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Effects of Pre-Treatments and Air Drying Temperatures on Colour and Antioxidant Properties of Gac Fruit Powder

Tuyen C. Kha, Minh H. Nguyen, and Paul D. Roach

Abstract

Gac fruit contains extraordinarily high levels of carotenoids that are well-known as strong antioxidants with an attractive yellow-orange-red colour. The aim of this study was to investigate the effects of different pre-treatments and air drying temperatures on colour characteristics, total carotenoid content (TCC) and total antioxidant activity (TAA) of resultant Gac fruit powder. Results showed that pre-soaking in solutions of ascorbic acid or bisulfite prior to air drying at low temperature of 40°C was effective in preserving TCC and TAA. Loss of TCC and TAA increased as drying temperatures increased (50, 60, 70, and 80°C). Moreover, the colour characteristics of Gac powder, such as chroma and hue angle, were not significantly affected by pre-treatments and air drying temperatures. The sorption isotherm curve of Gac aril powder has sigmoid shape.

KEYWORDS: Gac powder, antioxidant activity, carotenoids, air drying, pre-treatments, sorption isotherm

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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 (Bauernfield, 1971; Vuong, 2000; Aoki et al., 2002; Ishida et al., 2004). Gac fruits also contain significantly high levels of α-tocopherol (vitamin E) (Vuong et al., 2006) and of fatty acids (Vuong, 2000). In addition to Gac aril containing a very high nutritional content, it has also been used as a natural food colorant due to the attractive yellow-orange-red colour of its carotenoids. The use of carotenoid-based foods as natural colorant is currently receiving considerable attention from food manufacturers and consumers. It is very important therefore, to preserve or enhance these constituents in processed Gac fruit products, particularly colour, the high levels of carotenoids and the associated antioxidant activity.

At present, there are many drying techniques used for producing powders in the food industry. 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 and Yang, 2004). Compared to other drying techniques, such as spray drying, vacuum drying and freeze drying, hot air drying is well known as the simplest and cheapest drying method. The main benefits of powders made from fresh fruits and vegetables are the potential for long storage at ambient temperature and significant reduction in the costs for transportation and storage. This is particularly important for seasonal fresh fruits such as Gac fruit. Furthermore, powders are very convenient for use as flavours and colourings in food, including juices and dairy products (Fellows, 2000; Tang and Yang, 2004).

Pre-treatments prior to air drying are 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 and Hussein, 1994; Ramesh et al., 2001; Chen et al., 2007; Koca et al., 2007). Therefore, these pre-treatments should be tested to see whether they increase the retention of carotenoids in the resulting powders.

Total antioxidant activity (TAA) measurement methods are very useful to assess the relative antioxidant activities in foods and how they change after processing and storage (Arnao et al., 1998; Halliwell, 2002). TAA is also a useful tool for the provision of quality standards and comparison of different food

products (Shahidi and 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 and Singh, 2008; Moon and 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 demonstrate antioxidant activity (Schlesier et al., 2002; Moon & Shibamoto, 2009).

Papers have been published on process development of Gac powder by Tran et al. (2008) and spray drying of Gac aril by Kha et al. (2010). This paper reports on the effects of different pre-treatments and drying temperatures on the colour, total carotenoid content, total antioxidant activities and sorption isotherm of air dried Gac fruit powder.

2. Materials and Methods

2.1 Chemicals

All chemicals used in this research, being n-Hexane 95%, acetone ≥99.5%, carotene (approx. 2:1 of β:α) mixed isomers from carrots, ≥95% (HPLC) powder form, sodium bisulfite ≥99%, L-ascorbic acid 99%, potassium persulfate 99.99% metal basis, methanol spectrophotometric grade, Trolox ((S)-(-)-6-Hydroxy-2,5,7,8-Tetramethylchroman-2-Carboxylic acid, 98%), ABTS (2,2'-Azino-Bis(3-Ethylbenzthiazoline-6-Sulfonic acid) Diammonium), and DPPH (2,2-Diphenyl-1-Picrylhydrazyl), were purchased from Sigma-Aldrich Pty. Ltd.

