

Study on Extraction and Storage Stability of Watermelon Pulp Powder as Food Ingredient

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ABSTRACT

Watermelon fruit is well known source of carotenoids (lycopene (70-90%) and β carotene). The objective of this work was to study the extraction and storage stability of lycopene and β carotene rich watermelon fruit juice powder to use as a food ingredient. The effect of different pH levels (3.05, 3.45, 3.85 and 4.25) of the juice on extraction of the powder was studied. The juice was acidified with citric acid and stored at 2°C (2h) for rapid sedimentation of the pulp. There was insignificant ($p>0.05$) difference in the yield of dried powder extracted at different pH levels. However there were significant ($p<0.05$) differences in color, lycopene and β -carotene contents for powders extracted at different pH levels. Powder extracted at pH of 3.85 resulted in higher color values. Higher lycopene content was observed at pH 3.85 and 4.25. But better β -carotene content was observed at pH 3.05 and 3.45. On further work, 5 g potato puree was mixed with 0.1 g of extracted powder and a significant enrichment of the puree in lycopene (>83%) and β -carotene (91%) contents was obtained. Study on storage conditions indicated that presence of oxygen in storage container played a critical role on lycopene stability than storage temperature. However β -carotene was found relatively stable at low temperature (-40C) with/without oxygen. Extraction pH 3.85-4.25, storage in 9% carbon dioxide concentration and low temperature gave better powder extraction and storage stability.

INTRODUCTION

Watermelon (*Citrullus lantus*) is botanically a tropical fruit from watermelon vine plant. It is a member of the *Cucurbitaceae* plant family of gourds, related to the cucumber, squash, and pumpkin. The fruit has a smooth exterior rind and a juicy, sweet, usually red interior flesh. The percentage of water in watermelon fruit is very high;

and on average it has 92 % water by weight, the highest percentage that is found in any fruit. Mainly it is a good source of different carotenoids. Carotenoids are obtained by extraction from plants or by direct chemical synthesis or produced through fermentation using *Blakeslea tripsora* (FDA/WHO 2007). Some of the carotenoids in watermelon include lycopene, β -carotene, phytofluene,

phytoene, lutein, and neurosporene. Among these, lycopene makes up the majority (70-90%) (Gross, 1987) of the carotenoids. The mean lycopene concentration of watermelon (4868 µg/100 g) is about 40% higher than the year-round mean for raw tomato (3025 µg/100 g) (Holden *et al.*, 1999). In lycopene an open-chain unsaturated structure is responsible for the red color of watermelons. Several researches have reported an association between dietary lycopene consumption and lower incidence in diseases such as prostate and oral cancers and may also help reduce risks of cardiovascular disease (David, 2001; Oms-Oliu *et al.*, 2009). Besides its antioxidant property, lycopene extracted from different sources can be used as food colorants in the food industry (Bauernfeind, 1981).

Watermelon is a seasonal fruit and it is not available all year round. Because of its high nutritional value, it is beneficial if a watermelon product can be produced and made available throughout the year. Extraction of the pulp in different forms can be applied to turn the watermelon juice into powder form that has longer shelf life and can be used as a food ingredient which is readily available throughout the year. Up-to-date, the main dietary lycopene source is from tomato, so the development of lycopene-rich watermelon powder will provide the consumers with alternative choice. In order to obtain the above benefits, the fruit should be harvested at full maturity to ensure it has good quality in terms of its carotenoids and sugar content. The fruit does not develop internal color or increase in sugar content after being removed from the vine. Since it is harvested at full maturity, the majority of the losses are associated with the postharvest life of the fruit, mainly during storage and transportation. The optimum temperature for storage and transport of

watermelon is 10°C with market life of 21 days. Storage below 10°C results in chilling injury and with increasing storage temperature, market life is diminished with significant losses in lycopene and sweetness. In addition to this due to its big size, transportation cost is high and meanwhile significant losses are occurred due to cracking, splitting, and collapsing. The combined effects of these limitations result in loss of nutritious and economic benefits of the fruit. So far different attempts have been made to minimize the losses due to proper postharvest management practices and processing of the fruit juice. However, in this work, attempts were made to show additional alternative approach to preserve economically valuable part of the fruit through simplified method. Therefore the objectives of this work were (i) to elucidate a simplified approach on extraction of lycopene and β-carotene rich watermelon juice powder and (ii) to study storage stability of extracted powder under different storage conditions.

