>12.75±1.34b</td>
</tr>
</tbody>
</table>

The values in the same column followed by different superscripts (a-b) were significantly different (P<0.05)
Generally, in the samples of “Xoi Gac” the colour characteristics of lightness and hue angle were significantly affected by using different powders in the preparation (P<0.05), whereas a significant effect of the powders was not statistically observed for the chroma parameter (P>0.05). For the lightness and hue angle characteristics, there were no statistical differences between the “Xoi Gac” produced using FD powder and VD powder. Furthermore, “Xoi Gac” produced from SD powder was lighter and had a less orange-red colour, indicated by higher lightness and hue angle values, than that produced from FD and VD powders. Generally, the colour of “Xoi Gac” produced from these powders has an attractive orange-red colour.

Total carotenoid content and total antioxidant activity of “Xoi Gac” was statistically influenced by the different powders used in production, at P<0.05 and P<0.01, respectively. Similarly to the colour characteristics, there were no significant difference of TCC and TAA of “Xoi Gac” produced from FD and VD powders. However, their values were much higher than those for the samples produced from SD powder. Furthermore, according to a Pearson correlation test, the strong correlation between TCC and TAA (r=0.92, P<0.05) in “Xoi Gac” was positively established. This means that the high content of carotenoids in “Xoi Gac” resulted in a high level of TAA.

As can be seen from the results in Table 7.1, one serve of about 300 g of “Xoi Gac” contains approximately 5 to 9 mg of carotenoids, comprising mainly β-carotene and lycopene. The recommended daily intake levels of carotenoids, lycopene and β-carotene are 0.7 - 16.5 mg (Müller, 1996), 5 - 7 mg (Rao & Rao, 2007), and 0.2 - 9.7 mg (Müller, 1996), respectively. Furthermore, many studies indicate that consumption of carotenoid-rich fruits and vegetables, especially lycopene, has been linked with lower risk of prostate cancer (Guns & Cowell, 2005; Chan et al., 2009). Similarly, Vuong et al. (2002) report that the incidence of vitamin A deficiency was reduced by consumption of carotenoid-rich foods, particularly β-carotene. Therefore, having established that there are high levels of carotenoids and TAA in the “Xoi Gac”, this product is highly recommended.

7.5.2 Stability of powders for use in production of Gac fruit juice

7.5.2.1 Colour characteristics of Gac juice

Table 7.2 shows the colour characteristics and total colour difference (ΔE) of Gac fruit juice before pasteurisation (control) and after pasteurising at a “cold region” temperature of 90°C for 5 minutes and at a temperature of 80°C for 30 minutes.
The lightness, chroma and hue angle of Gac powder juice were not statistically impacted by heating pasteurisation at the temperatures of 90°C for 5 minutes and 80°C for 30 minutes (P>0.05). This means that the colour characteristic of the Gac powder juice did not change after heating at those temperatures and times. Similar results of a small increase in lightness and hue angle were also reported for pasteurised orange juice (Lee & Coates, 2003) and pasteurised red grapefruit juice (Lee & Coates, 1999). Generally, after pasteurisation, the lightness of the Gac juices slightly increased, indicating that the colour of the juices became lighter and brighter. The hue angle of FD and VD powder juices also slightly increased, indicating less red colour, whereas a slight decrease in hue angle of SD powder juice was insignificantly observed.

### Table 7.2 Colour characteristics of Gac juice

<table>
<thead>
<tr>
<th>Gac juice samples</th>
<th>Lightness</th>
<th>Chroma</th>
<th>Hue angle</th>
<th>ΔE</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>From FD powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>53.90±1.71a</td>
<td>25.12±1.16a</td>
<td>39.84±1.11a</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>54.98±1.04a</td>
<td>25.48±1.57a</td>
<td>40.17±0.23a</td>
<td>1.75±0.3a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>55.13±1.25a</td>
<td>22.92±0.49a</td>
<td>43.42±2.00a</td>
<td>2.97±1.31a</td>
</tr>
<tr>
<td><strong>From VD powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>52.72±1.48a</td>
<td>23.83±2.52a</td>
<td>39.59±2.28a</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>52.37±1.07a</td>
<td>24.40±0.69a</td>
<td>40.86±2.12a</td>
<td>1.36±0.26a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>53.75±2.07a</td>
<td>23.59±1.04a</td>
<td>41.67±0.35a</td>
<td>1.90±1.30a</td>
</tr>
<tr>
<td><strong>From SD powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>61.25±1.82b</td>
<td>20.19±1.72b</td>
<td>50.90±3.77b</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>61.83±0.30b</td>
<td>20.53±0.23b</td>
<td>50.64±0.23b</td>
<td>0.73±0.13a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>61.53±1.85b</td>
<td>19.51±1.25b</td>
<td>49.31±1.15b</td>
<td>1.83±0.06a</td>
</tr>
</tbody>
</table>

The values in the same column followed by different superscripts (a-b) were significantly different (P<0.05)

However, the results show that the different powders used in production significantly influenced the colour characteristics of the juice. There was no difference in the colour characteristics of the fruit juices produced from FD and VD powders. Moreover, there was no statistical interaction between powder and heating treatment (P>0.05). From
Table 7.2, it can be clearly seen that the colour of the fruit juice produced from SD powder was lighter and less red in colour than the other juices.

