

## Effect of Spray Drying on Recovery and Physicochemical Properties of Acid Lime Juice Powder

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Acid lime is known for its tart flavor and is commonly used for culinary uses. Market glut during its peak production and high demand in its off season has been a key issue in the post-harvest handling and management of acid lime. Processing this fruit into a powder form for its several food uses can be solution to avoid distress sale by farmer, making a ready to use product available to the consumers. Spray drying has been a novel technique for powder production from liquid foods without affecting its nutritional properties. Experiments were carried out for production of spray dried powder from acid lime juice using Maltodextrin as additive. The parameters viz., temperature of inlet air (140–160°C), concentration of maltodextrin (14–18%) and feeding rate (62.5–112.5 mL/h) were optimized with Response Surface Method. The optimized levels of temperature of inlet air, concentration of maltodextrin concentration and feeding rate were, 147.63°C, 16.27% and 90.94 mL/h, respectively for desirable recovery and quality of powder. The powder prepared at optimized conditions resulted maximum recovery (18.77%), water solubility index (94.788%), vitamin C (365.937 mg/100g) and total phenols content (183.695 mg/100g). Acid lime powder produced at optimized conditions showed mixture of heterogeneous particles (150 to 6000 nm). The scanning electron microscopy analysis presented variable shape particles with smooth surface as result of encapsulation.

**Keywords:** *Citrus aurantifolia* (Christm), Encapsulation, Maltodextrin, Phule Sharbati, Response surface methodology

### Introduction

Citrus fruits are most commonly grown in tropical and subtropical parts of southeastern Asia. Worldwide, Brazil occupies top most position in citrus production followed by China and United States contributing about 75% of the total production. There is tremendous variation in the cultivars of citrus fruits. The most common citrus fruits are sweet orange, lemon, mandarin, limes and grapefruit. Acid lime is important citrus cultivar grown in Asia, North Africa and Europe. India ranks first in the world in the production of acid lime.<sup>1</sup> There has been increase in the popularity and hence cultivation of acid lime in India. Adaptability to sustain diverse environmental conditions, perennial fruit production, extended shelf life and comparatively lesser input are the boosting factors in increase of cultivation of acid lime. India's cultivation area and production of lime is about 0.32 Mha, 3.83 MT, respectively.<sup>2</sup>

Acid lime fruits are more aromatic in flavor and scent than other citrus fruits. It is very popular for its

beautiful appearance, pleasing flavor and for excellent nutritional qualities. Fruits have their tangy flavor; therefore, they are generally used as a garnish in many different dishes. Being rich in ascorbic acid content, acid lime has been important part of meal. It is being uses in many kitchen preparations as a taste enhancer, garnishing salad. Further, it is also being consumed in the form of processed products like juice (*Sharbat*), Ready to Serve (RTS) drinks, pickle and cordial. Under the circumstances of COVID 19, the ascorbic acid rich citrus fruit including acid lime has gained tremendous importance in the human diet.<sup>3</sup>

Processing and bottling into juice and RTS beverages are some of the options for post-harvest management of acid lime. There are few industries in India which are processing the citrus products into juice and other beverages. However, juice also has limited storage life, therefore converting the acid lime juice into power by spray drying; drum drying etc. has been emerged as new technique. Reduced cost of packaging, handling, storage and extended shelf life from months to years based on packaging are the advantages of converting juice into powder form.<sup>4</sup>

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Increased surface area due to atomization of liquid juice droplets in drying chamber produces uniform spherical shaped powder particles.<sup>5</sup>

Quality of powder obtained with spray drying depends upon feeding rate, temperature of air and quantity of carrier agent used.<sup>4,5</sup> Drying air temperature and encapsulating agent expressively affects the physical quality of powder like moisture content, particle size etc.<sup>6,7</sup> Therefore, proper standardization of drying process parameters is of very importance to obtain quality powdered material.

There are several studies on production of powder using spray drying technique. Effect of this method on superiority of prepared powder is reported in the literature.<sup>4</sup> Application of spray drying technique for preparation of orange powder has been explored.<sup>8,9</sup> Powder yield and properties of powder affected by spray drying have been studied.<sup>10</sup> Spray-Vacuum drying of orange powder along with studies on physicochemical properties, glass transition and water sorption phenomenon has been reported.<sup>11</sup> Process of beet root powder production with spray drying has been optimized using response surface methodology. Temperature (160°C), feeding rate (400 ml/h) and maltodextrin quantity (15%) resulted into a desirable quality powder.<sup>12</sup> Mango powder has been prepared using spray drying technique along with its comparison with cast tape drying. The carotenoid content of mango powder with spray drying was reported higher (113 µm/g of dry mass) than the cast drying method.<sup>13</sup>

Temperature of glass transition (T<sub>g</sub>) is at which transition of amorphous material from its hard, brittle nature to molten, soft or rubbery like occurs. Above the T<sub>g</sub>, the powder material of amorphous nature changes from crystalline to rubbery stage which ultimately increases the stickiness of the powder. Higher T<sub>g</sub> is desirable for preparation of good quality free flowing powder and it can be achieved by incorporation of encapsulating material.

