Microencapsulation of lycopene rich cherry tomato powder using spray drying

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Abstract
An attempt was made to optimize the processing variables viz., inlet air temperature (140 and 160°C), feed flow rate (6 and 10 ml/min) and maltodextrin (10 and 20%) on efficacy of microencapsulation of cherry tomato juice powder. The results suggest, processing variables influence the physico-chemical qualities of the microencapsulated cherry tomato powder. The results on chemical parameters of the microencapsulated varied in different treatments viz., TSS recorded a range from 42.96 to 64.66 ºB; pH from 4.65-4.88; titratable acidity from 0.47-1.05; ascorbic acid from 51.84-111.11 mg/100g; lycopene content from 0.10 to 0.22 mg/100g; and antioxidant activity from 633.66 to 1182.83 µg/ml. With regard to physical parameters moisture content recorded a range from 5.77 to 7.84 %; water activity 0.31 to 0.50; L*: 83.36-99.87; a*: 0.80-4.13; b*: 20.57-30.51; and particle size from 516nm to 2682nm. Powder with lowest particle size resulted in good surface morphological characteristics with more homogenous capsules having smooth surfaces, few wrinkles and less agglomeration.

Keywords: cherry tomato, microencapsulation, spray drying, antioxidant activity, particle size

Introduction
Cherry tomatoes (Solanum lycopersicum var cerasiforme) are little utilised than normal tomatoes but are richer sources of several nutrients and antioxidants. They are small in size, sweeter in taste, have intense colour, flavour and generally round in shape weighing about 10 to 30 g. They are rich source of lycopene a carotenoid having antioxidant property and also contain ascorbic acid, vitamin E, flavanoids, phenolic acids, minerals and fiber. For more perishable produce like tomatoes, value addition is a necessity where it can be preserved without losing its nutritional benefits. As compared to normal tomato products in the market, innovative functional foods could be the better solution for present day lifestyle.

Spray drying is an efficient and cheap method of drying through which we can encapsulate the bioactive ingredient present in the produce. It can be defined as a single step process where the liquid feed is atomised in the hot air to simultaneously produce fine textured powder. This operation is economical because of the easiness of the process, easy availability of the equipment and comparative low cost. The dried powder obtained have advantage over liquid feed in having reduced weight, easy for packing, transportation and easiness in incorporation to other food matrix.

Encapsulation is a process to entrap active agents within a carrier material and it is a useful tool to improve delivery of bioactive molecules and living cells into foods (Fang and Bhandari, 2010) [9]. Spray drying is the most commonly used method for producing microencapsulated products by the food industry because of the quality of the finished product. Spray dried products have some problems regarding stickiness and hygroscopicity of product. This can be overcome by adding a carrier material which will act as a wall material to produce encapsulated product. Generally used carrier agents are polysaccharides like maltodextrin, gum arabic, tapioca starch etc.

The quality and efficacy of the process depends on many processing variables such as inlet temperature, feed flow rate and type and quantity of carrier materials. Hence, all these variables have to be optimised to obtain better powder quality and yield. The objective of this paper is to standardise the process condition for microencapsulation of cherry tomato juice powder by spray drying and to study the influence of spray drying parameters on physico chemical properties of cherry tomato juice powder.
Materials and Methods

Raw materials and preparation
Cherry tomato fruits were procured from Namdhari fresh, Bengaluru, Karnataka, India. Further the fruits were graded based on the uniformity in colour. The red ripe fruits were used for further experimentation after washing under clean running tap water and surface air dried by electric fan. The juice was extracted by crushing the fruits in a mixer grinder. The juice was filtered thrice through muslin cloth and used for spray drying after mixing with maltodextrin (10 and 20%) as a carrier agent using magnetic stirrer.

