

# Quality Attributes of Various Spray Dried Pulp Powder Prepared from Low Temperature Stored Calcium Salts Pretreated Guava Fruits

Renu Rahel, A. S. Chauhan, K. Srinivasulu, R. Ravi, V. B. Kudachikar

**Abstract**—The effect of calcium salts on the storage stability and on the quality attributes of both fresh and processed product (guava powder) of white flesh guavas (var 'Allahabad safeda') was studied. The pulp behavioral studies of fully ripened guava fruits indicated that fruits pretreated with 3% and 4.5% calcium chloride had the least viscosity. The guava pulp powder using spray drying technique was developed and its storage stability and the moisture sorption studies were carried out for product quality evaluation at normal storage condition (27°C; 65%RH). Results revealed that powder obtained from 3% calcium chloride pretreated guavas was found to be at par with the powder obtained from control guavas after 90 days of normal storage. Studies on microbiological quality of guava pulp powder indicated that among the treatments powder obtained from guava fruit pretreated with 3% calcium chloride to be the most effective through restricting microbial counts of total plate count, yeast, mold, *Staphylococcus* and *E. coli* below their permissible limit. Moisture sorption studies of guava powder revealed that foil laminate 12µm PET/9 µm foil/38-40 µm is the most suitable packaging material recommended.

**Keywords**—White flesh guava, calcium salts, spray drying, powder, storage stability.

## I. INTRODUCTION

GUAVA (*Psidium guajava* L.) is a known tropical fruit characterized by a low content of carbohydrates, fats, proteins, and high vitamin C (more than 100 mg/100 g fruit) and fiber content (2.8–5.5 g/100 g fruit) [1]. In addition to its nutritious properties, this fruit is very appetizing due to its sensory (flavor and color) properties [2]-[5].

In spite of the fact that there are a range of guava processed products, such as, marmalades, jellies, juices and soft drinks, this fruit is usually consumed in the fresh stage. However, the export of fresh guavas from producing countries has been restricted because this fruit is highly perishable and susceptible to tropical fruit fly attack.

Nowadays, the interest of the food industry in natural flavor and color-enriched additives has been increased significantly due to the consumer's non-preference for synthetic food

additives with potential short and long-term health risks. Most of the tropical fruits possess intense colors and flavors which make them excellent source of new and diverse natural additives. Therefore, an interesting alternative is to transfer those molecules responsible for sensory and/or bio-functional properties to a solid phase which will be able to enhance their stability and control their release in various intended food matrices. In this way, microencapsulation is a process in which tiny particles of an active principle or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix to give small capsules [6]. In this form, the active principle is protected from external conditions and its release is controlled. Among different methods to obtain microcapsules, spray-drying is the most used in food industry. This is a unit operation by which a liquid sample (solution, emulsion or suspension) is atomized in a hot gas current to instantaneously obtain a free flowing powder. At present, this technology is well established, rather inexpensive and straightforward [7]. Recently, attempts were made on the development of new and sensory-enriched processed products from tropical fruits in order to generate promising alternatives for producers, which let to reduce the loss of the production by postharvest handling, and give to consumer's new reliable food additives [8], [9]. A few attempts to produce powders from guava have been published.

Dehydration of guava pulp into powdered form with good rheological property that gives a considerable reduction in volume and is an effective method of prolonging the shelf life. Clarified guava juice powders were obtained by dehydrating the concentrated guava juice using freeze, spray and tunnel drying techniques [10] and found that the freeze-dried product exhibited superior sensory quality and the spray dried guava powder was stable and more economical to produce free-flowing guava powder with good stability. Instant guava-drink-powder may be prepared by grinding the dehydrated slices. Guava powder can be used in the preparation of guava juice, RTS beverage or milk shake [11]. It was reported that the maltodextrin serves as carrier and facilitate the drying [12]. In a previous study [13], the sensory properties of four guava extracts obtained by aqueous homogenization, osmodehydration with ethanol or sucrose and soxhlet extraction with ethanol were compared. It was found that aqueous extract exhibited the highest sensory similarity (color and flavor) with the fresh fruit and also the major vitamin C content.

Extending shelf life with least packaging requirements and

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reducing the bulk during transportation are the aims of drying food products while diversified fruit and vegetable powders can be used in many products such as instant soups, snacks, bakery, beverage, dairy, candy, ice cream, baby food, and pasta [14], [15]. Spray drying is a highly appropriate process for heat-sensitive products such as foods and results in powders with good quality, low water activity, and easier transport and storage [16]. It has been widely used in many fruits and vegetables such as guava [10], pineapple [17], cashew apple [18], pomegranate [15], mango [19]. Due to the enormous process variables influencing the quality of powders and in order to obtain products with better characteristics and process yield, it is important to optimize the drying process [20]. One of the main parameters in spray drying is feed characteristics [20]. Appropriate feed viscosity and solids concentration are necessary to easily pump the feed into a spray dryer and obtain acceptable yield [20], [21]. The common feed for spray drying of fruits and vegetables is concentrated juice or high solid puree while low solid content of the feed results in no or insignificant powder collection [10]. On the other hand, the main problem in the spray drying of fruits and vegetables is stickiness and a part of this problem can be solved by addition of some carriers such as maltodextrin (MD) [15], [21]-[25], which is the most common carrier used because of its neutral color, taste, and relatively low cost [22]. Maltodextrin is considered as hydrolyzed starch and it is obtained by the action of either acids or enzymes [26]. The addition of MD in food material prior to spray drying can reduce stickiness and agglomeration problems during storage of the end product [27]. MD negatively affects performance by increasing viscosity [17]. Viscosity reduction aids in the formation of fine spray [10]. Despite a large number of studies performed on optimization of spray drying of fruits and vegetables, there is a lack of sufficient knowledge on the development of spray dried powder from the calcium salts pretreated, low temperature stored white flesh guavas after extended shelf life and postharvest quality maintenance.

Thus, the objective of this work was to develop naturally flavored powders from calcium salts pretreated guava fruits after effective shelf life of 32 days at low temperature, by using the spray-drying technique and to evaluate their quality attributes such as physicochemical, rheological, microbiological and sensory properties as well as their stability under different humidity conditions, for suitability of packaging films and for shelf life predictions. The long term purpose of this work is to develop spray dried guava powder packed in suitable packaging film for higher shelf stability.

## II. MATERIAL AND METHODS

### A. Selection of Raw Material

Green, firm and optimally matured (10-11% soluble solids content) white flesh guava "*Psidium guajava*. L. cv. 'Allahabad safeda'" fruits were harvested from a commercial guava orchard from Bangalore, India. Sorted, graded for uniform size and color. Fruits were loaded into flexible plastic crates (FPC) with proper cushioning with paper strips at the

base and transported to CFTRI, Mysore, India, for 4 hrs duration and were transferred to pre-cooler room ( $2\pm 1^\circ\text{C}$ , 92-95% RH) for 6 hrs to remove field heat and to reduce the fruit metabolic activity.

### B. Pretreatment with Calcium Salts

The selected fruit lots were divided into five groups (60kgs/group), water washed, dipped in the solutions of various chemicals [3, 4.5% calcium chloride (T-1, T-2); 0.4%, 0.8% calcium propionate (T-3, T-4); and untreated control (T-0)] each for 15 minutes and surface of fruits was air dried under shade for 30 minutes.