Chroma value of all juice products slightly increased after pasteurisation at a temperature of 90°C for 5 minutes, indicating that the colour of the juice products was more intensive. By contrast, the colour intensity of the Gac powder juices decreased after pasteurised at a temperature of 80°C for 30 minutes. In a comparison of the different powders used in production, there was no significant difference of chroma value between FD and VD powder juices. Moreover, statistical analysis shows that these values were significantly higher than that of SD powder juice.

For the ∆E value, no significant difference between the fruit juices produced from the three powders and for the different heating treatments was statistically observed (P>0.05). The ∆E values, which indicate the magnitude of the colour difference between the control juice and the pasteurised juices, varied from 0.73±0.13 to 2.97±1.31. This is in agreement with the results of Lee and Coates (2003), who stated that the total colour difference of Valencia orange juice after pasteurisation at a temperature of 90°C for 30 seconds was 2.92±0.98. However, for all of the Gac powder juices, the total colour difference when pasteurised at the high temperature of 90°C for the short time of 5 minutes was less than that when processed at the lower temperature of 80°C for the longer time of 30 minutes, as indicated by lower ∆E values (Table 7.2). From these findings it can be concluded that the colour shift of the juices pasteurised at a temperature of 90°C for 5 minutes was minimal as compared to those treated at the temperature of 80°C for 30 minutes.

7.5.2.2 Total carotenoid content and total antioxidant activity of Gac juice

Total carotenoid content and total antioxidant activity of the fruit juices are presented in Table 7.3. It can be generally seen that TCC and TAA of the fruit juices produced from FD and VD Gac powders were higher than in the juice produced from SD Gac powder.

TCC and TAA of the fruit juices were statistically affected by the two tested factors, the type of powder used in production and the heating treatments (P<0.001 and P<0.05, respectively). In a comparison of the two pasteurisation treatments, there were no significant differences in TCC and TAA of the Gac powder juices when treated at the temperatures of 90°C and 80°C. However, the TCC of the samples pasteurised at the temperature of 80°C was significantly lower than in the control samples. The main
reactions causing loss of carotenoids in juice products are isomerisation and oxidation (Zepka & Mercadante, 2009). TCC differences between the Gac juice samples pasteurised at the temperature of 90°C and the control juices were not statistically observed. By contrast, TAA of all samples pasteurised at each of the two temperatures was statistically lower than the TAA of the control juices. Therefore, it can be concluded that significant loss of TCC and TAA of the fruit juices was observed when heating treatments were applied. However, the loss was greater as a result of pasteurisation at the lower temperature of 80°C with the longer time of 30 minutes as compared to the higher temperature of 90°C and short time of 5 minutes. Likewise, Lin and Chen (2005a) suggested that high temperature/short time (HTST) treatment should be applied for processing tomato juice due to the resultant high stability of carotenoids.

Table 7.3 Total carotenoid content and total antioxidant activity (ABTS assay) of Gac juice

<table>
<thead>
<tr>
<th>Gac juice samples</th>
<th>TCC  µg/ mL</th>
<th>TAA µmole TE/ mL</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>From FD powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>68.94±2.37a</td>
<td>28.36±2.75a</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>66.82±2.20ab</td>
<td>27.47±1.70b</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>63.69±5.70b</td>
<td>25.47±1.70b</td>
</tr>
<tr>
<td><strong>From VD powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>67.92±1.90a</td>
<td>27.49±1.34a</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>65.90±2.39ab</td>
<td>24.04±2.97b</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>64.34±3.15b</td>
<td>23.09±3.53b</td>
</tr>
<tr>
<td><strong>From SD powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>56.12±1.63c</td>
<td>22.29±3.08c</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>54.87±2.26cd</td>
<td>15.07±0.71d</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>48.39±4.50d</td>
<td>14.24±0.95d</td>
</tr>
</tbody>
</table>

The values in the same column followed by different superscripts (a-d) were significantly different (P<0.05)

In this study, thermal treatment at 90°C for 5 minutes did not cause significant reduction of total carotenoid content and its antioxidant activity in the pasteurised reconstituted Gac juices in comparison to the control samples. Similar results were also found by Lee and Coates (1999) who stated that β-carotene and lycopene content in red grapefruit juice did not change after pasteurisation at 91°C for 10 seconds. This is due to the high stability of these carotenoid components when subjected to heat processing. Similarly, Gama and de Sylos (2007) indicated that loss of TCC in
pasteurised Brazilian Valencia orange juice compared to the juice before pasteurisation was not significant, at around 13%.

However, TCC of the Gac powder juices significantly reduced when the juices were pasteurised at the lower temperature of 80°C for 30 minutes. In fact, different reduction behaviour for TCC has been observed depending on the thermal treatment applied and the different kinds of fruit juices tested. Lee and Coates (2003) showed that TCC of Valencia orange juice pasteurised at 90°C for 30 seconds decreased in comparison to juice before pasteurisation, at around 10% loss. Likewise, a significant loss of TCC (17%) in concentrated acerola fruit juice pasteurised at 110°C for 25 seconds was observed. The considerable loss increased further as the pasteurising time was increased up to 85 seconds (Mezadri et al., 2005).

As expected, significant loss of TCC in fruit juices after pasteurisation has been shown to lead to a reduction of TAA. In this study, a similar pattern to the loss of TCC in the Gac juices was observed for the TAA; however, the percentage loss of TAA in the juices was higher than that of TCC loss. After heating treatments, the loss of TCC in the juices varied from about 2% to 14%, whereas the reduction of TAA was from 3% to 36%. Furthermore, in a comparison of the juices produced from different Gac powders, the loss of TAA in FD powder juice was lowest, followed by the loss in the VD powder juice, with the SD powder juice showing the greatest loss of TAA. Thus, the loss of TCC in SD powder juice at each of the two pasteurisation temperatures was much higher than that of FD and VD powder juices.

