MATERIALS
&
METHODS
3. MATERIALS AND METHODS

3.1 Processing of Pineapple Juice

Mature, ripe giant Kew variety pineapples (*Ananas Sativus*) were procured (50 kg) from the local market. Crown portion of the fruit was removed manually and then it was washed in running water to remove surface contaminants. Washed fruits were cut into \(\frac{3}{2} \times \frac{3}{2}\)" cubes after peeling. Cut cubes were subjected to juice extraction in a screw type juice extractor (Model 579 Raylon metal works, Bombay) fitted with 60-mesh sieve. The extracted juice was filtered through muslin cloth to remove fibrous fruit particles. The acidity and brix of 3 kg juice batch was adjusted to 1.5% and 20\(^\circ\) respectively by adding AR grade citric acid and cane sugar. The resulting juice was pasteurised at 80\(^\circ\)C for 5 min in an electrically heated pasteuriser and cooled to room temperature. Remaining juice (18 kg) was also subjected to pasteurisation treatment as above without adding sugar and acid, and divided into 8 equal parts of 2.25 kg each. First 2.25 kg juice was kept as such separately, while for the remaining 7 parts; brix was adjusted to 20 by adding cane sugar. Each of the seven parts was taken separately for additives treatment viz., malic (1.5%), Fumaric (1.5%), anticaking agents (tricalcium phosphate, tricalcium silicate and calcium oxide, 0.25% each) beta cyclodextrin (0.25%) trehalose (0.5%) and sorbitol (0.5%) incorporated to improve the handling properties and the stability of juice powder. 500 g of juice from each treatment was kept aside for initial analysis. All the above treated juices were freeze dried.
3.2. Processing of Mango Juice

50 kg of mature, ripe Alphonso (*Mangifera indica*) variety mangoes were procured from the local market. Fruits were washed in running water and soaked in 0.15 % potassium permanganate solution for surface decontamination. After surface decontamination fruits were subjected to skinning and cutting manually using knife. During cutting stones were kept separately. Cut mango bits were subjected to pulping in a mango pulper. Around 500 g of pulp was kept aside for initial analysis. The remaining pulp was pasteurised in an electrical pasteuriser with constant stirring with help of a stirrer. Acidity and °brix of the pasteurised, cooled juice were adjusted to 1.5 and 20 respectively using citric acid and cane sugar and then freeze-dried.

3.3. Processing of Vegetables

Fifteen kg each of good quality pumpkin (*Cucurbita maxima*) carrot (*Daucus carota*), potato (*Solanum tuberosum*) and green peas (*Pisum sativum*) were procured from the local market. All the vegetables were cleaned in running water to remove surface contaminants and dust. Pumpkin was cut into 1"X2" pieces manually and the skin, seeds and inedible portions were also removed manually. In case of potato and carrot skinning was done using abrasive peeler (Gardners, New Delhi). After peeling the carrot, potato and pumpkin pieces were further diced to about 1 cm cubes in a mechanical dicer (Urschell dicer, Germany). The diced vegetables were blanched for 5 min at 70°C in a solution containing 300 ppm of potassium meta-bi-sulphite to
inactivate the enzyme. Blanched vegetables were subjected to different dehydration processes.

3.4. Processing of Herbs

Five kg of locally available Pudina (*Mentha spicata* linn) was procured from the local market. Roots and other fibrous stalks were removed manually and washed in running water to remove dust and soil. About 500 g of fresh leaves were kept aside for initial chemical analysis. Cleaned leaves were soaked in 0.2% sodium hydrogen carbonate solution for 30 min to neutralize plant acid and washed thoroughly in running water to remove excess of carbonates. Soaked leaves were treated with 0.4% potassium metabisulphite followed by soaking in 0.15% glycerol at chilling temperatures (5°C). Drained leaves were taken for further analysis and dehydration.