Drying of the cherry tomato juice for production of microencapsulated cherry tomato powder was carried out using Warrants laboratory spray dryer (LSD 48, Jay instruments and systems Pvt. Ltd. Bombay). The instrument consists of an atomizer nozzle which will atomize liquid feed into fine droplets, a drying chamber where feed liquid comes in contact with the hot air, two cyclone separators and a collector. The experiment was standardized according to trial and error method. Operating inlet temperature was tried at 140 and 160°C. Feed flow rate was tried at 6 and 10 ml/min. Outlet temperature was maintained constant in between 70-80°C. Eight treatments were tried with two different combinations of inlet temperature, feed flow rate and maltodextrin levels. The design of the experiment was completely randomized design (CRD) with three replications.

Powder analysis
Total soluble solids (%B)
Total soluble solids (TSS) of cherry tomato powder was measured by reconstituting the powder in equal amounts of water (1g/ml) using Atago pocket refractometer Pal-I (0-53%) and expressed as percentage.

PH
The pH of microencapsulated cherry tomato powder was determined by using Laboratory benchtop meter (model 86505/555), Symbiont Technologies, Bengaluru. After calibration, the pH of reconstituted juice from microencapsulated cherry tomato powder (1 g/10 ml distilled water) was determined.

Titratable acidity (%)
The acidity of the cherry tomato and reconstituted powders was determined by titration method. A known amount of powder sample (1g) was taken and reconstituted with water. The known volume of the reconstituted sample (10 ml) was taken and titrated against 0.1N standard NaOH using phenolphthalein indicator (Ranganna, 1986) [24]. The value was expressed in terms of citric acid as per cent acidity of juice (AOAC, 1984).

Ascorbic acid content (mg/100g)
Ascorbic acid content was estimated titrimetrically using 2,6-dichlorophenol indophenol dye as per the AOAC procedure. The powder is diluted with 4% oxalic acid. An aliquot of 10ml was titrated against 2, 6-dichlorophenol indophenol. The amount of the dye consumed was equivalent to the amount of ascorbic acid. The results were expressed as mg of ascorbic acid per 100g of fruit juice.

Lycopene content (mg/100g)
Lycopene content was estimated using acetone – petroleum ether extraction method. A known weight (1 g) of powdered cherry tomato sample is taken and crushed in acetone. This acetone extract is transferred into a separating funnel containing 10-15 ml petroleum ether and it is repeated until becomes colourless. These petroleum ether extracts were combined and a small quantity of anhydrous sodium sulphate is added to make it dry. Volume made up and OD of the solution at 503 nm is taken using petroleum ether as blank.

\[
\text{Lycopene content (mg/100g)} = \frac{3.120 \times \text{OD value at 503 nm}}{\text{weight of the sample} \times 100}
\]

Antioxidant activity (µg/ml)
DPPH (2, 2-diphenyl-1-picrylhydrazyl) radical scavenging capacity was assessed according to the method proposed by Brand-Williams et al. (1995) [3] with some modifications. The purple coloured DPPH is a stable free radical, which is reduced to 2, 2-diphenyl-1-picrylhydrazine (yellow coloured) by reacting with an antioxidant (Ivanisova et al., 2013) [14]. A concentration of 1mg/ml was prepared by adding sample in methanol. This spectrophotometric assay uses the stable radical DPPH as a reagent. Sample (0.1 ml) was added to 3 ml of DPPH solution. After 30 min incubation period at room temperature, the absorbance was read against blank at 517 nm. The percentage inhibition of free radical (DPPH) was calculated and expressed as IC50 (Inhibitory concentration) value (µg/ml), which is the concentration required to cause a 50% decrease in initial DPPH concentration.

\[
\text{Inhibition % (DPPH)} = \frac{A_{\text{blank}} - A_{\text{sample}}}{A_{\text{blank}}} \times 100
\]

Where, A = Absorbance.

Ash (%)
The known weight of cherry tomato powder samples (1 g) was transferred to muffle furnace (Metalab, Mumbai). The powder is dried at 525°C for 5-7 h and weighed and the per cent ash is calculated.