2.2 Raw Gac fruit sources

Whole fresh Gac fruits were purchased from a local market in Hochiminh City, Vietnam. The fruit was put inside an insulated hard plastic container to avoid light and temperature exposure during transport, and used on the same day.

2.3 Pre-treatments used prior to air drying

Each whole seed aril (0.5 kg) of fresh Gac fruit was scooped out and subjected to the following treatments before air drying (AD): soaked in ascorbic acid solution (0.002 kg ascorbic acid and 2 litres distilled water) for 1 hour, soaked in sodium bisulfite solution (0.002 kg sodium bisulfite and 2 litres distilled water) for 1 hour, blanching in stainless-steel steam cooker for 3 minutes, and untreated as control. The pre-treatments are based on the methods reported in section 1 and the range of parameters was chosen based on our preliminary trials.

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2.4 Air drying process of Gac fruit

After pre-treatment, the whole seed arils were dehydrated by hot air drying. The loading of material for AD process was 5 kg/m^2. The AD process was carried out in an oven at different temperatures of 40, 50, 60, 70 and 80 ^oC with 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. Three trays were left empty to ensure uniformly hot air distribution. The moisture content of the aril samples was determined every three hours. The AD drying process was terminated when the final moisture content of each sample was constant (approximately 6%). The seed separation was carried out after each drying run. The dried Gac fruit was powdered using a lab blender and sealed in 2 g quantities into high barrier vacuum bags, using a vacuum sealer.

The two factors (pre-treatment and drying temperature) were randomly designed to investigate the effect of the air drying conditions on the colour, TCC and TAA of fresh Gac aril powder. The drying runs were carried out in triplicate. A total of 60 runs was conducted.

2.5 Analytical methods

All analytical measurements were carried out in triplicate.

2.5.1 Moisture content

The moisture content of Gac samples were determined by drying at the temperature of 105^0C in a laboratory air oven until a constant weight was obtained.

2.5.2 Colour characteristics

The colour of Gac fruit powder sample 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 measurement, the powder samples were packed into a clear polyethylene pouch and measured.

Chroma, representing colour intensity was calculated by the formula $(a^{*2} + b^{*2})^{1/2}$. The hue angle (H^o) was calculated by the formula H^o=arctan(b*/a*). The hue angle values vary from 0^o (pure red colour), 90^o (pure yellow colour), 180^o (pure green colour) to 270^o (pure blue colour) (Duangmal et al., 2008). The desirable hue angle of Gac aril powder is about 45^o.

2.5.3 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. Approximately 0.1 g of Gac powder or 0.3g of fresh Gac aril was weighted in beaker extracted with 10 mL of the solvent, which is a mixture of n-hexane:acetone (v/v 3:2). The residue was then 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 analyzed within one day.

2.5.4 Determination of total carotenoid content

Carotene (from carrots) solution (0.0005–0.01 mg/mL) was used to construct the standard curve for the determination of total carotenoid content. 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 (mg/g of powder).

2.5.5 Determination of total antioxidant activity

2.5.5.1 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.

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2.5.5.2 DPPH assay

The DPPH assay was adapted from that used by Thaipong et al. (2006). The stock solution contained 24mg DPPH and 100 mL methanol. The working solution was obtained by mixing 10mL stock solution with 45 mL methanol to obtain an absorbance of 1.1±0.02 units at 515nm using the spectrophotometer. Gac sample extracts (0.15mL) were allowed to react with 2.85mL 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.

2.6 Moisture sorption isotherms

Air-dried Gac fruit powder was sieved using a lab sieve with a mesh opening of 0.0553 inches and used for constructing moisture sorption isotherms. About 2 g of powder was 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_3COOK), potassium carbonate (K_2CO_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 103^0C for 24 hours).

The monolayer moisture content M_0 (db) was calculated using the Brunauer Emmett Teller (BET) equation as follows:

$$\frac{A_w}{(1-A_w)MC} = \frac{1}{M_0 C}$$

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.

2.7 Statistical analysis

The experiments were carried out in triplicate and results were presented as mean values with standard deviations. Different mean values (two 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.