MATERIALS AND METHODS

Watermelon juice Preparation

Watermelons (*Citrullus lanatus*) fruits were purchased at commercial maturity from a local supermarket (Montreal, Canada). Fruits were cut into pieces and pulps were juiced using fruit extractor (Hamilton Beach/Proctor-Silex, Inc., Model 67900, Type CJ13, China). Juices from different fruits were bulked together to avoid variation among fruits.

pH reduction of the juice and separation of pallet

The average pH of the fresh juice was found to be 5.48±0.02. At this pH the juice had its natural color and no sedimentation of the red component was observed. In order to enhance the

separation of the red component from the water portion, the juice was divided in four glass bottles (750 ml) and the pH was adjusted to 3.05, 3.45, 3.85 and 4.25 using 10% (w/v) citric acid monohydrate (ACROS ORGANICS, USA). The pH was measured at 25°C using pH meter (Accumet Basic AB15, Fisher Scientific) with required calibration. Eventually samples were stored at 2°C to enhance rapid separation of pallet from the bulk water portion. After 2 h samples were withdrawn and the top portion was decanted to collect the pallets. Collected pallets were further centrifuged 7500xg, 4°C for 15 min (Thermo IEC, Model 120, and USA) to reduce excess water residues in the pallets.

Drying Process

The samples were spread on aluminum trays in a thin layer and convective air drying was performed in custom made cabinet dryer at 50°C. The residence time for the drying process was 15 h. Dried samples were eventually grounded using pestle and mortar and stored in desiccators till further analysis. Measurement of water activity was carried out using a water activity meter (Rotronic Instrument corp. Model HygroLab 3, USA).

Color measurement of dried samples

Visual total colour of samples was determined using CIE (Commission Internationale de L'Eclairage) L*a*b* colour space to evaluate the effect of extraction method on color of sample

powder using tri-stimulus colorimeter (Minolta camera CM-500d, Japan), which was calibrated using white tiles. The measurements were taken using D-65 illuminant and 10° observer. Total colour was expressed in terms of "L" value (lightness, ranging from zero (black) to 100 (white), "a" (redness) value ranging from +60 (red) to -60 (green) and "b" (yellowness) value ranging from +60 (yellow) to -60 (blue). Three measurements were made at different locations on the surface of each powder and the procedure was repeated twice to get the average values.

Quantitative analysis of lycopene and β-carotene

Lycopene and β-carotene content of extracted powders were estimated according to Nagata and Yamashita (1992). Briefly, 1 g of powder was homogenized using 10 ml of acetone and n-hexane (4:6) as an extraction solvent for 1 min with addition of 0.1% Butylated hydroxytoluene (BHT). The homogenized solution was allowed to stand for 60 s under dark condition. One ml of the supernatant was taken and diluted with 9 ml of the extract solution and analyzed using spectrophotometer (Novaspec II Visible, England). The optical density of the solution was measured at 663, 645, 505 and 453 nm versus extraction solution as reference. The lycopene and β-carotene concentrations were quantified as per Equations 1 and 2 respectively.

$$\text{Lycopene}(mg/100 g) = -0.0458A_{663} + 0.204A_{645} + 0.372A_{505} - 0.0806A_{453} \quad (1)$$

$$\beta\text{-carotene}(mg/100 g) = 0.216A_{663} - 1.22A_{645} - 0.304A_{505} + 0.452A_{453} \quad (2)$$

Where A_{663} , A_{645} , A_{505} and A_{453} are the absorbances at 663, 645, 505 and 453 nm, respectively.

Packaging and storage stability study

Part of extracted powers were packed and stored under different packaging and storage conditions. Samples were packed in storage anaerobic pouch with presence of ambient air (21% oxygen) and creation of 9% carbon dioxide and stored at three storage temperatures (-40, 2 and 25°C) in dark condition. Lycopene and β -carotene contents of samples were determined at 15 days interval for two months storage period.