### C. Storage Conditions

Both the treated and untreated fruits were stored at low temperature (LT) condition ( $7\pm 1^\circ\text{C}$ ; 90-95% RH).

### D. Sampling Method for Storage Experiment

For each treatment six replicates of samples with 10 kgs of guava per replicate were maintained. From each treatment three replicates of samples were labeled to record fruit colour on each sampling day. All these samples along with the remaining samples (three replicates of each treatment) were placed in FPC and stored at  $7\pm 1^\circ\text{C}$ ; 90-95% RH.

### E. Physicochemical Analysis of the Samples

For analyzing physicochemical quality parameters, samples were periodically (0, 8, 16, 24 and 32<sup>nd</sup> days) drawn. The data was obtained for each determinant in three replicates for each treatment and were used for analysis for physicochemical characteristics such as texture, pH, total soluble solids (TSS), titratable acidity and total sugars. The homogenate of pulp sample (6 fruits) was used for analysis of these chemical parameters. In addition, fully ripe guava fruits were characterized by their colour parameter, pH, total soluble solid content, acidity content (expressed as grams of citric acid per 100 g of fruit), sugars, ascorbic acid and total carotenoids as per the following standard procedures.

#### 1. Measurement of Penetration Force (N)

Fruit texture of the guava fruits was measured in terms of penetration test by using a Texture Measuring system (Lloyd, LR5K, Lloyd Industry Co. Ltd., UK) fitted with a needle probe (8 mm diameter) with load cell 1 Kilo Newton (KN) and cross speed of 50mm/min. The randomly selected fruits were placed at the base of the Texture measuring system. The force required to penetrate the fruit surface up to a depth of 5 mm and is expressed in terms of the Newton (N). All estimations were carried out in three replicates per treatment.

#### 2. Measurement of Colors (Hunter Values)

The color of the skin and pulp of the guava fruit was measured using color measuring system (Hunter Labscan XE, Hunter Laboratories, USA) at wavelength ranging from 400 to 700nm and expressed in terms of L, a and b (Hunter values). The Hue angle [ $= \tan^{-1} b/a$ ] and Chroma [ $= (a^2 + b^2)^{1/2}$ ] values were calculated by using the average of three replicates for 'a' and 'b' was considered for the calculation purpose for Hue angle ( $h^\circ$ ) and Chroma value [28].

### 3. Determination of Total Soluble Solids ( $^{\circ}$ brix)

The soluble solids content (SSC) of the homogenate sample was measured by using a digital refractometer (ATAGO-RX 5000, Japan) and was expressed in terms of  $^{\circ}$ brix.

### 4. Determination of Moisture Content (MC)

Moisture content of the sample was determined by using a calibrated Digital Moisture Analyzer (Denver instruments Germany, Model-IR 35, Germany) at  $110\pm 1^{\circ}\text{C}$ . A sample of 2-3 g in triplicate was exposed to the heated infra-red coils of the moisture analyzer and the analysis was completed in an automatic mode within 15-20 minutes.

### 5. Measurement of pH

The pH of the homogenated sample was measured by using a glass electrode pH meter (Model: APX 175, Control dynamics pH meter, India) at room temperature, which was calibrated using standard buffer solutions at pH 7.0 and 4.0.

### 6. Determination of Titratable Acidity (as % Anhydrous Citric Acid)

Acidity of the homogenate sample was determined by the standard method [29] and is expressed in terms of anhydrous citric acid in percentage.

### 7. Determination of Non-Enzymatic Browning (NEB)

Non-enzymatic browning (NEB) was estimated using standard procedures [29]. The sample of known weight 1g was added to the 10ml of 60% ethyl alcohol and kept at room temperature for 1 hour, then filtered through Whatman filter paper. Optical density of the sample was taken at 420nm using 60% alcohol as blank and OD was directly expressed to interpret the results.

### 8. Determination of Ascorbic Acid

The ascorbic acid content was determined using 2, 6 Dichlorophenol-Indophenol visual titration method [29] and expressed in mg/100g.

### 9. Determination of Total Carotenoids

Known weight of (10g) of homogenated sample was extracted and filtered with petroleum ether and then its OD was read at 450nm in a calibrated UV-Visible spectrometer (UV-1601, SHIMADZU, JAPAN) as per the standard procedure [29]. The total carotenoids were expressed in terms of  $\mu\text{g}/100\text{g}$ .

### 10. Determination of Sugars (%)

The reducing sugars (%), non-reducing sugars and total sugars (%) in the homogenate samples were estimated as per the Lane and Eynon method [30].

### 11. Measurement of Viscosity of Guava Pulps

The viscosity of the guava pulp was measured in a Brookfield Viscometer (Model: R1:3: M, M/s. Rheology International Ltd, Shannon, Ireland) at RT with the spindle No.7 at 100 rpm for one minute. The readings were expressed in mPas. The fruit pulp behavioral studies indicated that fruits pretreated with 3% and 4.5% calcium chloride showed the

least viscosity and therefore, the spray dried guava powders were developed from them were considered as the most suitable processed product. Further, detailed studies of these developed spray dried guava powders in terms of microbiological load, sensory analyses, moisture content, non-enzymatic browning, shelf life prediction, moisture sorption and rheology studies were carried out.

### F. Spray Drying of Guava Pulps for Powder Production

After effective storage life (of 32 and 24 days in pretreated and control respectively) of fruits stored at low temperature ( $7\pm 1^{\circ}\text{C}$ ), becomes soft, turned color to yellow and were allowed to fully ripen at RT ( $27\pm 1^{\circ}\text{C}$ ) for 3 days. Before processing, the fully ripe fruits (T-0: Control; T-1: 3% Calcium chloride; T-2: 4.5% Calcium chloride; T-3: 0.4% Calcium propionate; T-4: 0.8% Calcium propionate) were dipped into sodium hypochlorite (0.05%) solution to achieve an acceptable sanitary status and then processed. The guava pulp was collected separately by pulping in a fruit pulper (B.SEN Barry & Co, New Delhi, Mesh size 0.80mm) and 1.289g of the pulp combined with water and sugar in a ratio of 1/1 (w/w) (200g water + 200g sugar). The pulp was treated with 1% Pectinase enzyme mainly for liquefaction and reduction of viscosity. The formulation comprised of citric acid (0.25%), Tri-calcium phosphate (0.25%), and Maltodextrin (6%). The spray-drying process as illustrated in Fig. 1 was performed in a pilot scale Bowen spray-drier (BE 1216, Bowen Engineering Inc., USA). The nozzle of 2.0 mm diameter was coupled with the spray drier. The prepared feed (homogenized guava slurry,  $28\pm 1^{\circ}\text{C}$  temperature) was atomized through a nozzle into small droplets to the spray drier chamber (air velocity 1.0 to  $1.5\text{Kg}/\text{cm}^2$ ) at 2 bar air pressure in a co-current air flow system (total air flow 250 cubic feet meter). The feed (guava slurry) flow rate was regulated at 60 mL/min by peristaltic pump, and the inlet and outlet air temperatures were set at  $140\pm 2^{\circ}\text{C}$  and  $110\pm 2^{\circ}\text{C}$ , respectively. Thus, the guava spray-dried powders from various guava slurries (homogenized) prepared from all the pretreated guavas were obtained through a cyclone to the collection container of the spray drier. The guava powder obtained as illustrated in Fig. 2 (a), was hygroscopic in nature and therefore the powder was packed in aluminum foil pouches, sealed [Fig. 2 (b)] and was stored at RT ( $29\pm 1^{\circ}\text{C}$ ) for their storage stability and product quality evaluation studies. Further, moisture sorption behavior and storability studies of the spray dried guava powder were carried out for product quality evaluation at ambient storage ( $25\text{-}28^{\circ}\text{C}$ ) condition.