The carrier agent makes spray drying effective by elevating the T<sub>g</sub>, hence improving the recovery and product qualities. The normally used carrier agents in area arabic gum, starch and its derivatives like maltodextrin, gelatin, pectin, whey protein, sucrose etc.<sup>6,7</sup> Spray dried powder was prepared of lemon juice by means of spray drying with maltodextrin as drying aid in the concentrations between 10 and 20%. Further a Ready to serve (RTS) dry mixture was formulated with combination of lemon and aonla

powder using RSM.<sup>14</sup> Some of the biomaterial based drying agents such as ground mesocarp of citrus fruit in combination with maltodextrin had also been explored and proven to obtain good quality lemon juice powder.<sup>15</sup> Limited studies are reported on application of spray drying to obtain powder from acid lime. However, spray drying of citrus fruit like orange has been studied with different process parameters and drying aids.<sup>8,9</sup> Addition of carrier agent should be minimal as possible to obtain a powder of desirable properties and also to keep the cost of processing minimized.

RSM is an analytical technique used to augment the process that reduces the experiments and explores the response of parameters in an efficient manner.<sup>16</sup> It has been used for process standardization of powder production of beet root.<sup>17</sup> In general, the range of temperature and concentration of encapsulating material adopted are (120–180°C) and (7–20%), respectively.<sup>4</sup>

Effect of encapsulation material properties and dryer parameters on physicochemical attributes of powdered sample of different fruits prepared with spray drying is evident from the literatures.<sup>7,10,11</sup> So far acid lime has not been explored more to obtain a powder from its juice. There is glut in market during peak production of acid lime which leads to increased loss of fruit and farmer does not get good returns. On the other hand, there is increasing demand for the ready to serve/ready to eat products because of changing life styles and disposable income especially in the urban area. Value addition to acid lime fruit by processing into stable powder could address the issues of fruit loss, help to provide better returns to the farmers and will make the ready natural product available to consumers. Drying of acid lime juice without changing its nutritional properties needs proper optimization and control of the operating parameters during drying. The present investigation was therefore carried out on preparation of acid lime powder of spray drying process, optimizing the parameters viz., quantity of maltodextrin, temperature and feeding rate, for better powder recovery and desirable quality (vitamin C, solubility, phenolic content, and particle size) of acid lime powder.

## Materials and Methods

### Raw Material

Fruits of acid lime (*Citrus aurantifolia* (Christm) Cv. Phule Sharbati) were arranged from Research

Farm, Horticulture Department, M.P.K.V., Rahuri for the experimentation. The fruits were harvested at proper maturity indices viz., TSS of 8°B and when the color of fruits was pale yellow.

#### **Spray Dryer**

Spray dryer (Make: Labultima, Model: LU 222) was used for the experimentation. The water evaporation rate of the dryer at 250°C inlet temperature was 1000 mL/h.

#### **Sample Preparation**

The acid limes were washed properly under tap water and the juice extraction was done using juice squeezer. The juice was strained through strainer and used for analysis and experiments. The titratable acidity, pH, color and TSS of juice were measured using standard procedures.

#### **Selection of Carrier Agent and Operating Parameters**

Preliminary trails were conducted for selection of encapsulating ingredients such as Maltodextrin, Gum Arabic and combination of both with its different concentration levels. Among the materials, Maltodextrin was found better for production of powder from acid lime juice. The levels of temperature of inlet air and rate of feeding were also finalized based on random trails and literature. Accordingly, maltodextrin concentrations (14, 16 and 18%), temperature of inlet air (140, 150 and 160°C) and feeding rate (62.5, 87.5 and 112.5 mL/h) were selected as independent variables. The aspiration rate (50m<sup>3</sup>/h) was kept constant for the study. The powder

obtained in preliminary trails was examined for its recovery, color, stickiness and organoleptic properties. It was observed that at beyond and below the selected levels of process parameters and carrier agent concentrations, the quality was not desirable. In operation, after preparation of juice sample, the required concentrations of MD were added to the juice and stirred manually to homogenize the mixer. Further the sample was loaded to spray dryer and it was run with fixed operating parameters as per the experimental plan (Fig. 1). The prepared powder was packed in aluminum laminated bag (50 µ) and stored until further analysis.