Moisture content (%)
The moisture content of the powder was determined by drying up to constant weight using electronic moisture analyser (Sartorius MA 35) and expressed in terms of per cent.

Water activity (aw)
The water activity of the microencapsulated cherry tomato powders were measured using an electric water activity meter (Rotronic hydrolab).

Solubility (%)
The solubility of the powder was determined by Eastman and Moore (1984) [7] method. A total of 1 g of powder sample was mixed into 100ml of distilled water in a blender at high velocity (1550 rpm for 5 min). The moisture content of the powder was recorded; 1 g of powder was added to 100 ml distilled water. This solution was transferred to experimental tubes and centrifuged at 3000 rpm for 5 min and allowed to settle down completely for 30 min. An aliquot of 25 ml of the supernatant was transferred to pre-weighed petri dishes and immediately oven-dried at 105°C for 5h. The solubility (%) was calculated as the weight difference.

Colour (L*a*b*) values
The colour of the spray dried cherry tomato powder was measured using a Lovibond colour meter (Lovibond RT300, Portable spectrophotometer, The Tintometer Limited,
Salisbury, UK) fitted with 8 mm aperture and the instrument was adjusted at 10° observer and D65 primary illuminant. The instrument was calibrated using the black and white tiles provided. Colour was expressed in Lovibond units $L^*$ (lightness/darkness), $a^*$ (redness/greenness), $b^*$ (yellowness/blueness), $C$ (chroma) and $h^*$ (hue angle). Two measurements were performed for each sample and the values were averaged.

### Results and Discussion

#### Physico-chemical properties of cherry tomato pulp

Cherry tomato pulp recorded a moisture content of 94.17%; water activity ($a_w$): 0.702; TSS: 8.00; pH: 4.63; Acidity (%): 2.11; Ascorbic acid content (mg/100g): 533.33; Lycopene content (mg/100g): 1.79; Antioxidant activity ($\mu$g/ml): 560.16; $L^*$:44.44; $a^*$: 28.40; $b^*$: 33.18; $C$ (Chroma): 44.47; and $h^*$ (Hue angle): 48.53

#### Spray dried cherry tomato powder properties

**Total soluble solids**

TSS of cherry tomato powder was found to be in a range of 42.96 – 64.66B as shown in Table 1. Total soluble solids found to get increased after spray drying process which indicates that TSS value was higher in powder than in pulp of cherry tomato. This might be due to the evaporation of moisture from the juice and remaining solids. Total soluble solids were found to be not affected by the change in inlet air temperature but as the maltodextrin concentration increased, TSS found to be increased.

**pH**

pH of cherry tomato powder is shown in Table 1 and is in the range of 4 – 5, which is suitable for vegetable products to make it microbiologically safe. pH of the powder increased with inlet temperatures for different treatments.

**Titratable acidity**

Titratable acidity varied with a range of 0.36 – 1.05% in microencapsulated cherry tomato powder as shown in Table 1. Titratable acidity expressed in terms of citric acid decreased as the inlet temperature increased except for T2 (160°C+10 ml/min+10% MD). It may be because heat may act as a catalyst in chemical reactions therefore high temperature and longer duration may adversely affect the acidity. The result corroborates the early work of Singh et al. (2013) [27] in ber juice powder.

### Ascorbic acid (mg/100g)

Ascorbic acid is one of the antioxidant agents present in food products. Cherry tomato fruit pulp recorded ascorbic acid content of 533.33 mg/100g and it reduced drastically on spray drying. In powders, the value was in a range of 51.84 – 111.11 mg/100g as shown in Table 1. Increase in inlet air temperature leads to decrease in ascorbic acid content of microencapsulated cherry tomato powder for different treatments. This may be because of the heat reactions which will reduce the ascorbic acid content. These results was in accordance with Singh et al. (2013) [27] in ber juice powder, Lee et al. (2013) [17] in dragon fruit powder and Chiang (2011) [6] in orange juice. A decreasing trend was observed in ascorbic acid content with increase in maltodextrin concentration. This result was consistent with Singh et al. (2013) [27].