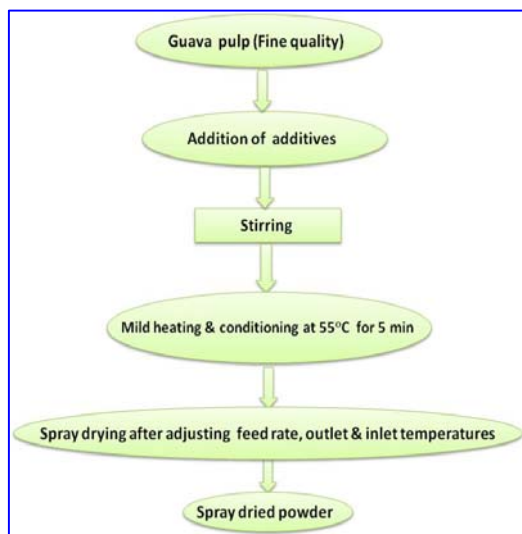


Fig. 1 Flow chart for the preparation of spray dried guava pulp powder



(a)



(b)

Fig. 2 (a) Spray dried guava pulp powder; (b) Spray dried guava pulp powder Packed in foil laminate

#### G. Rheology Testing of Powders

The guava powders were tested for their flow behavior employing a Powder Flow Analyzer (Stable Micro Systems, Surrey, UK). The Powder Flow Analyzer system constituted by a vertical glass container (120mm height and 50mm internal diameter) and rotating specific blade (48 mm diameter

and 10 mm height), which can provide rotational movement while traversing in vertical directions. The flow ability properties were evaluated during the displacement in a controlled manner of the rotating blade inside the container, filled with powder sample [31]. A fixed volume of spray dried guava pulp powder (180g) was poured into the container prior to testing. The instrument did a three cycle sample preparation step while moving vertically down and up with a tip speed of  $50 \text{ mms}^{-1}$ . Compaction property of powder was examined when the rotating blade moved downwards, while it measured cohesion property, while moving upward at a tip speed of  $50 \text{ mms}^{-1}$ . The test was repeated three times and the whole test procedure was repeated twice. The maximum force required for compaction studies was known from the peak value of force while moving downwards. The energy required for compression and decompression were calculated from positive and negative areas, respectively of the force–time curves by multiplying the area under the curve by the tip speed of the rotating blade. The rotation was measured over specific time period and a graph is plotted using force (in Newtons) and time (in seconds) parameters.

#### H. Moisture Sorption Behavior of Powders

Known quantity of the powder was taken in petri dishes and exposed to different levels of relative humidities (RHs) ranging from 11 to 92% built in desiccators using appropriate saturated salt solutions [32]. The samples were periodically weighed till they attained practically constant weight or showed signs of mould growth whichever was earlier. After equilibration, the moisture content (MC) of the product at different RHs was calculated by adding /subtracting % pick up/loss to/from the initial moisture content. The sensory remarks on their quality were taken and critical moisture content was fixed.

#### I. Sensory Evaluation

The guava powder prepared from both treated and control fruits were evaluated for sensory quality and acceptance of the product. The sensory properties were evaluated by a trained panel using a STD QDA scale [33]. Samples were randomly served to the panelists in white cups coded with 3 digit random numbers. The panelists were instructed to indicate their perceived intensity of the samples on the score card.

#### J. Microbiological Evaluation

The microbiological load of spray dried guava fruit powder (Initial and after 90 Days of storage) was determined by checking the fungal and bacterial growth in the product for safety of the consumers. Homogenous sample was prepared by mixing the contents. Portions (25g) of sample were taken and diluted in 225ml saline. Samples were further diluted serially if required and plated for quantitative analysis of aerobic mesophilic plate count, yeast and mold count, *Staphylococcus spp.* and detection of *E. coli* using PCA ( $37^\circ\text{C}/24 \text{ hr}$ ), PDA ( $25^\circ\text{C}/72 \text{ hr}$ ), BPA ( $37^\circ\text{C}/48 \text{ hr}$ ) and MCA ( $37^\circ\text{C}/48 \text{ hr}$ ). The colonies developed after incubation at  $37^\circ\text{C}$  for 24-48 hrs are counted and expressed as in  $\text{Log}_{10} \text{CFU/g}$  [34].

### K. Shelf Life Predictions of Guava Pulp Powders

The shelf life of the product was predicted using, M pack software developed in house prediction of the shelf life of moisture sensitive products [35]. The packaging schedule considered for predicting shelf life were Water Vapour Transmission Rate (WVTR) of Packaging material, 100g product packed in a pouch of dimensions of 11X16cms and storage conditions such as accelerated condition (38°C/90% RH) and Indian Standard condition (27°C/65% RH) and also moisture sorption isotherm of the product.

### L. Statistical Analysis

All determinations were conducted three times at least. An analysis of variance (ANOVA) of the data was evaluated by Statistical Analysis System (SAS). Duncan's Multiple Range Test was employed to determine the statistical significance ( $p \leq 0.05$ ) of differences between the means [36].

## III. RESULTS AND DISCUSSION

Results of guava storage studies revealed that the shelf life of fruits pretreated with 3% calcium chloride was extended up to 32 days as compared to 24 days in control fruits at low temperature (LT). Both calcium salts pretreated and control fruits after effective storage life were ripened at RT (28-32°C, 60-75% RH) respectively and were used for further studies in the present investigation.

### A. Quality Attributes of Guava Fruits (Calcium Salts Pretreated, Control and Fresh)

The physicochemical quality attributes of fully ripe guava fruits pre-treated with calcium salts are depicted in the Table I. Texture is an integral of any fruit and fruit product. At the end of fresh storage at LT (Table I), all the calcium pre-treated guava fruits have shown firm texture (penetration force in 'N') in comparison with the control samples such as T (fresh control) and T-0 (control after 24 days LT storage). Among all the calcium salt pre-treated guava fruit samples, T-1 (3% CaCl<sub>2</sub>) sample showed significantly highest firmness in terms of penetration force (N) as compared to T-2, T-3 & T-4 guava fruit samples.

Hue angle of the peel of calcium salt pre-treated LT stored guava fruit sample was more negative in T-3 sample followed by T-2, T-4, T-0 (control, LT stored) and T (fresh control) whereas less negative was observed in T-1 calcium salt pre-treated LT stored guava fruit sample. This may be correlated to the high concentrations of calcium propionate and calcium chloride salts, which might have accumulated in the skin of guava fruits and responsible for the change in fruit skin colour.

Chroma means color of fruit without lightness. It pertains to color quality of any fruit with respect to stage of ripeness in guava fruits and its change depends on pre-treatments and storage temperature. Chroma value of guava fruit peel of T-2 sample (32.99) was found highest followed by T-1, T-4 and T-3. Least chroma value was found in fresh control sample (T, 20.94) as compared to another control (T-0), which was stored at LT. It is revealed from the results that change in chroma of all the guava fruits was showing the stage of pale yellowness.