#### **Quality Analysis of Acid Lime Juice Powder**

##### **Powder Yield**

The powder yield was determined on % weight basis as a ratio of weight of powder received to weight of solution fed to the spray dryer (Eq. 1).

$$\text{Powder recovery (\%)} = \frac{\text{Weight of powder}}{\text{Weight of feed}} \times 100 \quad \dots (1)$$

##### **Water Solubility Index (WSI)**

The one gram powder sample of acid lime was intermingled in water (100 ml) with agitation in magnetic stirrer for a time of 5 min at a operating speed of 700 rpm. Prepared mixture was transferred to tube and centrifugation was done (3000 × g, 5 min). The solution (25 ml) was further taken in petri dish of known weight and heated inside oven at 103 ± 2°C, 5 h. WSI was determined on the basis of difference in weight using Eq. 2.<sup>(18)</sup>

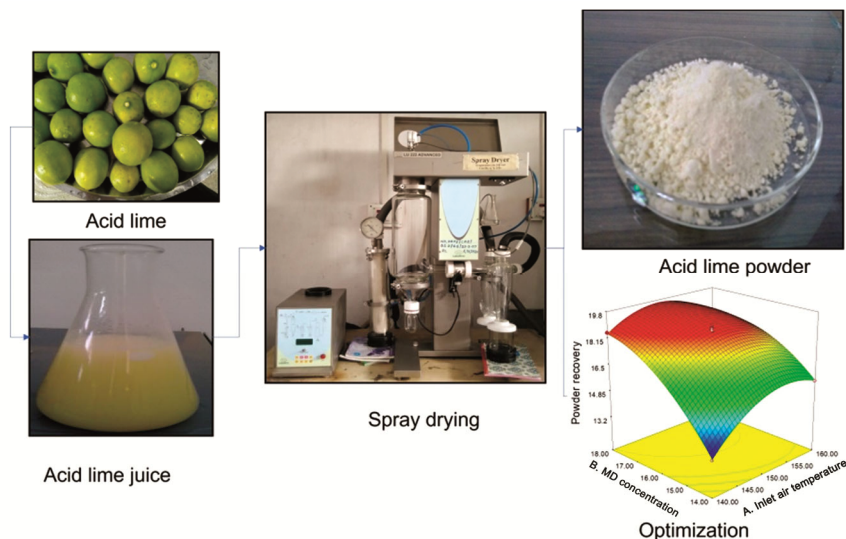


Fig. 1 — Developed process protocol for acid lime powder

$$\text{WSI (\%)} = \frac{(\text{msp} - \text{mp}) \times 100 / 25}{\text{mdb}} \quad \dots (2)$$

where, WSI= water solubility index (%), msp = petri dish mass and original sample weight (g), mp = empty petri dish weight (g), mdb = dry matter weight of sample (g).

#### **Vitamin C Content**

The visual titration method<sup>19</sup> was used for assessment of ascorbic acid of fresh acid lime and powdered sample using dye solution. A 50 ml of un-concentrated juice sample (in case of powder, an equivalent was made by adding powder to distilled water) was pipet out in volumetric flask (100 ml). The solution was further diluted with addition of 25 ml metaphosphoric acid (of 20% conc.) for stabilizing the solution. Sample (10 ml) was pipet out, 2.5 ml of acetone was added and filtered with indophenol dye until occurrence of faint pink color persisted for 15 sec.

$$\text{Vitamin C content} = \frac{\text{Titre} \times \text{Dye factor} \times \text{volume made of (100 ml)}}{\text{ml of aliquot} \times \text{weight of sample}} \times 100 \quad \dots (3)$$

#### **Total Phenol Content (TPC)**

Folic-Ciocalteu technique was used for estimation of TPC of acid lime juice and powder.<sup>20</sup> About 0.2 ml sample of acid lime juice (or for equivalent powder) was mixed with 1 ml Folic-Ciocalteu reagent in test pipette. Further sodium carbonate (2 ml, 20%) was mixed in a sample. Absorbance was dignified using Spectrophotometer (650 nm, Thermo Spectronic Machine, Model: 40001/4, USA).