7.5.2.3 Colour characteristics of Gac powder juices during storage

During the storage period of 30 days under refrigeration, the colour characteristics of the Gac powder juices pasteurised at a temperature of 90°C for 5 minutes were periodically evaluated. The results for lightness, chroma, hue angle, and total colour difference are shown in Figure 7.1a, 7.1b, 7.1c, and 7.1d, respectively.
Figure 7.1a Lightness of Gac juices during the storage period

Figure 7.1b Chroma of Gac juices during the storage period
Figure 7.1c Hue angle of Gac juices during the storage period

Figure 7.1d Total colour difference of Gac juices during the storage period

(ΔE calculated by comparing with the fresh juice before pasteurisation)
Generally, the colour characteristics (lightness, chroma and hue angle) of the Gac powder juice products were significantly affected by the type of powder used and the storage time. Moreover, increasing the storage time resulted in a slight increase in lightness, hue angle and \( \Delta E \) values of all juices; whereas a decrease in chroma value was observed. The lightness and hue angle values of the SD powder juice were highest, as indicated by a lighter and less red colour, followed by the FD powder juice and VD powder juice. However, no statistical difference of hue angle between FD and VD powder juices was significantly observed. A similar result was also found for \( \Delta E \) values. The chroma of juice samples produced from FD powder was highest, followed by that for VD powder juice and SD powder juice.

Similarly, Aguiló-Aguayo et al. (2008) and Odriozola-Serrano et al. (2009) found that the colour of pasteurised tomato juice was significantly lighter and less red, indicated by a higher L value and higher hue angle value, as storage time was increased over a test period of 56 days. Therefore, it could be suggested that the colour difference changed toward a less attractive visual appearance during storage due to the effects of environmental conditions such as light, oxygen and temperature.

However, in this study the colour difference of the Gac powder juices, stored under the refrigerated conditions for the 30 day period, can be considered small and the resultant juices identical in terms of visual observation, as indicated by \( \Delta E \) values which are lower than 3 units. According to Obón et al. (2009), the total colour difference between fresh and processed \textit{Opuntia stricta} (also known as prickly pear) fruit juices can be generally distinguished when the \( \Delta E \) value is higher than 5 units. Therefore, it can be concluded that the colour of the Gac juices insignificantly changed under storage at a temperature of 4\(^\circ\)C for up to 30 days.

7.5.2.4 Total carotenoid content and antioxidant activity of Gac juices during storage

Figures 7.2 and 7.3 show effects of different storage times on TCC and TAA of the fruit juices produced from the three types of Gac powder. Statistical results showed that different effects of three powders and storage times on total carotenoid content and total antioxidant activity was significantly indicated (P<0.01).

In general, TCC and TAA of the fruit juices produced from all powder types decreased as storage time increased. Furthermore, in terms of TCC, there was no significant
difference between the juice produced from FD and VD powders over the total storage period. However, TCC of SD powder juice was lower than that of both VD and FD powder juices. On the other hand, difference in TAA of the juices produced from the three powder types was significantly observed. Throughout the storage period, TAA of FD powder juice was highest, followed by that of VD powder and SD powder juices.

![Figure 7.2 Total carotenoid content of Gac juices during the storage period](image)

The loss of carotenoid content in the Gac powder juices during storage is mainly due to auto-oxidation, photo-oxidation and photo-isomerisation. These reactions can occur simultaneously and competitively in the presence of a catalyst, oxygen and light intensity. Furthermore, the oxidation severity depends on the carotenoid structure and environmental conditions (Chen et al., 1996; Odriozola-Serrano et al., 2009). Therefore, these oxidative reactions were the cause of a reduction of TCC and TAA of the Gac fruit juice samples during the storage period, and led to a loss of colour.

After refrigeration storage for up to 30 days, the loss of TCC in the Gac juices ranged from 18 to 24%, while the TAA loss was 26 to 41%. However, measurements for TCC of the Gac juices after 30 days of storage (55, 53 and 42 µg/mL for FD, VD and SD juices, respectively) are still higher than those reported for some other pasteurised fruit
and vegetable juices. For example, several studies reported that TCC of commercial tomato juices (Sánchez-Moreno et al., 2006), Caja juice (Hamano & Mercadante, 2001), concentrated acerola fruit juice (Mezadri et al., 2005) and orange juice (Lee & Coates, 2003) was approximately 32 (µg/mL), 17 (µg/g), 12 (µg/g) and 6 (µg/mL), respectively, which are much lower TCC amounts than in the experimental juices in this study. Another study found that TCC of commercial tomato juice in glass bottles ranged from 45 to 56 µg/g (Podsedek et al., 2003); the TCC in the Gac powder juice stored at 4±2°C for 30 days is comparative to these particular tomato juice products.

By contrast, according to Odriozola-Serrano et al. (2009), in their study the TCC in pasteurised tomato juices is about 114-116 µg/mL after storage at 4°C for 28 days. The explanation for these differences is due to different processing methods and concentrations of the various juices, which may differently affect the TCC of the products. In this study, the amount of Gac powder used to produce the juice samples for testing is known to be small. Furthermore, a loss of TCC may have occurred during the filtration step in the initial processing of the Gac juice. Therefore, it seems that further research could be performed to determine methods for reduction of TCC loss in Gac juices produced from powders.