Total soluble solids (TSS) have increasing trend in T-2 and T-3 calcium salt pre-treated LT stored guava fruit samples. The high concentration of calcium salt can make the fruit more firm and hard so guava fruits might have proceeded to more moisture loss; hence, increase in TSS of T-2 and T-3 samples were significantly different from other samples. The other samples like fresh control (T), T-1 and T-4 did not have any significant increase in the TSS content. The LT stored control sample (without calcium salt treatment) has shown the least TSS content. It was also observed that acidity (as % anhydrous citric acid) in control sample of guava fruit was less whereas another control sample (T-0) and calcium salt pre-treated guava fruit samples (T-1, T-2, T-3, T-4) stored at LT have shown increase in the content of acidity. Among all the pre-treated and LT stored guava fruit samples, T-1, T-3 and T-4 have shown non-significant level in acidity whereas T-0, and T-2 samples also showed similar kind of non-significant level in their acidity but found to be significantly different from the main fresh control sample (T), which may be due to the increase in the ascorbic acid content in the guava fruit samples. Our finding indicates that not only anhydrous citric acid is accumulating in the guava fruits but calcium salts also may contribute towards increase in the acidity of the guava fruits in the form of calcium ions. Similar kind of trend was found in pH values of all the control (T), control and calcium salt pre-treated guava fruits stored at LT. It could be due to the correlation of acidity with pH values.

Ascorbic acid is heat sensitive vitamin and considered to be very important from nutrition point of view. Hence, control samples (T and T-0) have shown degradation in ascorbic acid content. In case of all the LT stored calcium salt pre-treated guava fruit samples (T-1, T-2, T-3 and T-4) has shown increasing trend in their ascorbic acid content. Ripening enhances the ascorbic acid content during LT storage of calcium salts pre-treated guava fruits.

As far as sugars (total sugars and reducing sugars) are concern, there was a significant increasing trend among all the guava fruit samples (T-1, T-2, T-3 & T-4) in comparison with the control guava fruit samples (T and T-0). T-4 sample has shown highest total sugar (6.25%) whereas reducing sugar was found to be increased more in T-2 sample. Among all the LT stored guava fruit samples, T-1 sample has shown least total sugars and reducing sugars accumulation. The reason behind increasing trend of total sugars and reducing sugars could be attributed to the conversion of starch into sugars either monosaccharide or disaccharides.

Total carotenoids were found more in both the control guava fruit samples (87.27 µg/100g in T sample and 80.52 µg/100g in T-0 sample) due to fast progression of ripening as compared to LT stored calcium salt pre-treated guava fruit samples (T-1, T-2, T-3 and T-4). Among calcium salt pre-treated guava fruit samples stored at LT, T-2 sample showed highest total carotenoids and lowest in T-4 sample. The variations among the total carotenoids of LT stored guava fruit samples pre-treated with calcium salt may be because of combined effects of low temperature and calcium salts, therefore, ripening was delayed.

### B. Guava Pulps

The effect of calcium salts and influence of LT storage conditions on the physicochemical characteristics of fully ripen guava fruit pulps are shown in the Table II. Hue angle of the guava fruit pulp obtained from calcium salt pre-treated LT stored guava fruit sample was more negative in T-3 sample followed by T-4, T-2, T-1, T-0 (control, LT stored) and T (fresh control). This may be correlated to the influence of calcium salts and low temperature storage conditions on the guava fruits thus resulted in more negative hue angle in their fruit pulps. The calcium propionate has shown the more profound effect as compared to calcium chloride on the LT stored guava fruits, hence, resulted in more negative hue angle irrespective of its concentration. Results clearly revealed that the hue angle was negative in all the guava pulps which are the indicative of increase in lightness of all the guava pulps because of decrease in their hue angle.

Chroma defines the saturation level in color quality of any fruit and its product and it will not include lightness. It increases depending on the pre-treatments and storage conditions of the fresh fruits and that also will be reflected in the prepared pulp. Chroma value of guava fruit pulp of T-1 sample (18.65) was found highest as compared to their counterparts like T-0, T-3, T-0 (control, LT stored guava fruit sample) and least in T-4 sample. More chroma value of the prepared guava pulp is the indicative of good ripeness and good quality. All the guava pulps were having creamy color with slight tinge of pale yellowness.

Moisture was found more in both the control samples such as fresh control sample (T) (91.30%) and LT stored control sample (T-0, 84.58%). In case of calcium salts pre-treated LT stored guava fruit samples resulted in decrease of moisture content. Among calcium salts pre-treated guava fruit samples, T-4 sample had higher moisture content followed by T-1, T-3 and T-2 samples. The more decrease in moisture content of stored guava fruit pulps may be due to the influence of optimum concentration of calcium salt but increase in its concentration have resulted in decrease of moisture content of the guava fruit pulps because of desiccant nature of the calcium ions, which tends to reduce moisture depending on the concentration of the calcium ions accumulated in the pre-treated guava fruits.

A reverse trend was observed for solids of the pulps prepared from calcium salts pre-treated LT stored guava fruits. The trend clearly supports that more moisture content guava pulp resulted in less solids whereas less moisture content guava fruit pulp has shown more solids. The increase in solids of the guava fruit pulps might be due the stage of ripeness so starch conversion might have started to sugar. The less solids means ripening might have delayed in that particular sample, which is influenced by the pretreatments like calcium salts.

The rheological behaviors of the fruit pulp of fully ripe guava fruits treated with various pre-treatments were measured in terms of viscosity. Viscosity of the guava pulp (3806.50 mPaS) obtained from T-0 sample (control, LT stored guava fruits) was found more than the other samples such as T-3, T-4, T-2 and T-1. The pulp obtained from control sample

(T, fresh control: without treatment) has shown least viscosity (1015.80 mPaS). During pre-treatment with calcium salts, the calcium ions binds with the pectin molecules present in middle lamella of the cell wall and thus provides firmness to the LT stored guava fruits. Therefore, pulp prepared from the calcium propionate (0.4 %) pre-treated guava fruit has resulted in little higher viscosity as compared to their counterparts. The guava pulp obtained from T-0 sample (LT stored guava fruits) has shown more viscosity, which indicates more viscous nature of the pulp.

### C. Guava Pulp Powders (Spray Dried)

Table III shows the initial quality characteristics of various guava pulp powders (T, T-0, T-1, T-2, T-3 & T-4 samples) obtained from various guava pulps and compared with each other for evaluation purposes. As it has been reported earlier [12] for production of a grayish white powder after spray drying guava puree without an additive. They observed that the moisture content of all spray dried powders was lower than that of the freeze dried powders and the final moisture content in the spray dried powder was 2.24 %. Sugar was added to improve the retention of color and flavor and to improve drying characteristics of the final product. Similar observations were reported by other researcher [37]. There was increase in °brix and total titrable acidity and a decrease in pH following drying, which may be the result of concentration accompanied by release of sugars and acids from maltodextrin and citric acid during drying. Ascorbic acid was lost during drying as a result of the high temperatures and oxidation. The average loss of ascorbic acid was around 21%. The other researcher [12] found a similar loss of 19.1% ascorbic acid content in spray dried guava puree. The T-0 samples (untreated control) showed higher value of color 15.13 and 1.95% acidity when compared to the other pretreatments.

As depicted in Table IV, the titrable acidity decreased in guava fruit powder treated with 0.8% calcium chloride when compared with other treatments and control. There was no major difference due to treatments T-1 (3% calcium chloride) and T-3 (0.4% calcium propionate). The ascorbic acid content increased during LT storage. The total sugars, reducing sugar and nonreducing sugars in T-1 (3% calcium chloride) had lower values ( $P < 0.05$ ) when compared to other treatments. Similarly, the ascorbic acid content of the puree was reduced by spray drying [38].