#### **Particle Size**

It was measured using an equipment (Make: Beckman Coulter, Model: DelsaNano C), Malvern software. The powder sample was mixed in methanol analysis was carried out at 25°C. Size of particle and particle size distribution was expressed with respect to % intensity.<sup>21</sup>

#### **Microscopic Imaging (SEM)**

The powder was evaluated using microscopic imaging (SEM), (Hitachi, Model- S-3700N). Representative powder sample was located at SEM stubs and covered with fine coating of gold. The observations on coated sample were recorded using a scanning electron microscope.<sup>22</sup>

#### **Determination of Response Variables**

Experiments in triplicate were conducted as per experimental design using software (Design expert

(10.0.3.1, Statease Inc.), Box Behnken Design (3 factors and 3 levels) of response surface methodology.<sup>23,24</sup> Inlet air temperature (A: 140, 150, 160°C), concentration of maltodextrin (B: 14, 16, 18%) and feed flow rate (C: 62.5, 87.5, 112.5 mL/h) were taken as independent variables whereas powder recovery, WSI, vitamin C content and TPC were the responses. Total seventeen numbers of experiments were carried out including the center point.

The correlation among selected responses was developed as a second order equation (Eq. 4).

$$y = b_0 + b_1 * A + b_2 * B + b_3 * C + b_{12} * AB + b_{13} * AC + b_{23} * BC + b_{11} * A^2 + b_{22} * B^2 + b_{33} * C^2 + \epsilon \quad \dots (4)$$

where, y : response variable,  $b_{i_s}$  were regression equation coefficients Response and A, B, C are affecting variables viz., air temperature, maltodextrin, and feeding rate, respectively. Experimental data obtained in triplicate was statistically analyzed for variance (ANOVA) at 1% significance level with estimations of correlation and regression coefficients.

## **Results and Discussion**

#### **Physicochemical Properties of Acid Lime Fruit and Juice**

The content of moisture in fruit, peel and seed of acid lime was found to be  $87.40 \pm 0.59$ ,  $83.60 \pm 0.56$  and  $57.70 \pm 0.65\%$ , respectively. Freshly extracted lime juice contained TSS, pH, acidity, total sugars, reducing sugars, vitamin C content and TPC of  $8.07 \pm 0.31^{\text{B}}$ ,  $2.13 \pm 0.05$ ,  $6.25 \pm 0.09\%$ ,  $2.08 \pm 0.12\%$ ,  $1.12 \pm 0.05\%$ ,  $30.42 \pm 1.42$  mg/100 mL of juice and  $61.35 \pm 2.52$  mg GAE/100 mL of juice, respectively.  $L^*$ ,  $a^*$  and  $b^*$  values of juice were noted as 42.086, -2.717 and 4.640, respectively. The findings on properties of acid lime were in agreement of earlier results.<sup>25</sup>

#### **Effect of Independent Parameters on Powder Yield and Attributes of Powder of Acid Lime Juice**

The findings of powder yield and physico-chemical properties of acid lime juice powder are presented in Table 1 with ANOVA for each response variable and its statistical significance along with correlation coefficient. Desirability of fitted models was examined as per the maximum value of squared  $R(R^2)$ , minimum residual error and lack of fit as non-significant. It was observed that, fitted models showed significant with best  $R^2$  and minimal error. From ANOVA it is understood that, higher the F-value of the linear function of independent variables, higher

was the influence of that factor on dependent parameter. Statistically significant regression constant for linear, second order and interacting factors of variables showing the effect on all the response variables in coded form are presented in the Eqs (5 – 8). The positive linear, second order and interacting factors indicated the optimistic influence on the response variables i.e. rise in levels of independent parameters, increased the values of response variables, while negative coefficient indicates decreased values of the responses with surge in values of process parameters. Quadratic terms of process parameters indicated curvilinear nature of response variables. Response plots were generated for the fitted models with respect to any two process parameters, when the third parameter been its central point.

### Yield of Powder

Effects pertaining to spray drying parameters on powder yield from juice of acid lime have been depicted in Fig. 2a, 2b and 2c. The powder recovery ranged between 13.23% and 18.97% over the range of experiments. It could be seen that surging the temperature of air and maltodextrin resulted to increment in powder yield (Fig. 2a, 2b, 2c). The elevated drying temperature resulted to enhanced heat and mass transfer processes. Wang<sup>5</sup> observed similar results in spray drying of black mulberry powder. The findings of Tonon<sup>7</sup> and Goula and Adamopoulos<sup>9,26</sup> are also in conformity of the present results. The surge in temperature increased the powder yield up to certain limit, thereafter reduced with further rise in temperature. The reason might be melting of the dry solids at higher temperature.