3. Results and Discussion

3.1 Drying curve for air drying of fresh Gac fruit aril

Samples of the untreated fresh Gac aril were found to have original moisture contents ($MC_{w.b}$) ranging from 79.6 to 85.7%. The results for moisture content versus drying time are presented in Figure 1. Generally, the drying temperature had a direct effect on the moisture content and drying time. 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 hours respectively for air-dried samples at 40 ^{o}C to attain MC of 6%, while the drying time at 80 ^{o}C for air-dried samples was approximately 12 hours.

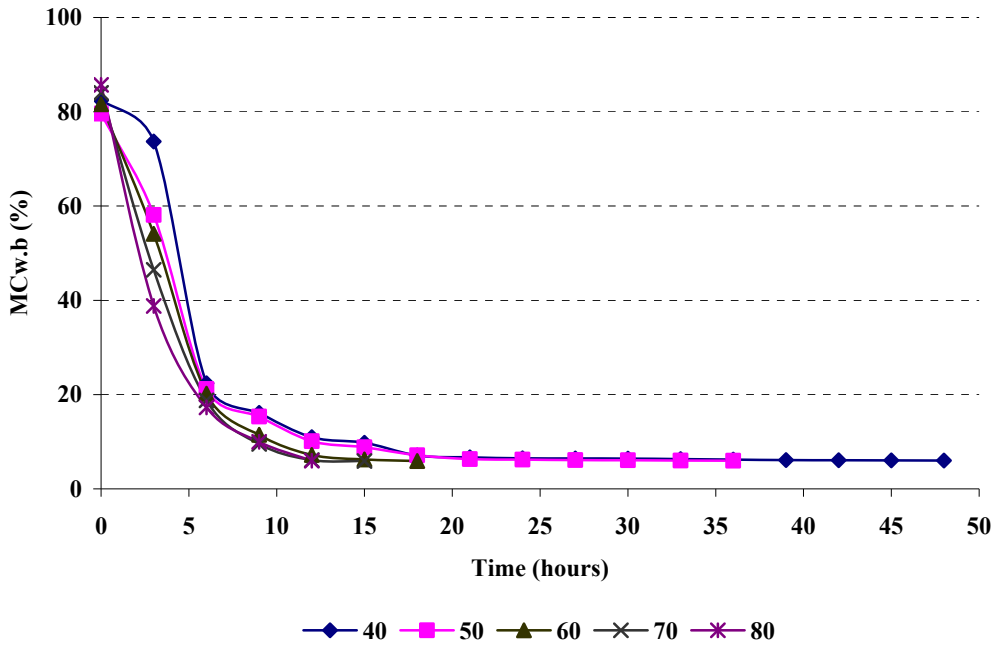

Figure 1. Drying curve of air drying at different drying temperatures

3.2 Effect of drying treatments on the colour characteristics of the Gac aril powder

The colour characteristics of powder samples from the fresh fruit aril at different pretreatments and air drying temperatures are shown in Table 1. The statistical results indicated that pre-treatments (soaking in 1% w/v bisulfite, ascorbic acid

solution and blanching) prior to air drying significantly affected the lightness of samples ($P<0.01$), however, no significant effect of drying temperature on the lightness of samples was observed ($P>0.05$). Moreover, the interaction between pre-treatments and drying temperatures on the lightness were statistically established ($P<0.01$).

In general, pre-treatments (soaking in ascorbic acid solution or blanching in steam water for 3 minutes) prior to drying did not improve the lightness of the powders in comparison with the control (untreated). This is because there was no significant difference in lightness among these samples ($P>0.05$). The highest lightness, indicating the highest L* value, was recorded in powder blanched in steam water for 3 minutes prior to drying at a temperature of 40^0C. This was consistent with the results of Piga et al. (2004), who reported that, the lightness value of blanched figs 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 higher lightness of untreated pumpkin samples was obtained. Therefore, it can be concluded that the lightness of different materials is differently affected by various pre-treatments before drying.