### D. Microbiological Quality of Guava Pulp Powders

The microbial load of the product was determined by checking the fungal and bacterial growth in the developed product for safety of the consumers. A low level of fungal and bacterial contamination was detected in all the processed guava products (Table V). The total plate count (TPC) ranged from 2.10 to 4.68 log CFU/g whereas yeast & mold count ranged from 2.46 to 2.62 log CFU/g. Similar results were reported in foam-mat dried mango [39]. In guava powder pretreated with T-3 (0.4% Calcium propionate), no *Staphylococcus* and *E. coli* was detected.

According to FSSAI regulation [40] fruit powder should contain less than 40,000 CFU/g and except control (T-0), all the samples were acceptable. As these products did not show any significant change in moisture (2.5 to 2.72%; 3.0 to 3.6%) during 90 days of storage, the microbial quality was assumed to be safe at this low level of moisture content.

The product pretreated with 3 % Calcium chloride restricted the growth of microbial counts like bacteria, yeast and mold to 2.10 (log CFU/g) and 2.46 (log CFU/g) respectively. The microbiological counts like *Staphylococcus* and *E. coli* were absent in all the guava powders whereas other microbiological counts like total plate count, yeast and mold count) were much below the specified limit of FSSAI [40] that may be because of less moisture content present in the product so microbes did not have enough moisture to multiply. Therefore, all the powders were found safe for consumption.

#### E. Sensory Quality of Guava Pulp Powders

Results on sensory evaluation of the guava pulps (Fig. 3) and powders (Fig. 4) showed that among the treatments the overall acceptability of T-1 (3% calcium chloride) was found higher than the other treatments. Sensory analysis showed that the use of calcium improved the textural characteristic of the guava powder during storage. The researcher [41] found that sensory evaluation of the samples treated with calcium propionate and calcium chelate (40mM) were taste free and did not impart a lip feel, while other researcher [42] did not

find significant differences in sensory attributes (browning, texture or off-flavors) between samples treated with calcium lactate and calcium chloride.

#### F. Rheology of Guava Pulp Powders

Powder flow is defined as the relative movement of a bulk of particles among neighboring particles or along the container wall surface. The forces involved in powder flow are gravity, friction, cohesion (inter-particle attraction) and adhesion (particle-wall attraction) [43]. The flow ability properties were evaluated during the displacement in a controlled manner of the rotating blade inside the container, filled with powder sample [31]. Sample flow curves of various guava powders during three successive compressions and decompressions in the powder rheometer are illustrated in Figs. 5 (a)-(d).

The sample flow curve obtained during the three cycle compression and decompression of guava powder in the powder rheometer. The similar pattern of graphs in three cycles of compression and decompression indicates the consistent behavior of powder during rheometric studies.

Increase in moisture content has been found to increase cohesiveness, wall friction and effective angle of wall friction [44]. In case of T-0 (Control), the peak (maximum) force offered by guava powder (moisture content 2.58%) during compression is in the neighborhood of at initial (0 day) was 5.9 N [Fig. 5 (a)] and at increased to 6.9 N at 90 days with 3% moisture content [Fig. 5 (b)].

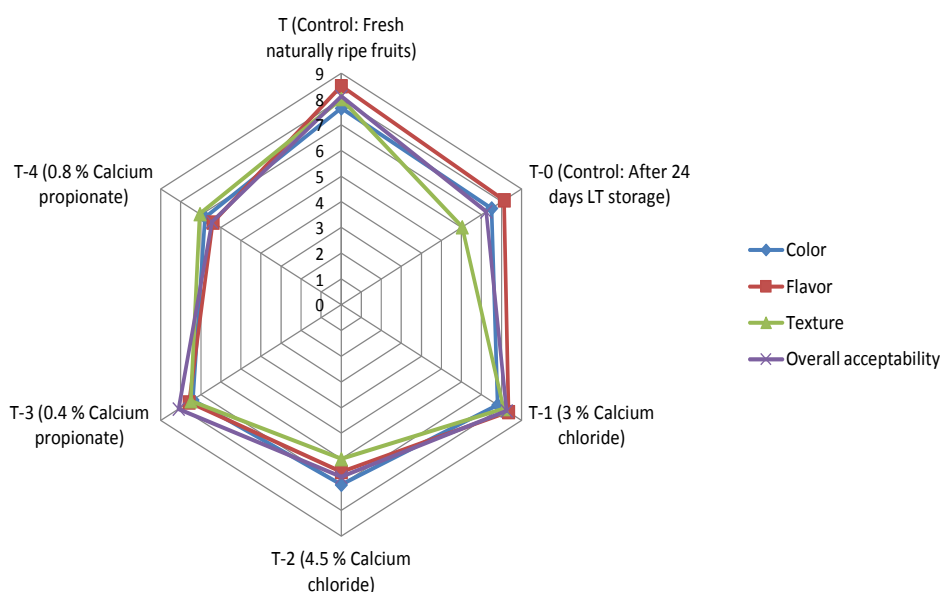


Fig. 3 Sensory profile of various guava pulps (n = 12)

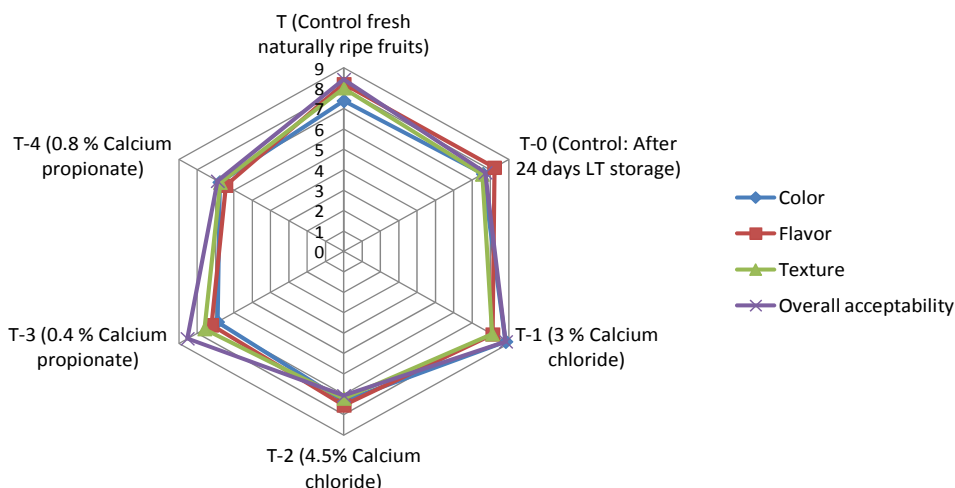


Fig. 4 Sensory profile of various guava pulp powders (n = 12)