3.5. Processing of Shrimp

50 kg of Shrimp (*Metapenaeus dobesoni*) was procured from Mangalore harbour and brought to laboratory in an ice container. Head, shell, tail and the vein (intestine) passing through the dorsal side of the abdomen were completely removed and the edible meat portion was used for processing. Peeled and de-veined shrimps were divided into 3 parts of equal weight and subjected to blanching in hot water containing NaCl and NaCl + Citric acid respectively at 70°C for 5 min. Blanching solution was kept aside to study the leaching of soluble solids. Blanched shrimps were subjected to freeze drying in SS trays.
3.6. Freeze Drying

Freeze drying was carried out in a pilot scale freeze dryer (Hull Corporation, USA), capable of rapid freezing. The samples were pre frozen to $-30 \pm 2^0 C$ in case of juices and $-20 \pm 2^0 C$ in case of vegetables and herbs for 2-3 hr in stainless steel trays. Drying was carried out under chamber pressure in the range of between 100-300 microns, using variable plate temperature. The plate temperature ranged from 35 – 70$^0 C$, the samples were dried to 1.2 to 6 % moisture depending on the type of the product. When the product was dried to required moisture level nitrogen gas was used to break the vacuum. The dehydrated products were removed from the drier and taken to the humidity and temperature controlled (20 ± 2 % RH and 25 ± 2$^0 C$) room for packing.

3.7. Hot Air Drying

Hot air drying of vegetables and herbs was carried out in a Kilburn cross flow cabinet hot air dryer (Model NO 012 E Motor capacity 0.25 HP, 3 phase, 440 v Heating load 3 kW). Samples were spread on drying trays (loading density 0.2 to 0.529 kg) and loaded into the dryer. The air velocity and the temperature of the dryer were maintained at 2.5 to 3 m/s and 60 ± 2$^0 C$ respectively. The dried product was taken to a low humidity room as above and packed in paper foil polyethylene laminate pouches without vacuum or nitrogen flushing.
3.8. Combination Drying

Both freeze drying and hot air drying techniques were employed for vegetables. Initially the freeze drying was carried out in a hull freeze drier as described above. About 2 kg quantities of each vegetable were removed periodically (2, 3, 4, 6 and 8 hrs) from the freeze drier and transferred to Kilburn cross flow cabinet hot air drier as described above for final drying. The product was unloaded from the dryer at 4 to 6 % moisture level and taken to a low humidity room as above for packing in paper foil polyethylene laminate pouches.

3.9. Simulated Model Studies

Sugar solutions (5 kg) similar to 14° brix pineapple juice were prepared using glucose / fructose / sucrose / and divided into 5 equal parts. The first portion was freeze dried as such while for the remaining portions acidity and brix were adjusted to 1.5 and 20° respectively by adding citric / malic / fumaric acids and sugar. All the solutions were freeze dried to observe the effect of acids and sugars on drying behaviour. Dried powders were packed separately to study the role of sugars and acids in hygroscopicity and thermal behaviour.

3.10. Controlled Crystallization

Freeze dried amorphous juice powders were subjected to critical relative humidity to convert into crystalline sugars. 2 kg of freeze dried juice powder was divided into 4 equal parts of 500g each and exposed to relative humidity ranging from 23 to 53% in a separate desiccators at 23 ± 2°C for 8 h.
After the exposure, the products were dried further in freeze drier by applying vacuum and temperature to remove surface water and powdered in a Raymond mill fitted with 60 mesh sieve. This product was subjected to hygroscopic and X ray diffraction studies to determine the degree of crystallization and hygroscopicity of the products.

3.11. Bulk Reduction or Compression of Juice Powder

Juice powder was mixed with cane sugar in the ratio of 1:2 and pulverized in a Raymond mill fitted with standard IS 44 mesh to get a uniform particle sized beverage powder. 30 g of beverage powder and freeze dried juice powder along with plasticizer was compressed in a laboratory model hydraulic carver press (Model 12-102T, 12 ton capacity) using 3/4" dia SS moulds by applying 100-110 Kgs/cm² pressure.

3.12. Analytical Methods

3.12.1. Moisture Estimation (Oven Drying Method)

The moisture contents of dried as well as fresh vegetables were determined using the standard AOAC (1984) procedures.