![Figure 7.3 Total antioxidant activity (ABTS assay) of the Gac juices during the storage period](image_url)
In a comparison of total antioxidant activity, a higher TAA in the experimental Gac juices was observed as compared to commercial tomato juice products. Mean values of TAA in FD, VD and SD powder juices after 30 day of storage were 19.61, 17.74 and 8.97, respectively (Figure 7.3). Podsdek et al. (2003) reported that TAA (using an ABTS assay) in commercial tomato juice products was about 1.2 to 2.1 µmole TE/g. Thus, although TCC in the tomato juices and those of the Gac juices in this study are very similar, TAA in the experimental Gac juices was found to about 4 to 16 times higher than results reported for the commercial tomato juice. The higher TAA results could be explained by the fact that Gac fruit also contains high levels of \( \alpha \)-tocopherol (vitamin E) (Vuong et al., 2006), which is well-known as a strong antioxidative compound that could be enhancing TAA in the Gac juices. Furthermore, several studies found synergism of different antioxidants being greater than the sum of individual antioxidant activities (Amao et al., 1998; Stahl & Sies, 2003). Therefore, synergism of these antioxidative compounds could be an explanation for a higher level of TAA in the Gac juices.

7.5.2.5 Kinetics of colour, TCC and TAA degradation of Gac juices during storage

Kinetic parameters of first-order degradation with respect to colour, TCC and TAA in the Gac juices are shown in Table 7.4, Table 7.5 and Table 7.6, respectively. As can be seen from the results in Table 7.4, the colour degradation rate, as indicated by lightness, was very low, leading to a high half life value. As such, this indicates that the Gac juice colour would be more stable over an extended storage time. However, poor correlation coefficients for chroma and hue angle were observed. These results mean that the colour degradation of the juices during storage was not fitted by a first-order reaction.

In contrast, the degradation of TCC and TAA of the Gac juices produced with all types of powder was best fitted by a first-order kinetic model, supported by a high correlation coefficient (R²>0.95). Similarly, Dhuique-Mayer et al. (2007) also reported that a first-order reaction for carotenoids in citrus juices was suggested by the results of their study. In a comparison of the three powders used for production of the Gac juice, the highest degradation rate of TCC and TAA and the lowest half life were found in the SD powder juice (Tables 7.5 and 7.6). Results indicated that the breakdown of carotenoids and antioxidant activity in SD powder juice was much faster than in VD and FD powder juices.
Table 7.4 Kinetic parameters of first-order colour degradation in Gac juice

<table>
<thead>
<tr>
<th>Gac juice samples</th>
<th>Degradation rate (day⁻¹)</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R²)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Lightness</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0007</td>
<td>990.21</td>
<td>0.9982</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0006</td>
<td>1155.25</td>
<td>0.7629</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0004</td>
<td>1732.87</td>
<td>0.8948</td>
</tr>
<tr>
<td><strong>Chroma</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0021</td>
<td>330.07</td>
<td>0.4691</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0007</td>
<td>990.21</td>
<td>0.7798</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0008</td>
<td>866.43</td>
<td>0.3876</td>
</tr>
<tr>
<td><strong>Hue angle</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0005</td>
<td>1386.29</td>
<td>0.2818</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0016</td>
<td>433.27</td>
<td>0.6415</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0002</td>
<td>3465.74</td>
<td>0.3202</td>
</tr>
</tbody>
</table>

Table 7.5 Kinetic parameters of first-order TCC degradation in Gac juice

<table>
<thead>
<tr>
<th>Gac juice samples</th>
<th>Degradation rate (day⁻¹)</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder</td>
<td>0.0064</td>
<td>108.30</td>
<td>0.9883</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0072</td>
<td>96.27</td>
<td>0.9599</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0085</td>
<td>81.55</td>
<td>0.9940</td>
</tr>
</tbody>
</table>

Table 7.6 Kinetic parameters of first-order TAA degradation in Gac juice

<table>
<thead>
<tr>
<th>Gac juice samples</th>
<th>Degradation rate (day⁻¹)</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder</td>
<td>0.0117</td>
<td>59.24</td>
<td>0.9851</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0104</td>
<td>66.65</td>
<td>0.9535</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0152</td>
<td>45.60</td>
<td>0.9903</td>
</tr>
</tbody>
</table>
7.5.3 Stability of powders for use in production of pasteurised Gac milk beverage

7.5.3.1 Colour characteristics of Gac milk beverages

The colour characteristics of pasteurised Gac powder milk beverages are shown in Table 7.7. Generally, the lightness, chroma and hue angle were significantly affected by type of powder used in production of the beverage (P<0.01), but not by the type of heat treatment (P>0.05). No significant difference in the colour characteristics between the pasteurised FD and VD Gac powder milk products was statistically found (P>0.05).