TABLE I  
FRUIT QUALITY ATTRIBUTES OF FULLY RIPE GUAVA FRUITS PRETREATED WITH CALCIUM SALTS

Fruit quality attributes	Treatments					
	T (Fresh, naturally ripe fruit)	T-0 (Control: after 24 days LT storage)	T-1 (3% calcium chloride)	T-2 (4.5% calcium chloride)	T-3 (0.4% calcium Propionate)	T-4 (0.8% calcium propionate)
Penetration Force(N)	6.76 <sup>b</sup>	5.91 <sup>a</sup>	7.11 <sup>c</sup>	6.61 <sup>b</sup>	6.74 <sup>b</sup>	6.85 <sup>b</sup>
Hue angle of peel (°)	-75.25 <sup>b</sup>	-76.28 <sup>b</sup>	-72.21 <sup>a</sup>	-76.70 <sup>b</sup>	-77.99 <sup>c</sup>	-76.47 <sup>b</sup>
Chroma value of peel	20.94 <sup>b</sup>	26.47 <sup>c</sup>	32.76 <sup>d</sup>	32.99 <sup>d</sup>	21.34 <sup>a</sup>	32.25 <sup>d</sup>
TSS (°Brix)	10.50 <sup>b</sup>	8.83 <sup>a</sup>	10.20 <sup>b</sup>	11.70 <sup>c</sup>	11.20 <sup>c</sup>	10.40 <sup>b</sup>
pH	4.10 <sup>a</sup>	4.42 <sup>c</sup>	4.25 <sup>b</sup>	4.20 <sup>b</sup>	4.32 <sup>c</sup>	4.52 <sup>d</sup>
Titration Acidity (%)	0.34 <sup>a</sup>	0.51 <sup>c</sup>	0.44 <sup>b</sup>	0.51 <sup>c</sup>	0.42 <sup>b</sup>	0.43 <sup>b</sup>
Ascorbic acid (mg/100g)	41.94 <sup>a</sup>	54.40 <sup>c</sup>	59.75 <sup>e</sup>	58.78 <sup>d</sup>	51.84 <sup>b</sup>	61.72 <sup>f</sup>
Total Carotenoids (µg/100g)	87.27 <sup>f</sup>	80.52 <sup>e</sup>	45.74 <sup>c</sup>	69.75 <sup>d</sup>	22.16 <sup>b</sup>	19.70 <sup>a</sup>
Total sugars (%)	4.35 <sup>b</sup>	3.69 <sup>a</sup>	5.10 <sup>c</sup>	5.98 <sup>d</sup>	5.11 <sup>c</sup>	6.27 <sup>d</sup>
Reducing sugars (%)	1.85 <sup>a</sup>	2.26 <sup>b</sup>	3.19 <sup>c</sup>	5.01 <sup>f</sup>	4.63 <sup>c</sup>	4.30 <sup>d</sup>
Non-reducing sugars (%)	2.26 <sup>f</sup>	1.43 <sup>c</sup>	1.68 <sup>d</sup>	0.97 <sup>b</sup>	0.46 <sup>a</sup>	1.97 <sup>e</sup>

Mean value in a row with different super script differ significantly at p<0.05 by DMRT (n=3).

TABLE II  
EFFECT OF PRETREATMENTS ON THE FRUIT PULP BEHAVIOR OF THE FULLY RIPE GUAVA

Treatments	Viscosity (mPaS)	Hue Angle (°)	Chroma value	Moisture (%)	Solids (%)
T (Fresh, naturally ripe fruit)	1015.80 <sup>a</sup>	-80.70 <sup>a</sup>	18.01 <sup>c</sup>	91.30 <sup>d</sup>	8.70 <sup>a</sup>
T-0 (Control: after 24 days LT storage)	3806.50 <sup>f</sup>	-80.71 <sup>a</sup>	17.68 <sup>b</sup>	84.58 <sup>e</sup>	15.42 <sup>b</sup>
T-1 (Calcium chloride: 3%)	1332.65 <sup>b</sup>	-81.18 <sup>b</sup>	18.65 <sup>d</sup>	80.86 <sup>b</sup>	19.14 <sup>c</sup>
T-2 (Calcium chloride: 4.5%)	1428.40 <sup>c</sup>	-82.04 <sup>c</sup>	17.42 <sup>b</sup>	76.32 <sup>a</sup>	23.68 <sup>d</sup>
T-3 (Calcium propionate: 0.4%)	2216.05 <sup>e</sup>	-88.64 <sup>d</sup>	17.92 <sup>b</sup>	77.13 <sup>a</sup>	22.87 <sup>d</sup>
T-4 (Calcium propionate: 0.8%)	1735.75 <sup>d</sup>	-88.03 <sup>d</sup>	16.21 <sup>a</sup>	81.70 <sup>b</sup>	18.30 <sup>c</sup>

Mean value in a column with different super script differ significantly at p<0.05 by DMRT (n=3).

TABLE III  
INITIAL QUALITY ATTRIBUTES OF SPRAY DRIED GUAVA PULP POWDER

Fruit Quality attributes	Treatments					
	T (Fresh, Control without storage)	T-0 (Untreated Control, after 24 day storage)	T-1 (3% calcium chloride)	T-2 (4.5% Calcium Chloride)	T-3 (0.4% calcium propionate)	T-4 (0.8% calcium propionate)
Moisture (%)	2.58 <sup>a</sup>	3.69 <sup>d</sup>	2.72 <sup>b</sup>	3.47 <sup>c</sup>	2.96 <sup>c</sup>	3.12 <sup>d</sup>
Solids (%)	96.99 <sup>b</sup>	96.33 <sup>a</sup>	97.28 <sup>b</sup>	96.69 <sup>a</sup>	97.03 <sup>b</sup>	96.89 <sup>b</sup>
Titration Acidity (%)	0.96 <sup>a</sup>	1.95 <sup>e</sup>	1.42 <sup>d</sup>	1.35 <sup>c</sup>	1.43 <sup>d</sup>	1.14 <sup>b</sup>
Ascorbic acid (mg/100g)	31.58 <sup>a</sup>	42.67 <sup>c</sup>	48.12 <sup>c</sup>	46.38 <sup>d</sup>	39.51 <sup>b</sup>	49.63 <sup>f</sup>
Total sugars (%)	5.74 <sup>c</sup>	6.30 <sup>e</sup>	5.97 <sup>d</sup>	5.44 <sup>c</sup>	4.85 <sup>a</sup>	5.23 <sup>b</sup>
Reducing sugars (%)	2.52 <sup>a</sup>	3.72 <sup>d</sup>	3.50 <sup>c</sup>	3.13 <sup>b</sup>	3.58 <sup>c</sup>	3.03 <sup>b</sup>
Non-reducing sugars (%)	3.21 <sup>c</sup>	2.58 <sup>c</sup>	2.77 <sup>d</sup>	2.58 <sup>c</sup>	1.27 <sup>a</sup>	2.20 <sup>b</sup>
Chroma value	9.17 <sup>b</sup>	15.13 <sup>c</sup>	7.16 <sup>a</sup>	12.20 <sup>d</sup>	11.60 <sup>c</sup>	11.40 <sup>c</sup>
Hue angle (°)	-86.82 <sup>a</sup>	-87.37 <sup>b</sup>	-86.08 <sup>a</sup>	-88.23 <sup>c</sup>	-88.65 <sup>c</sup>	-87.66 <sup>b</sup>
NEB (OD at 420nm)	0.09 <sup>a</sup>	0.19 <sup>b</sup>	0.43 <sup>c</sup>	0.39 <sup>d</sup>	0.24 <sup>c</sup>	0.28 <sup>c</sup>

Mean value in a row with different super script differ significantly at p<0.05 by DMRT (n=3).