### Lycopene content

Lycopene is the major carotenoid compound in tomatoes which gives the fruit its characteristic red colour (Fruscianti et al., 2007) [10]. Spray dried cherry tomato powder showed much reduced lycopene content than fresh fruit. It was found to be in range of 0.221 - 0.104 mg/100g as presented in Table 1. The lycopene content reduced with increased inlet temperature. A similar observation was made for spray drying of tomato pulp (Goula et al., 2004) [11] and for water melon powder (Quek et al., 2007) [23]. The reduction in lycopene content was likely due to thermal degradation and oxidation. Moreover, Goula et al. (2004) [11] reported that the spray dried powders produced at lower inlet temperature had a tendency to undergo agglomeration because of increased moisture content. Agglomeration would lower the exposure of powders to oxygen and therefore protect the lycopene from destruction. But the microencapsulation process here did not successfully concentrate the lycopene in the cherry tomato powder while antioxidant capacity increased after microencapsulating. However, here the maximum lycopene content was registered with less inlet temperature, less feed flow rate and higher maltodextrin concentration followed by high feed flow rate and less maltodextrin concentration at same inlet temperature which shows that maltodextrin helps in trapping lycopene content to some extent in cherry tomato powder.

### Antioxidant activity (IC$_{50}$ Value)

The antioxidant activity was determined in terms of the ability of the antioxidants in the fruit to inhibit oxidation. The free radical scavenging activity of each solution was calculated as per cent inhibition and was expressed as IC$_{50}$, defined as the concentration ($\mu$g/ml) of the test material required to cause a 50% decrease in initial DPPH concentration. Antioxidant activity of cherry tomato powder was found to be in a range of 1182.33 - 633.66 $\mu$g/ml (Table 1), where the highest value was found in first treatment. Increase in inlet temperature and maltodextrin concentration decreased the antioxidant activity of microencapsulated cherry tomato powder. This may be due to the degradation of bioactive compounds in higher inlet temperature but as the maltodextrin concentration (µg/ml) of the test material required to cause a per cent inhibition and was expressed as IC$_{50}$, defined as the concentration ($\mu$g/ml) of the test material required to cause a 50% decrease in initial DPPH concentration. Antioxidant activity of cherry tomato powder was found to be in a range of 1182.33 - 633.66 $\mu$g/ml (Table 1), where the highest value was found in first treatment. Increase in inlet temperature and maltodextrin concentration decreased the antioxidant activity of microencapsulated cherry tomato powder. This may be due to the degradation of bioactive compounds in higher inlet...
temperature and due to the dilution effect of the extract caused by maltodextrin.

**Ash (%)**

The ash content is a measure of the total amount of minerals present within a food. Ash content in cherry tomato fruit powder varied within a range of 24.33 – 50.00 % as shown in Table 1. It was observed that ash content tend to increase as the inlet temperature increases and also slight reduction was seen as the maltodextrin level increases even though there was no significant differences between the treatments. These results were in contrary with Patil et al. (2014) [23] and Hari et al. (2013) [26] on spray drying of guava powder and sugarcane juice powder respectively.

<table>
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<th>Parameters</th>
<th>TSS (°B)</th>
<th>pH</th>
<th>Titratable Acidity (%)</th>
<th>Ascorbic acid (mg/100g)</th>
<th>Lycopene content (mg/100g)</th>
<th>Antioxidant activity/ IC50 Value (µg/ml)</th>
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Values with different letters in the same column indicate significant differences at p = 0.05. Probability level according to Duncan’s multiple range test.

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<th>Water activity (at 25°C)</th>
<th>Solubility (%)</th>
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<th>a* values</th>
<th>b* values</th>
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Values with different letters in the same column indicate significant differences at p =0.05. Probability level according to Duncan’s multiple range test.