5 - 10 g of fresh or 2-4 g of dehydrated vegetables / herbs were taken in a previously heated and tared flat bottom aluminium dishes containing thin layer of asbestos. After weighing the dish cover was removed and kept for drying in an oven at 100°C for 6-8 h. The dried sample were cooled in a desiccator for 30-45 min and weighed. The moisture content of the samples was calculated as follows.
Moisture content \( = \frac{100(w_1 - w_2)}{(w_1 - w)} \)

where,
\[ w = \text{weight of empty dish} \]
\[ w_1 = \text{weight of dish before drying} \]
\[ w_2 = \text{weight of dish after drying}. \]

### 3.12.2. Moisture Estimation (Vacuum Oven Method)

Moisture content of freeze dried juice powders was determined using vacuum drying to a constant weight as per Ranganna (1995).

2 to 3 g of dried juice powder sample was taken in a previously tared flat bottom dish in a de-humidified room where relative humidity was maintained at 23 ±2% to avoid moisture uptake. Dish was covered with a lid and kept it in a desiccator containing \( P_2O_5 \) and transferred quickly to a vacuum oven for drying after uncovering the dish. The product was heated under vacuum for 12 to 16 h at 70° C. After drying, vacuum was broken through the trap containing calcium oxide. The product was taken from the oven and stored for 30 min in a desiccator and weighed. The moisture content of the sample was calculated as above.

### 3.12.3. Acidity

Titrable acidity was estimated as per the standard procedure described by Ranganna (1995). About 10 g of fresh or 5 g of dehydrated samples were
dissolved in distilled water and transferred to a clean 100 ml conical flask. The volume was made up using distilled water and filtered through Whatman filter paper No.41 to get clear solution. 10 ml of the filtrate was pipetted out into a clean dry 100 ml conical flask and titrated against standard 0.1 N sodium hydroxide solution using phenolphthalein indicator. From the titre value total acidity was expressed in terms of citric acid and calculated as follows.

\[
\text{Total acidity} = \frac{\text{Titre value x N of alkali x volume x Eq .wt of acid x 100}}{\text{Volume taken for titration x Weight of sample x 1000}}
\]

3.12.4. Sugars

Reducing and total sugar contents of the samples were also estimated as per Ranganna (1995).

20-25 g of fresh or 2-5 g of dehydrated samples were taken and dissolved in 50 ml water in case of juice and juice powder and soaked and blended with water in case fresh and dehydrated vegetables. After dissolving and blending the samples were transferred into cleaned 250 ml conical flask and the volume was made up to the mark with distilled water followed by filtration. The filtrate was titrated against standard Fehlings A and B solution using methylene blue as indicator. The entire titration was done under hot boiling conditions. From the titre value the reducing sugar content was calculated as follows.

\[
\% \text{ of reducing sugar} = \frac{\text{mg of inverted sugar x dilution x 100}}{\text{titre value x wt of the sample x 100}}
\]
25 ml from the above filtrate was taken in a 100 ml conical flask and hydrolysed with 5 ml of concentrated HCl at 60\(^{\circ}\) C for 30 min. The cooled solution was neutralized with 1 N Na OH to get a light pink colour and the volume was made up to 100 ml with distilled water. This was titrated against standard Fehlings A and B solution. From the titre value the total sugar content was calculated using the formula,

Non reducing Sugar content = % total invert sugar - % of reducing sugar x 0.95

Total sugar = % reducing sugar + sucrose

3.12.5. Determination of fat content

Fat content of the sample was determined as per AOAC (1990). The fat was determined by extracting 5 to 10 g of moisture free sample with petroleum ether for 6 to 8 hours in a Soxhlet apparatus. The solvent was evaporated by heating at 60\(^{\circ}\) C and the fat content was determined. The weighing was repeated until a constant weight was obtained. Increase in the weight of tared flask after complete evaporation of petroleum ether indicates the weight of fat.

\[
\text{% of FAT} = \frac{\text{Increase in weight of the flask} \times 100}{\text{Wt. of sample}}
\]
3.12.6. Free fatty acid content (FFA)

The FFA content of the samples was estimated as per AOAC (1990).