Statistical Analysis

All experiments were carried out at least in duplicates and the standard deviation and two ways ANOVA for replicated experiments were calculated using Microsoft Excel 2007 (Microsoft Corporation Version).

RESULTS AND DISCUSSION

Effect of pH levels on weight of extracted powder

Watermelon juice power treated at different pH levels showed no significant ($p>0.05$) difference on weight of powder extracted (Table 1). The effect of wide range of pH to induce sedimentation of the juice pulp showed

almost similar effect. This showed that the juice was sensitive for pH change and this could be taken as an opportunity to separate the pulp from the water portion without imposing significant pH reduction. The water activity of dried powders was determined at the end of drying period and statistically no significant ($p>0.05$) difference was observed among treatments. In relation to drying and storage of dried products, water activity is a measure of energy status of water in the system and is a major criterion for safety and quality. Basically water activity of 0.60 to 0.20 prevents microbial proliferation, enzymatic activity, nutrient loss and browning (Labuza 1979). Results in this work showed that water activity value of between 0.286-0.3 was achieved (Table 1) which could ensure the required safety and shelf stability. This range is in close agreement with results reported (0.2-0.29) by Quek *et al.* (2007) for watermelon powder extracted using spray drying. The slight variation might occur due to levels of drying temperature used in both drying methods.

Table 1. Weight of extracted powder and water activity values at different pH level

Extraction pH levels	Mass of powder extracted (g/750 ml juice)	Water activity (<i>aw</i>) dried powder
3.05	2.55±0.05†	0.288±0.005†
3.45	2.60±0.10	0.287±0.002
3.85	2.45±0.05	0.286±0.003
4.25	2.65±0.05	0.300±0.013
Significance level	NS	NS

†=Standard error, NS = non significant

Effect of extraction pH on color

One of the many attractive features of watermelon juice is its attractive color. This color should be maintained during extraction procedure or processing conditions. The results of color

measurement for powders were expressed on CIE color parameters (Figure 1 and Table 2). No significant ($p>0.05$) differences were observed on "L" and "b" values among extraction pH levels (Table 2). However there was

a significant ($p < 0.05$) variation in "a*" value, which was the most important color parameter for extracted powder (Table 2). Sample extracted at pH of 3.85 showed superior color value and followed by samples extracted at pH of 3.45. Since the CIE "a*" value represents the redness of the chromaticity dimension, it can be considered as one of the physical parameters to describe the color of extracted powder. The color values of spray dried watermelon powder reported in Queck *et al.* (2007) were in ranges of 67-75 (lightness), 15-20 (redness) and 16-24 (yellowness) which are in close agreement with redness and yellowness values of our work. However a huge discrepancy was observed in terms of lightness which might be explained by higher drying temperature ($\sim 75^{\circ}\text{C}$) used in spray drying and difference on variety of watermelon used.

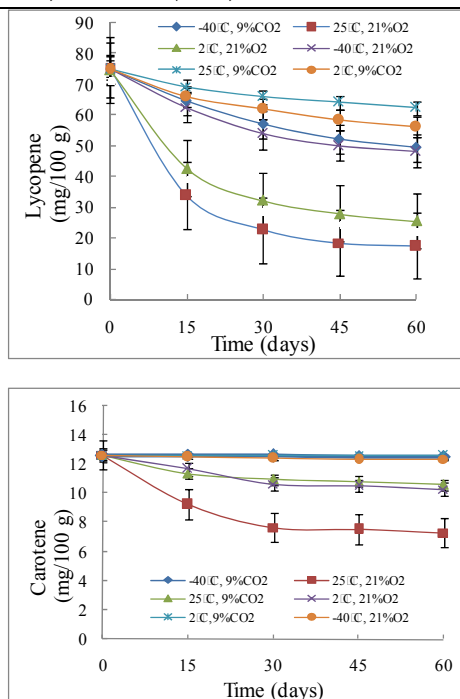


Figure 1. Effect of storage environment on stability of lycopene (a) and β -carotene(b) contents of extracted powder