<table>
<thead>
<tr>
<th>Pasteurised Gac milk beverage samples</th>
<th>Lightness</th>
<th>Chroma</th>
<th>Hue angle</th>
<th>∆E</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Freeze-dried powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>71.08±3.10a</td>
<td>34.80±0.98a</td>
<td>61.07±1.38a</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 minutes</td>
<td>71.12±2.19a</td>
<td>34.14±0.23a</td>
<td>61.82±1.69a</td>
<td>1.89±0.21a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 minutes</td>
<td>71.27±2.83a</td>
<td>34.54±2.93a</td>
<td>61.57±0.61a</td>
<td>2.92±0.11b</td>
</tr>
<tr>
<td><strong>Vacuum-dried powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>68.62±1.57a</td>
<td>35.47±2.15a</td>
<td>62.39±0.97a</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 minutes</td>
<td>69.65±0.90a</td>
<td>35.17±2.31a</td>
<td>62.41±2.30a</td>
<td>2.28±0.14a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 minutes</td>
<td>70.57±2.69a</td>
<td>34.50±4.34a</td>
<td>62.47±1.69a</td>
<td>4.27±0.27b</td>
</tr>
<tr>
<td><strong>Spray-dried powder</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>78.54±2.45b</td>
<td>27.47±3.92b</td>
<td>69.55±1.47b</td>
<td></td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 minutes</td>
<td>78.37±0.04b</td>
<td>27.99±0.27b</td>
<td>71.04±0.04b</td>
<td>0.97±0.14a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 minutes</td>
<td>81.33±1.61b</td>
<td>29.89±1.06b</td>
<td>70.27±2.05b</td>
<td>3.83±1.73b</td>
</tr>
</tbody>
</table>

The values in the same column followed by different superscripts (a-b) were significantly different (P<0.05)

As compared to the control Gac milk beverages, total colour difference of the pasteurised beverages was statistically influenced by the heating treatment (P<0.01), but not by the type of powder used (P>0.05). The ∆E value of the products heated at 90°C was significantly lower than those heated at 80°C. As shown in the results in Table 7.7, the ∆E value of the Gac milk beverage products pasteurised at 90°C varied from 0.97 to 2.28 units, whereas a ∆E value range of 2.92 to 4.27 was found for the products pasteurised at 80°C. Generally, a higher ∆E value is due to increased
lightness and hue angle values, indicating a product which is lighter and less red in colour. Thus, the results indicate that the colour of the Gac milk beverage was less changed when pasteurised at a high temperature of 90°C as compared to a lower temperature of 80°C.

There are a number of commercial fruit juice and skim milk mixture beverages, such as peach, apricot, carrot, guava and passion fruit, which are presently marketed in Spain. According to research by Zuleta et al. (2007b), these beverages come in a variety of different colours, indicated by a large range of hue angle values from 35.47 to 115.57. The fruit and vegetable juice-milk products which were typically observed to have a yellow-red colour exhibited hue angle values of less than 90. Therefore, from the commercial point of view, the mixture beverages of Gac fruit powder and milk, which have an attractive yellow-red colour, could be competitively marketed.

### 7.5.3.2 Total carotenoid content and total antioxidant activity of Gac milk beverages

The total carotenoid content and total antioxidant activity of the Gac fruit-milk mixture beverage products pasteurised at the different temperatures of 90°C and 80°C are shown in Table 7.8. A significant effect of the heating treatment and different powders used on TCC of the Gac milk mixture products was statistically observed, at P<0.05 and P<0.001, respectively. Statistical analysis also indicates that there was no significant difference in TCC between the products pasteurised at temperatures of 90°C and 80°C and the Gac milk beverage before heating. However, the TCC of the products pasteurised at temperature of 80°C was lower than that of the control. Moreover, no statistical difference between the TCC or the TAA of the FD and VD milk mixture products was indicated; and in each case these were higher than those in the SD milk mixture. In terms of TAA, the powder milk products were significantly impacted by the type of powder (P<0.01), but not by the heating treatment (P>0.05). No interaction between the powder type and the heating treatment was statistically indicated (P>0.05).
Table 7.8 Total carotenoid content and total antioxidant activity (ABTS assay) of pasteurised Gac milk beverages

<table>
<thead>
<tr>
<th>Pasteurised Gac milk beverage samples</th>
<th>TCC µg/ mL</th>
<th>TAA µmole TE/ mL</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Freeze-dried powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>60.49±1.24a</td>
<td>21.42±1.77a</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>58.14±2.31ab</td>
<td>19.15±2.17a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>57.15±3.08b</td>
<td>18.12±1.73a</td>
</tr>
<tr>
<td><strong>Vacuum-dried powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>59.29±2.29a</td>
<td>19.21±2.14a</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>57.59±0.94ab</td>
<td>17.41±1.72a</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>54.89±2.29b</td>
<td>17.19±1.27a</td>
</tr>
<tr>
<td><strong>Spray-dried powder</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>44.83±1.53c</td>
<td>15.06±2.00b</td>
</tr>
<tr>
<td>Pasteurised at 90°C for 5 mins</td>
<td>42.43±2.63cd</td>
<td>12.78±2.45b</td>
</tr>
<tr>
<td>Pasteurised at 80°C for 30 mins</td>
<td>41.89±1.87d</td>
<td>11.88±1.13b</td>
</tr>
</tbody>
</table>

The values in the same column followed by different superscripts (a-d) were significantly different (P<0.05)

A higher TAA in the experimental Gac milk beverages was observed, with a range from 11.88 to 19.15 µmole TE/mL, when compared with several commercial fruit juice and milk mixture beverages with a reported TAA range of 0.61 to 3.6 µmole TE/mL (Zulueta et al., 2007a). Furthermore, these authors reported that no significant difference in TAA between the commercial pasteurised products stored under refrigeration and the samples sterilised by UHT (Ultra high temperature) treatment and stored at room temperature was statistically indicated. However, the TAA in pasteurised samples was slightly higher than that of the UHT samples. From these results, it could be concluded that the pasteurised Gac powder milk beverage products are able to maintain nutritional value in terms of TAA.