A known quantity of fat extracted from the above procedure was taken in a 100 ml flask and 50 ml of hot neutralized alcohol was added followed by 1-2 ml of phenolphthalein reagent. The flask was shaken vigorously to dissolve all the fat content and titrated with 0.25 N NaOH solution to get a faint pink colour. From the titre value, FFA content was calculated as follows:

\[
\text{Free fatty acid (\% of oleic acid)} = \frac{\text{ml of alkali} \times N \text{ of alkali} \times 28.2}{\text{Weight of fat}}
\]

3.12.7. Peroxide Value (PV)

Peroxide values of the samples were determined as per AOAC (1984). 0.5 to 1 g of extracted fat was dissolved in 30 ml Glacial acetic acid and 20 ml chloroform into a clean 250 ml iodine flask. 0.5 ml of saturated potassium iodide was added to the flask and kept in dark after nitrogen fleshing for 15 min. 50 ml distilled water was added and titrated against 0.02N Sodium thiosulphate solution using starch as an indicator. From the titre value, PV of the sample was calculated as follows and expressed as milli equivalent of peroxide per kg of fat.

\[
P.V. = \frac{V \times N \text{ of } \text{Na}_2\text{S}_2\text{O}_3 \times 1000}{\text{Wt .of sample}}
\]

\(V = \text{Volume of sodium thiosulphate used for test}\)
3.12.8. Determination of Malonaldehyde content

Malonaldehyde content of the samples was determined by the method of Tarladgis et al. (1960). 10 g of fresh or equivalent weight of dehydrated sample was blended for 2 minutes in a blender. The mixture was quantitatively transferred into a round bottom flask by washing with an additional 47.5 ml of distilled water followed by 2.5 ml of 4 N HCl to bring the pH to 1.5. Distillation was carried out to collect 50 ml of distillate within 10 minutes. 5 ml of distillate was taken in to 50 ml stoppered test tube and added 5 ml of TBA reagent (prepared by dissolving 1.4415 g TBA in 500ml of glacial acetic acid). Stoppered tube was immersed in boiling water for 35 minutes. After cooling OD was taken at 538 along with water and TBA reagent blank. From the value, malonaldehyde content was calculated with the help of standard curve.

\[
\frac{\mu g \text{ of Malonaldehyde}}{\text{kg of sample}} = \frac{\mu g \text{ of Malonaldehyde} \times 1000}{\text{Wt of samples (100-Moisture)}}
\]

3.12.9. Estimation of Protein

Protein content of fresh as well as dehydrated samples was estimated as per AOAC (1984). 2g of fresh or 0.5 g of dehydrated samples were digested with 40 ml of concentrated sulphuric acid and 0.5g of digestion mixture in a Kjeldhal digestion flask till a clear solution is obtained. 5 ml of this digested solution was distilled with 20 ml of 40 %Na OH solution. The distillate (50ml) was collected in 10 ml of 2% boric acid solution and titrated against
N/70 HCl using mixed indicator. From the titre value crude protein was calculated as follows:

\[
\text{Protein} \% = \frac{\text{Titre value} \times V \times 100 \times 6.25 \times N}{\text{Aliquot of distillate} \times \text{weight of sample}}
\]

\(V\) = volume of digested material made up.
\(N\) = mg of nitrogen equal to 1ml of HCl

3.12.10. Estimation of Sodium Chloride

Sodium chloride content of the samples was estimated as per Ranganna (1995). Macerated 10 g of fresh or equivalent weight of dried sample was heated with 25 ml of 0.1 N of silver nitrate and 15 ml of concentrated nitric acid in a 250 ml conical flask for 10 minutes on a hot plate. The digested sample was cooled to room temperature and titrated against standard 0.1 N Ammonium thiocyanate solutions by adding 50ml of water, 5ml ferric alum indicator and 2 ml of nitro benzene. From the titer value sodium chloride content was calculated as follows:

\[
\text{Sodium Chloride} \% = \frac{(V_1 - V_2) \times 5.85 \times 100}{M \times 1000}
\]

\(V_1\) = volume in ml of silver nitrate
\(V_2\) = volume in ml of standard ammonium thiocyanate solution
\(M\) = Mass in g of the sample taken
5.85 = One ml of 0.1 N silver nitrate solution is equivalent to 5.85 mg of sodium chloride
3.12.11. Vitamin C Content