Table 2. Color values of extracted powder at different extraction pH conditions

pH	"L*" value	"a*" value	"b*" value
3.05	17.90 \pm 0.49 [†]	22.82 \pm 1.05 ^b	13.34 \pm 0.70
3.45	18.42 \pm 0.62	25.54 \pm 0.57 ^{ab}	15.23 \pm 0.60
3.85	18.82 \pm 0.51	27.10 \pm 0.43 ^a	16.10 \pm 1.10
4.25	17.40 \pm 0.29	25.31 \pm 0.26 ^b	13.83 \pm 0.33
Significance level	NS	*	NS

[†]=Standard error, NS = non significant ($p > 0.05$), * =significantly different ($p < 0.05$)

Effect of extraction pH on lycopene and β -carotene content

The main goal of this work was to extract watermelon fruit juice powder rich in lycopene and β -carotene. Lycopene and β -carotene extracted at different pH levels showed significant ($p < 0.05$) differences (Table 3). The lycopene content of powders varied from 67 – 83.5 mg/100 g powder (w.b),

which was in close agreement with Quek *et al.* (2007) for spray dried powders with a value of 72.4 – 95.4 mg/100 g. Powder extracted at pH 3.85 showed the highest lycopene content and followed by pH levels of 3.45 and 4.25. Powder extracted at the lowest pH showed a 20% decrease in lycopene content as compared to powder extracted at pH 3.85. This showed that

more acidic condition might induce degradation of lycopene during extraction time. Meanwhile β -carotene contents showed significant ($p<0.05$) differences among extraction pH levels. In contrary to lycopene the lowest value

of β -carotene was obtained from sample extracted at pH 3.85. Furthermore β -carotene content values observed in this work (10.34-17.46 mg/100 g) were higher than those reported in Quek *et al.* (2007) (2.3 – 3.1 mg/100 g powder).

Table 3. Lycopene and β - carotene contents of extracted powder at different pH levels

pH	Lycopene (mg/100 g) w.b	β -Carotene(mg/100g)w.b
3.05	66.98 \pm 0.57 ^c	17.46 \pm 0.77 ^a
3.45	70.62 \pm 0.81 ^b	16.46 \pm 1.4 ^a
3.85	83.49 \pm 0.32 ^a	10.34 \pm 2.56 ^b
4.25	75.43 \pm 0.38 ^b	14.72 \pm 0.51 ^a
Significance level	*	*

*=significant ($p<0.05$), numbers followed by different letters are significant

Many epidemiological studies showed that lycopene rich diet has beneficial effects on human health (Perkins-Veazie *et al.*, 2001, Edwards *et al.*, 2003, Perkins-Veazie and Collins, 2004). Lycopene as a main component of the fruit has been studied extensively for its antioxidant and cancer preventing properties (Giovannucci 1999). Enriching poor antioxidant source foods with such type of extracted product enhance the health benefits. The average lycopene content in commercial watermelon fruits is 4.8 mg/100 g(w.b) (Perkins-Veazie *et al.*, 2001). However extracted powder forms

showed significant increases in the concentration of lycopene, and hence can be used to enhance the antioxidant capacity of food formulations. As a follow up study, 0.1 gram of extracted powder was mixed with 5 gram of potato puree to evaluate the possibility to enhance lycopene and β -carotene contents of the puree. Table 4 shows a significant improvement in both components with the addition of small amount of the powder. Both lycopene and β -carotene contents were increased by more than 84 and 91 % respectively (Table 4).

Table 4. Lycopene and β -Carotene enriched potato puree from extracted pulp powder

Puree + powder from pH	Lycopene (mg/100g)	% Increase Lycopene	β -Carotene (mg/100 g)	% Increase β -Carotene
Pure potato puree	0.0093 \pm 0.0005 ^c	0	0.0022 \pm 0.0002 ^c	0
Puree+ pH 3.05	0.0605 \pm 0.0004 ^b	84.62	0.026 \pm 0.00072 ^b	91.65
Puree+ pH 3.45	0.059 \pm 0.0011 ^b	84.33	0.030 \pm 0.0019 ^{ab}	92.69
Puree+ pH 3.85	0.0712 \pm 0.0015 ^a	86.94	0.037 \pm 0.0007 ^a	94.16
Puree+ pH 4.25	0.076 \pm 0.0016 ^{ab}	87.77	0.039 \pm 0.0004 ^a	94.44
Significance level	*		*	