7.5.3.3 Colour characteristics of Gac milk beverages during storage

The differences in the colour characteristics of the pasteurised Gac milk beverages during the storage period of 30 days are presented in Figures 7.4a, 7.4b, 7.4c and 7.4d.
Figure 7.4a Lightness of pasteurised Gac milk beverage during storage

Figure 7.4b Chroma of pasteurised Gac milk beverage during storage
Figure 7.4c Hue angle of pasteurised Gac milk beverage during storage

Figure 7.4d Total colour difference of pasteurised Gac milk beverage during storage

(ΔE calculated by comparing with the fresh beverage before pasteurisation)
The lightness, chroma, hue angle and $\Delta E$ values of the pasteurised Gac milk beverages were significantly influenced by the different powders ($P<0.001$). In addition, the storage time also statistically influenced the lightness ($P<0.01$), hue angle ($P<0.01$) and $\Delta E$ ($P<0.001$), but not the chroma ($P>0.05$). Generally, for all samples, an increase in lightness, hue angle and $\Delta E$ was significantly observed as storage time increased. These results indicate that the samples were lighter, becoming pale red as storage time increased.

In terms of the different powders used for production of the Gac milk beverages, the highest lightness, the lowest chroma and the highest hue angle of samples were observed in the beverages produced from SD powder. Additionally, no significant difference in hue angle between the VD and the FD powder milk products was statistically observed. Measuring the total colour difference, after the storage time of 30 days, $\Delta E$ value of the SD powder milk product samples was lowest, indicating less difference of colour, followed by the FD and VD powder milk products. At the end of the storage period the mean of $\Delta E$ values of the Gac fruit-milk products ranged from 2.56 to 4.18 units, all of which are lower than 5 units. Therefore, similarly to the results indicated for the Gac juices, a visual colour observation of these Gac milk beverage products did not indicate a significant difference after storage of up to 30 days under refrigeration.

7.5.3.4 Total carotenoid content and antioxidant activity of Gac milk beverages during storage

As can be seen from the results in Figures 7.5 and 7.6, the TCC and TAA of the Gac milk beverages were statistically influenced by two factors, the type of powder used in production and the storage time ($P<0.001$). A significant reduction of TCC and TAA in the all of the products was found as storage time increased, regardless of the powder type used in processing. Furthermore, while the TCC and TAA of both the FD and VD powder milk beverages were significantly higher than those of SD powder milk beverage, there were no differences in TCC and TAA between FD and VD powder milk products during storage under refrigeration.

After storage under refrigeration for 30 days, the mean value of TCC in the three types of experimental Gac powder milk products was approximately 51, 50 and 26 µg/mL for products produced using FD, VD and SD Gac fruit powder, respectively. When compared to pasteurised commercial fruit juice and milk mixtures studied in other
research, these results were much higher than those for the commercial products, where TCC varied from 2 to 7 µg/g (Zulueta et al., 2007b).

The difference in the results between that study and the current research could be explained if the initial TCC in the commercial fruit juices added into milk is lower than that of the Gac powders. Furthermore, thermal pasteurisation conditions and storage conditions can also influence the TCC in the final products. Similarly to patterns recorded for TCC values, the TAA in the experimental Gac powder milk products was also much higher than the reported values for the commercial products after storage under refrigeration for up to 30 days (Zulueta et al., 2007a). Therefore, from the recorded results in this study, it seems that Gac fruit powders could be used for production of Gac fruit milk beverages to enhance TCC as well as TAA of the products.

![Figure 7.5 Total carotenoid content of pasteurised Gac milk beverage during the storage period](image-url)
7.5.3.5 Kinetics of colour, TCC and TAA degradation of Gac milk beverage during storage

As can be seen in Table 7.9, the lightness degradation of pasteurised Gac powder-milk beverages was strongly fitted by a first-order reaction, supported by the high correlation coefficient ($R^2>0.9$). A lower degradation rate and a higher half life of both FD and SD powder-milk products was observed as compared to VD powder-milk products. This indicates that the lightness of FD and SD powder-milk samples was more stable during storage.

It was also observed that the chroma of FD and SD powder milk beverage samples was poorly fitted by a first-order reaction, indicated by a low correlation coefficient ($R^2<0.5$). However, a high correlation coefficient was found for hue angle degradation of these samples, which means that the hue angle of the samples was strongly fitted by a first-order degradation reaction. In comparison to the samples produced using FD and SD powders, the hue angle of VD powder milk beverage was very poorly fitted by the degradation reaction ($R^2<0.2$).
Table 7.9 Kinetic parameters of first-order colour degradation in pasteurised Gac milk beverages

<table>
<thead>
<tr>
<th>Pasteurised Gac milk beverage samples</th>
<th>Degradation rate (day(^{-1}))</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Lightness</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0006</td>
<td>1155.25</td>
<td>0.9798</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0010</td>
<td>693.15</td>
<td>0.9967</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0006</td>
<td>1155.25</td>
<td>0.9139</td>
</tr>
<tr>
<td><strong>Chroma</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0003</td>
<td>2310.49</td>
<td>0.1312</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0009</td>
<td>770.16</td>
<td>0.7022</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0002</td>
<td>3465.74</td>
<td>0.4887</td>
</tr>
<tr>
<td><strong>Hue angle</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FD powder</td>
<td>0.0006</td>
<td>1155.25</td>
<td>0.9554</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0001</td>
<td>6931.47</td>
<td>0.1116</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0009</td>
<td>770.16</td>
<td>0.8278</td>
</tr>
</tbody>
</table>

From the results discussed above, it can be concluded that the colour of FD powder milk beverage strongly followed the first-order degradation reaction, and is thus more stable than SD and VD powder-milk beverages. This is supported by the lower degradation rate, higher half life value and higher correlation coefficient.