Table 1 — ANOVA of second order polynomial model for physicochemical properties of acid lime juice powder

Source	df	Powder recovery	Water Solubility Index (WSI)	Vitamin C	Total phenol content (TPC)
Model	9	306.62***	161.92***	34.38***	29.44***
A: Inlet air temperature	1	89.20***	210.81***	210.83***	48.43***
B: MD concentration	1	1574.35***	1006.73***	40.38***	115.45***
C: Feed flow rate	1	39.65***	74.18***	7.63*	22.27**
AB	1	95.08***	8.97*	16.76**	9.14*
AC	1	44.85***	7.35*	4.15	0.011
BC	1	1.22	25.96**	0.37	1.02
A <sup>2</sup>	1	421.91***	22.62**	10.54*	16.03**
B <sup>2</sup>	1	251.52***	48.06***	17.23**	42.42***
C <sup>2</sup>	1	150.63***	40.03***	0.21	4.46
Lasting	7				
Lack of fit	3	1.01	1.88	1.16	0.67
Error	4				
Total	16				
R <sup>2</sup>		0.998	0.995	0.978	0.974
R <sup>2</sup> , adjusted		0.994	0.989	0.949	0.941
R <sup>2</sup> , predicted		0.980	0.952	0.817	0.835
SD		0.140	0.230	2.380	5.140
CV (%)		0.790	0.240	0.660	2.840

Significance level; \*\*\* : 0.1%, \*\* : 1% and \* : 5%

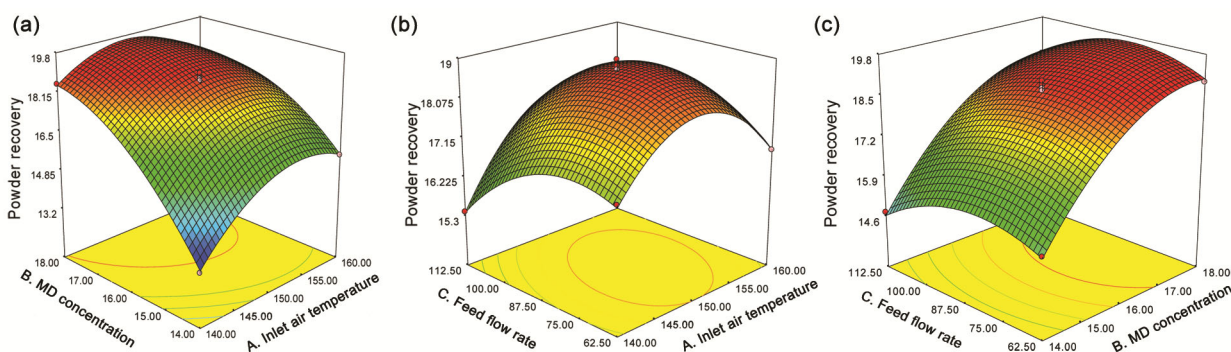


Fig. 2 — Yield of acid lime powder at different feeding rate and maltodextrin percent: (a) MD concentration vs. inlet air temperature, (b) feed flow rate vs. inlet air temperature, (c) feed flow rate vs. MD concentration

Increase in maltodextrin concentration from 14% to 18%, reported increase in the powder recovery. Maltodextrin helps for better encapsulation of basic sugars and organic constituents of the liquid extract during drying, which assisted the particles for easy flow in volume of drying chamber hence avoiding the stickiness in drying chamber.<sup>27</sup> The addition of encapsulating material during spray drying of liquid extract increases  $T_g$  of amorphous nature components. Wang<sup>5</sup> showed similar observations for black mulberry, raisin concentrate and orange juice. However, Tonon<sup>7</sup> reported decrease in yield as surge in encapsulating material quantity with a reason of increase in viscosity.

Rise in levels of feeding rate had an opposite influence on yield reporting reduced powder yield at elevated feed flow (Fig. 2b, 2c). The concentrated mixture at higher feed flow might have resisted the drying process resulting into stickiness in the chamber. Inadequate drying due to formation of large size droplet was also the cause of low powder recovery.<sup>28</sup> Higher feed flow rates also lead to dripping inside the drying chamber.<sup>7</sup>

The effluence of the operating parameters on powder yield of acid lime juice in terms of coded level of variables is described by Eq. (5).

$$PR = 18.77 + 0.45A + 1.91B - 0.03C - 0.066AB + 0.46AC + 0.075BC - 1.36A^2 - 1.05B^2 + 0.81C^2 \dots (5)$$

where, PR is the powder recovery of acid lime juice.