TABLE IV  
THE CHANGES IN QUALITY ATTRIBUTES OF SPRAY DRIED GUAVA PULP POWDER DURING NORMAL STORAGE

Quality attributes	Treatments			
	Initial (0 Day)		After 90 Days	
	T-0 (Control without Storage)	T-1 (3% Calcium Chloride)	T-0 (Control without Storage)	T-1 (3 % Calcium Chloride)
Titratable Acidity (%)	0.96 <sup>b</sup>	1.42 <sup>c</sup>	0.90 <sup>a</sup>	1.42 <sup>c</sup>
Ascorbic acid (mg/100g)	31.58 <sup>a</sup>	48.12 <sup>c</sup>	40.15 <sup>b</sup>	47.99 <sup>c</sup>
Total sugars (%)	5.74 <sup>a</sup>	5.97 <sup>b</sup>	5.71 <sup>a</sup>	5.76 <sup>a</sup>
Reducing sugars (%)	2.52 <sup>a</sup>	3.50 <sup>b</sup>	2.49 <sup>a</sup>	3.48 <sup>b</sup>
Non-reducing sugars (%)	3.21 <sup>c</sup>	2.77 <sup>b</sup>	3.22 <sup>c</sup>	2.28 <sup>a</sup>
Chroma value	9.17 <sup>c</sup>	7.16 <sup>a</sup>	8.15 <sup>b</sup>	9.73 <sup>d</sup>
Hue angle (°)	-86.82 <sup>a</sup>	-86.08 <sup>a</sup>	-88.57 <sup>b</sup>	-88.98 <sup>b</sup>
NEB (OD at 420nm)	0.09 <sup>a</sup>	0.43 <sup>c</sup>	0.38 <sup>b</sup>	0.37 <sup>b</sup>
Moisture (%)	2.58 <sup>a</sup>	2.72 <sup>a</sup>	3.00 <sup>b</sup>	3.60 <sup>b</sup>
Solids (%)	96.99 <sup>a</sup>	97.28 <sup>a</sup>	97.00 <sup>a</sup>	96.40 <sup>a</sup>

Mean value in a row with different super script differ significantly at  $p < 0.05$  by DMRT ( $n=3$ ).

TABLE V  
INITIAL MICROBIOLOGICAL PROFILE OF SPRAY DRIED GUAVA PULP POWDER

Treatment code	Total plate count (log cfu/g)	Yeast & mold (log cfu/g)	<i>Staphylococcus</i> (log cfu/g)	<i>E. coli</i> (log cfu/g)
T Control without storage	3.23±0.12 <sup>c</sup>	2.62±0.12 <sup>b</sup>	0	Absent
T-0 Untreated control (after 24 days)	4.68±0.15 <sup>d</sup>	2.52±0.10 <sup>a</sup>	0	Absent
T-1 Calcium chloride (3%)	2.10±0.17 <sup>a</sup>	2.46±0.12 <sup>a</sup>	0	Absent
T-2 Calcium chloride (4.5%)	2.71±0.12 <sup>b</sup>	0	<1	Absent
T-3 Calcium propionate (0.4%)	2.25±0.24 <sup>a</sup>	0	<1	Absent
T-4 Calcium propionate (0.8%)	2.81±0.10 <sup>b</sup>	0	<1	Absent

Values are expressed as mean ± standard deviation; Mean value in a column with different super script differ significantly at  $p < 0.05$  by DMRT ( $n=3$ ).

TABLE VI  
PREDICTION OF SHELF LIFE OF GUAVA PULP POWDER IN VARIOUS PACKAGING CONFIGURATIONS AND STORAGE ENVIRONMENTS

Product variant	wvtr (g/m <sup>2</sup> . d) at 38°C/90% RH gradient	Pkg wvtr (g at 38°C/90% RH gradient /pkg.d)	Shelf Life (in days)	
			65% RH/27°C	90% RH/38°C
Guava powder (T0: Control)	1.0	0.028	350	116
	0.5	0.014	701	234
	0.2	0.0056	>730	588
Guava powder (T-1: 3% calcium chloride)	1.0	0.028	444	150
	0.5	0.014	890	302
	0.2	0.0056	>730	>730

The sample curve of T-1 (3% calcium Chloride) also showed similar patterns of graphs in three cycles of compression and decompression indicates the consistent behavior of powder during rheometric studies [Figs. 5 (c) & (d)]. It is possible that these powders behave differently with calcium treatments as it shows the peak force offered by T-1 (3% calcium chloride) was 6.3N at initial day (moisture content 3%) (Fig. 5 (c) and 7.1N at 90 days with moisture content 3.60 [Fig. 5 (d)].

The area of compression zone is always higher than that for decompression zone for all the samples indicating that lesser energy is required for decompression of powder compared to compression. Because guava powder is moisture sensitive, the moisture content plays an important role in cohesiveness. As relative humidity of the surrounding air is increased, guava powder tends to absorb water molecules, which may form liquid bridges between powder particles and result in greater powder cohesion and reduced flow ability [Fig. 5 (d)].

#### G. Moisture Sorption Behavior of Guava Pulp Powders

Moisture Sorption isotherm: In order to design functional economic package for two variants guava fruit powders (T-0: control and T-1: 3% calcium chloride) which are highly hygroscopic, their moisture sorption characteristics were

studied. The moisture sorption isotherms as illustrated in Fig. 6 revealed that both formulations are almost similar and sigmoid indicating the typical of low fat starch rich products. The curves start raising high above 32% RH indicating the products deteriorates faster above 32% RH. The products (T-0: control and T-1: 3% calcium chloride) with an Initial moisture content of 2.58% to 2.72% on as is basis equilibrates to 16 and <11% RH respectively. These products equilibrating up to 22% RH in the moisture range of 3.34 to 3.93% was good and free flowing. Both these products equilibrated to 32% with moisture contents of 5.40 to 6.31% respectively, had a tendency for caking but could be broken by applying little pressure. These products equilibrated to 44% RH with a moisture content of 8.06 and 9.27% respectively had caked and at higher RH they became hard, watery and developed mould growth at 92 and 86% RH in 2 and 3 weeks' time. Hence, the moisture content equilibrating to 32% RH was taken as critical for both guava powder variants. The moisture tolerance of these products are 5.40-2.58=2.82% and 6.31-2.72=3.59% (The difference between initial and critical moisture contents) respectively. The spray dried powder from T-1 (3% calcium chloride) appears to be slightly better in storage stability due to its higher water holding capacity. Even though moisture tolerance is good, the critical RH being low,

it requires a high moisture barrier for extended shelf life. The sorption isotherm fits to the GAB equation as per GAB generalized equation for sorption phenomena [45] with  $R^2 > 0.98$ . The GAB constants are  $M_0=7.05$ ,  $C=2.8445$ ,

$K=0.9402$  for T-0 (control) Guava powder and  $M_0=7.1651$ ,  $C=4.0749$ ,  $K=0.9649$  for T-1 (3% calcium chloride) guava powder respectively.