Tables 7.10 and Table 7.11 show the kinetic parameters for first-order degradation of TCC and TAA, respectively, in the samples of Gac milk beverages. The results indicate that the degradation rates of TCC and TAA of the SD powder-milk beverage was much higher than that of the FD and VD powder-milk beverages. Therefore, the shelf life of SD powder-milk product in terms of retention of TCC and TAA is shorter than the others. Furthermore, all samples were strongly fitted by a first-order degradation reaction (R\(^2\)>0.9). These findings are consistent with the results for pasteurised Gac fruit juice.
Table 7.10 Kinetic parameters of first-order TCC degradation in pasteurised Gac fruit-milk beverages

<table>
<thead>
<tr>
<th>Pasteurised Gac milk beverage samples</th>
<th>Degradation rate (day(^{-1}))</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder</td>
<td>0.0048</td>
<td>144.41</td>
<td>0.9557</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0046</td>
<td>150.68</td>
<td>0.9536</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0125</td>
<td>55.45</td>
<td>0.9260</td>
</tr>
</tbody>
</table>

Table 7.11 Kinetic parameters of first-order TAA degradation in pasteurised Gac fruit-milk beverages

<table>
<thead>
<tr>
<th>Pasteurised products</th>
<th>Degradation rate (day(^{-1}))</th>
<th>Half life (days)</th>
<th>Correlation coefficient (R(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>FD powder</td>
<td>0.0132</td>
<td>52.51</td>
<td>0.9168</td>
</tr>
<tr>
<td>VD powder</td>
<td>0.0126</td>
<td>55.01</td>
<td>0.9857</td>
</tr>
<tr>
<td>SD powder</td>
<td>0.0182</td>
<td>38.09</td>
<td>0.9663</td>
</tr>
</tbody>
</table>

7.6 Conclusions

The effect of thermal pasteurisation on the colour characteristics, total carotenoid content and total antioxidant activity of Gac fruit juices and Gac milk beverages, produced using three types of Gac fruit powders, was investigated. Results showed that there was no significant difference in the colour of the beverage products as a result of applying different heating conditions during pasteurisation. However, when compared to samples of the beverage before heating treatment, the total colour difference of the Gac milk beverage pasteurised at 90°C for 5 mins was significantly less than the total colour difference of that pasteurised at 80°C for 30 mins. Moreover, TCC of the fruit juices and the milk beverages did not change after pasteurising at a temperature of 90°C, however, a significant loss was found in the products pasteurised at 80°C. In respect to the TAA of the samples after thermal treatments, a significant loss was observed in the fruit juices, but not in the Gac milk beverages.

Further testing was carried out periodically during a 30 day period when samples of all of the Gac fruit juices and Gac fruit milk beverages were stored under refrigeration at
4±2°C. The loss of TCC and TAA in the fruit juices and Gac milk products increased with increasing storage time. The degradation rate of TCC and TAA of the Gac fruit juice and Gac fruit milk beverage was best fitted by a first-order reaction.

Results of the experiments showed that freeze-dried, vacuum-dried and spray-dried Gac fruit powders were easily incorporated into the rice dish “Xoi Gac” and the pasteurised reconstituted fruit juice and Gac milk beverages. Examples of some of these experimental products produced from freeze-dried Gac powders are shown in Figure 7.7. The stability of these products was found to be satisfactory, in terms of retention of colour, TCC and TAA during the storage period of 30 days.
“Xoi Gac”
prepared with Freeze-dried powder

Pasteurised Gac fruit juice
from Freeze-dried powder,
before storage

Pasteurised Gac milk beverage
from Freeze-dried powder,
before storage

Figure 7.7 Samples of food and beverage products from Gac powders
Chapter 8
CONCLUSIONS AND RECOMMENDATIONS

8.1 Conclusions

Fresh and frozen Gac fruits were analysed in terms of colour, total carotenoid content (TCC) and total antioxidant activity (TAA) were dried using a variety of drying processes to produce powders. The effects of different pre-treatments and drying processes on the physicochemical and antioxidant properties of the resultant Gac fruit powders were investigated.

In a comparison of air, vacuum and freeze drying processes, the freeze drying process resulted in superior powder products in terms of colour parameters, TCC and TAA. Results showed that pre-treatments, of soaking the Gac aril in 1% w/v ascorbic acid or 1% w/v sodium bisulfite solution, prior to air drying or vacuum drying at a low temperature of 40°C, preserved the TCC and TAA of Gac fruit powder. Vacuum drying at temperature of 40°C for 45 hours, after pre-soaking in 1% w/v ascorbic or bisulfite solution, was just as effective in preserving colour, TCC and TAA as freeze drying. Furthermore, a significant loss of TCC and TAA was found as the drying temperature increased from 40 to 80°C. Therefore, to preserve carotenoid content and its antioxidant activities, the pre-treatment of Gac aril with ascorbic acid or bisulfite and drying (air drying and vacuum drying) at low temperature are highly recommended. The final choice for the most suitable drying method for the production of a high quality Gac powder may be influenced by the costs involved in the drying method including the availability of equipment and facilities, and the consequent costs to consumers.