#### Index of Water Solubility (WSI)

WSI of prepared powder varied from 89.33 and 96.80% with the average value of 93.92%. The results showed that rise in temperature if inlet air led to enhancement in WSI of powder (Fig. 3a, 3b). Higher temperature produces the powder of lower density hence leading to rise in WSI.<sup>5,26</sup> Augmentation of maltodextrin (from 14% to 18%) also resulted increase in WSI (Fig. 3a, 3c). Maltodextrin poses higher solubility in water and hence leads to increased WSI of spray dried powder sample.<sup>18</sup>

At constant levels of inlet air temperatures, the WSI of acid lime juice powder reduced with surge in the feeding rate of juice (Fig. 3c). Higher feed flow rate leads to inadequate drying of powder particles with higher moisture content. Powder with higher moisture content has poor rehydration property and hence reduced WSI. The findings of Muzaffar<sup>29</sup> are in agreement of this conclusion who studied the spray drying of pomegranate juice powder.

Regression equation (Eq. 6) is the empirical equation for prediction of WSI of acid lime powder.

$$WSI = 94.86 + 1.17A + 2.56B - 0.70C - 0.34AB + 3.10AC + 0.58BC - 0.53A^2 - 0.77B^2 - 0.71C^2 \dots (6)$$

#### Vitamin C/Ascorbic acid

The value of ascorbic acid ranged between 342.20 and 378.30 mg/100 g with the mean value of 360.17

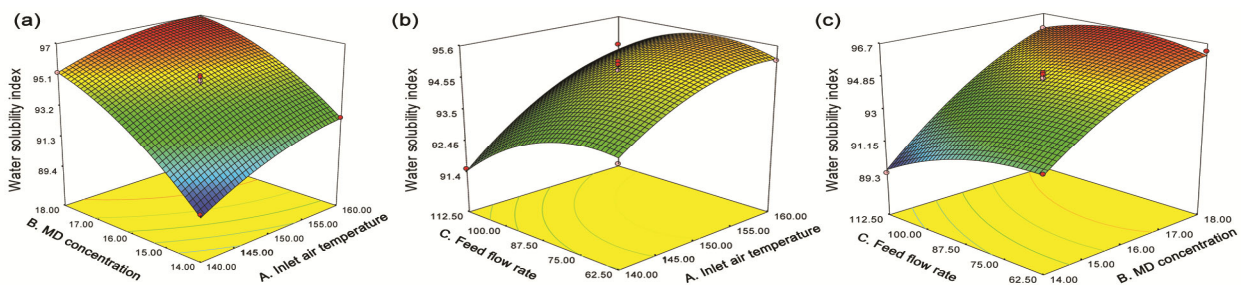


Fig. 3 — Water solubility index (WSI, %) of powder at different feeding rate and maltodextrin percent: (a) MD concentration vs. inlet air temperature, (b) feed flow rate vs. inlet air temperature, (c) feed flow rate vs. MD concentration

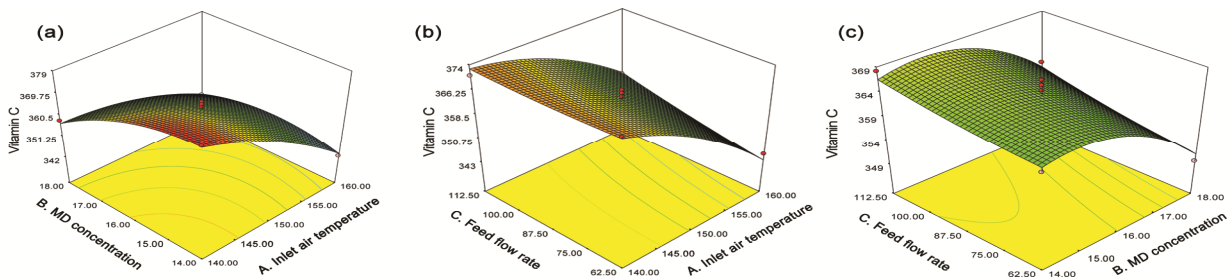


Fig. 4 — Vitamin C content (mg/100 g) of acid lime powder at different feeding rate and maltodextrin percent: (a) MD concentration vs. inlet air temperature, (b) feed flow rate vs. inlet air temperature, (c) feed flow rate vs. MD concentration

mg/100g. It was reduced with increase in temperature and maltodextrin content (Fig. 4a, 4b, 4c). Thermal degradation and accelerated oxidation might be the causes of loss of vitamin at increased air temperature. Patil<sup>30</sup> found that the ascorbic acid content guava powder dropped with rise of temperature and also with elevation of maltodextrin content. Findings of Quek<sup>27</sup> for lycopene in tomato and Chauhan<sup>31</sup> in case of guava pulp powder are in conformity of present results.