Table 1. Colour characteristics of Gac aril powder affected by air drying treatments

		Lightness	Chroma	Hue angle
Pre-treatment (PT)	Control	43.95±5.61[a]	28.07±2.54	41.57±3.58
	Ascorbic acid	44.30±5.08[a]	29.77±3.13	42.69±6.07
	Sodium bisulfite	38.94±4.22[b]	26.32±4.37	37.91±5.45
	Blanching	44.54±5.12[a]	28.01±4.23	41.05±4.27
Air drying temperature (ADT)	40	42.98±5.15	29.20±3.79	39.64±4.66
	50	40.00±4.86	28.44±4.94	40.28±6.44
	60	44.78±5.08	27.56±2.45	40.55±3.23
	70	43.81±7.18	28.57±4.41	42.05±5.03
	80	43.08±4.07	26.44±2.58	41.53±6.19
Significant interaction			**Significance**	
PT		**	N.S	N.S
ADT		N.S	N.S	N.S
PT x ADT		**	N.S	N.S

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 ($P<0.05$)

The chroma of the Gac aril powders was not significantly affected by different pre-treatments and drying temperatures ($P>0.05$). The chroma of blanched sample prior to air drying at 40 ^{o}C was the highest, whilst the lowest was observed in the sample soaked in bisulfite solution before air drying at 80 ^{o}C.

As indicated by the hue angle, the tonality of samples was insignificantly influenced by pre-treatments and drying temperatures ($P>0.05$). Generally, the sample soaked in a bisulfite solution prior to air drying had the higher hue angle value, indicating less red colour (Table 1). This is inconsistent with the results of Latapi and Barrett (2006) who stated that tomatoes pre-treated in sodium metabisulfite solution resulted in a redder colour. However, the samples soaked in ascorbic acid solution before air drying had lower hue angles than those of control samples, indicating redder colour. A similar result was also found by Carvajal et al. (1997) who stated that the redness of ascorbic acid treated paprika pepper samples was higher than others. Therefore, pre-treatment in ascorbic acid solution prior air drying is highly recommended in terms of red colour retention.

3.3 Effect of drying treatments on the total carotenoid content of Gac aril powder

The total carotenoid content (TCC) of powder products versus different drying treatments is presented in Figure 2. In general, the total carotenoid content of samples was statistically impacted by different pre-treatments ($P<0.001$) and drying temperatures ($P<0.05$). Statistical results indicated that there was no significant difference in TCC between samples soaked in ascorbic acid and bisulfite, or between blanched sample and control (untreated sample). There was significantly more TCC in the soaked samples than in the control ones.

Contradictorily, many studies have shown that the blanching process can be used to effectively maintain nutrient components in general and carotenoid contents in particular. Koca et al. (2007) found that the β-carotene content in dehydrated carrots was enhanced by blanching before drying at 60 ^{o}C. Similarly, Ramesh et al. (1999) reported that TCC 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. Furthermore, a study on lycopene retention of carrots when blanching at temperatures of 50 to 90 ^{o}C for 15 minutes and in oxygen-free condition was performed. The results showed that lycopene was unchanged during blanching process (Mayer-Miebach and Spieβ, 2003). However, Vedrina-Dragojevic et al. (1997) found that 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.

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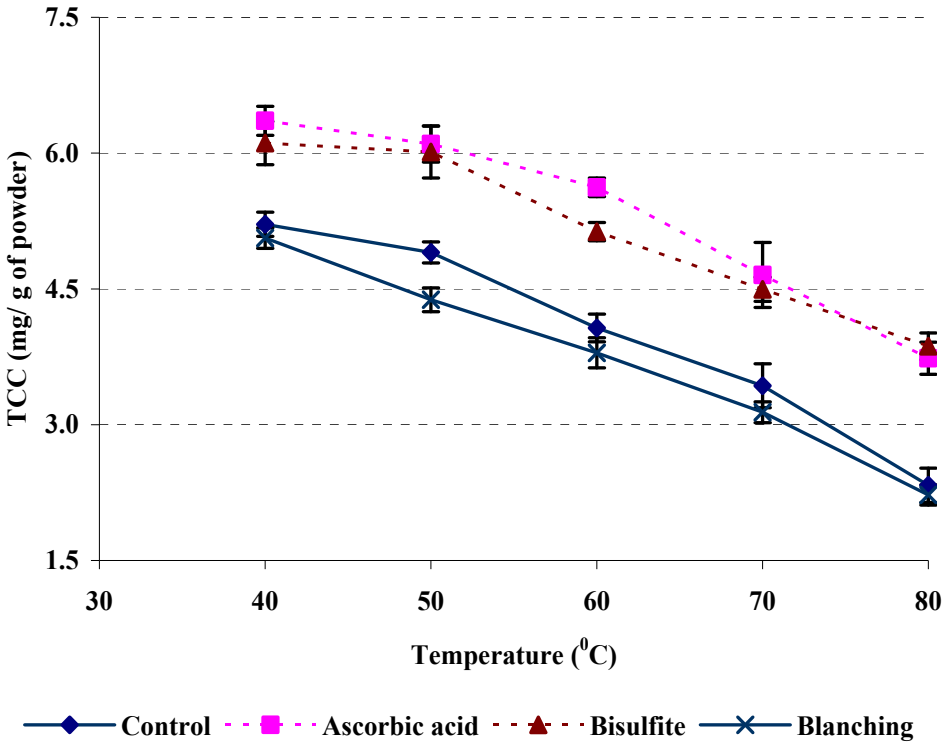