**Moisture content**

Moisture content is an important powder property which is related to the drying efficiency. Moisture content of a microencapsulated product plays an important role in determining its flowability, stickiness and storage stability (Shrestha et al., 2007). Moisture content of spray dried cherry tomato powder is shown in Table 2 and is in the range of 5.77 to 7.84%, which was sufficient to make the powder biologically safe. The moisture content of the powder did not register significant results.

**Water activity**

Water activity is the parameter which can directly relate with the powder stability with respect to microbiological contamination. Water activity is the availability of free water in a food system responsible for any biochemical or microbiological reactions and hence shorter shelf life (Quek et al., 2007) [23]. The encapsulated cherry tomato powder obtained in all the treatments registered water activity in a range of 0.30 to 0.50 (Table 2), thus they can be considered biochemically or microbiologically stable. Similar observation on water activity of 0.30 was reported in spray dried water melon powder by Quek et al. (2007) [23], acai powder by Tonon et al. (2009) [26] and Santhalakshmy et al. (2015) [26] in jamun juice powder.

**Solubility**

Solubility is the most dependable criteria to evaluate the behaviour of powder in aqueous solution (Caparino et al., 2012). Solubility of cherry tomato powder ranged from 78.87% to 89.69% as shown in Table 2. Generally, solubility used to increase with increase in inlet temperature because increasing the drying air temperature generally produces an increase in particle size and so decrease in time required for powder to dissolve. The same result was observed here and is in accordance with Phuongchandang and Sertwasana (2010) in ginger powder; Izidoro et al. (2011) in spray dried banana starch; Santhalakshmy et al. (2015) [26] in jamun juice powder; Goula and Adamopoulos (2008) [23] in tomato powder. Further higher maltodextrin concentration (20%) has resulted in high solubility (89.69%) value when compared to low maltodextrin concentration of 10% (78.87%). This increase can be explained as maltodextrin which is an amorphous and non-crystalline material which is having superior water solubility.

**Colour values**

Colour attributes of cherry tomato powder are shown in Table 2. Lightness value indicated that the cherry tomato powders produced by different treatments are lighter in appearance (>)50). Moreover as the inlet temperature increased, the lightness value tends to increase. This result is in accordance with Santhalakshmy et al. (2015) [26] in jamun juice powder. Contrarily Quek et al. (2007) [23] reported that higher inlet temperature decreased the lightness value. It was also observed that an increase in lightness of powder was obtained by increasing the maltodextrin concentration from 10-20 per cent which was consistent with the result of Kha et al. (2010) [16] in gac powder. This may be due to the higher concentration of malt dextrin present in it. Lightness value can be correlated with the lycopene content of the cherry.
tomato powder. The \( L^* \) value decreased with increased lycopene content of the powder which indicates that lower the temperature higher the lycopene retention and lower the lightness value.

The increased concentration of maltodextrin reduced the redness value of cherry tomato powder. The \( b^* \) values increased as the inlet temperature increased in different treatments. As the maltodextrin concentration increased, the \( b^* \) values show an increasing trend at low inlet temperature but the result was opposite at higher inlet temperature of 160°C.

In cherry tomato powder, as the inlet air temperature increased chroma value increased. The result was in accordance with the values of water melon powder (Quek et al., 2007) [23]. As maltodextrin concentration increased, chroma value of cherry tomato powder also increased. This result was consistent with that of blackberry powder (Ferrari et al., 2012) [9].

The hue angle values for cherry tomato powder showed that, increase in inlet temperature tends to increase the hue angle. This indicates that there was a decrease in colour intensity of cherry tomato powder when the inlet temperature was increased. These changes in hue angle might be caused by the destruction of lycopene at higher temperature (Quek et al., 2007) [23]. A similar result was reported by Santhalakshmy et al. (2015) [26] in jamun juice powder and Mestry et al. (2011) [18] in carrot and watermelon. The increased concentration of maltodextrin decreased the hue angle and leading to the formation of more intense coloured powders. This can be attributed to the better pigment retention and higher antioxidant activity of powders produced with maltodextrin as the carrier agent.