In a comparison between frozen and fresh Gac arils, a redder colour and lower total carotenoid content of the powder produced from the frozen aril was observed. By contrast, the TAA in the aril powder produced from the untreated fresh fruit was lower than that in frozen aril powder when analysed using an ABTS assay, but no difference was statistically found when using a DPPH assay. Therefore, from the results of the study, it is suggested that either frozen aril or fresh aril can be used equally successfully for the production of Gac powder.
A method to successfully deal with the difficulty of grinding the dried Gac aril, due to its stickiness, was established during the study. When a paste of maltodextrin and fresh Gac aril was air-dried at a temperature of 60°C, it was easily ground to form powder. The quality of the resultant powder in terms of colour, TCC and TAA was significantly improved by the addition of maltodextrin (0.5 or 1 g maltodextrin/ g of total fruit solids).

Untreated skin and yellow pulp were found to be easily air-dried and ground to form powders. Therefore, since high levels of carotenoids were found in Gac skin and yellow pulp powders these products can be utilised at least as animal feed. This solution may also prevent disposal problems and also enhance the overall commercial value of the Gac fruit.

In relation to the spray drying process, it was clearly established by the study results that Gac aril can be spray-dried with the addition of maltodextrin to produce a valuable powder. The preservation of colour, TCC and TAA of the resultant Gac powder was most significant when the aril was spray-dried at an inlet temperature of 120°C with an added-maltodextrin concentration of 10%. These parameters were found to be lower than those of air-dried, vacuum-dried and freeze-dried powders due to higher drying temperature and high maltodextrin concentration. Additionally, several variations of the parameters of inlet temperature and maltodextrin concentration were investigated and the results showed that pH, A_w and WSI of the Gac powder were insignificantly influenced by the different spray drying conditions. However, a significant effect of the combination of inlet drying temperature and maltodextrin concentration on the moisture content, bulk density, colour characteristics, TCC, EE and TAA was statistically observed. Therefore, spray drying of Gac aril at low inlet temperature of 120°C and maltodextrin of 10% is recommended for better preserving physicochemical and antioxidant properties in the resultant powder.

Compared with commercial Gac powders, the Gac powders produced in the laboratory were significantly better quality in terms of higher levels of TCC and TAA and better colour characteristics. Therefore, it is highly recommended that the Gac aril powders produced under the suitable conditions of pre-treatment with ascorbic acid or bisulfite solution before air drying or vacuum drying at low temperature should applied for commercial production. Additionally, production of Gac aril powder from spray drying at an inlet temperature of 120°C and maltodextrin of 10% is also recommended. As a result, it is again indicated that the commercial value of fresh Gac fruit will be enhanced through processing. Depending on the suitability of processing conditions and the
desired quality of final product, a choice of the specific drying process will be determined.

In the examination of the Gac powders under the storage conditions of the study, a significant progressive degradation of colour, TAA and TCC of the FD and SD Gac aril powders was found as storage temperature increased from 10°C to 20°C, and then to 37°C, and over a longer period. The TAA of Gac aril powders packed into laminated aluminium bags was higher than that of the powders in non-laminated bags. In addition, the degradation of TCC and TAA during the 8 months storage period for FD powder and the 3 month period for SD powder was best fitted by a first-order reaction. Therefore, it can be concluded that preservation of colour, TCC and TAA in the Gac aril powders was more effective when packed into laminated bags and stored at a temperature of 10°C or lower.

The sorption isotherm curves for a variety of the Gac fruit powders were also constructed at room temperature. Results indicated that the curves of all powders have sigmoid shapes. The highest hygroscopicity was observed the in the air-dried yellow pulp powder produced at a temperature of 60°C, followed by, respectively, the freeze-dried fresh aril powder produced at a condenser temperature of -46°C, the fresh aril air-dried at 60°C, the fresh aril vacuum-dried at 60°C, the skin air-dried at 60°C, and the aril spray-dried at 120°C with added maltodextrin of 10%. For preventing hygroscopicity, air tight packaging should be applied for the Gac powders, together with desiccant if possible.

Finally, Gac fruit aril powders (specifically FD, VD and SD powders) were found to be easily incorporated into "Xoi Gac", pasteurised Gac powder juice and pasteurised Gac milk beverage. The colour, TCC and TAA of these products were evaluated before and after processing, and at intervals during a storage period. Under refrigerated storage, the reduction of TCC and TAA in the Gac fruit juices and Gac milk beverages increased with an increase in storage time. Furthermore, the degradation rate of TCC and TAA was best fitted by a first-order reaction. It can be concluded that during the storage of 30 days under refrigeration the juices and the milk beverages were found to be satisfactory in terms of maintaining an attractive colour and providing a sufficient daily intake of carotenoids. Therefore, Gac fruit aril powder can be used successfully as a natural colorant and a nutrient supplement.
8.2 Recommendations

In this thesis, the results of different drying processes and conditions for Gac fruit, the effect of different storage conditions, the stability of Gac fruit powders over time, and the incorporation Gac fruit powders into processed foods and beverages were investigated. Further investigation into the effects of encapsulating agents, other than maltodextrin, on retention of the physicochemical and antioxidant activity of spray-dried Gac powder may be carried out. In this project, the storage study was limited to only FD and SD Gac powders, therefore, further study for storage of other types of powder such as VD and AD should be performed. In addition, air drying is likely to be the cheapest process, so cost and economic evaluation for different drying processes and packages could be carried out. To encourage industrial adoption, further research into the incorporation of Gac aril powder in other processed food products, such as yoghurts and beverages, including an evaluation of the microbial and sensorial qualities of such products should be performed. Future measurement of the carotenoids by a more sophisticated approach (e.g. HPLC) will allow determination of trans/cis isomers. This information could then be used to interpret/support some of the findings discussed in this thesis.
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