Figure 2. Effects of air drying treatments on the total carotenoid content of Gac aril powders

From our 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 SO_2; hence, carotene deterioration caused by molecular oxygen is prevented by the presence of SO_2. This is in agreement with the results by Mohamed and Hussein (1994) and Chen et al. (2007). They pointed out that the carotenoid content of dehydrated carrots or mangoes was effectively preserved when pre-treated with sodium metabisulfite solution before air drying.

As can be seen in Figure 2, 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 treatment. This is in agreement with Shi et al. (1999) who reported 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.

Compared to different drying processes studied by Tran et al. (2008), at the same drying temperature of 60 ^{o}C, TCC in Gac powder soaked in an ascorbic acid solution was slightly higher than those dried by both air drying and vacuum drying methods without pre-treatment. However, TCC in Gac samples soaked in a sodium hydrogen sulfite solution or an ascorbic acid solution before air drying at low temperature of 40 or 50 ^{o}C were much higher than the samples reported by Tran et al. (2008). Furthermore, TCC in the experimental samples were also much higher than that of spray dried Gac powders investigated by Tran et al. (2008) and Kha et al. (2010). The lower TCC in the samples reported by Kha et al. (2010) is due to the dilution effect of addition malto dextrin. Accordingly air drying process at low temperature can be considered as alternative method for Gac powder processing in terms of lower processing cost and comparable quality (refer to section 1).

3.4 Effect of air drying treatments on the antioxidant activity of the Gac aril powders

The total antioxidant activity (TAA) of samples affected by different pre-treatments and drying temperatures is illustrated in Figure 3a and Figure 3b. The results showed that the effects of different pre-treatments ($P<0.001$) and drying temperatures ($P<0.05$) on TAA (both in ABTS and DPPH assays) were significantly observed. In both assays, no significant difference of TAA was statistically observed in samples soaked in ascorbic acid and bisulfite solutions, or between blanched and control samples ($P>0.05$). Moreover, the highest TAA (in both ABTS and DPPH assays) was recorded for samples soaked in ascorbic acid solution before air drying at 40^{0}C (that is, 0.37 and 0.33 mmole TE/g of powder, respectively), while the lowest was found for air-dried untreated samples at a drying temperature of 80^{0}C (that is, 0.15 mmole TE/g of powder). Further, the loss of TAA increased when increasing drying temperature from 40 to 80 ^{o}C.

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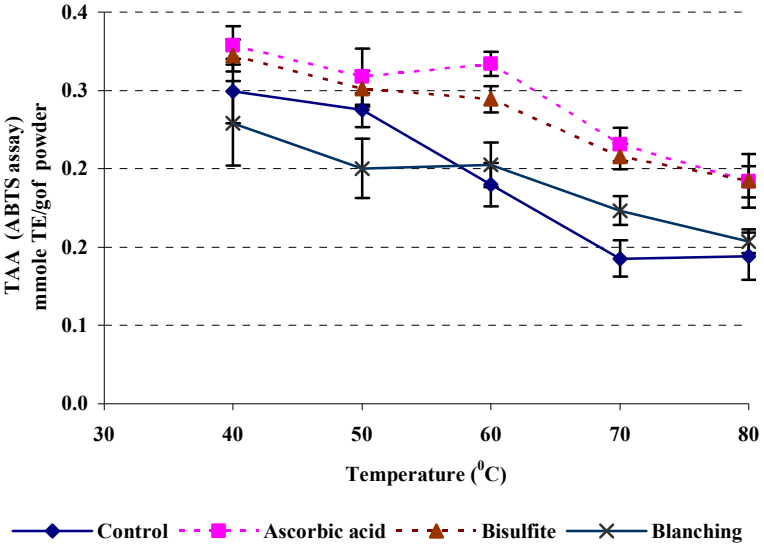