**Particle size**

The effect of process parameters on the size of particles is analysed by Zetasizer system which gives the average size of particles in the developed microencapsulated cherry tomato powder. The sizes of the particles in cherry tomato powder were in the range of nano scale and are presented in Table 2. The average size of particles produced was within the size range of particles produced by atomization, which was in a range from 5 to 150 µm, according to Thies (1995). The particles produced in this study, however was smaller which varies from 516 to 2682 nm (0.5-2.6 µm). In present study, there was no particular effect of inlet temperature on particle size but as the maltodextrin level decreased the particle size increased. The presence of larger particles may be attributed to an incipient agglomeration process, where the formation of irreversible link bridges leads to the production of particles of greater size (Tonon et al., 2009) [30]. The larger particle size was found in samples with higher concentration of solids. These results are logical because the high concentration increased the amount of solid in each droplet exiting the nozzle. Therefore, when the water in each droplet evaporates, more the particles remain (Obon et al., 2009). Particle size distribution of the powder showed 3-4 distinct peaks (Fig.1) in all the treatment combinations, each one representing a predominant size varying from 0.1 to 10µM. It was also observed that higher percentage of particles is towards a size of 10 µM. Spray dried acai juice powder recorded similar size ranging from 9.01 µM (Tonon et al., 2010) [30].

**Surface morphology**

The analysis of surface morphology of powder particles was performed using Scanning Electron Microscopy (SEM) and the resulted images were shown in Fig 2. Most of the particles produced were spherical in shape, which is typical of materials produced by spray drying (Tonon et al., 2009) [30]. Treatment combination, 160°C+10 ml/min+10% MD (T_7) registered lower particle size resulted in the formation of more homogenous capsules with smooth surfaces, few wrinkles and less agglomeration whereas, particles produced in T_6 (160°C+6 ml/min+20% MD) showed more agglomeration with irregular shapes and several indentations. The shrivelled surface is due to slower heat transfer and resulting in particles with more pliable and collapsed crust (Allamilla-Beltran et al., 2005) [1]. The more regular and spherical particles were seen at higher temperatures. Similar behaviour was verified for cactus pear (Saenz et al., 2009) [23], acai (Tonon et al., 2009) [30] and milk powder (Nijdam and Langrish, 2006).
Fig 1: Particle size distribution of microencapsulated cherry tomato powder at different processing variables

T7 - 160°C + 10 ml/min + 10% MD

T6 - 160°C + 6 ml/min + 20% MD
Conclusion
Spray drying operational variables has significant influence on powder physico-chemical properties. Increased maltodextrin concentration increased TSS of the powder and pH got increased with higher inlet temperature. Acidity value decreased with increasing inlet temperature. Ash content found to be least affected by the operational parameters of spray drying. Increased inlet temperature reduced ascorbic acid content, lycopene content, antioxidant activity and total sugars. Reduced ascorbic acid content and antioxidant activity; and higher total sugar content was found with higher concentration of maltodextrin. Powder produced at higher temperature and lower concentration of maltodextrin with 10 ml/min feed flow rate was found to be the best with smaller particle size and good morphological characteristics. Combination of variables like 140°C inlet temperature, 6 ml/min feed flow rate and 20% MD was found to be good while considering the chemical parameters of microencapsulated cherry tomato powder while, treatment involving 160°C+10 ml/min+10% MD found to be better considering particle size and morphology.

Acknowledgement
The research was conducted in Kittur Rani Chennamma college of Horticulture, Arambhati and College of Horticulture, Bengaluru, Karnataka, India. I acknowledge all the teachers especially my guide Dr. Suresha G J, staff and students of Dept. of Post-harvest technology of both the colleges for guiding and helping me throughout my work.

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