Vitamin C content of the samples was determined using the procedure described by Ranganna (1995). 5 ml of ascorbic acid solution (10 mg/50ml) prepared in 3% metaphosphoric acid was titrated against (0.0025%) 2, 6 – dichlorophenol- indo phenol dye solution to a pink colour, which persisted for 15 sec. Dye factor, mg of Ascorbic acid per ml of dye was determined using the formula:

\[
\text{Dye factor} = \frac{0.5}{\text{Titre value}}
\]

10 g of fresh samples or 2-5 g of dehydrated samples were blended with 20 ml of 3 % metaphosphoric acid and made up to 100 ml using the same solution. About 10ml of the above filtrate was pipetted out into a clean conical flask and titrated against standard 0.0025% 2,6 – dichlorophenol- indophenol dye to a pink colour which persisted for 15 sec. From the titre value ascorbic acid content was calculated as follows:

\[
\text{Ascorbic acid mg/100g} = \frac{\text{Titre value x dye factor x volume made up x 100}}{\text{Aliquot taken x wt. of the sample}}
\]

3.12.12. Carotenoids

Total carotenoids content of fresh and dehydrated samples was determined as per the standard (AOAC 1990) Procedure. About 25-50 g of fresh samples or 5 to 10 g of dehydrated samples were ground in a blender using 50 ml water and transferred into a clean separating funnel. About 50 ml
acetone was added and shaken for few min. After thorough shaking 20 to 30 ml hexane was added by shaking and the pigment was dissolved in hexane layer. Finally hexane layer was separated out and transferred to a clean dry conical flask. The extraction was repeated thrice, the extract was dried over anhydrous sodium sulphate and transferred to a 250 ml volumetric flask after filtering through Whatman No 41 filter paper and made up to the level. The OD of the filtrate was measured at 450 nm and the total carotenoid content was calculated as follows:

\[
\text{Total Carotenoid content} = \frac{\text{OD} \times \text{Volume made up} \times 1000}{\text{Weight of the sample} \times 250}
\]

Molecular extension co-efficient of carotene = 250 at 450

3.12.13. Chlorophyll

Total chlorophyll content of the samples was estimated as per Ranganna (1995). About 5 to 10 g of fresh or 2-5 g of dehydrated samples was taken in a clean pestle and mortar along with a pinch of magnesium carbonate. Extraction of pigment was carried out with 50 ml of 85 % acetone by thorough grinding. The supernatant liquid was decanted. The extraction was repeated with acetone till the residue became colourless. The extract was filtered in to a 250 ml volumetric flask and volume was made up with acetone.

About 50 ml of diethyl ether was taken in a separating funnel and 50 ml acetone extract was added along with the addition of water until the water
layer was free of all the soluble pigments. The ether layer was washed 3 to 5 times with distilled water and transferred into a clean dry flask. The extract was treated with anhydrous sodium sulphate and filtered through Whatman 41 filter paper to a 100 ml volumetric flask and made up to volume with ether. The OD of the filtrate was measured at 660 nm and 642.5 nm. From the OD chlorophyll a, chlorophyll b and the total chlorophyll contents were calculated as follows:

\[
\text{Total chlorophyll mg/lit} = (7.12 \times \text{OD at } 660) + (6.8 \times \text{OD at } 642.5)
\]
\[
\text{Chlorophyll a mg/lit} = (9.93 \times \text{OD at } 642.5) - (0.777 \times \text{OD at } 642.5)
\]
\[
\text{Chlorophyll b mg/lit} = (17.6 \times \text{OD at } 642.5 \text{ nm}) - (2.81 \times \text{OD at } 660\text{nm})
\]

### 3.12.14. Pyruvate

Pyruvic acid content in fresh as well as dehydrated onions was estimated as per the standard procedure of Sigmund and Weston (1961). 2-5 g of fresh or 1to1.5 g of dehydrated onions was blended with 100 ml of distilled water in a blender for 2-3 minutes. The extract was filtered and 2 ml of the filtrate was taken in a test tube and treated with 1ml of 0.125% 2,4 DNPH prepared with 2 N HCl. The test tube was kept at 37°C for 10 min and 5 ml of 0.6N NaOH was added. After shaking, OD was measured at 420 nm. The concentration of pyruvic acid content in the samples was calculated with the help of standard graph constructed using standard sodium pyruvate.
3.12.15. Flavour Strength