Storage stability of lycopene and β -carotene

Storage stability of lycopene and β -carotene from powder extracted at pH 3.85 were studied at different storage temperatures in combination to ambient air and 9% carbon dioxide concentration. Result in Figure 1 shows that fast lycopene degradation rate was observed for the first 15 days of storage at storage conditions of 2 and 25°C in presence of ambient oxygen. The same trend was followed by β -carotene stored at ambient condition. In both cases oxygen is the major determinant factor for the degradation of the compounds rather than storage temperature. Ax *et al.* (2003) reported that under saturated oxygen supply for pure lycopene dissolved in oil-water emulsion (25°C), 80% degradation of lycopene was observed. In similar work, lycopene exposed to saturated oxygen at 30°C showed 90% loss within few hours (Henry *et al.*, 1998). Furthermore Giovanelli and Paradiso (2002) observed the effect of oxygen during storage of tomato lycopene powder by comparing samples stored in N₂ and air, and the result showed that oxygen is a critical factor for lycopene retention during storage. The instability of lycopene under presence of oxygen is mainly because oxidative degradation of its unsaturated bonds (13 double bonds). As compared to β -carotene, the

sensitiveness of lycopene to oxygen was very high and even it was found that it degrades in a faster rate as compared to unsaturated fatty acids (Boon *et al.*, 2008). Storage temperature also showed significant impact ($p < 0.05$) on the stability of lycopene. Figure 2 shows the effect of storage temperature under identical concentrations of oxygen and carbon dioxide. Powder stored at room temperature resulted in the lowest and highest lycopene content in presence of oxygen (21%) and carbon dioxide (9%) respectively (Figure 2). Study of Sharma and Maguer (1996) showed that after 4 months' of storage at room temperature in dark, lycopene loss in tomato pulp reached 79%. Similar pattern was observed in our case for powders stored at 2°C. However powders stored at minus 40°C showed almost equal stability of lycopene regardless of composition of gases. Fish and Davis (2003) reported that a 30-40% and 5-10% loss of lycopene was observed for fresh samples stored at minus 20 and minus 80°C for a year period. Our result is in agreement with their work regardless of water activity level in fresh tissue of their samples and dried powder in our case which was found more stable at minus 40°C. In general lycopene appears to be degrading in a faster rate than β -carotene during storage which is in consensus with observations of Lee and Coates (2002).

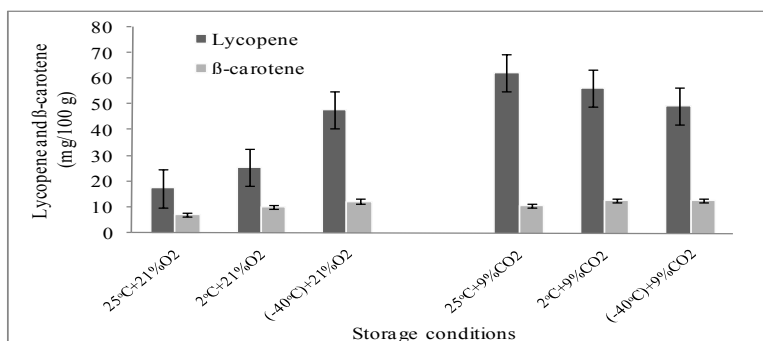


Figure 2. Effect of storage temperature on stability of lycopene and β -carotene contents of extracted powder at constant gas composition.

CONCLUSIONS

Watermelon is a fruit rich in antioxidant components. However due to limitations associated with seasonality of the fruit, transportation and post harvest handling, its market availability is not consistent. With extraction and concentration of antioxidant rich part of the pulp, extracted powder can be used as an additive in antioxidant deficient foods. Based on this study, juice powder extracted at pH of 3.85 showed better color and lycopene contents. Powder stored in the presence of ambient air oxygen showed least stability in lycopene and β -carotene content. However samples stored at lower temperatures under 9% of carbon dioxide concentration showed better stability.

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