Figure 3a. Effect of different drying treatments on total antioxidant activity (ABTS assay) of powders from fresh Gac fruit aril

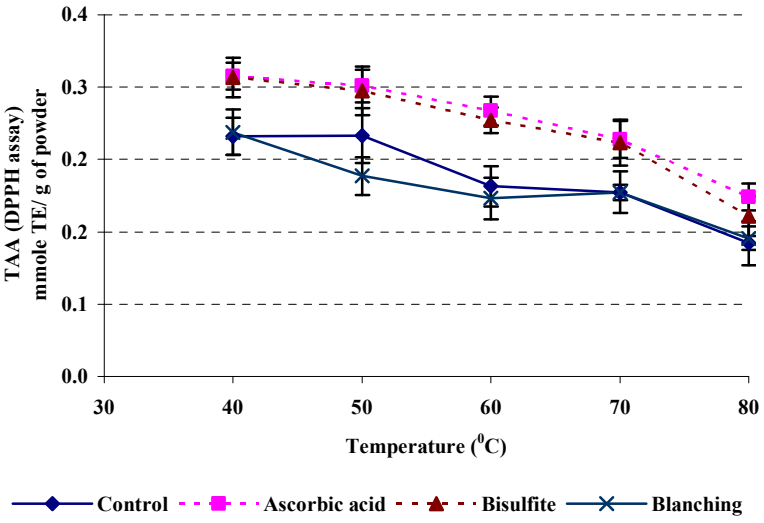

Figure 3b. Effect of different drying treatments on total antioxidant activity (DPPH assay) of powders from fresh Gac fruit aril

In this study, it is evident that pre-treatments and drying temperatures significantly affected the TAA of powder products. These results indicated that

the TAA of blanched samples prior to drying was lower than that of other treatments such as ascorbic acid and bisulfite. 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 ascorbic acid and sucrose solution.

It can be clearly seen that increasing drying temperature resulted in a significant loss of TAA (both in ABTS and DPPH assays) in powder samples. It is suggested that the thermal treatment destroyed antioxidant components, which led to a reduction of antioxidant activity. This is in agreement with the research of Chantaro et al. (2008) who studied effects of various hot air drying temperatures on the antioxidant capacity of carrot peel powder. Their results indicated that an air drying temperature of 60 ^{o}C could preserve antioxidant activity in the powder whereas the antioxidant activity significantly reduced at temperatures of 70 ^{o}C or higher.

In contrast, several studies in tomato, have found that a low drying temperature decreases antioxidant activity in comparison with a high temperature (Kerkhofs et al., 2005; Toor and Savage, 2006). These inconsistencies are likely to be due to different raw materials and drying process conditions examined for the various studies. In addition, as Gac aril is rich in lycopene, the formation of *cis*-isomers from all-*tran*s-lycopene is promoted by thermal treatment as suggested by Shi and Le Maguer (2000) and Stahl and Sies (1992) for tomato. Cis-isomers are higher antioxidant activity than that of all-*trans* isomers (BÖhm et al., 2002).

3.5 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 4 shows the sorption isotherm curve constructed for Gac fruit powder sample at room temperature. 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 curve of the tested Gac powder exhibited a sigmoid shape and similar trends. Generally, EMC values at the constant temperature increased with increase in ERH.

As can be seen clearly from Figure 4, 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. In contrast, 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.