Flavour strength of fresh and dehydrated onion samples was determined as per Shankarnarayana et al. (1981). 50 g of fresh or 7.5 g of dehydrated onion samples were homogenised with 100 ml water in a blender for 5 min and kept for 30 min. The slurry was transferred to a 2 lit round bottomed flask followed by washing and adjusting the volume to 500ml with water. Antifoam and glass beads were added to prevent frothing and ensure uniform boiling. Finally distillation head and condenser were connected and the steam was let into the flask. Around 250 ml of the distillate was collected by dipping the tip of the condenser tubing into a flask containing 35 ml of 10 N H₂SO₄, 15 ml water and 20 ml chloromine-T (0.2 N). This was titrated against 0.1 N sodium thiosulphate using KI and starch as an indicator to determine un-reacted chloromine-T. From the titre value volatile oil content was calculated as follows:

\[
\text{Total volatile oil content \%} = \frac{V \times N \times 100}{W \times F}
\]

V = Blank - sample titre value
N = Normality of thiosulphate
W = Weight of the sample
F = Factor (experimental value of chloramine-T per gram of volatile oil = 84)

3.12.16. Non-Enzymatic Browning

Non-enzymatic browning of the samples was determined as per the procedure of Ranganna (1995). About 1-2 g samples were soaked in/blended
with 66 % alcohol and made up to a known volume followed by filtration. The OD of the filtrate was measured at 420 nm.

3.12.17. Rehydration Properties

Rehydration properties viz., rehydration time, rehydration ratio and rehydration co-efficient of the dehydrated vegetables were determined as per Ranganna (1995). 5 g of dehydrated samples were soaked in 100 ml of distilled water in 250 ml graduated beaker. When the sample attained maximum absorption the time was recorded and excess water was removed by filtration under mild suction. The weight of the rehydrated sample was recorded and the rehydration ratio, rehydration co-efficient and percentage water in rehydrated material were calculated as follows:

\[
\text{Rehydration Ratio} = \frac{\text{wt of rehydrated sample}}{\text{wt of the dehydrated sample}}
\]

\[
\text{Rehydration Coefficient} = \frac{\text{Drained weight of dehydrated sample} \times 100 - \text{Moisture content of the sample before drying}}{\text{Weight of the dried sample taken for rehydration} - \text{Amount of moisture present in the dried sample taken for rehydration} \times 100}
\]

\[
\text{% of water in rehydrated material} = \frac{\text{wt of rehydrated sample} - \text{Dry matter in the sample}}{\text{Dry Weight of the rehydrated sample}}
\]
3.12.18. Total Volatile Content

Total volatile content of the samples was estimated as per Weurman (1969). 500 g of fresh or 100 g of dehydrated samples were taken in a simultaneous steam distillation evaporation flask and connected to a distillation head. 100 ml of pentane was taken in a 500 ml round flask and connected to receiving end of the distillation flask. The distillation was carried out for two h and the solvent containing flavour compounds was dried over anhydrous sodium sulphate, evaporated and weighed. The total volatile content was calculated as follows:

\[
\text{Total volatile content mg /kg} = \frac{1000 \times \text{wt of the volatile}}{\text{Weight of sample}}
\]

3.12.19. Total Ash

Total ash content of the samples was determined as per the procedure of Ranganna (1995). 5 – 10 g of the samples were taken in a previously weighed silica dish and ignited on a Bunsen burner. Ignited samples were kept in a muffle furnace and ashing was done at 600°C for 8 to 10 h. After sufficient cooling, sample was removed from the muffle furnace, kept in a desiccator for 30 to 40 min and weighed. The difference in the weight was taken and expressed as total ash content as follows:

\[
\text{Total ash %} = \frac{\text{Ash content}}{\text{Wt of the sample}} \times 100
\]
3.12.20. Sorption Characteristics/Hygroscopic Behaviour

Hygroscopic behaviour or sorption characteristics of dehydrated samples were determined as per the method of Resnik et al. (1984). 2 to 5 g of material was taken in a 3" dia petri dish and exposed to different relative humidity conditions ranging from 0 to 75 % using various salt solutions. Changes in their weight were recorded at hourly intervals in the first 24 hours and later at one-day intervals up to 7 days followed by weekly intervals until equilibrium was achieved. These data were used to calculate the equilibrium moisture content, water activity, monolayer moisture content and hygroscopicity of the samples (Brunauer et al. 1938).