The increase in feeding rate increased the vitamin C of powder (Fig. 4c). The reason might be the higher particle size at higher feed rate, thus less surface area exposed to hot air and retention of maximum vitamin C in the powder. Similar results were also observed by for lemon juice powder,<sup>14</sup> for pomegranate powder.<sup>29</sup>

The following regression equation (Eq. 7) shows that the process conditions were the most influencing factors which affect the content of vitamin C.

$$\text{Vitamin C} = 363.96 - 12.23A - 5.35B + 2.33C + 4.88AB + 2.42AC + 0.73BC - 3.77A^2 - 4.82B^2 + 0.53C^2 \quad \dots (7)$$

#### Total Phenol Content (TPC)

The TPC of acid lime juice powder varied from 164.47 to 188.55 mgGAE/100 g over the range of experiments with a mean value of 177.976 mg GAE/100 g of powder. Rising temperature and maltodextrin content resulted in decreased value of TPC in powdered product (Fig. 5a, 5b, 5c). Thermal degradation of phenolic compounds at higher temperature caused decrease in its content in powder. Similar effect on TPC was observed for lemon juice powder<sup>14</sup> and bayberry.<sup>32</sup> The dilution effect of maltodextrin causes decrease in TPC of spray dried product.<sup>11</sup> Further addition of carrier agent causes the oxidation of phenolic compounds.

The flow rate of feed had positive effect on the TPC of final product (Fig. 5b, 5c). Higher feed rate leads to larger particle size of powder, thus reduces its exposure to heat and retains the TPC. The findings of effect of feed flow rate for sumac extract are in agreement of present study.<sup>33</sup>

$$\text{TPC} = 183.59 - 4.77A - 7.37B + 3.24C + 2.93AB + 0.10AC - 0.98BC - 3.78A^2 - 6.16B^2 - 2.00C^2 \quad \dots (8)$$

#### Numerical Optimization and Model Validation

The numerical optimization of independent parameters was carried out by setting the desired goals of responses. The optimum condition for spray dried acid lime juice powder was determined to obtain maximum powder recovery, WSI, vitamin C content and TPC content. The numerical importance value of responses was decided based on the significance of responses for desired quality of output product. Acid lime juice was the excellent source of vitamin C, therefore maximum importance (5) was assigned to the amount of vitamin C of prepared powder followed by powder recovery, TPC and WSI (4) and minimum but equivalent rank (3) was assigned to all remaining responses.<sup>29</sup> At a predicted optimal level of independent values viz., inlet air temperature (147.63°C), concentration of maltodextrin (16.27%) and feeding rate (90.94 mL/h) the dependent variables as powder recovery, WSI, Vitamin C content and TPC were 18.77%, 94.79%, 365.649 mg/100g and 183.702 mgGAE/100g of powder, respectively. Actual experiment at 145°C temperature of entering air, 16% maltodextrin and 87.5mL/h feed flow rate was conducted to validate the model. Experimental value of powder recovery, WSI, vitamin C and total phenol content obtained were 19.45%, 96.67%, 365.36 mg/100g powder and 177.99 mgGAE/100g powder, respectively. Validation of model was done

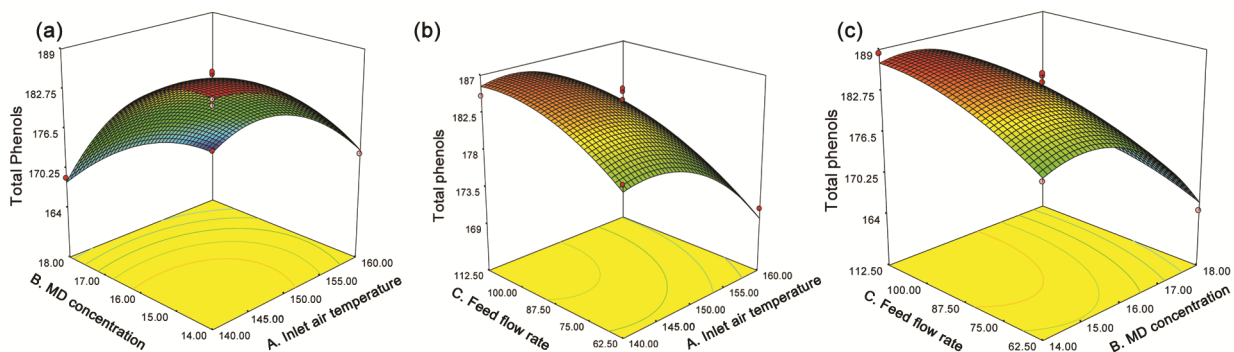


Fig. 5 — Total phenol content (TPC, mgGAE/100 g) of powder at different feeding rate and maltodextrin percent: (a) MD concentration vs. inlet air temperature, (b) feed flow rate vs. inlet air temperature, (c) feed flow rate vs. MD concentration