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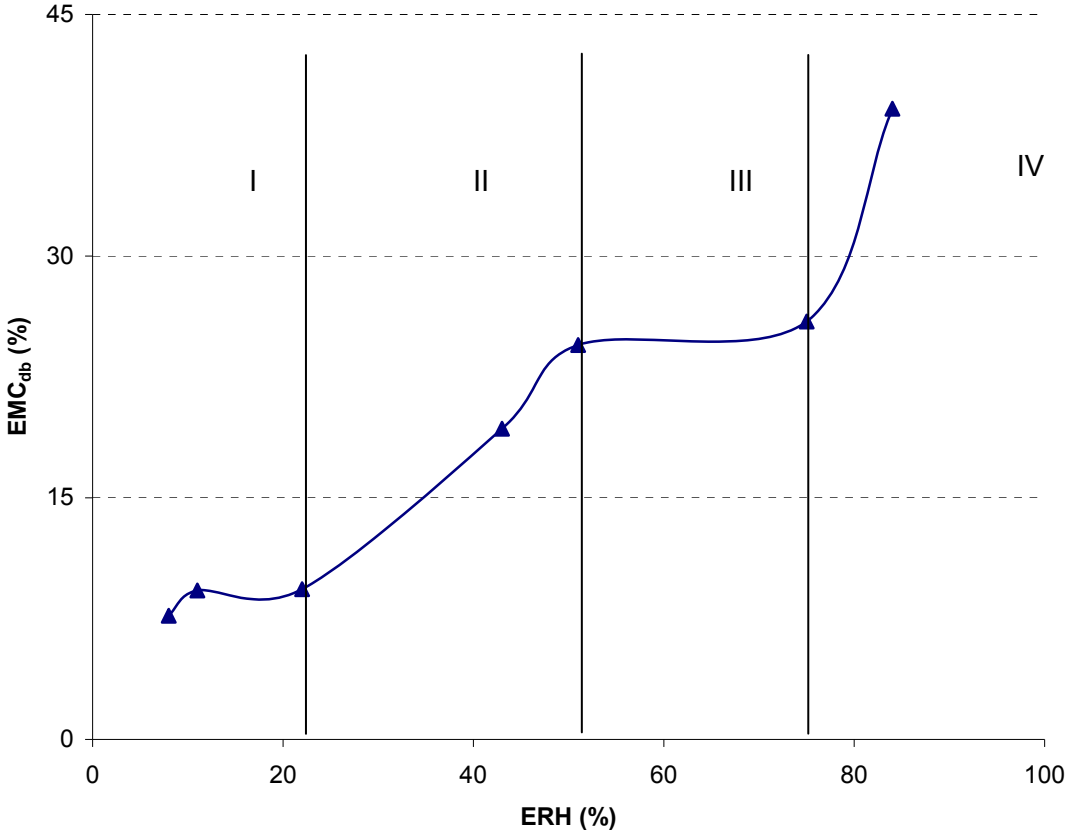

Figure 4 Moisture sorption isotherm of Gac fruit powders

According to the BET equation, the monolayer moisture content, M_0 (%, d.b.) in the powder was 6.88%. Generally, the M_0 of a dried food product is considered to be the safest level for good storage capability (Fellows, 2000). This is because at higher moisture content levels, deterioration of food occurs due to promotion of Maillard browning, enzymic and microbiological activities. Below M_0, the rate of lipid oxidation is again increased, so it is not desirable to go there. Moreover, drying food to below M_0 value requires extra heat of evaporation. Therefore, the final 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 Figure 1, the MC of the Gac powder was close to M_0, indicating that the sample could be stored for a longer time.

4. Conclusion

The colour characteristics, carotenoid contents and antioxidant activities of the powder products from Gac fruits were significantly affected by pre-treatments and drying temperatures. Pre-soaking in solutions of ascorbic acid or bisulfite prior to air drying at low temperature of 40^0C was effective in preserving TCC and TAA. Moreover, loss of TCC and TAA was lowest in the powder dried at a temperature of 40 ^{o}C, with increasing losses observed at increased drying temperatures (50, 60, 70, and 80 ^{o}C). As consumers tend to avoid sulfur dioxide, it is suggested that pretreatment with ascorbic acid before air drying at an economically suitable low temperature should be applied for Gac fruit processing.

The sorption isotherm curve for air-dried Gac aril powder was constructed at room temperature. Results indicated that the curve of the powder has sigmoid shape. Air tight packaging together with desiccant or vacuum packaging if possible, should be applied for the Gac powder to avoid hygroscopicity.

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