Differential scanning calorimetry was used to determine the onset of endothermal change in specific heat (Tg) and isothermal crystallization of juice powders containing various additives and other dehydrated vegetables using the Differential Scanning Calorimeter, Model 2010 fitted with graphic plotter and the Thermal Analyser (TA instruments, USA) as per the procedure described by Jagannath et al. (1999 b) for Tg and Krause et al. (1984) for isothermal crystallization experiments. 5-10 mg samples were weighed into standard aluminium pans in a dehumidified room to avoid moisture up take along with empty pan. The hermetically sealed sample pan was held at a temperature, low enough to ensure no crystallization for 1 min. The sample was then ramped up to a maximum temperature of 320° C. The final isothermal holding temperature where it was held for 20 minutes during which time crystallization was observed and crystallization curves were recorded.
with respect to time. Taking the end of the induction period as zero time, the peaks were integrated with respect to time. Degree of crystallization was plotted against time for each peak. The results were analysed using the avrami equations (Avrami 1939) as follows:

\[ Q(t) = 1 - e^{-k t^n} \]

Where,

\( Q(t) \) is the relative crystallization at time \( t \), and \( k \) is the isothermal crystallisation rate, \( n \) is the Avarami index. Using the method of least squares \( K \) and \( n \) values were obtained.

### 3.12.22. X-ray Diffraction

The X-ray diffraction pattern of the freeze dried juice powders, subjected to controlled crystallization and juice powders exposed to different relative humidity were measured on a STOE/STADI-P X-ray Powder Diffractometer with a graphical interface of Microsoft Windows NT or Windows 95 software – Win XPOW. The samples were collected on a STOE/STADI-P powder X-ray diffractometer with Germanium monochromated CuKa (25 mA, 30 kV \( \lambda = 1.540598 \text{ Å} \)) radiation in transmission mode. The samples were rotated during data collection to reduce orientation effects and the data were recorded using a curved position sensitive detector (PSD). The X-ray data were measured in the range of \( 2\theta = 3 \) to \(-57^0 \) at steps of 0.03 at room temperature with \( 6^0/ \text{min scan rate.} \)

Freeze-dried juice powders, juice powders subjected to rehumidification at different relative humidities and other dehydrated samples were sprayed on to double stick cellophane tape pasted on copper stubs used for mounting specimens for scanning electron microscopy. All the above operations were carried out in a dehumidified room where the RH and temperature were maintained at 23 % + 2° and 26%+ 2° C respectively. The samples on the copper stubs were then coated with about 20 nm gold in a sputter coater (EMS-550). Observations and photography were made with a JEOL 100 CX –II electron microscope fitted with a scanning attachment (ASID-4D) at 20 KV.


Metals in food samples were determined using AAS as per the method of Jacob (1958). Powdered food sample (20 g) was digested by heating in a Kjeldahl flask with concentrated HNO₃ and H₂SO₄ mixture for oxidation of carbonaceous matter in each sample, a blank was prepared simultaneously by taking similar amounts of HNO₃ and H₂SO₄ mixture. Care was taken during heating so that no excessive foaming took place. Concentrated HNO₃ in small amounts was added until all the organic matter was oxidised. This point was reached when no further darkening of the solution occurred on continuous heating and a clear solution was obtained. It was cooled and transferred to 100ml volumetric flask and the volume was made up with de-ionised water. The concentration of various metals was determined using AAS (Model AA670, Shimadzu, Kyoto, Japan) by aspirating the solution into
the oxygen-acetylene flame. The instrument was calibrated using standard solutions (1-6 mg/l) of various metal salts.

13.12.25. Sensory evaluation

Sensory evaluation of dehydrated as well as rehydrated samples was evaluated as per the Hedonic scale by an experienced panel of judges.


The data obtained from the experiments were subjected to the analysis of variance using suitable statistical software like MS-Excel.

All the chemicals and standards used in the above investigations are of highest purity from the reputed firms.