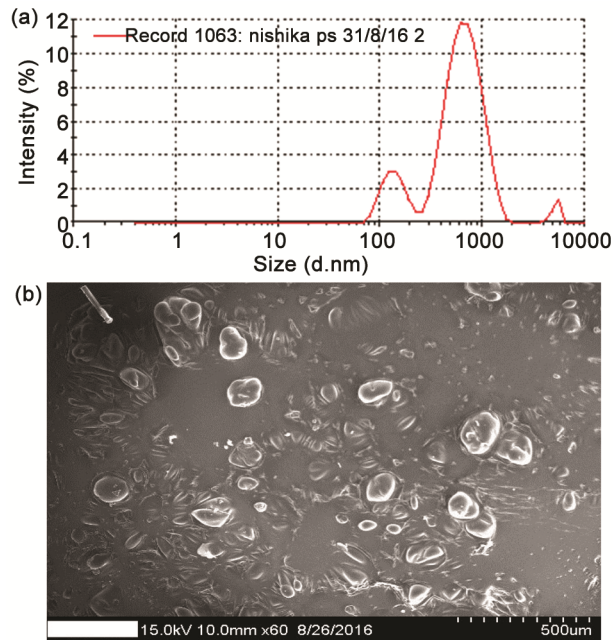


Fig. 6 — Morphology of spray dried lime juice powder prepared at optimized condition: (a) Particle size distributions (by intensity) of powder, (b) Scanning electron microscopy (SEM) of powder particles

by comparison of projected values of the model and actual values of responses with percentage error recorded as 3.48%, 1.95%, 0.078% and 3.21% for powder recovery, WSI, Vitamin C content and TPC, respectively. The error value states that the fitted model is suitable to predict the values of all the responses. A model with error values is less than 5% is considered as best fit model.<sup>24</sup> Process of numerical optimization for spray drying has also been followed for pomegranate<sup>29</sup> and guava powder.<sup>30</sup>

#### Size of Particle

The distribution of size of spray dried acid lime powdered particles prepared at optimized conditions has been shown in Fig. 6a. Particle size distribution was represented as number of particle (in percent) having a specific size in comparison to total number of particles. Three different intensity weighted peaks could be observed in particle size distribution graph having average size of particle of 150 nm, 800 nm and 6000 nm at weighted intensity values of 3, 11.7 and 1.5%, respectively. The trimodal size distribution of particle depicts that powder of acid lime is heterogeneous in texture. Bimodal particle size of powder is desirable because in heterogeneous powder mixture, the void spaces are fulfilled by small particles thus improving the bulk density and flow

ability of powder.<sup>21,34</sup> The variation in formation of droplet size is the reasons for heterogeneous production of spray dried powder.<sup>35</sup>

#### Microscopic Analysis (SEM) of Powder

Microstructure of spray dried acid lime particulates produced at optimized conditions has been shown in SEM image (Fig. 6b). The image depicts heterogeneous mixture of particle size as discussed in particle size analysis. Diverse shape of particles ranging from uniform spherical to elongate could be observed in the microstructure. The surface of particles seems to be smooth which is a required attribute of powder. The oxygen and water vapor transmission of smooth surfaced powder materials are better.<sup>35</sup> Spray dried particle with smooth surface has a protective coating on its surface by means of encapsulation and plays key role in properties such as solubility, water activity of powder.<sup>36</sup> The study on microscopic analysis of lemon juice powder at maltodextrin content of 15% with varying air temperature reported that production of smooth surfaced powder particles with no effect of drying air temperature on surface smoothness.<sup>14</sup>

#### Conclusions

The present investigation describes the process of production of acid lime powder using a spray drying method. The operating parameters of spray dryer viz., temperature of inlet air (147.63°C) maltodextrin content (16.27%) and feeding rate (90.94 mL/h) were optimized using RSM for a powder with better quality. At these operating parameters the quality of powder in the form of recovery, WSI, amount of vitamin C and TPC were found to be 18.77%, 94.79%, 365.937 mg/100g and 183.695 mg GAE/100g, respectively. Polynomial model of second order was best tailored for process optimization of spray drying of acid lime powder. Spray drying of acid lime juice powder produced heterogeneous particle distribution with smooth surfaced particles. Production acid lime powder at industrial scale would avoid the losses of acid lime during its peak production and will also be a boon for the entrepreneurs to setup a business.

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#### Declarations:

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