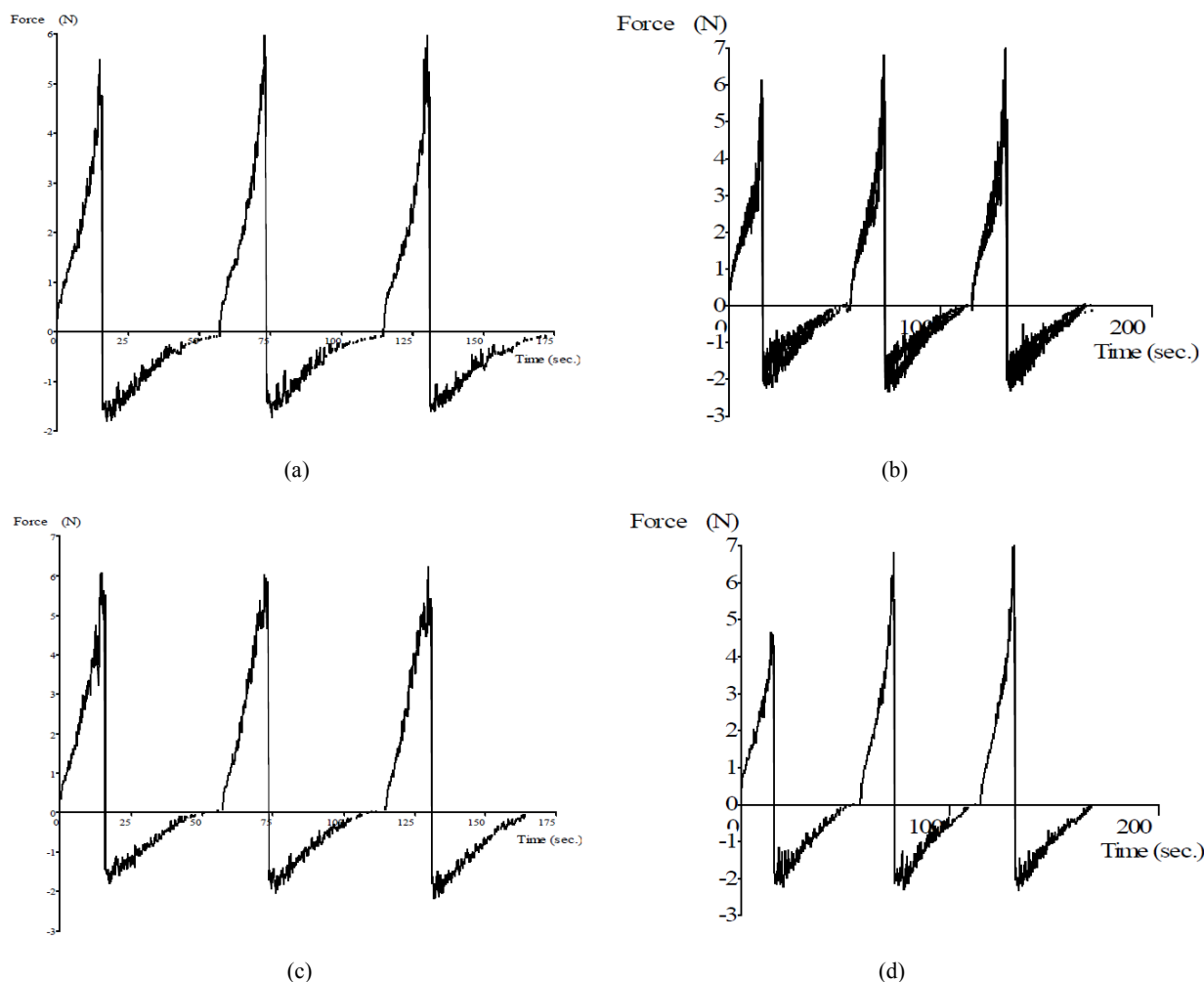


Fig. 5 (a)= T0-Control (0 Day); (b)= T0-Control (90<sup>th</sup> Day); (c)= T1-3% Calcium Chloride (0 Day); (d)= T1-3% Calcium Chloride (90<sup>th</sup> Day) illustrate flow curves of various guava powders during three successive compressions and decompressions in the powder rheometer

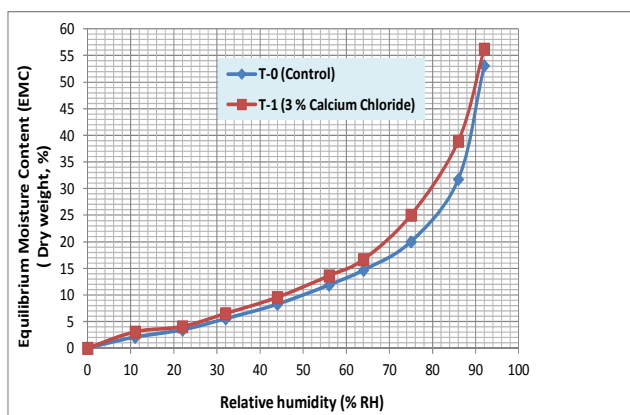


Fig. 6 Moisture sorption isotherm of various guava powders at 27°C

#### H. Packaging Studies for Suitability of Films and Shelf Life Predictions of Powders

The shelf life of the product was predicted using, M pack software developed in house to “prediction of the shelf life of moisture sensitive products” using GAB parameters and packaging Schedule. The developed method has been validated with a biscuit which is also a Moisture sensitive product as per Prediction of Moisture changes in packed glucose biscuits stored under variable Temperature and RH conditions. As depicted in Table VI, the predicted shelf life of two guava powder variants (T-0: control, T-1: 3% calcium chloride) are <1 year, 2 years and > 2 years when packed in packaging material of WVTR 1.0, 0.5 and 0.2 respectively under normal (27°C; 65%RH) storage condition. When many available packaging materials were screened 12µm Met PET/38µmPE of Optical Density (OD)>2.5 has WVTR close to 1.0 and 15µm Met BOPP/ 45µm CPP and 12µm PET/ 9 µm

Al foil/ 38  $\mu\text{m}$  LDPE laminates have WVTR of about 0.5 offer about 700 days shelf life under 65%RH/27°C it fails to offer > 1year shelf life under 90%RH/38°C. However, 9 $\mu\text{m}$  Al Foil laminate offers >2 year shelf life under 65%RH/27°C and >1 year shelf life under 90%RH/38°C and also from permitted moisture from outside. The calculation is based on 100 g unit pack (in cm area) pouch. However, the moisture content of the product will increase due to NE and also due to high moisture tolerance. The increase in moisture content in 90 days being <0.5% when kept in foil laminate, at least one year shelf life can be expected under normal storage condition.

#### IV. CONCLUSIONS

Results revealed that the shelf life of optimally matured guava fruits pretreated with 3% calcium chloride was extended up to 32 days at LT (7 $\pm$ 1°C; 90-95% RH) condition as compared to control fruits for 24 days. These fruits were fully ripened at RT (28-32°C, 60-75% RH) respectively. The fruit pulp behavioral studies indicated that fruits pretreated with 3% and 4.5% calcium chloride showed the least viscosity and therefore, the guava powder using spray drying technique was developed from them as most suitable processed product. Further, the storage stability studies of the guava powder were carried out for product quality evaluation at normal storage conditions. It is evident from the RT storage studies that guava powder obtained from 3% calcium chloride pretreated guavas were found to be on parity with the control after 90 days of normal storage. The initial microbial counts (TPC and yeast and mould) in guava powder pretreated with T-3 (0.4 % calcium propionate) was very low and pathogens were absent. Moisture sorption studies of guava powder revealed that foil laminate (12 $\mu\text{m}$  PET/9  $\mu\text{m}$ ) or foil (38-40  $\mu\text{m}$ ) is the most suitable packaging material for guava powder. The guava powder (spray dried) developed in this study represents an innovative and natural processed product from 3% calcium chloride pretreated and LT stored guava fruits, which can be incorporated into different food products due to its nutritional (ascorbic acid and sugars) and sensory quality attributes.

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